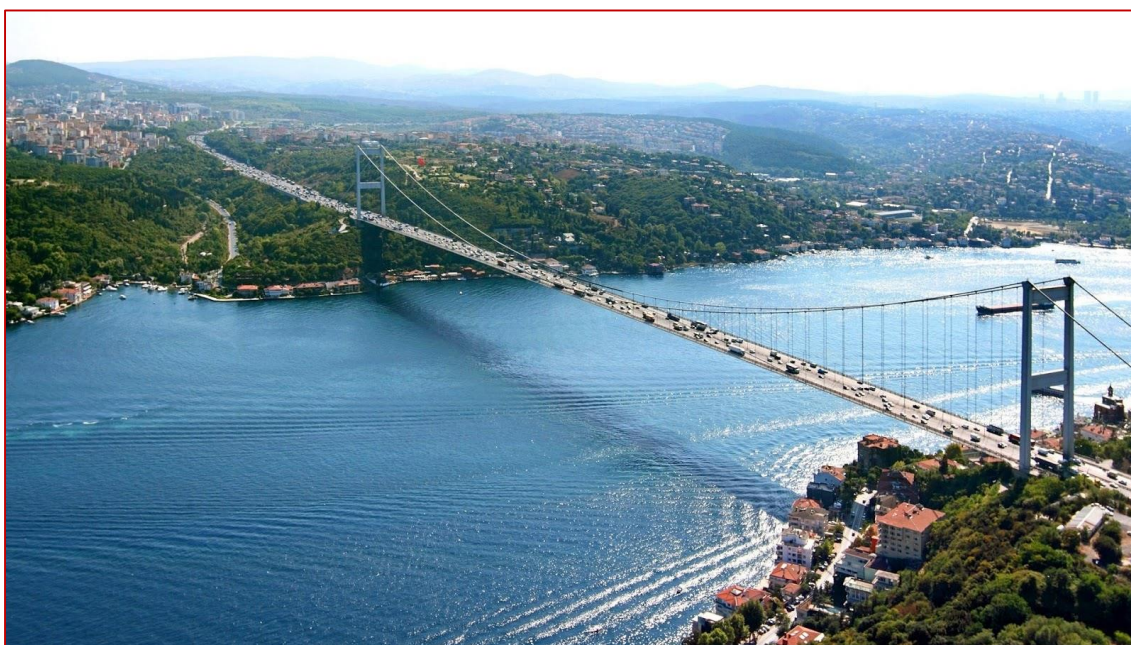




**8th Black Sea Basin
Conference on Analytical Chemistry**

PROCEEDING BOOK

Editor: Mehmet Yaman



9-11 May 2018 Sile/Istanbul-Turkey

BBCAC 2018

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Preface

We are very pleased because the “8th Black Sea Basin Conference On Analytical Chemistry (8th BBCAC)” Conference was held successfully in Sile/Istanbul, Turkey, in May 09-11, 2018. The 8th BBCAC is a biennial series of conferences that started in 2001 and covers all areas of Analytical Chemistry and applications of Chemical Analysis.

The scientific conference program consists of 15 sessions that include **16 invited and 49 oral** presentations as well as **128 posters** to be presented in the respective sessions. In addition, researchers of Academia (**54 universities from 11 countries**) and Research Institutes will present up-to-date development on analytical chemistry as well as applications to a wide range of environmental, biological and food matrices.

We strongly believe that the discussions and the exchange of ideas among the participants during the 3 days of the meeting will make 8th BBCAC a brilliant platform to initiate new research collaborations, particularly in favor of the young scientists participating in the conference.

We wish you all to enjoy this conference as all the previous BBCAC conferences since 2001 and have a pleasant stay in Sile/Istanbul, hoping to meet you again during the next BBCACs.

With our best regards

The Chair (on behalf of Organizing Committee)

Prof. Dr. Mehmet YAMAN

Firat University, Science Faculty, Department of Chemistry, Elazig-Turkey

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INVITED SPEAKERS

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Prof. Dr. Gokce KAYA-Firat U.

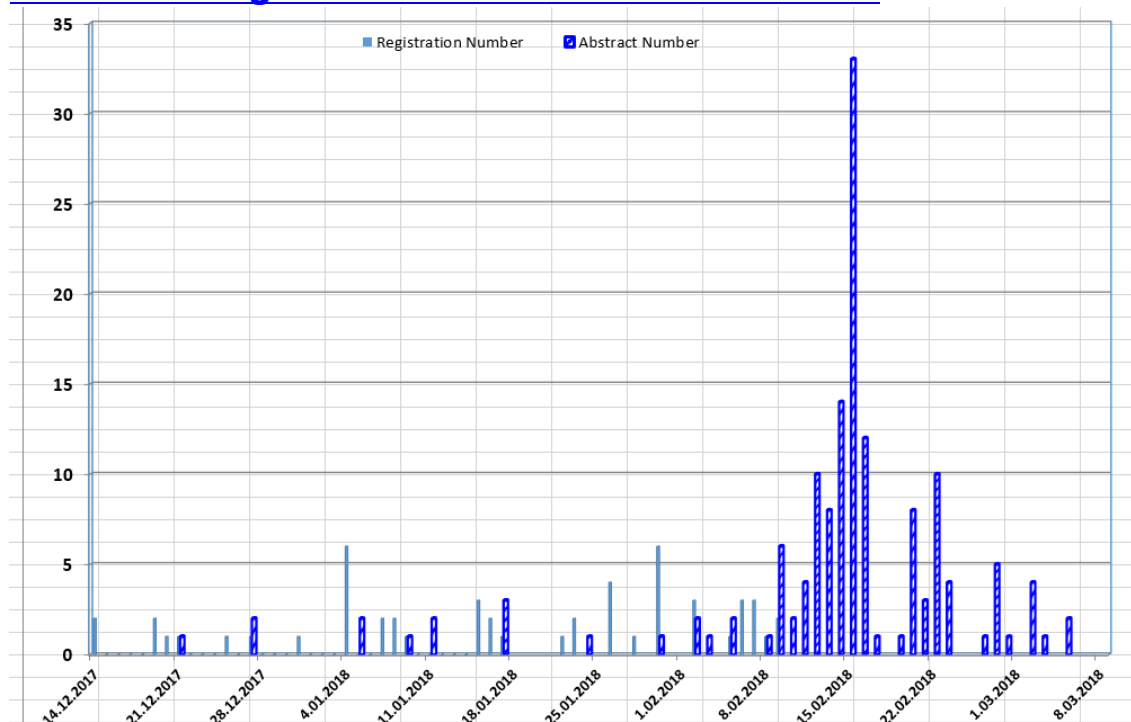
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Prof. Dr. Mehmet YAMAN
Firat University

Local Organizing Committee-Secretariat

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Statistic of registration and abstract submissions



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GENERAL INFORMATION

Introduction

The 8th International Conference “8th Black Sea Basin Conference On Analytical Chemistry” will be held on 9-11 May 2018 in Sile/Istanbul-Turkey is a three-days scientific meeting covering all areas of Analytical Chemistry and applications of Chemical Analysis. For the last 17 years, BBCAC has provided an excellent framework for the presentation of new concepts, instruments, methods, and applications in the area of modern chemical analysis. Researchers and scientists from Universities, Research Institutions, State Organizations, and the Industry come together during the meeting to present and discuss the current state of the art in the area of chemical analysis. At the same time, it provides the grounds for the graduate and post graduate students to present their projects, discuss scientific collaborations with other groups, as well as to explore employment opportunities. An exhibition of analytical instruments and accessories will be also organized in the conference place whereas a ship tour and social events are planned to be included in the program of the BBCAC 2018.

Topics

Current trends on Spectrochemical, Electrochemical, Chromatographic, Microscopic and Thermal analysis methods, Hyphenated techniques, Speciation analysis, Bioanalytics, Trends on sample handling and preparation, Chemical and bio-sensors, Field analysis-Mobile analytical instruments, Laboratory information management systems (LIMS), Miniaturized analytical systems (chips), Quality control-quality assurance on analysis, Commercial developments and markets, Flow and micro-flow methodologies, Immunoassays, Electrophoretic separation techniques, Sampling techniques and strategies.

Applications: Food Analysis, Environmental analysis, Biomedical (Clinical, Ecotoxicological) analysis, Pharmaceutical analysis, Material science (Nanomaterials), Archaeometry analysis, Industrial analysis.

Contributions from commercial organizations, including detailed descriptions of new instrumentation, Specific applications, Assessment of future commercial trends and opportunities.

Previous conferences

The recent past conferences were held in Odessa / Ukraine (2001), Sile / Istanbul / Turkey (2003), Constanta/Romania (2005), Golden Sands/Bulgaria (2007), Fatsa/Turkey (2009), Trabzon/Turkey (2013), and Varna / Bulgaria (2015).

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Location of Conference

BBCAC-2018 will be held in Sile in south-coast of the Black Sea-Istanbul province. Şile, a holiday district, is around 70 km away from the Istanbul city center. Ağlayan Kaya and Kumbaba are most popular beaches in Şile. Others are Şile Public Beach, Uzunkum Beach, Ayazma Beach and Aqua beach. Coastline of Şile is the second longest in the world. Şile is famous for its cloth (Şile Bezi). It is slight, comfortable and above all it never makes you sweat. That's why it is usually used for clothes. Every July, there's a Şile Bezi festival which takes place in Şile and it's possible to see various things made from Şile Bezi. Agva is small holiday town located in Sile. It's between the Goksu River, a well-known for boat excursion, and Yeşilçay stream. Agva is amazing getting away destination together with its natural beauty and silence. Hacılı village, only 12 km away from Agva, is famous for its caves and waterfalls.

The airport of Sabiha Gokcen-Istanbul is about 80 km away. The easiest way to go from Istanbul is taking bus (number 139) from Şemsipaşa, Üsküdar-Istanbul.

Papers presentation

Scientific program will include Invited Speakers, which will provide an up-to-date presentation of modern trends of Analytical Chemistry as well as of related subjects of chemical analysis-interest. Oral Presentations will be presented between 10-15 min. Contributed papers describing original research work will be also presented as posters in order to promote efficient discussion on new scientific ideas and results. The presenting authors should hang their posters before 13:30, and remove them in the evening of the corresponding day. The preferable dimensions for posters should be 70 x 100 cm (width x height). All posters are required to conform to portrait orientation. Posters should be clear and easy to read. Type size should be sufficiently large to allow people to read from 2-3 meters. All presentations should be in English. Poster and oral presentation will be accepted if at least one of the authors is registered and present at the conference for personal communication.

Best poster certificate

A competition for the best poster among the young scientists in each poster session will also take place. These certificate will be given to recognize excellence in research and presentation. The winners will be announced during the Welcome Cocktail and Gala Dinner. on 09-10 May, 2018.

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CONFERENCE PROGRAM

8th Black Sea Basin Conference On Analytical Chemistry

9-11 May, 2018, Sile-Istanbul/Turkey

Detailed Program

	9 May, 2018
8.30 – 10.00	Registration -Sile Resort Hotel, Sile-Istanbul-Turkey The registration desk will be open everyday during conference hours
10.00 – 11.00	➤ Welcome Ceremony ➤ Respect-Silence of Independence and Opening Speeches: Prof. Dr. Mehmet Yaman (Chair) Prof. Dr. Şeref Gucer (on behalf of continuation committee) Honorable
11.00-11.15	Inv. 1: Prof. Dr. Michael A. Bolshov - Absorption Spectrometry With Diode Lasers As A Technique For Contactless Measurements Of The Parameters Of Hot Zones- Moscow State U.
11.00-11.15	Tea/Coffee break
	Session 1 -Chairs: Sibel Ozkan/Bekir Salih
11.15-11.45	Inv. 2: Prof. Dr. Bezhan Chankvetadze - Enantioselective analysis of chiral compounds by using capillary electromigration techniques- Tiflis U.
11.45-12.00	01: Levent Pelit -Development of a Noninvasive Method for Biomarker Determination in Human Breath for Early Diagnosis of Lung Cancer-Ege U.
12.00-12.15	02: Emirhan Nemutlu - Mass Spectrometry Based Targeted and Untargeted Metabolomic Analysis for Early Diagnosis of Breast Cancer Using LC-ESI-qTOF-MS and LC-ESI-MS/MS-Hacettepe U.
12.30-13.30	Lunch
	Session 2- Professor Dimitar TSALEV/Speciation session Chairs: Durisehvar Unal/Seref Gucer
13.30-14.00	Inv. 3:Prof. Dr. Irina Karadjova - Smart Materials for Mercury speciation- Sofia U
14.00-14.15	03: Asli Erdem Yayayuruk -Speciation of chromium in waters using phosphomethylated polystyrene-divinylbenzene hairy brushes prior to inductively coupled plasma mass spectrometric determination-Ege U
14.15-14.30	04: Orhan Gezici -“Cryogel-cemented” Humic Acid as an Alternative Separation Medium-Nigde U.
14.30-14.40	05: Sezen Sivrikaya - Selective Determination of Cr(VI) by a Novel FI-SPE-FAAS Method Using Tris(2-aminoethyl)amine-Functionalized Silica Gel-Sakarya U.
14.40-14.50	06: Gokce Kaya - Determination of Trace Elements In Different Tissues Of Green Tiger Shrimp (<i>Penaeus semisulcatus</i>) from Eastern Mediterranean, Turkey-Firat U
14.50-15.05	Tea/Coffee break
	Session 3 -Chairs: Gularen Alsancak/Alex Zacharia
15.05-15.35	Inv 4: Prof. Dr. Trajče Stafilov - Heavy Metals Environmental Pollution Studies In The Republic Of Macedonia-Skopje U.
15.35-16.05	07: Mustafa Ersoz -COST-Nanoporous Polymer Membranes by Blockcopolymer Technique and Their Analytical Applications-Selcuk U.
16.05-16.15	08: K. Volkan Ozdokur - Simultaneous Determination of Levodopa, Dopamine, Homovanilic Acid and 3,4-Dihydroxyphenylacetic Acid by using HPLC-UV system- Erzincan U
16.15-16.25	09: Ilknur Bagatir Erbas - Fast, easy and green method employing magnetic dispersive solid phase extraction for the determination of pesticide residues- Ege U.
16.25-16.35	Tea/Coffee break
16.35-18.10	Session 4:Poster session (P1-64) Chairs: Agnese Osite/Aysem Uzer Arda/ Nilgun Sen/Elif Tumay Ozer/Levent Pelit/Yusuf Dilgin

	Session 5- Separation-Microextraction-Preconcentration Session Chairs: Serife Tokalioglu/ Mustafa Culha
18.10-18.40	Inv. 5: Prof. Dr. Yuriy Stetsyshyn - Fabrication of the temperature and pH-responsive grafted polymer brushes for stimuli-modulated bioseparations and orientations-Lviv U
18.40-18.50	O10: Mustafa Tuzen - A Simple and Green Deep Eutectic Solvent Based Air Assisted Emulsification Liquid-Liquid Microextraction of Palladium from Water and Environmental Samples-Gaziosmanpaşa U.
18.50-19.00	O11: Mustafa Imamoglu - Determination of Cu(II) Ions at Trace Level in Some Vegetable Samples by a Novel SPE-FI-FAAS Method-Sakarya U.
19.00-19.10	O12: Irem Aydın - Development of PPy-CNT-SPME Fiber Development and Their Use in Pesticide Determination in Apple Juice by GC-MS-Ege U
19.10-19.20	O13: Dotse Selali Chormey - Quadrupole Isotope Dilution Mass Spectrometry-Dispersive Liquid-Liquid Microextraction Using Multivariate Optimization: A Novel Analytical Approach for the Determination of Parathion Methyl in Water-YTU
20.00-22.00	Dinner-Welcome Coctail-Music

	10 May, 2018
	Session 6-LC and MS Session Chairs: Nevin Erk/Bezhan Chankvetadze
08.30-09.00	Inv. 6: Prof. Dr. Bekir Salih - Determination of Site Specific Protein-PEGylation by MALDI-MS and IM-MS- Hacettepe U.
09.00-09.15	O14: Mehmet Atakay - Monitoring Conformational Features of Protein-Polyelectrolyte Complexes Using Ion Mobility-Mass Spectrometry-Hacettepe U
09.15-09.30	O15: Emine Akyuz Turumtay - Hepatoprotective extracts of <i>Cuscuta campestris</i> Y. and Their Best Known Anticancer Flavonols Isolated by Preparative HPLC-Recep Tayyip Erdogan U.
09.30-09.45	O16: Engin Koçak - Analysis of Human Plasma Proteome by Using UPLC/MS Method- Hacettepe U
09.45-09.55	O17: Yilmaz Ugur - The Phenolic Compounds of Some Cornelian Cherry (<i>Cornus mas</i> L.) Genotypes Grown in Turkey-Inonu U.
09.55-10.05	O18: Tugba Yavuz -Simple, Fast and Reliable Method for Determination of H ₂ O ₂ in Aqueous Samples-Ege U
10.05-10.20	Tea/Coffee break
	Session 7- Professor Trajče Stafilov Session Chairs: A. Nur Onar/ Mustafa Tuzen
10.20-10.50	Inv. 7: Organizing committee: Prof. Dr. Şeref GUCER - Geography and Analytical Chemistry: Trace elements Analysis for Geographic Origin- Uludag U.
10.50-11.05	O19: Elif Tümay Ozer - Molecularly Imprinted Nanoparticles for Selective Tetracycline Adsorption-Uludag U.
11.05-11.20	O20:Sema Bagdat - A New Off-line Solid Phase Extraction Method for Antimony using Silica Functionalized with N,N'-bis(4-methoxysalicylidene)-1,3-propanediamine-Balikesir U.
11.20-11.35	O21: Umran Seven Erdemir - Fractionation and Bioaccessibility Studies: Are they Meet the Demand for the Risk Assessments of Some Elements in Baby Foods?- Uludag U.
11.35-12.15	SEM LAB Presentation
12.30-13.30	Lunch
	Session 8-Chairs: Irina Karadjova /Michael Bolshov
13.30-14.00	Inv 8: Prof. Dr. Mustafa Çulha - Surface-enhanced Raman Scattering for Living Single Cell Analysis-Yeditepe U.
14.00-14.15	O22:Burak Ulgut - The importance of Ion pairing and polarity effects Electrochemical Double Layer in Ionic Liquids: Experimental and Modeling Study-Bilkent U
14.15-14.30	O23: Nilgun Sen - CocrySTALLIZATION of Energetic Materials with Enhanced Sensitivity-Edinburgh U. UK

14.30-14.40	O24: Hasan Ilhan: <i>SERS Based E. coli Enumeration in Urine With Using Casein Bound Magnetic Nanoparticles</i> -Hacettepe U.
14.40-14.50	O25: Can Berk Uzundal- <i>pH in Unconventional Electrolytes: Acid Containing Lyotropic Liquid Crystalline Mesophases</i> - Bilkent U.
14.50-15.05	Tea/Coffee break
	Session 9-Spectral Imaging/Preconcentration Session Chairs: Belgin Izgi/ Hasan Kucukbay
15.05-15.35	Inv 9: Prof. Dr. Rawil Fakhrullin- <i>Dark-Field Microscopy and Hyperspectral Imaging for Detection and Identification of Nanoscale and Microscale Particles in Cells, Tissues and Live Organisms</i> -Kazan Federal U.
15.35-15.50	O26: Feyzullah Tokay- <i>The Usage of N,N'-bis(4-methoxysalicylidene)-1,3-propanediamine Loaded Silica Gel for Simultaneous Preconcentration of Ba, Cd, Co, Cu, Mn and Ni Ions</i> - Balikesir U.
15.50-16.00	O27: Birgul Celik- <i>A New Analytical Strategy for the Determination of Indium</i> -Dicle U.
16.00-16.10	O28: Imran Guney Afacan- <i>Adsorption of VOCs onto Activated Carbon and Biochar Obtained From Textile Wastes for Their Determination by GC-MS</i> - Ege U
16.10-16.20	O29: Cagdas Buyukpinar- <i>Development of an Accurate and Sensitive Analytical Method for the Determination of Cadmium Using Hydrogen Assisted T-Shape Slotted Quartz Tube-Atom Trap-Flame Atomic Absorption Spectrophotometry</i> -YTU
16.20-16.30	Tea/Coffee break
16.30-18.15	Session 10: Poster session (P65-128) Chairs: Zeynep Aydogmus/ Sema Bagdat/ Nina Djapic/Mustafa Imamoglu/Emirhan Nemutlu/Durisehvar Ozer
	Session 11-Chairs: Julide Hızal/ Trajce Stafilov
18.15-18.40	Inv. 10: Prof. Dr. A. Zacharia- <i>The Analytical Chemistry For Industrial Needs And Scientific Laboratory Accreditation</i> -Odesa U.
18.40-19.00	Inv. 11: Dr. Agnese Osite- <i>Pine Tree as Environmental Pollution Indicator: X-Ray Fluorescence Studies</i> - Latvia U.
19.00-19.15	O30: Erdal Yabalak- <i>Purification of olive mill wastewater by subcritical water oxidation using hydrogen peroxide: Application of response surface methodology</i> -Mersin U
19.15-19.25	O31: Muge Gemili- <i>Stability constants of cobalt (II) complexes of octahydropyrrolo [3,4-c]pyrrole N-benzoylthiourea derivatives</i> -Mersin U
20.00-22.00	Dinner-Gala Dinner-Music
	11 May, 2018
	Session 12-Chairs: Zehra Kucukbay/ Rawil Fakhrullin
08:30-09.00	Inv. 12: Prof. Dr. Resat Apak- <i>Design Principles of Optical and Electrochemical Sensors for the Determination of Antioxidant Ability and Reactive Species</i> - Istanbul U
09.00-09:30	Inv. 13: Prof. Dr. Shahabuddin Memon- <i>Challenges and Strategies for the Removal of Toxicants from Water</i> - Sind U.
09.30-09.45	O32:Gorkem Yalcin- <i>Change of Some Physical Parameters Alter the Antioxidant Capacity of Pine Honey</i> -Ege U.
09.45-10.00	O33-Abdullah Taner Bisgin- <i>Spectrophotometric Determination of Allura Red (E129) in Foodstuffs after Cloud Point Extraction</i> -Nigde U.
10.00-10.10	O34:Zafer Ocak- <i>Solvent and Molecular Structure Effects on Acidity Strength in Non-Aqueous Medium</i> -Kafkas U.
10.10-10.20	O35: Fatma Ozge Gokmen- <i>Optimization of Poly (Acrylic Acid-co-N-vinyl 2-pyrrolidone) / SiO2 Nanocomposite Hydrogels</i> -Bilecik U.
10.20-10.30	O36:Elif Ozdemir- <i>Spectrophotometric determination of cefpodoxime proxetil in Pure and Pharmaceutical Preparations</i> - Yeniyuzyl U.
10.30-10.45	Tea/Coffee break

	Session 13- Electroanalytical Session Chairs: Hasan Ertas/Yurij Stetsyshyn
10.45-11.15	Inv 14: Prof. Dr. Yucel Sahin- From Graphite to Graphene: A Novel Electrochemical Approach- YTU
11.15-11.30	O37:Lokman Liv- Voltammetric Determination of Boron in Dried Fruits and Nuts- TUBITAK UME
11.30-11.45	O39:Sukriye Ulubay Karabiberoglu- Electrochemical Determination of Bisphenol-A Based on Copper Oxide-Zinc Oxide/Graphene Oxide Modified Electrode- Ege U.
11.45-12.00	O40:Metin Gencten- Organic Based Additives for Vanadium Redox Battery Systems-YTU
12.00-12.15	O41:Mohammed A. Zabara- Electrochemical Impedance Spectroscopy Based Modelling Approach for Primary LiSOCl ₂ Battery- Bilkent U
12.15-12.25	O42:Ceren Kusci- Development of Cobalt Oxide Modified Electrodes and Their Investigation in Electrocatalytic Applications-Ege U.
12.30-13.30	Lunch
	Session 14- Chairs: Ramazan Gurkan/Firat Aydin
13.30-14.00	Inv 15: Prof. Dr. O. Yavuz ATAMAN- Misnomers and oxymorons
14.00-14.10	O38: Oya Irmak Sahin- Spectrophotometric Determination of Bioactive Compounds in <i>Spirulina platensis</i> -Yalova U.
14.10-14.20	O43: Cemil Can Eylem- Dynamic Phosphometabolomic Profiling of Caco-2 cell lines by 18O-assisted GC-MS- Hacettepe U
14.20-14.30	O44:Buse T. Zaman- Determination of Ultratrace Cadmium by a New Combination: Solid Phase Microextraction by Stearic Acid Coated Magnetic Nanoparticles Prior To Batch Type Hydride Generation Atomic Absorption Spectrometry- YTU
14.30-14.40	O45: Murat Celiker- Temporal Changes in Trace Element Concentrations in Spring, Shallow and Deep Well Waters From the Uluova Plain, Elazig- Firat U.
14.40-14.50	Tea/Coffee break
	Session 15- Chairs: Isil Aydin / Shahabuddin Memon
14.50-15.20	Inv 16: Prof. Dr. Yusuf Dilgin- Developments and applications in enzyme based photoelectrochemical biosensors- Canakkale 18 Mart U
15.20-14.30	O46: Zeynep Kalaycioglu- Separation and Determination of Rosmarinic and Carnosic Acid Contents of Anatolian Salvia Species by Capillary Electrophoresis: Correlation Between Ingredient Contents and Plant Antioxidant Activities-ITU
15.30-15.40	O47: Halil Ibrahim Ulusoy- Development of an Analytical Method for Determination of Tetracycline Residues in Food Samples-Cumhuriyet U.
15.40-15.50	O48: Veselina Adimcilar- Determination of Khellin and Visnagin Contents of Amni Visnaga and Amni Majus by Capillary Electrophoresis with UV Detection-ITU
15.50-16.00	O49: Oznur Karaoglu- Optimization Of Solid-Phase Extraction By Experimental Design Methodology For Separation Of Sterol And Tocopherol In Sunflower Oil Deodorizer Distillate Using Zeolite As A Novel Sorbent-TUBITAK-MRC
16:00	Closing
	12 May, 2018 Social program (Optional) Istanbul Bosphorus-ship tour

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^A Ege University Science Faculty Chemistry Dep. Bornova İzmir, TURKEY	27
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FULLTEXT PRESENTATIONS (FT)

FT1–Multivariate Optimization for Removal of Cu(II) and Pb(II) from Aqueous Solutions Using Synthesized Hybrid and Calcined Materials

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Abstract

Hybrid materials have been receiving attention most especially due to their unique properties and offer probability for many new applications in variable fields. One of the application areas of these materials is heavy metal removal from aqueous solutions. In this study, organic–inorganic hybrid material on the basis of metal alkoxide $\text{Al}(\text{O}^t\text{Bu})_3$ -GPTS-KOBut-Hydrolyzate ($\text{Al-GPTS-KO}^t\text{Bu-H}$) and its calcined form (KAlSiO_4) were used to remove Cu(II) and Pb(II) ions from aqueous solutions by adsorption process. Different parameters such as contact time, initial pH, adsorbent amount, and initial metal ion concentration were studied, and CCD was applied for each metal ion and each material to optimize metal ions removal. According to results of some optimum adsorption conditions; with a determination coefficient (R^2) value greater than 97%; for Cu(II) and Pb(II) respectively, with hybrid material 91.82%-93.59%; and with calcined material 85.95%-86.36% metal removal values have been reached.

Keywords: Hybrid material, Heavy metal removal, Central composite design

INTRODUCTION

Hybrid materials with organic–inorganic character have been receiving attention most especially due to their unique properties and offer probability for many new applications in variable fields and industrial applications^{1,2}. One of the application areas of these materials is heavy metal removal from aqueous solutions. Especially in developing countries, industrial applications of toxic heavy metals in the last few decades have increased at fields as chemical manufacturing, painting and coating, mining, extractive metallurgy, nuclear and other industries. Because toxic heavy metal pollution has resulted in serious environmental problems for soils, water and living organisms as plants, animals and humans, removal of heavy metals has become a more important and wide working subject. Due to heavy metal removal process's characteristics are very complex, detecting the optimum values of removal conditions become significant to get the optimum removal efficiency. By way of this base, it is thought that process optimization is vital to determine the values of design parameters at which the response reaches its optimum. Response Surface Methodology (RSM) involves the multiple parameters and appertaining to reduced number of experimental trials needed to evaluate multiple parameters and their interactions; and Central Composite Design (CCD) is the most frequently used method of RSM^{3,4}.

MATERIALS AND METHODS

Reagents and Equipment

$\text{Al-GPTS-KO}^t\text{Bu-H}$ was used as prepared in literature¹. The stock metal ion solutions (1000 mg.L^{-1}) of Cu(II) and Pb(II) were prepared from their nitrate salts (Merck). For pH adjustment to experiment medium 0.1 mol.L^{-1} or/and 0.01 mol.L^{-1} hydrochloric acid and sodium hydroxide solutions were used. All the used chemicals were of analytical grade. A Perkin Elmer model Flame AAnalyst 800 Atomic Absorption Spectrometer was used for metal quantity analysis.

2.2. Adsorption process

For adsorption studies Cu(II) and Pb(II) metal ion's stock solutions were prepared as 1000 mg.L^{-1} using deionized water. Hybrid material and its oxide were used as adsorbents. On

the purpose of determine the utility of these materials as adsorbents, basic batch method was applied. The concentrations of the heavy metal ions in the filtrate were measured using flame atomic absorption spectrophotometer (FAAS), and requisite calculations were made according to equations below.

Removal percentage of adsorption, R (%), was calculated by using the following Eq. (1).

$$R(\%) = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (1)$$

The experiments were done with two replicates. The Minitab 16 computer software was used for regression analysis of the data obtained and to estimate the coefficients of the regression equations.

RESULTS AND DISCUSSION

The effects of factors such as initial pH, initial metal ion concentration and adsorbent amount on the removal efficiencies of the materials were investigated using RSM according to the CCD. Second-order models were generated to determine the removal efficiency of calcined and novel original organic-inorganic hybrid material for these ions and given below for the hybrid and calcined material, respectively.

Cu (%) = $55.149 + 14.269 \cdot pH - 11.500 \cdot C_0 + 6.770 \cdot m - 6.629 \cdot pH^2 - 4.978 \cdot m^2 - 3.953 \cdot pH \cdot C_0 - 4.384 \cdot$

$pH \cdot m$ Pb (%) = $84.409 + 17.122 \cdot pH - 3.761 \cdot C_0 + 3.394 \cdot m - 13.042 \cdot pH^2 + 3.517 \cdot pH \cdot C_0^2 + 4.156 \cdot pH \cdot m$

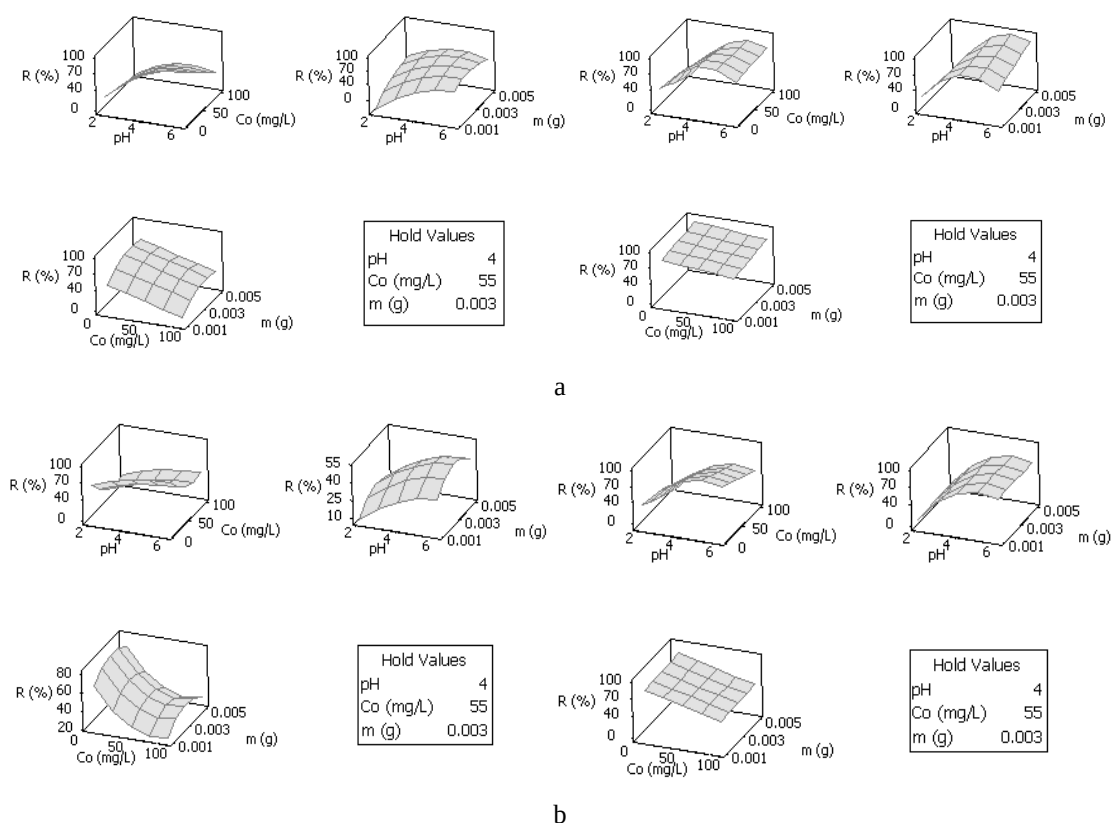


Figure 1. Response surface plot graphics for a) Hybrid Material with Cu(II) and Pb(II) b) Calcined Material with Cu(II) and Pb(II)

Cu (%) = $43.591 + 8.253 \cdot pH - 12.451 \cdot C_0 + 1.596 \cdot m - 2.442 \cdot pH^2 + 6.120 \cdot C_0^2 - 4.443 \cdot m^2$

Pb (%) = $70.066 + 17.802 \cdot pH - 9.963 \cdot C_0 + 0.593 \cdot m - 9.867 \cdot pH^2$

The shape of the fitted overall response was best summarized in graphs and was generated response surface plots for the fitted model (Figure 1). Response surface plots that graphically illustrate the effects of variables on the removal efficiencies of metals were drawn to determine the individual and cumulative effects of variables and the mutual interactions between them. In this study, pH was found to be the most important parameter and the largest positive main effect in hybrid and calcined materials for nearly all metals. This may be explained by the increase the number of negatively charged active sites on the surface of adsorbents at higher initial solution pH. It reveals that the metal removal decreased with increase in initial metal concentration and increased with increase in the pH up to 5.0 for almost all metals and materials. However, because C_0 has generally negative effect, it obtains lower metal removal at higher C_0 values.

References

- [1] A. Kayan, J Inorg Organomet Polym Mater. **25**, 6 (2015)
- [2] Y-H. Han, A. Taylor, M.D. Mantle, K.M. Knowles, J Non Cryst Solids. **353**, 3 (2007)
- [3] S. Pandey, B.S. Mishra, J Sol-Gel Sci Technol. **59**, 1 (2011)
- [4] D. Bingöl, Fresen Environ Bull. **20**, 10 (2011)

FT2-The Role of High-Energy Milling Applications on Leaching Performance

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ABSTRACT

Typical metallurgical methods used for processing laterite ores cause chemical consumption in large quantities in addition to challenging reaction conditions and thus environmental pollution¹. Therefore, new and more economical processing technologies should be developed for nickel recovery. This study was included a mechanical activation of lateritic ore then followed by an acid leaching, and aimed to overcome the disadvantages of the conventional metallurgical methods to achieve high-efficiency nickel recovery from lateritic ore at lower temperature and lower acid concentration. The optimization conditions for both of mechanical activation and atmospheric leaching processes were evaluated by response surface method (RSM). The results showed that the changes in ore structure during mechanical activation had a positive effect on acid leaching and high nickel recovery could be achieved in a shorter leaching time and lower acid concentration due to high-energy milling applications.

Keywords: Mechanical activation, Laterite ore, Leaching, RSM approach

INTRODUCTION

Mechanochemical processing, chemical reactions and phase transformations take place because of mechanical energy applied in high-energy milling. Mechanical activation generated by high-energy milling may break down crystals. Thus, fresh and active surfaces expose, which both enhance mass transfer to initiate chemical reactions and provide the required contact for solid reaction partners². Nowadays, mechanical activation by high-energy milling is an innovative procedure that improves the efficiency of mineral processing³. In this study, the effect of mechanical activation (MA) on the acid leaching (AL) was investigated for high efficiency nickel recovery from lateritic ore. For this purpose, mechanical activation and acid leaching studies were evaluated by RSM that was used to identify the optimal parameter settings, evaluate the results and efficiency of processes.

MATERIALS AND METHODS

Lateritic Ore

A low-grade nickel laterite provided from Kayseri (*Uzunpınar*)/Turkey was chosen to investigate the effect of the high-energy milling on leaching performance for nickel recovery.

Determination of MA and AL Conditions by Response Surface Method Approach

MA to be applied as a pretreatment and AL studies were carried out in accordance with the experimental plan generated by the CCD for the specified factors in order to obtain high efficiency nickel from the ore.

Table 1. Coded and actual levels of selected factors for CCD in MA

Factors	Symbols	Factor levels				
		$-\alpha$ (–1.68)	–1	0	+1	$+\alpha$ (+1.68)
time, t (min)	X_1	30	83	135	188	240
Ball/Ore, (B/O) ratio	X_2	5	10	15	20	25
Mill rotation speed, (RS) (rpm)	X_3	200	313	425	538	650

Table 2. Coded and actual levels of selected factors for CCD in AL after MA

Factors	Symbols	Factor levels				
		$-\alpha$ (-1.68)	-1	0	+1	$+\alpha$ (+1.68)
Acid concentration, (Co) (mol/L)	X_1	0.50	0.8	1.2	1.7	2.00
acid/ore ratio, (L/S oranı) (mL/g)	X_2	10	28	55	82	100
Temperature, (T) (°C)	X_3	25	39	60	81	95

RESULTS AND DISCUSSION

Statistical Evaluation of Results with Experimental Design Approach

This section contains the results of studies evaluated using RSM approach using Minitab® 17 software. The experimental data for MA and AL procedures were evaluated by RSM approach with variance analysis (ANOVA) in order to determine the statistical significance both the main effects and all possible interactions on the "Ni (%)" response variable of the investigated factors. The second-order model equations showing the relationship between the response variable and the factors were obtained with the reduced model obtained by neglecting the statistical significance of less than 95% (p -value greater than 0.05).

Optimization of Mechanical Activation (MA) Conditions

As a result of evaluation of experimental data for MA by ANOVA the binary interactions $X_1 \cdot X_2$ and $X_1 \cdot X_3$ were ignored at 95% confidence level.

Table 3. ANOVA results for MA

Terms	Coefficients	Standard Error	t-value	p-value	VIF
Constant	46.27	1.86	24.89	0.000	
time (X_1)	0.26	1.23	0.21	0.833	1.00
B/O ratio (X_2)	0.15	1.23	0.12	0.901	1.00
RS (X_3)	-1.47	1.23	-1.19	0.243	1.00
time*time (X_1^2)	-5.58	1.20	-4.65	0.000	1.02
B/O ratio*B/O ratio (X_2^2)	-3.80	1.20	-3.17	0.004	1.02
RS*RS (X_3^2)	-8.29	1.20	-6.90	0.000	1.02
time*B/O ratio (X_1 * X_2)	-0.52	1.61	-0.32	0.750	1.00
time*RS (X_1 * X_3)	-2.68	1.61	-1.66	0.107	1.00
B/O ratio*RS (X_2 * X_3)	-9.66	1.61	-5.99	0.000	1.00
$S = 6.45$	$R^2 = 71.81\%$	$R^2 (pred) = 56.62\%$	$R^2 (adj) = 78.32\%$		
The significant coefficients according to the reduced model:					
$S = 1.93$	$R^2 = 97.35\%$	$R^2 (pred) = 95.76\%$	$R^2 (adj) = 96.77\%$		

In this case, the second-order model equation obtained with the coded values according to the reduced model shows that the mill rotation speed is the most important factor on Ni (%).

$$\text{Ni (\%)} = 46.27 + 0.26X_1 + 0.15X_2 - 1.47X_3 - 5.58X_1^2 - 3.80X_2^2 - 8.29X_3^2 - 9.66X_2X_3 \quad (1)$$

As a result of activation conditions obtained by CCD in order to reach higher nickel recoveries, time (X_1), ball/ore ratio (X_2) and mill rotation speed (X_3) were found as 135 min, 15 and 425 rpm, respectively. Model and experimentally obtained nickel recovery were determined as $46.28\% \pm 1.93$ ($N=2$) and $45.99\% \pm 2.04$, respectively.

Optimization of Atmospheric Leaching Conditions

The ANOVA results given in Table 4 show that all experimental factors selected for AL are statistically significant on Ni(%), except for the $X_2 \cdot X_3$ binary interaction.

According to the reduced model temperature was determined as the most effective factor to achieve high yield nickel with AL.

$$\text{Ni (\%)} = 65.20 + 2.43X_1 + 1.57X_2 + 14.19X_3 + 0.92X_1^2 + 1.20X_2^2 + 2.99X_3^2 + 1.97X_1X_2 + 2.73X_1X_3 \quad (2)$$
 Optimum AL conditions for raw ore were found as 3.08 M H₂SO₄ concentration, 27.9 mL/g L/S ratio and 95 °C at 240 min. with estimated and experimental nickel recoveries were determined as 99.96%±1.18 and 97.99%±1.83, respectively. However, optimum AL conditions for mechanically activated ore were determined as 1.30 M H₂SO₄ concentration, 100 mL/g L/S ratio and 85 °C at 30 min with estimated and experimentally nickel recoveries 98.76%± 0.10 and 97.92%±1.83, respectively.

Table 4. ANOVA results for nickel recovery with AL after MA

Terms	Coefficients	Standard Error	t-value	p-value	VIF
Constant	65.20	0.687	94.85	0.000	
Co (mol/L) (X_1)	2.43	0.456	5.33	0.000	1.00
L/S ratio (X_2)	1.58	0.456	3.45	0.002	1.00
T (°C) (X_3)	14.19	0.456	31.11	0.000	1.00
Co (mol/L)*Co (mol/L) (X_1^2)	0.92	0.444	2.08	0.046	1.02
L/S ratio*L/S ratio (X_2^2)	1.20	0.444	2.70	0.011	1.02
T (°C)*T (°C) (X_3^2)	2.99	0.444	6.73	0.000	1.02
Co (mol/L)*L/S ratio (X_1X_2)	1.97	0.596	3.31	0.002	1.00
Co (mol/L)*T (°C) (X_1X_3)	2.73	0.596	4.59	0.000	1.00
L/S ratio*T (°C), (X_2X_3)	0.13	0.596	0.22	0.826	1.00
S= 2.38 R^2 = 97.32% R^2 (pred)= 94.15% R^2 (adj)= 96.52%					
The significant coefficients according to the reduced model:					
S= 0.10 R^2 = 100.00% R^2 (pred)= 99.99% R^2 (adj)= 99.99%					

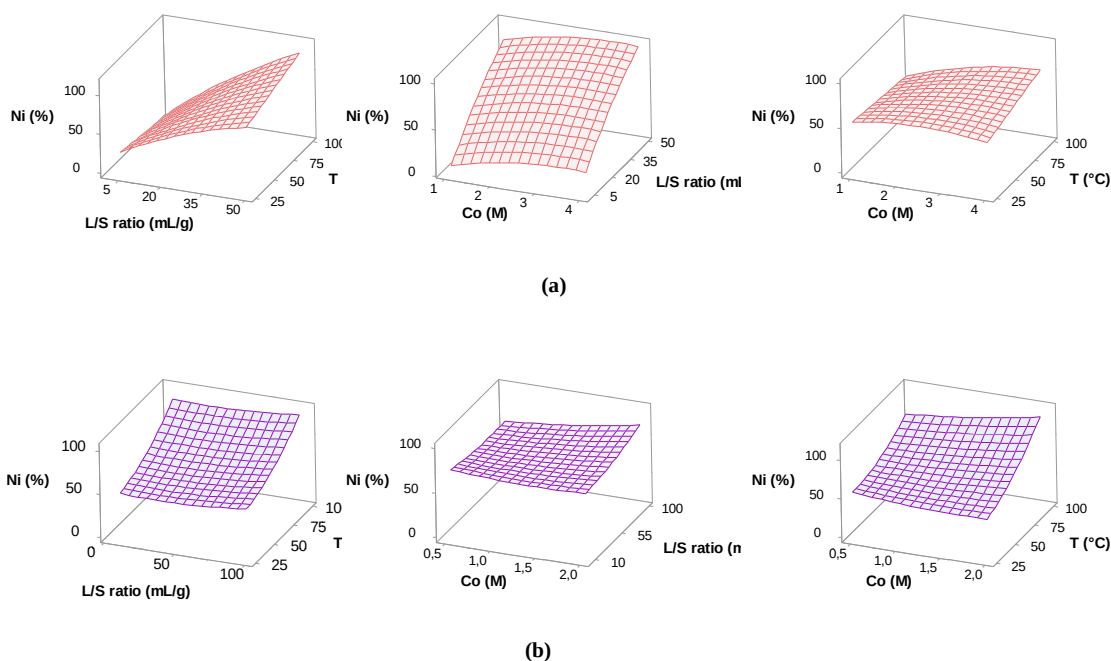


Figure 1. Surface plots for nickel recovery from (a) raw ore and (b) activated ore

CONCLUSION

The model equations for the MA and AL processes were obtained using the RSM approach. The high determination coefficients ($R^2 > 95\%$) of models indicated that CCD is a suitable design method for both MA and AL processes. The optimization results showed that the changes in ore structure during mechanical activation had a positive effect on acid leaching. High nickel recovery could be achieved in a shorter leaching time and lower acid concentration due to this effect.

Acknowledgement: We are grateful to TUBITAK (Project Number:116M076) for financial support.

REFERENCES

1. Gao J., Yan Z., Liu J., Zhang M., Guo M., A novel hydrometallurgical approach to recover valuable metals from laterite ore, *Hydrometallurgy* (2014) 150 161-166.
2. Baláž P., Baláž M., Bujňáková Z., Mechanochemistry in technology: from minerals to manomaterials and drugs, *Chem. Eng. Technol.* (2014) 37 747-756.
3. Apaydin F., Atasoy A., Yıldız K., Effects of Mechanical Activation on the Carbothermal Reduction of Chromite with Metallurgical Coke, *SAÜ. Fen Bilimleri Dergisi* (2010) 14 33–38.

FT3- Graphene and Molecularly Imprinted Polymer Based Sensor Production with One Step Electrochemical Procedure for Trinitrotoluene Detection

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Abstract

It is aimed to detect trinitrotoluene (TNT) using an electrochemical sensor based on graphene and molecular imprinted polymer (MIP). Picric acid served as a model molecule for TNT. The reduction of the synthesized graphene oxide and polymer formation were carried out electrochemically using cyclic voltammetry by scanning 15 cycles in the potential range of -1.0 to + 1.2V (vs Ag / AgCl) as a one step process. The methanol-acetic acid mixture was used to remove the repressed picric acid from the sensor and the experimental conditions were optimized. After the sensor was developed, square wave voltammetry, cyclic voltammetry, SEM and electrochemical impedance measurements were used for characterization. The prepared new composite film possessed the advantages of both polyaniline and reduced graphene oxide with a synergistic effect, which has excellent electrocatalytic activity.

Keywords: molecularly imprinted polymer, graphene, trinitrotoluene, picric acid.

INTRODUCTION

Conducting polymers which present π -conjugated structures are characterized by a high electrical conductivity¹. As specificity is concerned, molecular imprinting technology can be applied to the manufacture of these conducting synthetic polymers with pre-determined molecular recognition properties. MIPs are obtained through polymerization in the presence of a target molecule². Hence, highly specific cavities are created into the polymeric matrix, originating from shape and chemical functionality. After target removal, molecularly imprinted polymers show strong recognition properties towards the analyte.

Graphene, a two-dimensional form of carbon atoms organized in a closely packed hexagonal lattice structure, has been considered as one of the most important materials in the current century because of its unique nanostructure and extraordinary properties³.

The advantages of using molecularly imprinted polymer and graphene composites as sensing material can be expressed with respect to increased surface area, higher numbers of analytical recognition sites, low resistance and improved environmental stability, due to the unique properties of both components⁴. Most of the graphene is produced from exfoliated graphite oxide (GO) and its reduction thereof (rGO). Such rGO is very promising electrode material due to its heterogeneous electron transfer properties, unlike in high quality graphene⁵.

MATERIALS AND METHOD

Graphene oxide (GO) was prepared using modified Hummers method⁶. The reduction of synthesized graphene oxide and polymer formation were carried out simultaneously using cyclic voltammetry by scanning in the potential range of -1.0 to +1.2 V (vs Ag / AgCl) as a one step process. Square wave voltammetry, cyclic voltammetry were used for measurements between -1.2 and 1.0 V. The methanol-acetic acid mixture was used to remove the repressed picric acid from the sensor and the experimental conditions were optimized.

After the sensor was developed, voltammetry and SEM were used for characterization. Then, the electrode was tested for the sensitivity to picric acid and TNT.

A conventional three-electrode system was used for all electrochemical measurements: a carbon rod electrode (4 mm in diameter) served as the working electrode, an Ag/AgCl reference electrode, and a platinum wire was as the counter electrode. (GAMRY Instruments Reference 600 BAS Electrode stand CGME).

The reduction of the synthesized graphene oxide and polymer formation were carried out electrochemically using cyclic voltammetry by scanning in the potential range of -1.0 to + 1.2V (vs Ag / AgCl) as a one step process in 0.1 M phosphate solution (pH 7.0).

Electrochemical measurements to characterize the MIP film were carried out in the supporting electrolyte of 5 mmol L⁻¹ K₃ [Fe (CN)₆] solution containing 0.1 mmol L⁻¹ KCl at room temperature (25 °C). Cyclic voltammetry was performed from -0.2 to 1.0 V at a scan rate of 0.1 V s⁻¹. Square wave voltammetry (SWV) was used for measurements between -1.2 and 1.0 V. The methanol-acetic acid mixture was used to remove the repressed picric acid from the sensor. All experimental steps were optimized.

RESULTS AND DISCUSSION

Graphene and/or RGO are able to be prepared by an electrochemical method is a more convenient, effective and green route to produce RGO for preparing electrochemical sensors, since it can be performed at room temperature without any dangerous chemicals and RGO can be produced directly on the surface of nanostructured electrochemical devices.

Graphene oxide reduction and electropolymerization of a monomer in the presence of template molecule are usually performed by separate procedures. Here in this work electropolymerization of aniline and electrochemical reduction of graphene oxide were taken place simultaneously. Thus, we report a facile one step electrochemical method to prepare the polyaniline/graphene composite film modified carbon electrode.

Picric acid served as a model molecule. Carbon electrodes were used as the electrode instead of the commercial glassy carbon electrode.

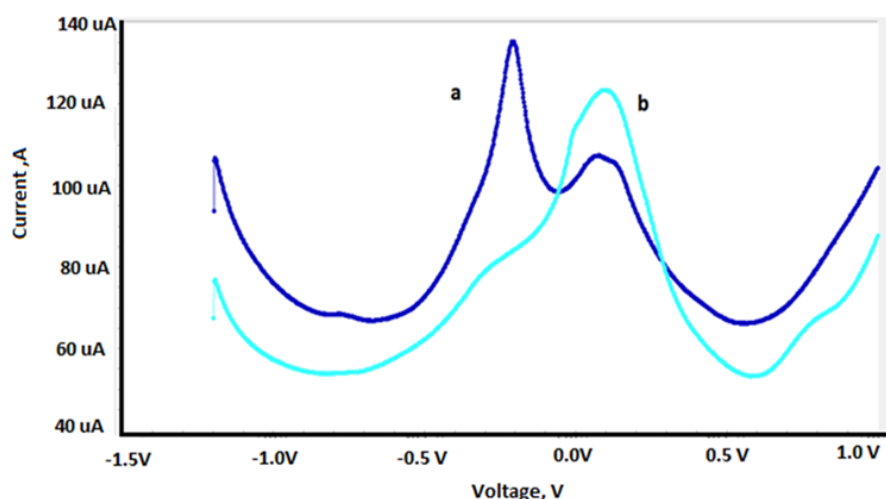


Figure 1. SWV measurements of graphene and molecularly imprinted polymer based sensor in 0.1 M phosphate buffer (pH 7.0) after incubation for one minute, a) Picric acid (10⁻³ M), b) TNT (10⁻³ M)

The prepared new composite film possessed the advantages of both polyaniline and reduced graphene oxide with a synergistic effect, which has excellent electrocatalytic activity.

Acknowledgment: This work is supported by Ondokuz Mayıs University (Project Number PYO.FEN.1904.17.001)

References:

1. Electroactive Polymer Electrochemistry. Part 2. Methods and Applications, M.E.G. Lyons (Ed.), Plenum Press, N.Y, 1996.
2. K. Haupt, K. Mosbach, Chem. Rev. 100 (2000) 2495.
3. M.J. Allen, V.C. Tung, R.B. Kaner, Honeycomb carbon: a review of graphene, Chem. Rev. 110 (2010) 132–145.
4. Wang, Y.; Li, Y.; Tang, L.; Lu, J.; Li, J. Application of graphene-modified electrode for selective detection of dopamine. Electrochem. Commun. 2009, 11, 889–892.
5. K.P. Loh, Q. Bao, G. Eda, M. Chhowalla, Graphene oxide as a chemically tunable platform for optical applications, Nat. Chem. 2 (12) (2010) 1015e1024.
6. Zhang, F. Zhang and H. Yang, Langmuir, 26, 6083 (2010).

FT4- Simultaneous Determination of Levodopa, Dopamine, Homovanilic Acid and 3,4-Dihydroxyphenylacetic Acid by using HPLC-UV system

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Abstract

A rapid, easy and non-laborous HPLC method was developed for the simultaneous determination of Levodopa, dopamine, 3,4 dihydroxyphenylacetic acid and homovanilic acid in artificial cerebrospinal fluid without any extraction process. The method exhibited a linear range between 0.05 to 2.0 mgL⁻¹ and limit of detection and quantification were calculated as 0.016 and 0.05 mgL⁻¹ for all the analytes. Overall results indicated that the method can be used for clinical purposes.

Keywords: HPLC, Catecholamines, Artificial Brain Fluid

INTRODUCTION

A wide range of neural pathways originated from catecholamines employ biogenic amines as neurotransmitters. The neurotransmitters in cerebrospinal fluid can be an indicator of neuronal functioning in nearby diencephalon structures. Hence, it is very crucial to quantify neurotransmitters and their metabolites level in the extracellular fluid for monitoring neurotransmission process [1].

Levodopa (L-Dopa) is the precursor of catecholamines which is widely used in the treatment of Parkinson's disease. Dopamine (DA) is responsible for the communications of nerve cells and one of its metabolites is 2-(3,4-Dihydroxyphenyl)acetic acid (DOPAC) in metabolic pathway to produce Homovanilic acid (HVA). A number of methods including spectrophotometry, gas chromatography with mass detection (GC-MS), high performance liquid chromatography (HPLC) coupled with various detection system, chemiluminescence, potentiometric, amperometric and voltammetric determination, radioimmunoassay and flow injection analysis (FIA) have been reported for their determination in various biological samples and pharmaceutical preparations. Among them HPLC provides a practical tool for sensitive, selective and accurate method for the quantification of dopamine precursor and metabolites [2-3].

This study describes an HPLC-UV method developed for simultaneous determination of levodopa, dopamine along with their metabolites 3,4 dihydroxyphenylacetic acid (DOPAC) and homovanilic acid (HVA). Optimization studies were conducted in order to achieve better peak resolution and peak symmetry and several types of analytical columns (C-18, C-8 and HILIC) have been used for this purpose. The effect of the pH and composition of mobile phase and dilution solvent on the peak characteristics have also been investigated. Then, the method was validated by using artificial cerebrospinal brain fluid.

EXPERIMENTAL

Reagents: Levodopa, Dopamine, DOPAC, Homovanilic Acid, Trifluoroacetic acid (TFA) and Acetonitrile are of analytical grade and used without further purification. Stock solutions (1000 mg.L⁻¹) of all analytes were prepared and stored at -20°C. The standard solutions were prepared freshly by diluting the stock solutions prior to study.

Chromatographic conditions: Initial studies included the use of several types of columns such as Phenomenex C18 Luna octadecylsilane 3 column (4.6 mm $\times$ 150 mm, 3 μ m) and Atlantis HILIC column (2.1 mm $\times$ 150 mm, 5 μ m) and Zorbax C-8 (4.6 mm $\times$ 150 mm, 5 μ m). The mobile phase consisting of 0.05% (v/v) TFA in water and acetonitrile was prepared from HPLC grade reagents. Experiments have been carried out by using gradient flow with a rate of 1.0 mL min⁻¹. The gradient elution was programmed as follows; 0 to 1.50 min 10% ACN, 1.50 to 9.0 min ACN ratio was increased to 30% and 9.0 to 11.50 min ACN increased to 50% and finally 11.50 to 15.0 min ACN was decreased 10%ACN. The analytes detected at 274 nm. The autosampler kept at 10°C and injection was 20 μ L.

Artificial Cerebrospinal Fluid Preparation: Artificial Cerebrospinal Fluid (ACF) was prepared according to former study [4]. The content of the ACF is 126 mM NaCl, 2.5 mM KCl, 2.0 mM CaCl₂ 2.0 mM MgCl₂, 1.25 mM NaH₂PO₄, 26 mM NaHCO₃ and 25 mM D-Glucose. The mixture was kept in +4°C.

Results and Discussion

Initial studies have been included the injection of mixed standard solution containing 5 mg L⁻¹ each analyte into the HPLC system packed with three different columns namely C-18, C8 and HILIC with aid of aqueous TFA solution and ACN with a flow rate of 1.0 mL min⁻¹. The best resolution was obtained with C-18 column for all the analytes while the other two columns have resulted badly convoluted peaks. Further studies were conducted with C-18 column for optimization of other chromatographic conditions including the composition of mobile phase. Here, trifluoroacetic acid is selected since it has less UV cut-off level but, its percentages in the mobile phase will change the pH which has an impact on the chromatographic peaks. Therefore, the chromatograms by using aqueous solutions of 0.01%, 0.05%, 0.075% and 0.1% TFA have been recorded by HPLC-UV system set at 274 nm. The best results in terms of peak resolution have been obtained with 0.05% TFA solution as given in Fig.1.

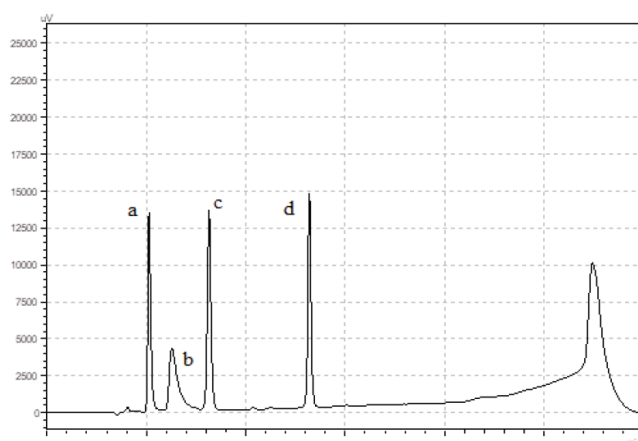


Figure 1. HPLC–UV chromatograms recorded for 5 mg L⁻¹ a) L-Dopa, b) DA, c) DOPAC and d) HVA by using C-18 column with a mobile phase solution of 0.05% TFA.

Following the optimization step, calibration studies have been carried out in sub ppm to ppm regions and the graphs have been found linear in 0.05 to 2.0 mgL⁻¹ concentration ranges with correlation coefficient close to unity. Method validation studies have been carried out in artificial cerebrospinal fluid. Stability studies were also conducted and up to 10 hours, no significant change in the signal was observed. The overall validation results were given in the Table 1.

Overall results have been evaluated as the method developed provides a selective and fast way for simultaneous analysis of the targeted analytes in clinical samples. Since the validation studies of the method have been carried out in artificial cerebrospinal fluid, it can be concluded that the method offers a practical tool for their determination in real samples.

Table 1. Analytical Merits of the Method

<i>Analytical Parameter</i>	<i>Levodopa</i>	<i>Dopamine</i>	<i>DOPAC</i>	<i>HVA</i>
Linear Range (mg L ⁻¹)	0.05 – 2.0	0.05 – 2.0	0.05 – 2.0	0.05 – 2.0
LOD (mg L ⁻¹)	0.016	0.016	0.016	0.016
LOQ (mg L ⁻¹)	0.05	0.05	0.05	0.05
Reproducibility (% at 0.075 mg L ⁻¹)	5.11	5.84	8.67	4.67
Recovery (% for 0.075 mg L ⁻¹)	88.9	93.3	91.2	82.4

Acknowledgements

This study was supported by the scientific and Technological Research Council of Turkey (TUBITAK) with a project number 114 Z 153.

References:

1. Bugamelli F., Marcheselli C., Barba E., Raggi M.A., Determination of L-dopa, carbidopa, 3-O-methyldopa and entacapone in human plasma by HPLC–ED, J. of Pharm. and Biomed. Anal. 54: 562–567
2. Goldstein, D.S., Kopin, I.J., Sharabi, Y., Catecholamine autotoxicity. Implications for pharmacology and therapeutics of Parkinson disease and related disorders, Pharmacology & Therapeutics 144: 268–282
3. Issa et al., 2011. “Application of high performance liquid chromatographic method for the determination of L-Dopa, carbidopa, and entacapone in tablet dosage forms” Issa, Y. M., Hassoun, M. E. M., Zayed, A. G. J. of Liquid Chrom & Related Technologies, 34:2433–2447.

FT5- Development of PPy-CNT-SPME Fiber Development and Their Use in Pesticide Determination in Apple Juice by GC-MS

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Abstract

Present study describes a direct immersion solid-phase microextraction (DI-SPME) method for the determination of selected pesticides used in apple production namely bifentrin, propargite, tebuconazole and tebufenpyrad prior to the GC-MS analysis. The SPME fiber was fabricated by using a stainless steel wire dipped into the voltammetric cell containing pyrrole monomer, tartrate solution and carbon nanotube mixture where it was subjected to electropolymerization by cycling the potential in a range of -0.5 - 2.0 V. The multiwall carbon nanotubes (MWCNT), amino-functionalized multiwall carbon nanotubes (NH₂-MWCNT), carboxylated multiwall carbon nanotubes (COOH-MWCNT) and fullerenes have been used for this purpose. Fiber surfaces have been characterized by SEM measurements and the analyses have been performed with GC-MS system in SIM mode.

Keywords: pesticide residue analysis, SPME, GC-MS, polypyrrole, carbon nanotube

INTRODUCTION

Pesticides are widely used in agricultural production and due to their harmful effects for the environment and human health; accurate and reliable methods are required for their analysis. Searching for a greener alternative to the traditional sample preparation techniques has prompted the development of solid phase microextraction (SPME) based on adsorption of organic analytes from liquid samples onto a polymeric film coated on a fused-silica fiber [1].

Commercial SPME fibers are available for a variety of analytes but, the fiber can be fabricated by electropolymerization of conducting polymers for this purpose [2]. In addition to their inherent porous structure fiber surfaces can also be modified with carbon based nano structures to increase the adsorption capacity [3,4].

Present study aims to develop a HS-SPME/GC-MS method for the analysis of four pesticides used in apple production namely bifentrin, propargite, tebuconazole and tebufenpyrad. Their maximum residue levels (MRL) have been reported as less than 0.3, 2.0, 1.0 and 0.1 mg/kg, respectively. Operational parameters effecting the extraction including the medium pH, adsorption time, desorption time and temperature have been investigated by using central composite design (CCD).

EXPERIMENTAL

Reagents: All the reagents used were of analytical reagent grade. Ultrapure water (18.2 MΩ/cm) from a MilliPore Milli-Q Gradient water purification system was used. Pesticides standards; bifentrin, propargite, tebuconazole and tebufenpyrad were purchased from Dr. Ehrenstorfer GmbH and stored at 4°C prior to use. Their stock solutions were prepared in acetone. Multiwall carbon nanotube (MW CNT) and its amino and carboxyl derivatives along with fullerenes have been purchased from Nanografi (Anara, Turkey). Apple juice samples have been obtained from local market and stored at 18°C before the analysis. The pH adjustments were made by using Orion 4 Star pH-meter.

Instrumentation: Electropolymerization studies have been carried out using a Metrohm Autolab PG Stat 101 voltammetric analyzer. The three-electrode system were completed with a stainless steel wire (316 type, i.d. 0.3 mm) as the working electrode, a platinum wire as the auxiliary electrode and a Ag/AgCl electrode as the reference electrode. The SPME holder for manual sampling was from Supelco (Bellefonte, PA, USA). Scanning Electron Microscopy (SEM) measurements of prepared SPME fiber were made by Zeiss Gemini Sigma 300VP. A heating magnetic stirrer, purchased from IKA-RCT (Germany) was used with a 10 mm stirring bar. Transsonic 460/H was used for ultrasonication purposes.

GC analyses have been carried out with Agilent 7890B Series coupled and 5977E MS detector systems and a HP 5MS column (30 m $\times$ 0.25 mm I.D. and 0.25 μ m film thickness). Helium (99.999%) was used as carrier gas. The injection port and detector were operated at 190°C and 250°C, respectively. The fiber was introduced into the chromatographic columns using splitless injection mode. Lab-made PPy-MWCNT modified SPME fiber inserted to GC injection port and GC analysis was started manually. The flow rate of carrier gas was adjusted at 1 mL/min. The separation of pesticides on GC-MS was performed by using a temperature program starting from 50°C for 5 min and increasing the temperature to 150°C at a rate of 25°C/min and then, to 220°C at a rate of 10°C/min and holding for 5min.

Fiber coating and conditioning: Polymeric composite films have been directly electrodeposited on the surface of a stainless steel wire electrode by using cyclic voltammetry. The wire surfaces have been subjected to ultrasonication in saturated solution of oxalic acid for 15 min and then, placed in acetone for 15 min for further cleaning.

The coating solution includes aqueous 0.35 M pyrrole and 0.2 M tartrate solution. In modification with carbon nanotube materials, 0.7 mg MWCNT, NH₂-CNT, COOH-CNT or fullerene have been added into the solution and the mixture was well dispersed under ultrasonication for 15 min. Electropolymerization was carried out by cycling the potential from -0.5 V to 2.0 V subsequently for 5 cycles at a rate of 50 mV/s. The fiber was then connected to SPME holder and then, the fibers have been conditioned at 200°C for 30 minutes.

The procedure for DI-SPME measurements: Apple juice sample was placed in a vial where the pH is adjusted to 8.0 by adding citrate buffer solution. Then, 10.0 mL of this sample was transferred into a 20.0 mL of a vial which was placed in a thermostatic cell set at 35°C. The SPME fiber was immersed in the solution for 10 min. After this step, the fiber was withdrawn into the needle and immediately introduced into the injection port set at 190°C for 22 min. All the measurements have been performed by tree replicates.

RESULTS AND DISCUSSION

First of all, surface morphology of polymeric composite coated SPME fiber was investigated by using SEM measurements at different magnifications. Figure 1 shows the close images of electrochemically prepared composite films on the stainless steel wire after five successive cycles in the potential range of -0.5 V to 2.0 V. As it is evident from Figure 1, all the fiber surfaces have displayed a rounded structure sized about 1 μ m while MWCNT modified PPy-SPME fiber has exhibited a thread-like structure more suitable for adsorption.

Initial studies include the selection of the medium pH and the fiber type according to their performances. The pH of apple juice samples was changed in a range of 3.0 to 9.0 by adding citrate buffer to adjust the desired pH and after extraction at 35°C for 10 min, the analyses have been performed with GC-MS system by desorbing the analyte at 210°C

for 5 min. According to the peak areas, pH 6.0 was selected and then, the performances of the four fibers developed have been compared under these conditions by spiking the pesticide standards into the apple juice sample. It was evident that MWCNT/PPy has given the best results for all the analytes. Further studies include chemometric approach by using this fiber.

In optimization studies, CCD was applied where the factors were chosen as pH, desorption temperature and time. Adsorption time was kept as 10 min. The surface plots between desorption temperature and pH; desorption time and pH and desorption time and temperature has revealed that maximum extraction efficiency was obtained when the extraction is performed in pH 8.0 medium and desorption was completed at 190°C for 22 min.

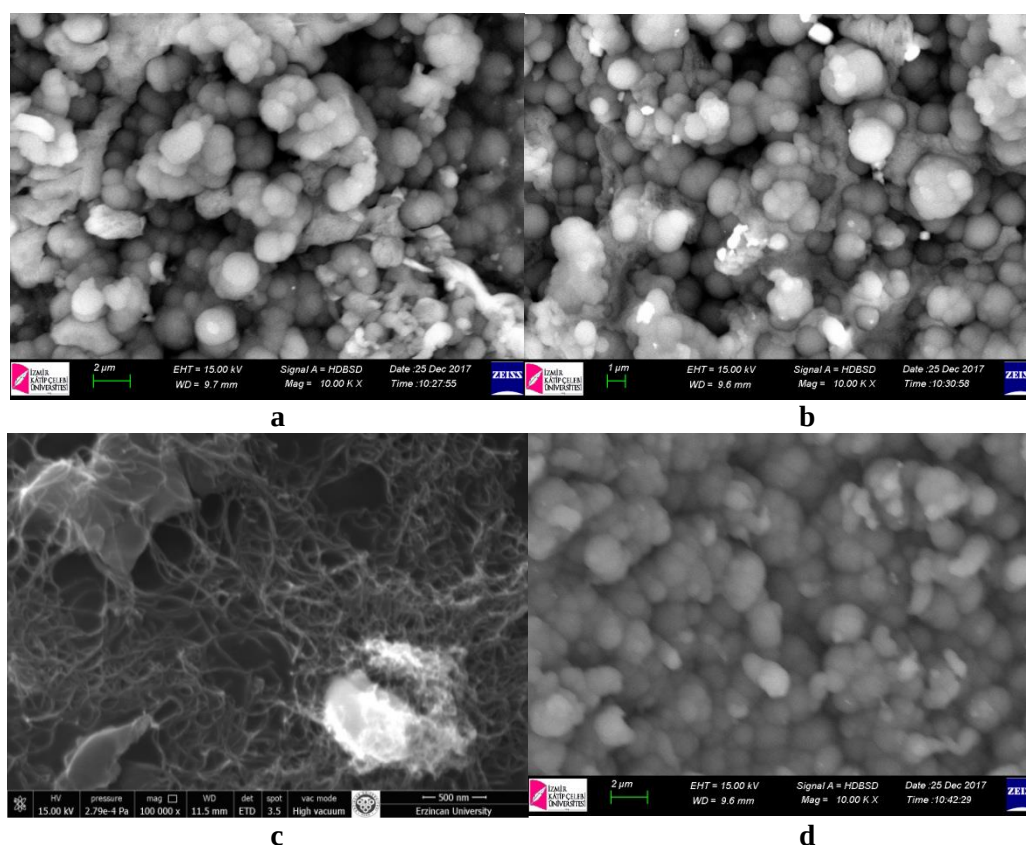


Figure 1. SEM images of a) COOH-MWCNT/PPy, b) NH₂-MWCNT/PPy, c) MWCNT/PPy and d) Fullerene/PPy coated on a stainless steel wire (x10 000).

Then, chromatograms recorded in apple juice sample after addition of pesticide standard and calibration graphs have been constructed for each pesticide. Among the pesticides studied, propargite has given two separate peaks and each was evaluated. Table 1 summarizes the analytical merits of the method.

Conclusion

The developed method was proven to be sensitive and LOD and LOQ have been calculated sub ppb levels for bifenthrin. Satisfactory recovery values have been obtained for all the pesticides in a range of 88.8-97.9 %. CNTs enhance the extraction capacity of SPME fibers probably due to the increased surface area. The calibration graphs drawn were found linear and LOD values were calculated at ppb levels. The method provides an economic and sensitive alternative for the determination of selected pesticides in apple juice samples.

Table 1. Analytical merits of the method developed for pesticides in apple juice

Pesticide	t _R (min)	m/z	Linear range (ng/mL)	LOD (ng/mL)	LOQ (ng/mL)	Recovery (%)
Bifenthrin	18.9	181, 165	0.5-15	0.17	0.56	5 ng/mL : 91.1 15 ng/mL : 97.9
Tebuconazol	17.9	125, 70, 83, 250	5-100	1.14	3.81	15 ng/mL : 88.8 50 ng/mL : 94.2
Propargite (1+2)	13.8/18.1	135	2.5-50	0.90	3.01	7.5 ng/mL : 92.3 25 ng/mL : 96.3
Tebufenpyrad	19.1	171, 318, 333	2.5-75	2.83	9.45	25 ng/mL : 95.2

Acknowledgement: Authors would like to thank Ege University Research Project (2015 FEN 032) for financial support.

References:

1. Arthur, C. L., Pawliszyn, J. "Solid phase microextraction with thermal desorption using fused silica optical fibers". Anal. Chem., 62 (1990) 2145-2148.
2. Korba, K., Pelit, L., Pelit, F. O., Özdokur, K. V., Ertaş, H., Eroğlu, A. E., Ertaş, F. N. "Preparation and characterization of sodium dodecyl sulfate doped polypyrrole solid phase micro extraction fiber and its application to endocrine disruptor pesticide analysis", J of. Chrom. B, 929 (2013) 90– 96.
3. Sun, M., Feng, J., Bu, Y., Wang, X., Duan, H., Luo, C. "Graphene coating bonded onto stainless steel wire as a solid-phase microextraction fiber" Talanta, 134 (2015) 200–205.
4. Chen, J. Zou, J., Preparation and evaluation of graphene-coated solid-phase microextraction fiber, Anal Chim Acta (2010) 678 44-49.

FT6- Development of Cobalt Oxide Modified Electrodes and Their Investigation in Electrocatalytic Applications

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Abstract

Recently, transition metal oxides are drawing interest due to their unique electronic and electrocatalytic characteristics. Cobalt oxides attract a special attention mainly due to their excellent electrocatalytic activity toward various compounds. This study concerns with electrochemical preparation of cobalt oxide decorated with platinum nanoparticle composite electrodes on a GCE surface in the pursuit of its catalytic applications. The parameters affecting the deposition conditions have been evaluated by monitoring oxygen reduction reaction (ORR) in pH 5.0 acetate buffer. Under optimal conditions, the electrode was used as a platform for a biosensor and polyphenol oxidase enzyme was immobilized onto the electrode surface for phenol detection. The medium pH and the enzyme amount have been optimized as pH 7.0 and 0.4 mg, respectively. Chronoamperograms were recorded for phenol detection in micromolar levels.

Keywords: Cobalt oxides, platinum nanoparticle, phenol detection, oxygen reduction reaction, Polyphenol oxidase

INTRODUCTION

Transition metal oxides are drawing special interest in the last two decades due to their unique electronic and electrocatalytic characteristics. Cobalt oxides attract considerable attention in view of their potential applications in energy conversion devices [1]. In addition, combination of hyper d-electronic transition metals with hypo d-electronic transition metals have displayed pronounced synergetic effect for both anodic and cathodic reactions [2].

Several methods have been developed for the preparation of cobalt oxide and among them; electrochemical method offers a practical way for tuning its catalytic properties. In the last decade, pulsed electrodeposition (PD) was emerged as an efficient technique since the pattern of applied potential determines the compositions and morphologies of metal oxide film [3].

This study concerns with electrochemical preparation of cobalt oxide and platinum nanoparticle composite electrodes on a GCE surface in the pursuit of its catalytic applications. Electrochemical deposition of the metal-metal oxide film structure has been carried out by both pulsed deposition and cyclic voltammetry. The parameters affecting the deposition conditions have been evaluated by monitoring oxygen reduction reaction (ORR) in pH 5.0 acetate buffer. Under optimal conditions, the electrode was shown to be utilized as a platform for a biosensor over ORR.

Phenol detection via biosensors is usually carried out by using purified enzymes such as tyrosinase also known as polyphenol oxidase. It is a copper-containing enzyme that plays a crucial role in melanin biosynthesis and in enzymatic browning of fruit and vegetables.

EXPERIMENTAL

Reagents and Apparatus: All the reagents used were of analytical grade. Ultrapure water (Milli Pore with 18.2 MΩ resistivity) was used throughout the study. Cobalt chloride hexahydrate (CoCl₂·6H₂O), sulfuric acid (H₂SO₄), acetic acid and sodium

hydroxide (NaOH) was purchased from Carlo Erba. Potassium tetrachloroplatinate was purchased from Aldrich. Chloroacetic acid buffer solution (CAA) was prepared from 0.1 M CAA and pH was adjusted as pH 3.0 with 3.0 M NaOH. Acetate buffers were prepared by using 0.08 M acetic acid and adjusting the pH to 5.0. Phenol was purchased from Merck. Glutaraldehyde (GA) solution (%25, v/v) was obtained from Sigma Aldrich.

Cyclic voltammetric measurements were performed by using Autolab PGSTAT with a three electrode system consisting of Ag/AgCl (sat. KCl) as the reference electrode, Pt wire as the counter electrode and GCE (BASi, 3 mm diameter and a surface area of 0.07 cm²) as the working electrode. Morphological characterization of the surface was made by using Philips XL 30 SFEG scanning electron microscope (SEM).

Preparation of CoOx modified Electrode: Prior to each experiment, the GCE was polished with 0.3 micron alumina slurry and treated with water and ethanol mixture in an ultrasonic bath. After rinsing with ultrapure water, the GCE surfaces have been modified with metal oxide film by using cyclic voltammetry or pulse deposition procedure. The metal oxide deposit was accumulated at the electrode surface by simply cycling the potential in the range of 1.0 –(-1.2) V for 40 times at a scan rate of 50 mV/s. The cell content was 0.1 M Co(II) solution in pH: 4.0 CAA/Acetate buffer. The electrode obtained (CoO_x/GCE) was then immersed into 1×10⁻³ M PtCl₄²⁻ solution prepared in 0.01 M H₂SO₄ solution and potential was cycled between -0.25 and 1.05 V with a scan rate of 10 mV/s for 20 times. The electrode obtained by this means was denoted as Pt/CoO_x/GCE. For comparison, Pt-GCE was also prepared from a solution containing 1×10⁻³ M K₂PtCl₄ in 0.01 M H₂SO₄ solution by cycling the potential with a scan rate of 10 mV/s for 20 cycles.

Similarly, pulsed deposition (PD) was applied in the accumulation step and resulting Pt/CoO_x/GCE was first immersed into the cell containing 0.1 M Co(II) in pH: 4.0 CAA/Acetate and the pulsed deposition program was applied where the potential was set -1.2 V for 3 s and then 1.2 V for 3 s sequentially. This process was repeated for 30 times. In the second step, CoO_x/GCE developed was immersed in 1×10⁻³ M PtCl₄²⁻ solution and a similar electrodeposition process was applied where the potential was set -0.25 V for 2 s and then 1.05 V for 2 s sequentially for 50 times. Then, the modified electrodes were placed in pH 5.0 acetate buffer de-aerated with nitrogen gas for background response and then, the buffer solution was saturated with O₂. The ORR signal is recorded by cyclic voltammetry in a potential range of 0.8 - -0.5 V at a rate of 50 mV/s.

Procedure in Biosensor Studies: 1.0 mg gelatin was weighted and solved in 25 µL pH 7.0 PBS by heating. Upon addition of 0.3 mg enzyme into the tube, it was dropped onto the Pt/CoO_x/GCE surface. The biosensor was allowed to dry at room temperature for four hours and then, it was exposed to a 2,5% (v/v) glutaraldehyde solution for 10 min at room temperature and rinsed with phosphate buffer solution. The performance of the electrode was tested by using synthetic waste solution spiked with phenol standard solution.

RESULTS AND DISCUSSION

Initial studies have been devoted to the optimization of the parameters affecting on the modification process of CoOx electrode by monitoring ORR signals. Then, the performance characteristics of the electrode developed have been compared with other electrodes to reveal any synergic effect of the composite structure.

The solution related parameters such as medium pH and Co(II) concentration were optimized. The medium pH used in CoOx deposition step was studied in a range of 2.0 - 6.0 by mixing CAA-Acetic acid and NaOH solution. Then, 0.1 M Co(II) solution was placed in the cell and either cyclic or pulsed deposition program was applied. The electrode (Pt/CoO_x/GCE) was transferred to pH 5.0 acetic acid buffer solution where the

ORR signal is recorded by cyclic voltammetry. It was concluded that the pH of CoO_x deposition solution has a strong effect on the subsequent formation of the ORR signal. The reduction peak currents of oxygen were plotted against the pH in CoO_x deposition step and it was observed that peak potentials shifted towards more positive potentials. The peak currents at 0.21 V have given a maximum at pH 4.0 and this pH was chosen for further studies. The other parameter is the Co(II) concentration which can determine the film thickness on the electrode surface. In a concentration range of 0.01- 0.5 M, 0.1 M Co(II) was selected as the optimum concentration.

Further studies have been conducted to see the effects of deposition conditions on the ORR signal. First, the metal oxide deposit on the electrode, denoted as CoO_x/GCE , was accumulated at the surface by simply cycling the potential in the range of 1.0 –(-1.2) V for 40 times at a scan rate of 50 mV/s. Then, the film electrode was immersed into 1×10^{-3} M PtCl_4^{2-} solution prepared in 0.01 M H_2SO_4 and the potential was cycled between - 0.25 and 1.05 V with a scan rate of 10 mV/s for 20 times. Similarly, in PD technique, the number and duration of the potential pulse applied to the electrode for deposition of CoO_x optimized as 30 pulses for 3 s and subsequent deposition of Pt np was and 50 pulses for 2 s, respectively.

The last parameter to be optimized is the scan rate on the ORR signal formation. As the rate was changed from 2 – 1000 mV/s, the ORR peak currents have linearly increased at lower scan rates and then, leveled off at high scan rates while the peak potential was shifted from 0.27 V to 0.128 V. Similar change can be observed with square rate of scan rate indicating a diffusion controlled current formation. The observed decrease in the ratio of peak current/ $v^{1/2}$ at higher scan rates is characteristic to the catalytic reaction.

Under optimal conditions, the surface morphology of the modified surface was also studied by using pencil lead electrode. The SEM images of the CoO_x/PLE and $\text{Pt}/\text{CoO}_x/\text{PLE}$, respectively at different magnification have revealed a homogeneous distribution of the platinum nanoparticles over the oxide film. The performance of the $\text{CoO}_x\text{-Pt}/\text{GCE}$ prepared by PD technique has been compared with bare GCE, CoO_x/GCE , Pt/GCE electrodes.

Figure 1 illustrates the cyclic voltammograms recorded for ORR in pH 5.0 acetic acid solution at all the electrodes for comparison. It is clear from the figure that bare and CoO_x deposited GCE has shown a poor effect towards ORR signal, but Pt np deposited GCE has given a high peak at 0.2 V as expected. On the other hand, depositing the Pt nanoparticles over the CoO_x film, obtained $\text{Pt}/\text{CoO}_x/\text{GCE}$ has given the best results. The enhanced catalytic activity of the electrode can be attributed to the high dispersion of Pt nanoparticles on the porous film surface. Interestingly, the composite electrode prepared by cyclic voltammetry has displayed higher ORR peaks than the electrode prepared by PD (Figure 1C). This can be attributed to the deposition of more platinum onto the electrode surface during slow scanning the potential in cyclic conditions as it is evident from the SEM images.

Finally, the potential of the electrode for bioanalytical applications have been searched. In phenol detection, the electrode developed was placed in a cell and the potential was set at 0.1 V where the oxygen signal can be traced. The oxygen left over from the reaction of Tyr with phenol was monitored and phenol can be determined indirectly. Before starting these experiments, the pH of the medium was checked and pH 7.0 PBS was selected as the optimum.

Upon addition of phenol standards into the cell the chronoamperogram recorded has displayed a series of sharp decreases in the ORR signal and calibration graph was constructed by measuring the current change. A linear range ($R = 0.999$) was obtained in micromolar levels of phenol and the RSD was calculated as 6,05% for 1×10^{-5} M. The

method was applied to the artificial waste water samples and linear graph was constructed between 5,0 – 35 μM phenol with an RSD level of 4.1% for low (5 μM), 4,7% for medium (20 μM) and 1,36% for high (30 μM) levels of phenol concentration. LOD and LOQ have been calculated as 0.39 μM and 1.18 μM , respectively.

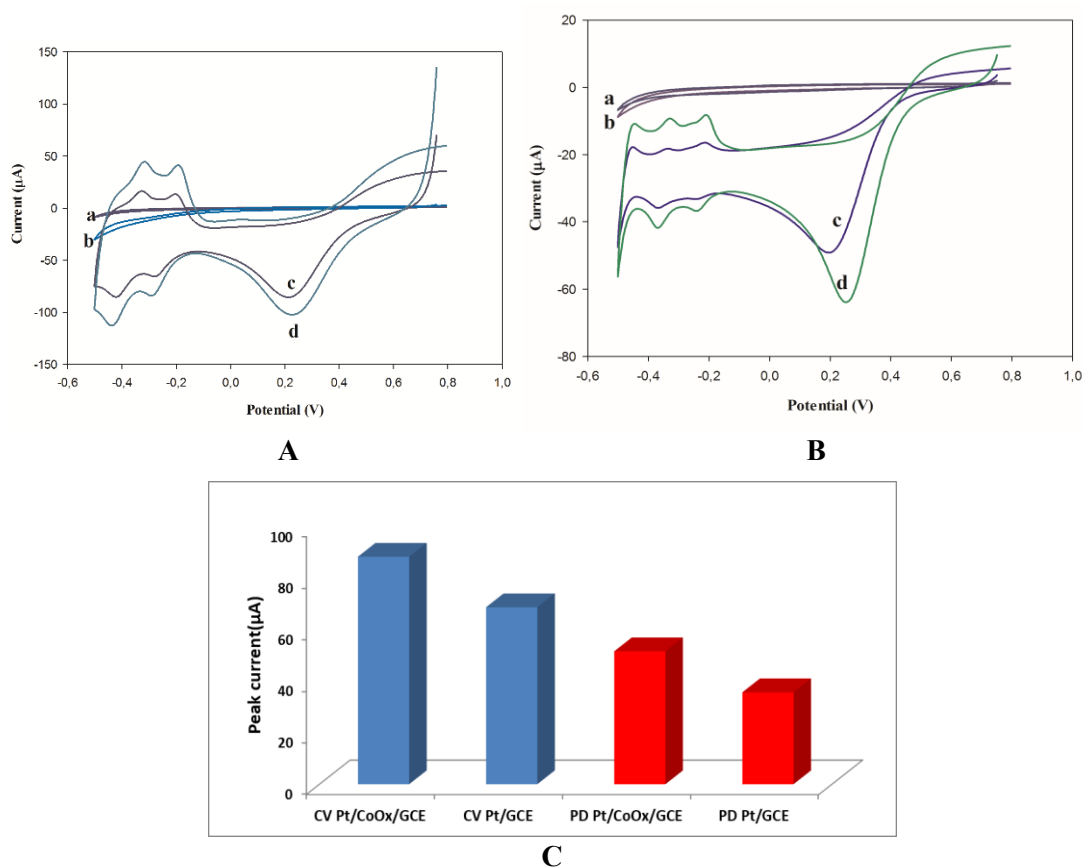


Figure 1. Cyclic voltammograms recorded for ORR in pH 5.0 Ac buffer at a) bare GCE, b) CoOx/GCE c) Pt/GCE and d) Pt/CoOx/GCE prepared by A) cyclic voltammetry, B) pulsed deposition, C) Comparison of the performances of the electrodes prepared by CV and PD

In conclusion, catalytic activity of the Pt/CoOx/GCE electrode towards reduction of dissolved oxygen was found better with an electrode prepared by subsequent deposition of CoOx and Pt by cycling the potential rather than the pulsed deposition. As the electrode was used as a platform for tyrosinase immobilization, the biosensor obtained has given very sensitive results for phenol detection.

Acknowledgement: Authors would like to thank E.U. Scientific Research Project (2017 FEN 061) for financial support.

References:

1. Casella, I.G., J. of Electroanal. Chem., 520 (2002) 119–125
2. Jordanov, S. H., Popovski, P.O., Dimitrov, A., Slavkov, D., Bull. of the Chem. and Tech. of Macedonia, 23 (2004) 101–112
3. Lee, J., Jeong, B., Ocon, J.D., Current Applied Physics 13 (2013) 309–321

FT7-Synthesis, Characterization and Swelling Behavior of Poly (Acrylic Acid-co-N-vinyl 2-pyrrolidone) Copolymeric Hydrogels

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Abstract

In this work, acrylic acid and vinyl pyrrolidone were preferred as monomers because of their biodegradability. Hydrogels were obtained in the presence of a radicalic initiator (APS). Therefore, copolymeric hydrogels were obtained under optimum conditions and synthesized by free radical polymerization technique. Swelling (%S) values of the hydrogels were measured during 56 h. The surface morphologies of the hydrogels were shown by SEM analysis.

Keywords: Hydrogel, Acrylic acid, N-vinyl 2-pyrrolidone, swelling behavior.

INTRODUCTION

Hydrogels are preferred in many application fields because of their porous and water-loving structures. An approach to strengthening polymeric hydrogels and enhancing their functionality. The water content in the hydrogels affects different properties like permeability, mechanical properties, surface properties, and biocompatibility¹. Hydrogels have physical properties similar to that of living tissue, and this similarity is due to the high water content, soft and rubbery consistency, and low interfacial tension with water or biological fluids^{2,3}. The polymerization is initiated by UV, by redox initiator system or thermally⁴.

EXPERIMENTAL

Preparation of Hydrogels

The crosslinked cylindrical hydrogels were prepared by using AA, VP monomers in various ratio, in PVC pipettes. Each hydrogel was obtained by selecting 3 different monomer-monomer quantities (75:25, 50:50, 25:75 (v:v)). The solution of hydrogel was heated with temperature-controlled heater on 80°C, 2 hours. Then, the pipettes were carefully stored in equal volumes (3-4 mm) cut into cylindrical discs. The hydrogels were dried in a room conditions before vacuum oven (35°C).

RESULTS and DISCUSSION

Swelling Behavior

Swelling behavior of copolymeric hydrogels were given in Fig.1. Best swelling behavior at the end of the constant value (48 h) was seen in 75:25 (AA:VP) copolymeric hydrogel.

SEM Results

Morphological structure of hydrogels were characterized by SEM images. Before and after lyophilization images of 75:25 (AA:VP) copolymeric hydrogels were shown in Fig.2.

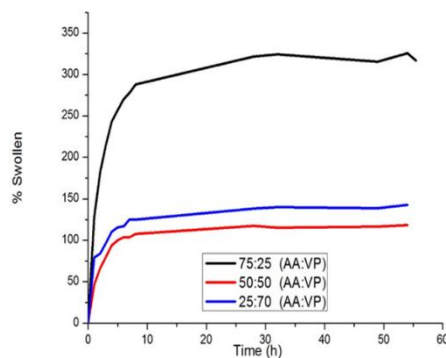


Fig.1. Swelling behavior of copolymeric hydrogels

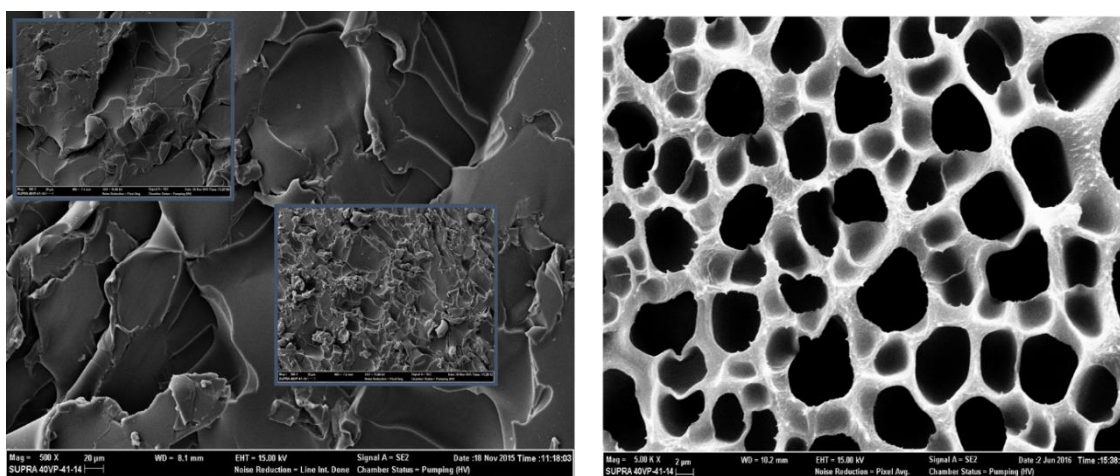


Fig. 2. SEM pictures of AA hydrogel (before and after lyophilization)

CONCLUSION

The swelling behaviors and SEM images improved the synthesis of 75:25 (AA:VP) copolymeric hydrogels. Best swelling, elasticity and flexibility was obtained in this hydrogel. To better understanding, lyophilization process was applied to hydrogels. Therefore, the pore diameters were seen clearly. The pores of hydrogels distributed homogenously. In the future, this copolymeric hydrogels will be modified with inorganic materials to obtain for various applications.

ACKNOWLEDGMENTS

This work was supported by Scientific Research Project Commission of Bilecik Seyh Edebali University (project number is 2017-01.BŞEÜ.28-01). FT-IR and FESEM measurements were performed in Bilecik Seyh Edebali University Central Research Laboratory.

References

- ¹Gökmen, F. Ö, Pekel-Bayramgil N., Eur. Chem. Bull. (2017) 6(11) 514-518.
- ²Blanco, M. D., Garcia, O., Trigo, R. M., Teijon, J. M. and Katime, I., Biomaterials, 1996, 17 (11), 1061-1067.
- ³Ahmed, E. M., J. Adv. Res., 2015, 6 (2), 105-121.
- ⁴Hamidi, M., Azadi, A., Rafiei, P., Adv. Drug Del. Rev., 2009, 60 (15), 1638-1649.

FT8-Adsorption Of VOCs Onto Activated Carbon And Biochar Obtained From Textile Wastes For Their Determination By GC-MS

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Abstract

Present study deals with the adsorption of selected VOCs onto the activated carbon and biochar materials obtained from textile wastes prior to their GC-MS determination. Adsorptive performances of activated carbon and biochar from cotton/polyester and acrylic wastes have been compared with commercially obtained activated carbon and nanocellulose material. The adsorbents have been exposed to the mixed gas standards including toluene, ethylbenzene and xylene in a vial kept at 25°C for 15 min and then, the VOC content of adsorbent was thermally desorbed in the autosampler set at 60°C for 5 min. The sample withdrawn from the head space of the vial was injected into the GC-MS system for subsequent separation in a wax column. The chromatograms recorded have revealed that cotton/polyester biochar adsorbent has given the best response for the majority of analytes according to their peak areas. Calibration graphs constructed for all the targeted analytes has revealed that very sensitive analysis can be performed well below the short term exposure level (STEL) values determined for workplaces including the paint sector.

INTRODUCTION

Volatile organic compounds (VOCs) are considered as a fundamental parameter for assessing the air quality because of their toxicity and precursor role in photochemical smog. BTEX refers to the chemicals benzene, toluene, ethylbenzene and o-, p-, and m-xylenes, all severely threaten human health and the environment due to their toxic, mutagenic, and carcinogenic effects [1]. Monitoring of their levels in workplace atmosphere is of great importance in terms of occupational health and safety. Adsorptive enrichment on several adsorbents is often used to combine active or passive sampling. A great deal of research has been conducted to investigate the adsorption properties of VOCs onto a variety of materials including zeolite, polymeric or carbon based adsorbents [2]. Engineered carbonaceous materials have been reported to display excellent VOC adsorption ability because of their large surface area, plentiful pore structure, high stability, as well as relatively low cost [3]. Activated carbon (AC) is produced from carbon-rich materials such as lignite, wood etc. by the processes of carbonization and activation while biochar is produced from biomass by slow pyrolysis at the temperatures lower than 700°C under inert atmosphere [4]. Although biochar has been widely used in various fields, a limited number of researches have been done for VOC vapor adsorption [3].

Present study deals with the use of novel carbon materials as adsorbents for selected VOCs Activated carbon and biochar obtained from cotton/polyester and acrylic wastes along with commercial activated carbon and nano cellulose materials have also been used for comparison purposes.

EXPERIMENTAL

Apparatus and Reagents: All the reagents used were of analytical grade. Mixed standard solution (500 mg/L) of toluene (Sigma Aldrich), ethyl benzene (Acros Organic) and xylene isomers (Alfa Aesar) were prepared in methanol (Sigma Aldrich). Activated carbon and biochar materials from textile wastes were produced in a 1 L volume of fixed bed reactor made of stainless steel. For biochar production, 15-25 g of cotton/polyester polyester (%50:%50, w/w) and acrylic wastes cut in pieces about 2 cm was loaded into the reactor and then heated at a rate of 5°C/min

up and held at 350–400°C for 30 min under nitrogen atmosphere. Volatile products passed through ice-bath at where liquid products collected and remaining gases was collected in a Tedlar bag. Activated carbon was obtained from biochar by chemical activation. The biochar was impregnated with ZnCl_2 solution at a ratio of 1:1 and after drying, the chars was carbonized at 500°C under nitrogen atmosphere for 2 hours. The char was boiled with 200 mL of 10% HCl solution for 60 min, filtered in a vacuum flask, and washed with hot water and finally with cold water to remove the chloride ions and other inorganics. After washing, activated carbon samples were dried at 110 °C for 24 h and weighed. Commercial activated carbon (Merck) and nano cellulose materials (Nanografi Inc.) were also used for comparison. Close images of the adsorbents has been recorded by using the SEM (Carl Zeiss 300 VP). Adsorption studies have been carried out by using Nüve BM 402 cryostat. Gas chromatographic analyses were performed with Agilent Technologies Model 7890B Series GC system equipped with PAL RSI 85 autosampler and 5977E MS detector systems. A DB-Wax (60m x 0.25mm x 0.15 μm) column was used. Helium was used as carrier gas with a flow rate 1.5 mL/min. The inlet and detector have been operated at 200°C and 250°C, respectively. The gas sample (1 mL) was injected in split mode (1:50) and the temperature program was started by holding at 40°C for 5 min and then, rising to 150°C by a heating rate of 5°C/min.

Procedure: Each adsorbent weighed as 20 mg in a small glass cap was placed in a 20 mL GC vial where it was exposed to gas standards evaporated from injected 10 μL of mix standard solution at 25°C for 15 min. Then, the cap was transferred into the of autosampler vial for thermal desorption of the VOC content at 60°C for 5 min. 1 mL of sample withdrawn from the head space of the vial was injected into the GC-MS system for subsequent separation in a wax column. Head space gas sample was injected into the GC-MS system.

RESULTS AND DISCUSSION

Initial studies have been devoted to the calibration studies of gas phase standards injected into the GC-MS system. Well separated peaks were observed for all the analytes 125 mg/m^3 each of which was withdrawn from the headspace of standard solution incubated at 60°C for 5 min. Table 1 lists the retention times (t_R) observed and mass to charge ratios (m/z) used for the analytes. Calibration graphs were constructed in 1.25–25 mg/m^3 range for all of the analytes with a correlation coefficient close to unity. Then, the experiments were repeated in the presence of the adsorbents.

The procedure given in experimental section was applied for targeted analytes at their short term exposure levels (STEL). The blank corrected peak areas obtained for each analyte were plotted against the adsorbent types as shown in Figure 1. Interestingly, the performances of the commercial and lab-made- activated carbons for VOC adsorption were found to be poor in comparison to the biochar and nanocellulose materials.

Table 1. Chromatographic characteristics of targeted analytes by direct injection method

	Toluene	Ethylbenzene	p-Xylene	m-Xylene	o-Xylene
t_R (min)	8.723	11.343	11.578	11.789	13.146
m/z	91.92 39.65	91.106 51.65	91.106 105.77	91.106 105.77	91.106 105.77

Generally, large specific surface area and small pore size facilitate the adsorption but, it should be noted that surface chemical functional groups are also crucial factor for VOC adsorption. Therefore, the biochar rich in functional groups on rather large pore size might result in enhanced adsorption of analyte depending on its polarity. For the majority of analytes, the best results were obtained by cotton/polyester biochar and this adsorbent was used for further calibration studies. Analytical performances of the HS/GC-MS method were evaluated for all the analytes (Table 2).

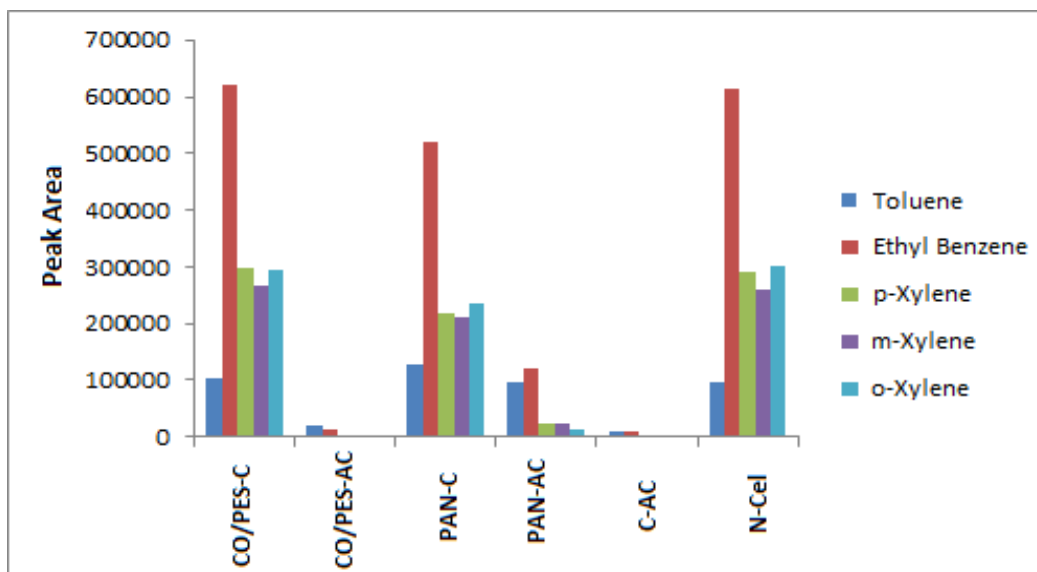


Figure 1 Performance comparison of the adsorbents used for 15 min exposure to selected VOCs at 25°C and desorbed at 60°C for 5 min.

Table 2 Analytical merits of the HS/GC-MS method carried out with cotton/polyester biochar

VOC type	Dynamic Range (mg/m ³)	R ²	RSD (%) (75 mg/m ³)	LOD (mg/m ³)	LOQ (mg/m ³)	STEL for 15 min (mg/m ³)
Toluene	75-175	0.9781	19	22.52	68.26	384
Ethyl Benzene	75-175	0.9926	22	13.02	39.47	884
p-Xylene	75-175	0.9922	19	13.32	40.36	442
m-Xylene	75-175	0.9923	16	13.24	40.14	442
o-Xylene	75-175	0.9960	19	9.55	28.95	442

According to the results of HS/GC-MS method, the biochar adsorbent used has displayed a superior adsorption performance for the BTEX analysis. However, another important aspect of the adsorbent to be considered is the quick desorption ability for subsequent measurement. Therefore, it was tested upon withdrawing a second and third injection from the headspace of the adsorbent after thermal desorption. The peak area values have been compared and it was calculated that only a less than 0.4% of the peak is remained on second injection indicating the complete removal of the VOC from the adsorbent by thermal desorption.

Conclusion

Carbon-based adsorbents obtained from textile wastes can serve as an economic and renewable material for sensitive and selective analysis of VOCs. The biochar materials have great potential to be used in VOC adsorption because of its excellent adsorption efficiency and low cost. These materials can also be used as passive sampler systems for monitoring the BTEX level in workplace atmosphere which is of great importance in terms of occupational health and safety.

Acknowledgement: Authors would like to thank Ege University Scientific Research Project (2016 FBE 013) for financial support.

References

1. EPA, 2017. <https://www.epa.gov/indoor-air-quality-iaq>, Indoor Air Quality (IAQ), Volatile Organic Compounds' Impact on Indoor Air Quality Access date: 19 Jan 2017.
2. CATC Technical Bulletin Choosing An Adsorption System For VOC: Carbon, Zeolite, Or Polymers? EPA-456/F-99-004 May 1999
3. X. Zhang, B. Gao, A.E. Creamer, C. Cao, Y. Li, Adsorption of VOCs onto engineered carbon materials: A review, *J. of Hazardous Mat.* 338 (2017) 102–123
4. M. Ahmad, A.U. Rajapaksha, J.E. Lim, M. Zhang, N. Bolan, D. Mohan, M. Vithanage, S.S. Lee, Y.S. Ok, Biochar as a sorbent for contaminant management in soil and water: a review, *Chemosphere* 99 (2014) 19–33.

FT9-Development of a Noninvasive Method for Biomarker Determination in Exhaled Breath for Early Diagnosis of Lung Cancer

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Abstract

Early diagnosis of lung cancer is one of the most important strategies to reduce morbidity rate and improve the survival rate. Invasive techniques alternative to the current screening techniques are being searched extensively. Exhaled breath analysis is a relatively new strategy for early cancer screening and detection of molecular bio markers specific for lung cancer however; direct sampling of breath does not provide the necessary detection limits. Therefore; enrichment techniques are being searched for development of reliable sampling techniques. Present study describes a new sensitive and reliable method for the determination of biomarkers of lung cancer in human breath sampled by a solid phase microextraction (SPME) fiber. For this purpose, exhaled breath samples of the lung cancer and asthmatic control groups were collected on the lab-made SPME fiber and then, injected into GC-MS system for thermal desorption and subsequent analysis. Following the optimization studies, the VOCs were assessed using a chemometric approach using principle component analysis (PCA). A good classification of cancer cases and healthy volunteers for lung cancer diagnosis has been obtained. It has been shown that SPME fibers prepared in our research laboratory can be used for early detection of lung cancer.

Keywords: Breath analysis, SPME, Chromatography, Lung Cancer

INTRODUCTION

Lung cancer is the most common kind of cancer in the world and it is a leading cause of cancer death by late diagnosis. Early diagnosis is one of the most important strategies to reduce lung cancer morbidity rate and improve the survival rate. To improve the overall survival, many screening methods including tomography and radiography have been used. However, current screening techniques for lung cancer are always expensive and not comfortable, even harmful for patients.

The detection of molecular bio markers specific for lung cancer in exhaled breath is a relatively new strategy for early cancer screening and detection. This technique is non-invasive, painless, easy to perform and no risk to patients. Exhaled breath contains about 3500 different kinds of volatile organic compounds (VOCs) [1,2].

Breath analysis has taken a long time to be a useful tool because of the lack of standardized breath collecting methods and the low concentrations of VOCs which are present in nano or pico-molar level. On the other hand, the direct sampling does not provide the necessary detection limits therefore; there is a need for development of reliable sampling techniques for breath analysis.

Present study was designed to develop a new sensitive and reliable method for the determination of biomarkers of lung cancer in human breath by SPME sampling. Lab-made fibers were placed in the breath sampling device to enrich the VOCs in exhaled breath samples from of the lung cancer and asthmatic control groups. Then, the fibers have been injected into the GC-MS system for subsequent analysis. Considering the

extensive data obtained, principle component analysis (PCA) has been used to classify the cases.

EXPERIMENTAL

Apparatus and Reagents: Chromatographic experiments have been performed by using an Agilent Technologies Model 7890B Series GC system and 5977E MS detector systems. A DB-624 (60m x 0.25mm x 0.15 μ m) column was used. Helium was used as carrier gas with a flow rate 1.5 mL/min. The inlet has been operated at 250°C. The temperature program was started as followed 35°C for 5 min rising to 280°C by ramp of 25°C/min and hold for 5 min.

SPME Fiber Production: Stainless steel wire was cleaned by ultrasonication for 15 min. in acetone and placed in a voltammetric cell where three electrode system was completed by Ag/AgCl electrode as the reference and Pt spiral as the auxiliary electrode. Thiophene was added into the cell 0.1 M of LiCl containing acetonitrile solution and purged with nitrogen gas for 5 min. Electroplating of the steel wire was maintained by cycling the potential between -0.2 to 2.2 V for subsequent 50 scans at a rate of 50 mV/s.

Subject Distribution and Procedure: Exhaled breath samples of 70 lung cancer and 106 control groups were taken in cooperation with Ege University, Faculty of Medicine and total 176 cases have been evaluated. A lab made sampling device was used for the VOC samplings. 30 L exhaled breath passed thorough fiber at 38°C. Desorption of the adsorbed VOCs was maintained at 250°C for 5 min.

Data Analysis: Partial Least Squares- Discriminant Analysis (PLS-DA) was applied to entire data. By using the coefficients obtained from PLS-DA model, determination of the peaks having high contribution to classification became possible.

RESULTS AND DISCUSSION

Two different SPME sampling procedure namely constant time and constant volume were applied. The classification between lung cancer and healthy wasn't obtained by constant time SPME sampling. Therefore, studies were carried out by constant volume SPME sampling. A sample chromatogram of SPME-GC-MS results was given in Figure 1.

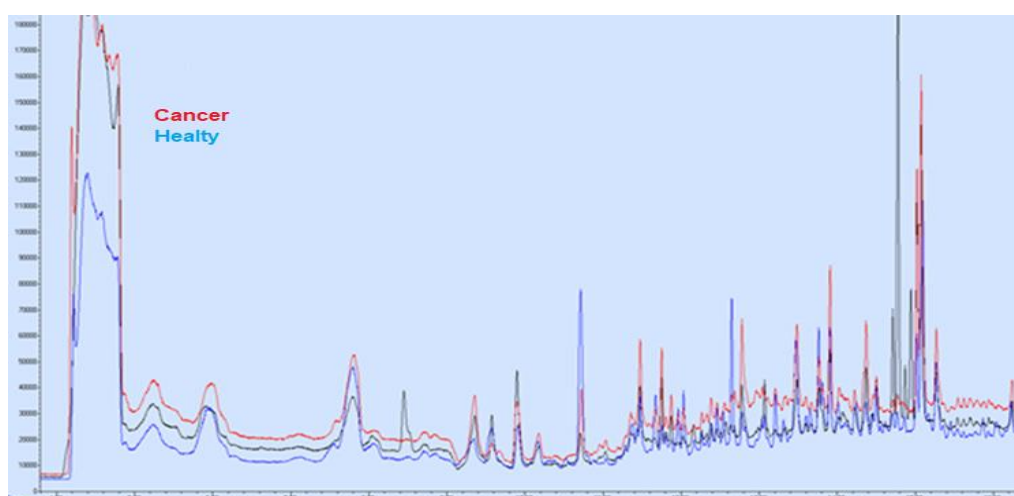


Figure 1. Sample chromatogram of SPME-GC-MS

As can be seen from the Figure 1, variety of compounds were identified in the chromatograms. All MS spectrum of healthy control and lung cancer patients were classified by PCA analysis. After contracting PLS-DA model using full spectrum, the contribution of each data point was calculated. As can be seen from the Figure 2, there are lots of potential biomarkers. The sample chromatogram and its distribution of potential biomarker for healthy control and lung cancer patients was given in Figure 3.

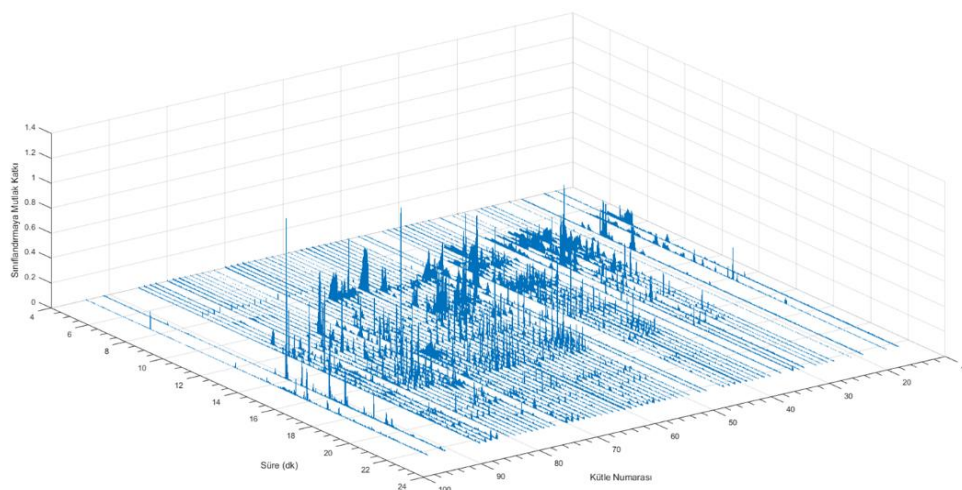


Figure 2. PLS-DA analysis of potential biomarkers

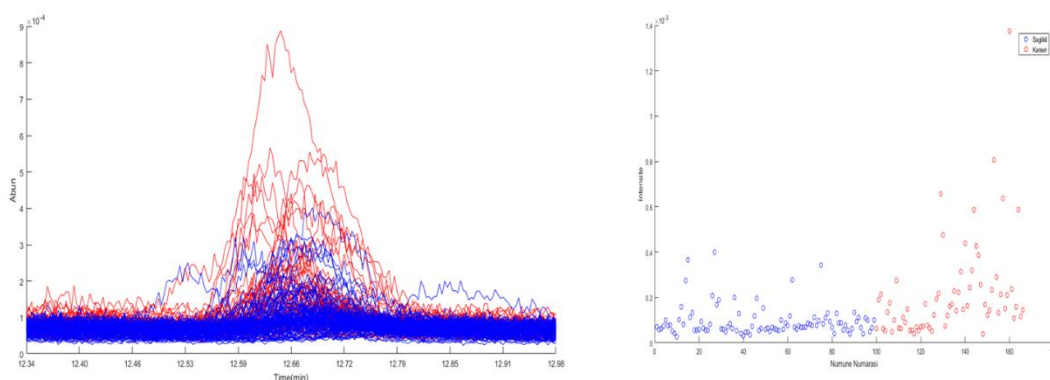


Figure 3. Sample chromatogram and its distribution of potential biomarker for healthy control and lung cancer patients

A good classification of cancer patients and healthy volunteers for lung cancer diagnosis has been obtained. Classification accuracy for healthy controls and lung cancer patients were 72%. It has been shown that SPME fibers prepared in our research laboratory can be used for early detection of lung cancer.

This study was supported by Tübitak (113Z672).

References:

- 1 B. Buszewski, M. Kêsy, T. Ligor, A. Amann, Biomed. Chromatogr. **2007**, 21, 553-566.
- 2.T.H. Risby, Ed. A. Amann, D. Smith, World Scientific, **2005**.

FT10- Fast, easy and green method employing magnetic dispersive solid phase extraction for the determination of pesticide residues

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Abstract

Pesticides are widely used in agricultural production nevertheless, their residues in food and environmental samples are harmful to human health because of their potential mutagenicity and carcinogenic properties. Their extensive use in world-wide agricultural practice in addition to industrial emission during their production has led to substantial occurrence of pesticide residues. For these reasons, the evaluation of pesticide residues is nowadays a priority objective to ensure safety and quality.

In this work the information about novel green extraction methodological approach including magnetic solid phase extraction (MSPE) is presented. A $\text{Fe}_3\text{O}_4/\text{Ni}/\text{Ni}_x\text{B}$ nanocomposite magnetic adsorbent was synthesised [1] and used for the determination of Bromopropylate (BRP) and Lambda-Cyhalothrine (LMD) pesticides in well water, wine and grape juice samples by MSPE method. Characterisation of the adsorbents was performed using scanning electron microscopy combined with energy dispersive X-ray spectroscopy (SEM-EDX). The parameters related to the extraction efficiency were screened and the performance of extraction of pesticides were compared by using gas chromatography with electron capture detector.

Keywords: Magnetic dispersive, Green chemistry, Pesticide, Chromatography

INTRODUCTION

The presence of very low levels of endocrine disruptor pesticides in environmental samples requires a preliminary concentration of the analytes before the analysis, so that the target analytes can be detected accurately and sensitively. Processes in which pre-concentration processes are carried out are generally techniques for determining overall non-specific components of target analyte. The fact that the selected pre-concentration technique is not specific to the relevant pesticide species may cause the amount of the target analyte to be detected not accurately due to reasons of interferences. The monitored analytes are generally adsorbed on a solid support, separated from the matrix environment and then analyzed with the appropriate device. The most critical step is the adsorption step where the target analyte is attached onto a solid support during the sample preparation step. The higher the polarities and the polarities of the adsorbent, the more selectively the target analytes are adsorbed.

Different extraction techniques namely, solid phase extraction (SPE) [2] liquid-liquid extraction (LLE) [3] dispersion liquid liquid micro extraction (DLLME) [4] solid phase micro extraction (SPME) [5] were developed for the extraction of environmental pollutants in different samples to meet the above mentioned requirements.

When we look at the classical extraction techniques used from these methods; the liquid phase (LLE), solid-liquid (SPE) based techniques have been developed and nowadays popular with solid phase micro-extraction (SPME) techniques have limited capacity. The sensitivity of the last two techniques depends on the affinity, polarity and pore structure of the cartridge or fibers used for sample preparation, in short, their physical and chemical properties.

As these adsorbents, which are usually prepared in one type, address only chemical groups with similar properties, alternative surfaces should be developed for structures with different chemical properties. In order to solve such problems, magnetic solid phase extraction (MSPE) was developed and it is one of the new procedure of micro solid phase

extraction (μ -SPE) based on the use of magnetically modified adsorbents. This technique has gained much attention due to easy separation under an applied external magnetic field, minimum solvent consumption and the extraction efficiency. In this novel sample preparation method, a series of magnetic particles have been used for the extraction of organic pollutants including, nano Fe_3O_4 , Fe_3O_4 -grafted graphene and graphene [6], carbon nanotubes and Fe_3O_4 - carbon nanotubes [7] and so on.

In this study, a novel $\text{Fe}_3\text{O}_4/\text{Ni}/\text{Ni}_x\text{B}$ nanocomposite magnetic adsorbent was synthesised and used for the enrichment of pesticides from the samples.

EXPERIMENTAL

Apparatus and Reagents:

Bromopropylate (97.5%) and Lambda-cyhalothrin (98.5%) were purchased from Dr. Ehrenstarfer-Schafers (Augsburg, Germany). Stock solutions of these pesticides were prepared in acetone and stored at 4°C before use. Wine and grape juice samples were purchased in local supermarkets and stored at 5°C. Gas chromatographic analyses were performed with a gas chromatograph from Agilent Model 7820A Series, equipped with HP ECD detector systems. The analytical column used was a DB-5-MS column (30m x 250 μm I.D. and film thickness 0.25 μm). Helium and nitrogen (99.99%) were used as carrier and make-up gas, respectively. The oven temperature program used for the analyses was as follows: 50°C for 5 min increased to 150°C at a rate of 25°C min⁻¹ and increased to 220°C at a rate of 10°C min⁻¹ and increased to 280°C at a rate of 5°C min⁻¹ with a total run time of 33 min.

μ -SPE Procedure:

7 mL of sample (pH:7.0) was placed in a centrifuge tube containing 5.0 mg of $\text{Fe}_3\text{O}_4/\text{Ni}/\text{Ni}_x\text{B}$ nanocomposite material. Adsorption process was made by using a shaker for 30 min. After this process, centrifugation was made and nano particles were isolated from the sample by placing a strong magnet at the bottom of the tube. The preconcentrated target analytes were desorbed with 75 μL ethyl acetate by ultrasonication for 15 min. After centrifugation process for 1 min, 50 μL of the extract was placed in an insert containing 1 mg of Na_2SO_4 for the removal of excess amount of water. Inserts were centrifugated in a micro centrifuge and at the end, 20 μL of sample was placed in a vial and 1 μL was injected to the gas chromatography system.

RESULTS AND DISCUSSION

In order to reveal the major effects of the extraction, eleven factors were selected and Plackett-Burman Design was constructed. The parameters effecting the extraction, mainly, solvent polarity, solvent volume, pH, sample volume, adsorption amount and time, desorption time, centrifuge time, mini centrifuge time, ultrasonication time and salt amount were optimized. The significant parameters effecting on the extraction efficiency were found as the solvent polarity, pH and sample volume and the results of this study were further used in Box Behnken design. Optimum values for the analysis was obtained as pH: 2, sample volume: 5 mL, adsorption temperature: 80°C and time: 20 min, desorption temperature: 250°C and time: 5 min, stirring rate: 1000 rpm and salt amount: 0.5 g.

The regression coefficients relating to linearity were at least 0.99. Under optimized conditions the linear range was found between 0.1 – 50 ng mL⁻¹ for BRP and 0.06 – 50 ng mL⁻¹ for LMD, and the detection limits for BRP and LMD were calculated as 0.03 and 0.02 ng mL⁻¹, respectively. Recoveries from spiked samples range from 89 to 108% and RSDs were no higher than 15% in the most unfavourable case. Enrichment factors were calculated as 30.3 and 393 for BRP and LMD, respectively.

Hence, this developed micro extraction method shares the advantages of the classical extraction methods such as excellent enrichment performance, easy operation and ability to employ a wide range of “green” extraction way.

This study was supported by Ege University (2017/Fen/069).

References:

- [1] T.D.Çiftçi, E.Henden **Powder Tech.** 269 (2015) 470-480
- [2] W-L. Chen, G.-S. Wang, J.-C. Gwo and C.-Y. Chen, **Talanta**, 89 (2012) 237–245.
- [3] Y. Niu, J. Zhang, Y. Wu, B. Shao, **J. Chromatogr. A**, 1218 (2011) 5248–5253.
- [4] B. Mokhtari, N. Dalali, K. Pourabdollah, **Chromatographia**, 76 (2013) 565–570.
- [5] X. Ma, Q. Li and D. Yuan, **Talanta**, 85 (2011) 2212–2217.
- [6] X. Cao, J. Chen, X. Ye, F. Zhang, L. Shen, W. Mo, **J. Sep. Sci.** , 36 (2013) 3579–3585.
- [7] B. Maddah, M. Hasanzadeh, **Int. J. Nanosci. Nanotechnol.**, 13:2 (2017) 139-149.

FT11- Development of an Analytical Method for Determination of Tetracycline Residues in Food Samples

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Abstract

In this study, a pre-concentration and determination method for tetracycline residues in food samples was developed based on cloud point extraction and HPLC-DAD analysis. Tetracycline molecules was extracted to surfactant phases of poly ethylene glycol (PEG-6000) in the presence of pH:5.0 buffer and high electrolyte concentration. The samples were centrifuged in order to increase phase separation and then diluted and filtered by 0.45 μm membrane filter prior to HPLC determinations. Experimental variables were optimized and examined such as concentration of electrolyte and surfactant, pH, time effect. All analytical merits were determined by using experimental results such as detection limit, linear range, and pre-concentration factor.

In development method, determination of tetracycline molecules in the surfactant rich phase was carried out by isocratic elution of pH:4 oxalate buffer; methanol;acetonitrile solvents and absorbance measurements of DAD detector at 276 nm and 358 nm. By optimization all experimental parameters, linear ranges for both wavelengths were calculated as 10-1000 ng mL⁻¹ and 25-800 ng mL⁻¹, respectively. Detection limits were 2.98 and 7.46 ng mL⁻¹ while relative standard deviations (RSD%) were lower than 4.20 % for 100 ng mL⁻¹ tetracycline. Finally, the developed method was applied to food samples including milk, liver and breast tissues of chicken.

Keywords: Tetracycline, PEG-6000, HPLC, Cloud point extraction, Food samples

INTRODUCTION

Antibiotics are active biological molecules, both in the field of medicine and in veterinary medicine. Although drugs have been forbidden to use as a growth promoter in animal feed in the European Union since 2006; As a treatment and growth accelerator in livestock production, can play an important role in livestock, industry and modern agriculture as feed additive in fish farms and to prevent damage of products by bacteria [1,2]. In recent years HPLC-MS / MS has been widely used to analyze antibiotics at trace levels in environmental samples. Nowadays, UPLC systems, which are a shorter response system for analyzing antibiotics, have been developed. Since 2006, UPLC / MS-MS has been applied to analyze drugs in wastewater and surface waters. But, the concentration of antibiotics is very low in most of real samples and sample medium is so complex for a simple analysis. So, a pre-concentration and separation method is mostly preferred in this type analysis [3,4].

The method of cloud point extraction (CPE) is applied to biological samples, organic contaminants and metals for the separation and determination of vitamins, hormones, enzymes and proteins.

This study proposes a sensitive analytical methodology for trace determination of tetracycline molecules by using cloud point extraction and HPLC-DAD system. All parameters were optimized and the developed method was applied to food samples successively.

Instrumental System

The HPLC system used for detection of quercetin consists of a inertsil C18 column (250 mm×4,6×5 μm) for chromatographic separation. a pump a thermostatic oven (CTO-10 AS, Shimadzu), (model LC20-AD, Shimadzu, Tokyo, Japan), a DAD detector (Model SPD-M20A, Shimadzu) and auto sampler (SIL-20Ac, Shimadzu). In order to obtained data from detector to computer, an LC solution software (Shimadzu) was used. pH measurements were conducted by a pH meter with a glass-calomel electrode (Selecta, Spain).

The proposed method

An aliquot of the sample or standard solution containing tetracycline molecules in the range of 10–1000 $\mu\text{g L}^{-1}$, PEG-6000 (2.0 mL of 20.0 (v/v) %), sodium sulfate (11.0 mL of 20.0 (v/v) %), and buffer solutions (1.5 mL, pH:5.0) were mixed in a centrifuge tube having 15 mL of final volume and kept in a thermostatic water-bath at 50 °C for 50 min. The phase separation was accelerated by centrifuging at 5000 rpm for 5 min. Then, the mixture was cooled in a refrigerator for 20 min in order to increase the viscosity of the surfactant-rich phase and facilitate the removal of the aqueous phase. After then, the aqueous phase was easily separated from surfactant-rich phase by inverting the tube. 400 μL of Methanol 50 % was added to the surfactant-rich phase to reduce its viscosity before delivering for HPLC-DAD system. Finally, the tetracycline concentrations were determined by making either direct calculation from the calibration curve obtained by HPLC-DAD or using standard additions approach.

RESULT AND DISCUSSION

The obtained chromatogram was given in Figure 1 by directly determination tetracycline before CPE. As can be seen in Figure 1, a good shaped peak was obtained for analysis of model solutions.

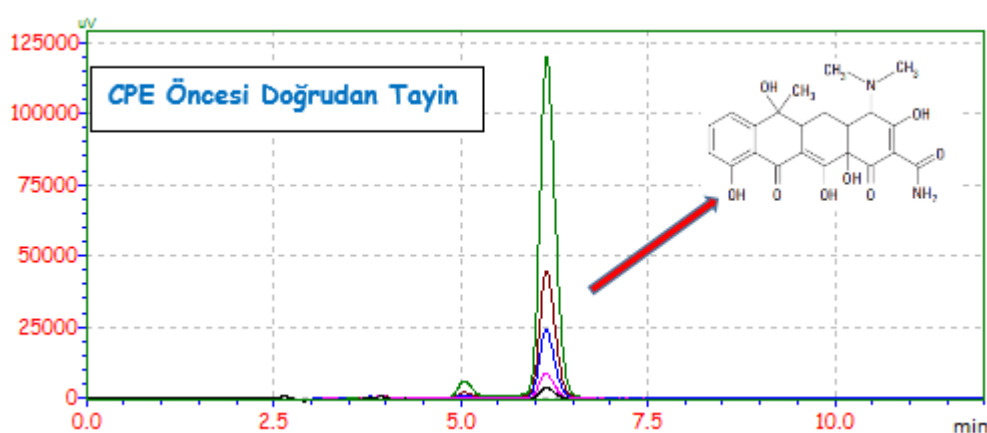


Figure 1. Directly determination of tetracycline before CPE

HPLC conditions were also submitted in Table 1. The most sensitive results were obtained by using these parameters. As expected, the determined concentrations by

directly method are so limited and generally upper than 1.0 mg/L. Due to low concentrations of drug molecules in real samples, studies on development of sensitive and versatile analytical method are continuing in many of research laboratory.

Table 1. HPLC conditions for tetracycline analysis

Parameter	Value/Conditions
HPLC Mode	Isocratic
Mobile Phase	%70 0.01 M pH:4.0 Oxalic Acid Buffer % 20 Acetonitril % 10 Methyl Alcohol
Eluent Rate	1 mL/min
Run Time	18 min
Column	C18- Inertsil ODS-3 (250 mm×4.6×5.0 µm)
Column temperature	40 ° C
Injection Volume	20 µL
Total Pressure	150 bar

Conclusion

All variables were optimized in CPE and finally a sensitive method was developed for trace analysis of tetracycline molecules. Table 2 summarizes the analytical merits of developed method. Because tetracycline molecule has two sensitive wavelengths, two separate methods was developed for each wavelength. As can be seen in the table, the proposed method presents so good analytical results for a new method. Without any complex instrumental system, sensitive analysis can be applied by this method. Application of new methodology on food samples were carried out and quantitative results and recovery values were obtained in this experiments.

Table 2 Analytical Merits of new developed method

Parameter	Before CPE	After CPE	
		276 nm	358 nm
Lineer range	1-50 µg mL ⁻¹	10-1000 ng mL ⁻¹	25-800 ng mL ⁻¹
LOD ^a	0.35 µg mL ⁻¹	2.98 ng mL ⁻¹	7.46 ng mL
LOQ ^b	1.01 µg mL ⁻¹	9.83 ng mL ⁻¹	24.63 ng mL ⁻¹
RSD (%) (250 ng mL ⁻¹)	2.75	4.20	3.80
Slope	0.00077	0.052	0.045
(R ²)	0.9972	0.9969	0.9988
Preconcentration factor	-	37.5	37.5

Enhancement factor	-	67.5	59.0
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^a Based on statistical $3S_{\text{blank}}/m$ -criterion for ten replicate blank absorbance measurements

^b Based on statistical $10 S_{\text{blank}}/m$ -criterion for ten replicate blank absorbance measurements

^c Preconcentration factor is defined as the ratio of the initial solution volume (50 mL) to the volume of surfactant rich phase (500 μL)

^d Enhancement Factor is defined as ratio of slope of calibration before and after CPE

Acknowledge

This study has been supported by Cumhuriyet University Scientific Research Projects Commission as the research project with the ECZ-052 code. This submitted manuscript includes some results from master thesis of K. Sarıdal. All optimizations of new methods and other detailed results are being prepared as full research article for an international journal.

References

1. David Moreno-González, Ana M.García-Campaña, Salting-out assisted liquid–liquid extraction coupled to ultra-high performance liquid chromatography–tandem mass spectrometry for the determination of tetracycline residues in infant foods, *Food Chemistry* (2017) 221 (1763-1769).
2. Michael Pérez-Rodríguez, Roberto Gerardo and et al. An overview of the main foodstuff sample preparation technologies for tetracycline residue determination, *Talanta* (2018) 182 (1-21).
3. J. Fritz, Y. Zuo, Y. Simultaneous determination of tetracycline, oxytetracycline, and 4-epitetracycline in milk by high-performance liquid chromatography, (2007). *Food Chemistry* 105, 1297–1301.
4. S. Kools, J.F. Moltmann, T. Knacker, Estimating the use of veterinary medicines in the European union. *Regulatory Toxicology and Pharmacology*, (2008). 50(1), 59-65.

FT12-Sensitive Determination of Hydrogen Peroxide in Water Samples by High Spin Peroxo Complex

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Abstract

In this study a simple, sensitive and selective spectrophotometric method based on high spin complex of hydrogen peroxide (H_2O_2) was developed for the determination of H_2O_2 in real water samples. The method is based on the complexation of hydrogen peroxide with the Fe(III)-EDTA mixture in the alkaline medium. A purple color of low stable peroxo-iron(III)-EDTA complex was formed in the method and it has a maximum absorbance peak at 525nm. Variety of parameters such as Fe(III), EDTA and ammonia concentrations were optimized.

Under optimized conditions, limit of detection and limit of quantification was found as 2.5×10^{-6} and 8.5×10^{-6} mol/L. Intraday and interday relative standard deviation of the proposed method for 2.0×10^{-4} mol/L of H_2O_2 were found as 1.5% and 6.1% respectively. The proposed method was successfully applied to real water samples namely drinking water, tap water and seawater with acceptable recovery values between 90% and 118%. Nearly none of common ions except Fe(II) showed interfering effect to proposed method so, the method can be easily acceptable to real water samples without any sample preparation step.

Keywords: Hydrogen peroxide, Spectrophotometry, Peroxo-iron complex

INTRODUCTION

H_2O_2 is a reactive oxygen species, which has a great importance in chemistry, biochemistry, and in the field of life sciences. H_2O_2 , which is an unstable compound cause radical formation in some cases and may have carcinogenic effects to human [1]. H_2O_2 can be occurred in real water samples based on industrial application or natural reaction. On the other hand, the ways for the natural production of H_2O_2 in environment are photochemical reaction of dissolved organic matter [2], wet deposition [3], dry deposition [4] or biological reactions [5]. Because of the rapid degradation of H_2O_2 , monitoring and identification of it with a fast technique is so important analytical problem. In the literature different methods were used for the determination of H_2O_2 such as titrimetric [5] spectrophotometric [6], fluorometric [7], chemiluminescence [8] or electrochemical [9] techniques. However, most of them do not have access to adequate sensitivity and these are time consuming methods.

In this study a simple, rapid and sensitive spectrophotometric method is described for the determination of hydrogen peroxide using iron(III) and EDTA as a reagent.

EXPERIMENTAL

Apparatus and Reagents: Spectrophotometric measurements were carried out by using a CARY 100 Bio UV-Visible Double-Beam spectrophotometer. UV absorption spectra were recorded at room temperature and Hellma Analytics high precision quartz cells (111-QS) were employed for analyses.

Solutions of EDTA were prepared from solid $Na_2(H_2EDTA)$ (Merck). $S_2O_3^{2-}$ solution were prepared from solid $Na_2S_2O_3$ (Kimetsan). Fe(III) solution were prepared from solid $FeCl_3 \cdot 6H_2O$ (Merck). H_2O_2 standard solutions were prepared by dilution of a 35 % (w/w) stock solution of H_2O_2 (Merck). 25 % (w/w) concentrated solution of ammonia (Merck) was used in analysis. Stock solutions of interfering ions were prepared by dissolving suitable salt in water. All of the other reagents and solvents used were of analytical reagent grade.

All solutions were prepared in ultrapure water [Millipore Milli Q system (18.2 MΩ). 0.1 mol/L $\text{Na}_2\text{S}_2\text{O}_3$ solution was standardized iodometrically against to IO_3^- primer standard. Stock solutions of H_2O_2 (0.1 mol/L) were freshly prepared and standardized iodometrically against to thiosulphate.

Preparation of complexing reagent

Complexing reagent was prepared freshly before use. For this purpose, solid FeCl_3 transferred to a beaker and a few milliliters of ultrapure water added and mixed well. Then solid $\text{Na}_2\text{H}_2\text{EDTA}$ added to this mixture and stirred until all compound dissolved. After that 10 mL of 25 % NH_3 stock solution added to mixture to get alkaline media. Finally, solid $\text{Na}_2\text{S}_2\text{O}_3$ added to the mixture and diluted to 25 mL in a volumetric flask.

Spectrophotometric Analysis Method of H_2O_2

First of all the cell was treated with acetone and then was dried completely two remove residual water from the cell. After that, 2.0 mL of H_2O_2 containing solution was added to cell then 1.2 mL of concentrated NH_3 added to the medium. Finally, 300 μL of complexing reagent (containing 0.5 mol/L EDTA, 0.03 mol/L Fe(III), 5 mol/L NH_3) was added to final solution. Final concentration of the EDTA, Fe(III) and NH_3 in the cell were 5.0×10^{-2} mol/L, 3.0×10^{-3} mol/L and 5.0 mol/L respectively. Purple colored peroxo complex formed immediately and the colored solution was shaken well to get homogenous solution. The spectrophotometric measurement was done as fast as possible after mixing the solution.

Sample Analysis Method

The water samples were filtrated by 0.25 μm PTFE filter to remove the particles from water samples before analysis. Then sample analysis was carried out according to part 2.4. After filtration, water samples except seawater directly analyzed with proposed method. The sea water sample were 5 times diluted by pure water before analysis.

RESULTS AND DISCUSSION

Absorption spectra of aqueous solutions of Fe(III), Fe(III)-EDTA, Fe(III)-EDTA- NH_3 and colored complex of Fe(III)-EDTA- NH_3 - H_2O_2 were recorded between 800-400 nm without baseline correction (Figure 1).

As can be seen from the Figure 1, a very sharp charge transfer band started around (500 nm) (Zena, 2013). After addition of EDTA into the Fe(III) solution sharp charge transfer band shifted to more short wavelengths (450 nm) because of the complex formation of Fe(III) with EDTA (Figure 1-c). A similar absorption spectra was observed by the addition of NH_3 into the Fe(III)-EDTA solution (Figure 1-c). Except a small absorption band was observed at $\lambda_{\text{max}} 475$ nm. When H_2O_2 was added to the Fe(III)-EDTA- NH_3 mixture a purple colored complex was observed immediately and a peak appeared at $\lambda_{\text{max}} 525$ nm due to the formation of the Fe(III)-EDTA- H_2O_2 complex, as shown in Figure 1-d.

The color of Fe(III)-EDTA- NH_3 solution and its peroxo complex were also presented in Figure 2.

It was difficult to get reliable absorbance of peroxo complex because of the high decomposition rate. Therefore, the effects of varieties of compounds on decomposition rate were investigated for 3 minutes. The best result was obtained with $\text{Na}_2\text{S}_2\text{O}_3$ was selected as stabilizator reagent for further experiments.

Variety of parameters (ie Fe(III), EDTA concentration) was optimized. Optimum parameters were summarized in Table 1.

Under optimized conditions calibration curve was performed. Good obedience to Beer's law is obtained in the range of 3.6×10^{-6} and 4.08×10^{-3} mol/L. Calibration curve for

the determination H_2O_2 is presented in the Figure 3. The increase in concentration of H_2O_2 shows a linear increase in the absorbance.

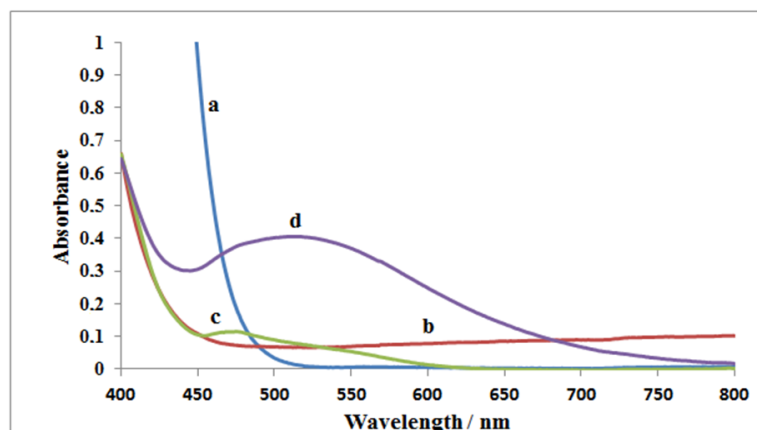


Figure 1. The spectra of a) Fe(III) solution, b) Fe(III)-EDTA solution, c) Fe(III)-EDTA- NH_3 solution and d) Fe(III)-EDTA- NH_3 - H_2O_2



Figure 2. The color of a) Fe(III)-EDTA- NH_3 and b) Fe(III)-EDTA- NH_3 - H_2O_2 complex.

Table 1. Optimum parameters

Parameters	Condition
Fe(III) concentration	0.003 mol/L
EDTA concentration	0.05 mol/L
$\text{S}_2\text{O}_3^{2-}$ concentration	0.05 mol/L
NH_3 concentration	5 mol/L

The proposed method successfully applied to real water samples namely drinking water, tap water and seawater samples. The precision of the proposed method is evaluated by the three replicate analysis of water samples containing at three different concentrations H_2O_2 and are presented in Table 2. Two different commercial bottled waters (A and B) were analyzed by proposed method. The H_2O_2 concentrations of all samples were blow the LOD value.

In conclusion, we have developed a spectrophotometric method for the determination of H_2O_2 by using colored peroxo-iron(III)-EDTA complex in basic solutions. Stability of peroxo-Fe(III)-EDTA was not good enough for the reliable determination of H_2O_2 and stability of peroxo complex was enhanced by adding $\text{S}_2\text{O}_3^{2-}$ to medium as a stabilizator reagent. Developed method provided a sensitive, simple, rapid and inexpensive method for the determination of H_2O_2 in aqueous sample. Developed method allowed detection of H_2O_2 in a range from 5.0×10^{-6} and 4.08×10^{-3} mol/L with high repeatability (%R.S.D: 1.6% for intraday, 6.5% for interday).

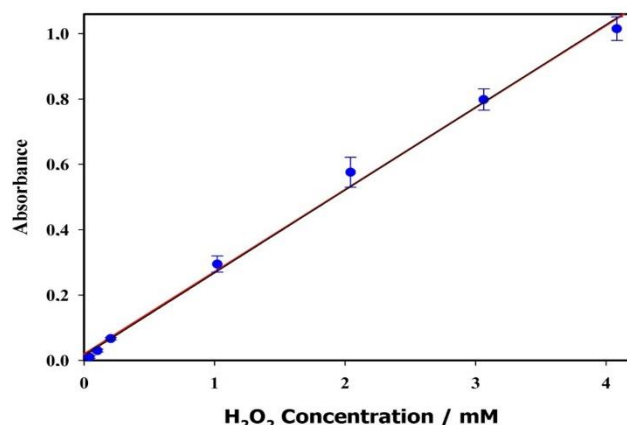


Figure 3. Calibration curve of the proposed method.

Table 2. The recovery values of the sample applications

Concentration (M)	Recovery (%)				
	Found	Bottled Water, A	Bottled Water, B	Sea Water	Tap Water
1.0×10^{-5}	<LOD				
5.0×10^{-5}	<LOD	9	9	9	1
1.0×10^{-4}	<LOD	1	1	1	9

The colored complex formation between H₂O₂ and Fe(III)-EDTA is very sensitive and can be applied for the determination of H₂O₂ in real water samples without any further sample preparation step. The LOD value of the proposed method is good enough for the determination of H₂O₂ in real water samples with acceptable recovery values. This method can be applied to other aqueous samples such as lake, river, well and mineral water samples.

This study was supported by Ege University BAP (2015FEN062).

References:

- [1] Demirkol O, Cagri-Mehmetoglu A, Qiang Z, Ercal N, Adams C., Impact of food disinfection on beneficial biothiol contents in strawberry, *J Agric Food Chem.* (2008); 56: 10414-10421.
- [2] W.J. Cooper, R.G. Zika, R.G. Petasne, J.M.C., Photochemical formation of hydrogen peroxide in natural waters exposed to sunlight, *Plane Environ. Sci. Technol.*, 22 (1988), pp. 1156-1160
- [3] D.W. Gunz, M.R. Hoffmann, Atmospheric chemistry of peroxides: a review, *Atmos. Environ.*, 24A (1990), pp. 1601-1633.
- [4] H. Sakugawa, I.R. Kaplan, Comparison of H₂O₂ and O₃ content in atmospheric samples in the San Bernardino mountains, Southern California, *Atmos. Environ.*, 27 (1993), pp. 1509-1515.
- [5] The Japanese Pharmacopoeia, Sixteenth ed., Hirokawa Publishing Co., Tokyo, (2011), pp. 505–506.
- [6] Cheng-Chih Hsu *, Yuan-Rong Lo †, Yu-Chian Lin †, Yi-Cen Shi † and Pang-Lung Li
A Spectrometric Method for Hydrogen Peroxide Concentration Measurement with a Reusable and Cost-Efficient Sensor, *Sensors*, (2015), 15, 25716-25729.
- [7] Odo, K. Matsumoto, E. Shinmoto, Y. Hatae, A. Shiozaki, *Anal. Sci.*, 20 (2004), pp. 707-710.
- [8] N. Yamashiro, S. Uchida, Y. Satoh, Y. Morishima, H. Yokoyama, T. Satoh, J. Sugama, Determination of hydrogen peroxide in water by chemiluminescence detection, (I) flow injection type hydrogen peroxide detection system, *J. Nucl. Sci. Technol.*, (2004), 41, pp. 890-897.
- [9] K. Nakashima, K. Maki, S. Kawaguchi, S. Akiyama, Y. Tsukamoto, K. Imai, *Anal. Sci.*, (1991), 7, pp. 709- 714.

FT13- DETECTION OF Cd AND Pb IN BB CREAMS

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ABSTRACT

In this study the levels of Cd and Pb in different brands of BB creams (beauty balms/blemish balms) marketed in Turkey were investigated. Samples were prepared by wet digestion using some acids and/or acid combinations, and lyophilization (freeze drying), then analyzed by using Flame Atomic Absorption Spectroscopy (FAAS) via standard addition calibration. Table 1 shows Cd and Pb concentrations of samples.

Keywords: AAS, trace metals, creams

INTRODUCTION

BB creams are one of the most popular cosmetic products nowadays. BB creams are basically a tinted moisturizer, antiaging and skin care cream in one product [1]. They are not only lighter than foundation and colorful than skin creams but also they offer to neutralize skin tone, correct imperfections, moisturize, protect your skin from UV, and correct your skin.

Various undesirable organic and inorganic components end up in the final product through the raw materials and additives used in the preparation of skin products. In particular, heavy metals may be found in colored cosmetics as impurities due to the persistent nature of these substances and the fact that they are found in natural environment [2].

Acceptable level of metals in the cosmetic products according to cosmetic regulations are declared as 1 ppm for Hg, 5 ppm for As and Cd, 10 ppm for Sb, and 20 ppm for Pb [3].

MATERIALS & METHODS

Commonly used BB cream samples were purchased from cosmetic markets. Working conditions of Thermo Scientific–SOLAAR Flame Atomic Absorption Spectrometer for Cd and Pb are selected as 228.2 and 217.0 nm wavelength, 0.5 nm slit width, 7 and 11 mm flame height, respectively. As an atomization gas C₂H₂-air was used. LOD for Cd is 0.02 µg g⁻¹ and Pb is 0.41 µg g⁻¹. The first three of cream samples were also lyophilized and then wet digested. The calibration curves are lined via standard solutions from Cd and Pb stock standard 1000 mg L⁻¹. Sample preparation procedures are given below:

1st GROUP (HNO₃)

1. Approximately 5.00 g of sample was dissolved in 10 mL HNO₃ and heated on a hot plate to near dryness, when it's cooled a few drops of distilled water added.
2. Each digested sample was filtered through a filter paper.
3. The digested sample was transferred into volumetric flask and diluted with distilled water up to 30 mL.

2nd GROUP (HNO₃ + H₂O₂)

1. Approximately 5.00 g of sample was dissolved in 10 mL HNO₃ and 1 mL of H₂O₂, heated on a hot plate to near dryness, when it's cooled a few drops of distilled water added.

2. Each digested sample was filtered through a filter paper.
3. The digested sample was transferred into volumetric flask and diluted with distilled water up to 30 mL.

RESULTS AND DISCUSSION

Obtained results for Cd and Pb concentrations ($\mu\text{g g}^{-1}$) of BB creams purchased from cosmetic stores is shown in the Table 1.

Table 1. Cd and Pb concentrations ($\mu\text{g g}^{-1}$) of BB creams

SAMPLE ID	WET DIGESTED				LYOPHILIZED & WET DIGESTED			
	HNO ₃		HNO ₃ + H ₂ O ₂		HNO ₃		HNO ₃ + H ₂ O ₂	
	Cd	Pb	Cd	Pb	Cd	Pb	Cd	Pb
BB 1	1.95±0.68	2.69±1.21	1.83±0.37	1.34±0.61	0.28±0.03	<LOD	0.44±0.20	4.90±2.69
BB 2	3.26±1.14	2.31±1.04	2.39±0.48	3.43±1.54	1.00±0.10	3.10±1.71	1.00±0.45	4.91±2.70
BB 3	0.23±0.08	<LOD	0.59±0.12	0.54±0.24	<LOD	1.77±0.98	<LOD	<LOD
BB 4	0.20±0.07	3.24±1.46	0.11±0.02	2.16±0.02				
BB 5	0.62±0.22	1.10±0.50	0.54±0.11	1.44±0.11				

Acceptable levels of Cd and Pb in the cosmetic products are 5 and 20 ppm ($\mu\text{g g}^{-1}$), respectively. According to obtained results Cd and Pb levels of selected BB Creams are below the acceptable limit. In generally, BB creams has complex formulation that's why the results of its metal content has not statistically correlated. According to these results, it is obvious that to obtain the more accurate results the more samples should be studied.

REFERENCES

- 1) Turkish Medicines And Medical Devices Agency. "Turkish Medicines And Medical Devices Agency Guideline on Heavy Metal Impurities in Cosmetics." [ONLINE] Available at: <https://www.titck.gov.tr/PortalAdmin/Uploads/UnitPageAttachment/AOZpWpNn.pdf>. [Last update date 10 May 2016].
- 2) Trace determination of lead in lipsticks and hair dyes using microwave-assisted dispersive liquid-liquid microextraction and graphite furnace atomic absorption spectrometry. K. Sharafi, N. Fattahi, M. Pirsahab, H. Yarmohamadi and M. Fazlzadeh Davil, International Journal of Cosmetic Science, 2015, 37, 489–495.
- 3) Çağlar, A. B., Saral, S., "Kozmetolojide Toksisite Sorunu" Turk J Dermatol, (2014) 4:248-51.

FT14- Optimization of Poly (Acrylic Acid-co-N-vinyl 2-pyrrolidone) / SiO₂ Nanocomposite Hydrogels

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Abstract

Copolymeric hydrogels have better mechanical stability and durability than homo polymeric hydrogels. The monomers (acrylic acid and N-vinyl 2-pyrrolidone) were used in this study, optimized of the monomer / monomer ratio and reinforcement material which shows the best strength property selected from this copolymeric hydrogels were carried out in study. Nano SiO₂ particles (20-30 nm) was commercially purchased and used as a reinforcing material for synthesis of nanocomposite hydrogels. % Conversion (%C), % crosslinking (%CL) and % swelling (%S) values of the obtained hydrogels were calculated for determine swelling behavior. The chemical bonds of the hydrogels were clarified by FT-IR analysis. The surface morphologies of the hydrogels were shown by SEM images.

Keywords: nano SiO₂, nanocomposite, hydrogel, acrylic acid, vinyl pyrrolidone

INTRODUCTION

Hydrogels are three dimensional hydrophilic polymer networks capable of swelling in water or biological fluids, and retaining a large amount of fluids in the swollen state their own structures. Several techniques have been reported for the synthesis of hydrogels. The first approach involves copolymerization/crosslinking of co-monomers using multifunctional co-monomer, which acts as crosslinking agent. The polymerization reaction is initiated by chemically. The polymerization reaction can be carried out in bulk, in solution, or in suspension. The second method involves crosslinking of linear polymers by irradiation, or by chemical compounds¹. Nano SiO₂ has the features of small particle size, narrow particle size distribution, porous and large surface area. Also, owns a large number of hydroxyl groups and unsaturated residual bonds on its surface and shows high reflectivity to long wave, visible light and ultraviolet ray. Although hydrogels have attracted the attention of researchers as a field of study on their own, there has been more interest in "Nanocomposite Hydrogels" recently. Nanocomposite hydrogels are being increasingly evaluated for various biomedical applications. The enhanced surface interactions between the nanoparticles and the polymer chains result in material properties that may be useful for various biomedical applications².

EXPERIMENTAL

Amount of monomer/monomer in volume ratio for acrylic acid (AA)-vinyl pyrrolidone (VP) 3-1; 2-2 and 1-3 were used. In the presence of optimized 1% cross-linker (molar ratio) and radical initiator (APS), by mass of free radical polymerization method 0.05%; 0.5% and 1% nano SiO₂ added AA-co-VP nanocomposite hydrogels were synthesized. % Conversion (%C), % crosslinking (%CL) and % swelling (%S) values of the obtained hydrogels were calculated. The chemical bonds of the hydrogels were clarified by FT-IR analysis. The surface morphologies of the hydrogels were shown by SEM analysis.

RESULTS and DISCUSSION

Swelling Behaviour

It is important that to determine of swelling behavior of hydrogels due to ability of response in water. First, the copolymeric hydrogels were synthesized by using free radical polymerization technique. Then, best hydrogel structure was selected for obtain SiO₂ added nanocomposite hydrogels. Swelling properties of various monomer:monomer ratio of hydrogels were given in Table 1. According to swelling behaviors, best conversion of polymerization to hydrogels, best crosslinking or gelation and best swelling behavior of hydrogels was AA-co-VP (3:1) hydrogel (326 %Swelling). For this reason, AA-co-VP (3:1) hydrogel was selected for optimized nano SiO₂ amounts.

Table 1. Swelling properties of hydrogels

Plain AA			AA-co-VP (3:1)			AA-co-VP (2:2)			AA-co-VP (1:3)		
C%	CL%	S%	C%	CL%	S%	C%	CL%	S%	C%	CL%	S%
95,42	95,34	370	95,50	81,34	326	104	74,76	117	91,83	63,16	142

FT-IR Results

0.05%; 0.5% and 1% nano SiO₂ added AA-co-VP (3:1) nanocomposite hydrogels were characterized for selected optimized best chemical harmony in structures. Chemical interactions of nanocomposite hydrogels were characterized by FT-IR spectroscopy. In Fig. 1, FT-IR spectra of copolymeric hydrogels and nano SiO₂ added copolymeric nanocomposite hydrogels were given. In this results, low differences were seen in structures because of small amount of SiO₂. However, ideally amount of SiO₂ nanoparticle was shown in 0.5 % (w:w). Also, the result was matched by swelling value in SiO₂ doped hydrogels (%250, %300 and 230, %0.05 SiO₂, %0.5 SiO₂ and %1 SiO₂ added hydrogels respectively).

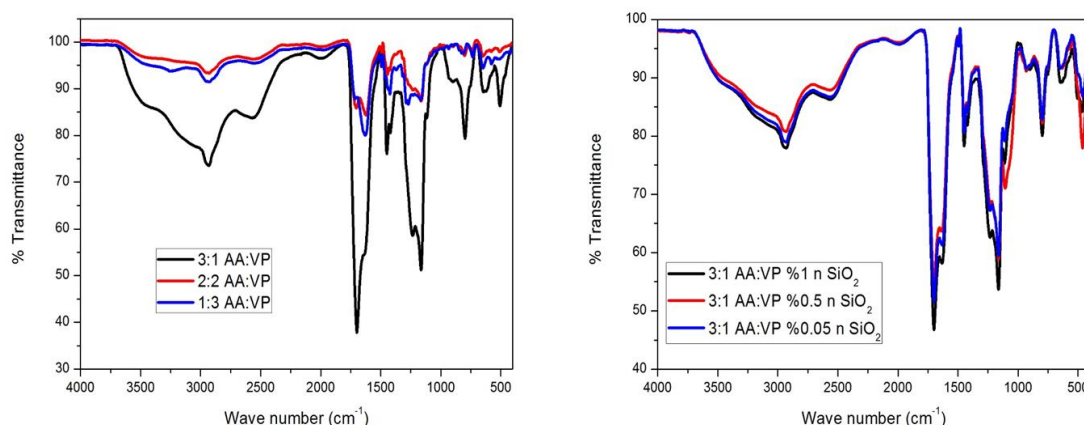


Fig.1. FT-IR spectra of copolymeric hydrogels and nano SiO₂ doped copolymeric nanocomposite hydrogels

SEM Results

Morphological structure of hydrogels were imaged by SEM. For better understanding, lyophilization process was applied to hydrogels. Therefore, the pore structure with and without nano SiO₂ was shown clearly. SEM pictures of nano SiO₂ undoped (left) and doped (right) copolymeric nanocomposite hydrogels were given in Fig.2.

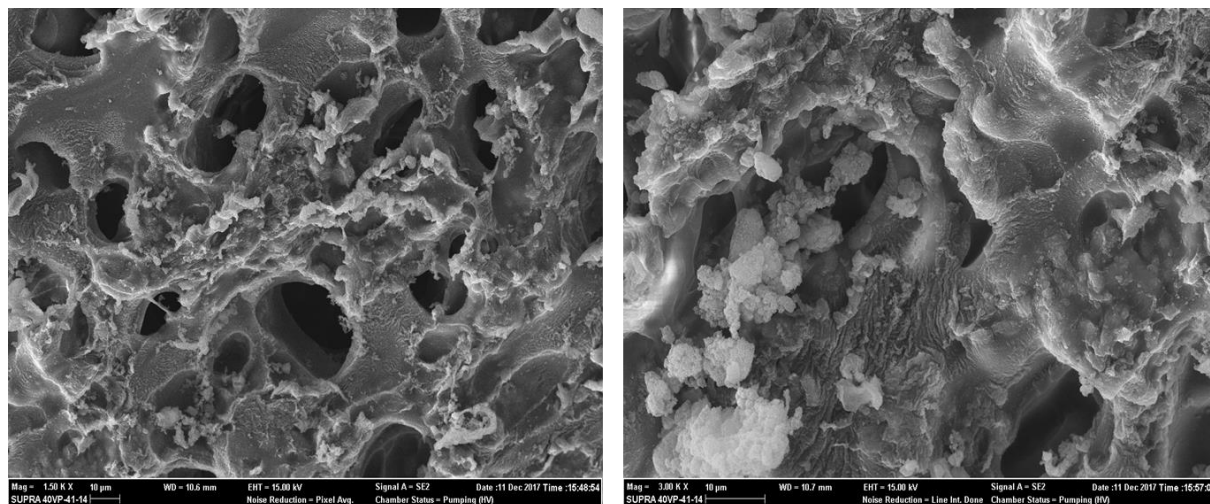


Fig. 2. SEM pictures of nano SiO₂ doped copolymeric nanocomposite hydrogel

CONCLUSION

In this study, synthetic monomers were used, nano SiO₂ added, crosslinked disk shaped nanocomposite hydrogels were produced while the monomers were polymerized by free radical polymerization technique. The nano SiO₂ used as reinforcement material was commercially purchased. When synthetic based nanocomposite hydrogels synthesized, the vinyl pyrrolidone and acrylic acid have been preferred because of their biocompatibility.

Considering the behavior of the SiO₂ reinforcements in composite hydrogels during the planning of target-focused work with SiO₂ doped nanocomposite hydrogels is important in interpreting the results to be achieved in the target applications.

ACKNOWLEDGMENT

This work was supported by Scientific Research Project Commission of Bilecik Seyh Edebali University (project number is 2017-01.BŞEÜ.28-01). FT-IR and FESEM measurements were performed in Bilecik Seyh Edebali University, Central Research Laboratory.

References

- ¹Fatma Özge Gökmen, Nursel Pekel Bayramgil, synthesis and characterization of n-[3-(dimethyl-amino)propyl] methacrylamide/(nano-SiO₂, amine-modified nano- SiO₂ and expanded perlite) nanocomposite hydrogels, Eur. Chem. Bull. (2017) 6(11) 514-518.
- ²A.K. Gaharwar, N.A. Peppas, A. Khademhosseini; Biotechnology and Bioengineering 2014, 111 (3), 441-453.

FT15-Temporal Changes in Trace Element Concentrations in Spring, Shallow and Deep Well Waters from the Uluova Plain-Elazig, Turkey

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Abstract: In this study, our aim was to examine temporal changes in trace element (As, Al, Fe, Mn, Cr, Ni, Pb, Zn, Cu, Cd, Ba, B, V, Se, Sb, Co) concentrations at three different locations, including spring, shallow and deep well waters, in a monthly frequency between November 2012 and October 2013. The water sampling sites are in the middle of Uluova plain near Elazig city. The results obtained suggest that arsenic (As) levels in most of the studied water samples are higher than the maximum concentration allowed (MCL) in drinking water. However, the concentrations of other elements were lower than the permissible levels. Climatic conditions change the trace element concentrations, especially in water samples taken from spring and shallow well waters.

Keywords: Uluova, Trace Elements, Arsenic

INTRODUCTION

Assessment of trace, particularly toxic trace, elements has high popularity due to widespread occurrence of pollution resulting from industrial and agricultural activities. In the past several decades, our understanding of water quality has expanded with our increased understanding of the importance of the hydrologic cycle for groundwater quality conditions. Additionally, other natural factors, including geology, tectonic structure and geomorphology, also control the timing and amount of surface and groundwater flow and the transport of waterborne constituents, including contaminants. Furthermore, natural complexity is increasing because of changes in climate, resulting in new patterns of seasonal precipitation, runoff, and spatial and temporal distribution of snow versus rain [1, 2].

MATERIALS AND METHODS

Study Area

This study was conducted in spring, shallow and deep well from three different locations of Uluova sub-basin. Uluova sub-basin is located in the southeast part of Elazig Province, Turkey. This sub-basin is located between longitude 39° 00" - 39° 35" E and latitude 38° 25" - 38° 45" N.

Based on their age, the rocks found in the Uluova sub-basin can be classified into eight units. Geological units consist of the Upper Cretaceous Elazig magmatic rocks, Upper Maastrichtian Harami Formation, Upper Maastrichtian - Middle Eocene Hazar Group, Middle Eocene Maden Group, Middle–Upper Eocene Kırkgeçit Formation, Upper Miocene–Lower Pliocene Karabakır Formation, Upper Pliocene – Pleistocene Palu Formation and the Quaternary alluvial deposits.

The water sampling sites are in the middle of Uluova plain near Elazig city. There are industrial factories and Elazig airport near the study area. Furthermore, agricultural activities are intense in the region where the aquifer beneath the plain provides drinking water of Elazig city with a population of 600 000.

Most of the precipitation falls in the spring and winter months. The mean annual total precipitation (1929 - 2013) is 426.3 mm. The highest monthly rainfall was observed at 66.4 mm in April and the lowest at 1.17 mm in August. According to Elazig Meteorology Station observations (1938-2013), mean temperature is 13 °C and monthly maximum temperature (42.2°C) is measured in August, minimum temperature (-22.6 °C) in January.

Sampling and Measurement: Water samples ($n = 36$) were collected from three different locations, including spring, shallow and deep well water in a monthly frequency between November 2012 and October 2013.

The trace elements (As, Al, Fe, Mn, Cr, Ni, Pb, Zn, Cu, Cd, Ba, B, V, Se, Sb, Co) were analyzed by ICP-MS (inductively coupled plasma-mass spectrometer) method and compared to the values of maximum concentration allowed (MCL) in drinking water.

RESULTS and DISCUSSION

The graphs showing the results of the analysis together with the mean rainfall are shown in Figure 1a – 1p. The concentration of arsenic in the groundwater ranges from 2.74 to 367.2 ppb. It was low in shallow well water in July and high in shallow well water in October. The concentration of aluminum in the groundwater range from 0.08 to 162.4 ppb. It was low in deep well water in September and high in spring water in October. The concentration of iron in the groundwater range from 0.03 to 57.88 ppb. It was low in deep well water in December and high in deep well water in November. The concentration of manganese in the groundwater varied from 0.27 to 240.7 ppb. It was low in shallow well water in April and high in spring water in October. Chromium ranged between 0.94 to 8.25 ppb. It was low in shallow well water in June and high in spring water in October. Average nickel results ranged from 0.65 to 49.15 ppb with minimum value at spring water during March and maximum value at sampling shallow well water during July. Lead concentration in the Uluova Basin at the studied locations ranged from 0.31 to 4.70 ppb. The minimum value was recorded at deep well water (December) while the maximum value was recorded at spring water (August). Zinc was recorded in 35 samples during the investigation period. It ranged from 0.16 to 156.9 ppb. The maximum value and the minimum value were recorded during October (at shallow well water) and May (at spring water), respectively. Copper ranged between 0.08 and 9.14 ppb with minimum concentration at deep well water during June and maximum concentration at spring water during December. Cadmium concentrations for the water samples under study varied between 0.05 and 4.35 ppb. It was low in spring, shallow and deep well waters in November and maximum in spring water in August. The concentration of boron in the groundwater varied from 92.12 to 22180 ppb. It was low in shallow well water in September and high in deep well water in February. Barium ranged between 8.22 to 101.8 ppb. It was found low in spring water in December and high in shallow well water in January. The concentration of vanadium in the groundwater range between 0.60 and 14.04 ppb. It was low in spring water in May and high in shallow well water in January. Selenium ranged between 0.27 and 28.08 ppb with minimum concentration at deep well water during November and maximum concentration at spring water during September. The concentration of antimony in the groundwater varied from 0.10 to 12.33 ppb. It was low in deep well water in December and high in deep well water in October. Cobalt was recorded in 31 samples during the investigation period. It ranged from 0.01 to 4.75 ppb. The maximum value and the minimum value were recorded during February (at spring water) and November (at shallow well water), respectively.

CONCLUSION

Climatic conditions change the trace element concentrations, especially in water samples taken from spring and shallow well waters.

In the spring water samples: In November, January, February, and June; arsenic, manganese and boron, in December, April and July; arsenic, manganese, boron and selenium, in March; manganese, boron and selenium, in May; manganese and boron, in August; manganese, boron, selenium and antimony, in September; only manganese, in October; arsenic, manganese, boron, vanadium and antimony were detected in concentrations that were higher than the MCL.

In the shallow well waters: In December; arsenic, boron and antimony, in March, April, June and August; only selenium were detected in concentrations that were higher than the MCL.

In the deep well water samples: In the sampling periods, except November only arsenic was detected in concentration that were higher than the MCL. In November, arsenic, iron, boron and vanadium were detected in concentrations that were higher than the MCL.

REFERENCES

1. Matthew, C., Larsen, Pixie, A. Hamilton, William H. Werkheiser, Water Quality Status and Trends in the United States, Monitoring Water Quality (2013), 19-57.
2. Larsen M.C. Water Resources Impact. American Water Resources Association (2012), 14(5), 3–7.



Figure 1. Graphs showing the temporal changes in elemental concentrations together with the mean rainfall.



Figure 1. Continued.

INVITED SPEAKERS (IS)

IS1-Absorption Spectrometry with Diode Lasers as A Technique for Contactless Measurements of the Parameters of Hot Zones.

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Temperature is the critical parameter of any combustion process. In particular, temperature of the combustion zone in mixing flows of oxidant and fuel characterizes the efficiency of a jet and fuel consumption. Absorption spectrometry with tunable diode lasers (TDLAS) is a powerful tool for contactless measurements of gas concentration and temperature in hot zones. The technique is based on the registration of the experimental transient absorption spectra of water molecules and fitting of the experimental spectra by the simulated ones constructed using the spectroscopic data bases. The temperature is inferred from the ratio of the integral intensities of the absorption lines with different low energy levels.

Two types of DLAS sensors were designed and tested for different types of combustion at low and high pressure conditions - plasma-assisted combustion in air-fuel mixing supersonic flows ($P < 1$ atm.) and combustion in a test chamber of high-speed ramjet air-breathing engines ($P > 1$ atm.). In case of relatively low total pressure the absorption lines are narrow, so that one can select a spectral range with several resolved or slightly overlapping absorption lines within a tuning range of a single DFB laser ($\sim 1.5 - 3$ cm⁻¹). For such a case the fitting can be adequately performed. The efficiency and potentials of the developed H₂O sensor is tested by detection of the parameters of the hot tail of combustion in the mixing supersonic flows at reduced pressure (250 – 400 Torr).

In case of high gas pressure (> 1 atm) the H₂O absorption lines are broadened which makes the selection of the resolved lines within the narrow tuning range of a single Distributed Feedback (DFB) laser very problematic. The alternative approach for the high pressure sensing of the hot zones is the using of two DFB lasers radiating in different spectral ranges. This approach extends the possibility to select the optimal strong absorption lines from different spectral ranges. Different combinations of the excitation wavelengths are theoretically examined with the emphasis on the attainable precision of the temperature evaluation.

The new TDLAS sensor for the measurements of the temperature up to 2500 K and gas pressure up to 3 atm is developed. The peculiarities of the TDLAS technique for the case of high pressure (up to 3.5 atm.) and temperature, as well as the algorithms for data processing will be shortly discussed in the talk. The efficiency of the developed technique was exemplified in the first set of the experiments on real test engine.

IS2- Enantioselective Analysis of Chiral Compounds by Using Capillary Electromigration Techniques

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Capillary electrophoresis (CE) is very useful technique for analytical scale separation of enantiomers. It is clear that even only at the expense of higher peak efficiency CE may allow to observe enantioseparation for certain chiral analyte-selector pairs where the separation power of HPLC is insufficient for achieving this goal. In addition, chiral CE offers almost unlimited possibility from the viewpoint of adjustment of separation factor. Together with aforementioned conceptual advantages CE offers some favorable technical characteristics for achieving high separation selectivity. Thus, chiral stationary phases (CSPs) in HPLC contain commonly limited and predefined amounts of a chiral selector, whereas the concentration of a chiral selector is easily variable and just limited by the solubility (for charged selectors also with Joule heating) of a chiral selector in a CE buffer. In addition, the combination of two or more (chiral) selectors is technically much easier and not associated with instrumental difficulties in CE compared to column-coupling in HPLC. Again, two columns are coupled with given amounts of the chiral selectors in HPLC whereas the ratio of chiral selectors in a combination can be easily optimized in CE. Thus, chiral CE offers really enormous flexibility from the viewpoint of the adjustment of the separation selectivity. This in combination with the inherently high separation efficiency makes chiral CE a very powerful technique for enantioseparations. Chiral CE is a powerful technique not only for separation of enantiomers but also for understanding fine mechanisms of enantioselective selector-select and interactions. However, in order to achieve this goal CE must be used in combination with other instrumental (for instance, nuclear magnetic resonance spectroscopy) and calculation (molecular modeling, molecular mechanics) techniques.

This presentation discusses most recent examples from our research on understanding of chiral recognition mechanisms of cyclodextrins by combined use of CE, nuclear magnetic resonance spectroscopy and molecular modeling calculations [1, 2]. Some new developments in enantioselective capillary electrochromatography (CEC) will be also discussed.

Keywords: separation of enantiomers, capillary electrophoresis, capillary electrochromatography.

Acknowledgment:

This project was supported financially in part by the Rustaveli National Science Foundation (RNSF) of Georgia (Project No. 217642).

References:

1. A. Gogolashvili, E. Tatunashvili, L. Chankvetadze, T. Sohajda, J. Szeman, A. Salgado, B. Chankvetadze, Separation of enilconazole enantiomers in capillary electrophoresis with cyclodextrin-type chiral selectors and investigation of structure of selector-selectand complexes by using nuclear magnetic resonance spectroscopy, *Electrophoresis* (2017) 38, 1851-1859.
2. A. Salgado, E. Tatunashvili, A. Gogolashvili, F. Gago, B. Chankvetadze, Structural rationale for the chiral separation and migration order reversal of clenpenterol enantiomers in capillary electrophoresis using two different β -cyclodextrins, *Phys. Chem. Chem. Phys.* (2017) 19, 27935-27939.

IS3- Smart Materials for Mercury speciation

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All chemical species of mercury are global pollutants due to their extremely high toxicity, ability to bioaccumulate and mobility. Both total Hg determination and Hg speciation analysis are analytical challenges because of very low environmental Hg concentrations. Nonchromatographic procedures for Hg species separation and enrichment combined with sensitive instrumental measurement by AFS or ICP-MS are widely used as an analytical approach for both total Hg quantification and Hg speciation analysis. The application of highly selective and efficient smart materials as sorbents for solid phase extraction (SPE) of Hg species ensures development of analytical methods with low determination limits and good reproducibility.

The comparison of extraction efficiency of different types of smart materials used for Hg species separation and enrichment is discussed in the present study. The comparison is in terms of the selectivity, limits of determination achieved, reproducibility of the analytical method and of the material synthesis procedure, simplicity of the analytical procedure, possibility for the application of sorption procedure during sampling. The compared smart materials include: ion imprinted polymers and silver nanoparticles (AgNPs) and composite materials based on them. Nanosized ion imprinted polymers (IIP) are smart materials with high selectivity toward the template species and ensured both selective separation of different Hg species and their enrichment. Silver nanoparticles combined both high surface/volume ratio and high chemical activity and in this way allowed Hg species reduction, amalgamation and separation. However, separation of nanosized sorbent by filtration and centrifugation is difficult and composite materials based on IIP incorporated on the surface of silicagel or Ag NPS incorporate in chitosan film are preferable materials for SPE of Hg species. In this way analytical methods developed are fast, simple and suitable for routine laboratory practice.

Keywords: smart materials, ion imprinted polymers, silver nanoparticles, Hg speciation

IS4- Heavy Metals Environmental Pollution Studies in The Republic of Macedonia

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Anthropogenic environmental changes, associated mainly with chemical pollution, lead to a degradation of the natural human environment. Among all chemical pollutants, trace elements are of a special ecological, biological and health significance. The production of energy and the consumption of natural resources are the main source of trace elements as contaminants. However, agricultural activities, especially the application of fertilizers and pesticides, also contribute significantly to trace metal pollution in the environment. Soil, as a part of the ecosystem, is vital for the survival of mankind which is closely connected to its productivity. Therefore, the surveys of the pollution with heavy metals of soil, waters, sediments, air and food on the whole territory of the Republic of Macedonia were performed. For that purpose, various spectrometric (atomic absorption spectrometry, inductively coupled plasma – atomic emission spectrometry, and inductively coupled plasma – mass spectrometry) and radioanalytical (neutron activation analysis) techniques were applied. Air pollution was investigated by the application of moss biomonitoring and dust samples (attic dust and house-hold dust). The pollution with heavy metals in the particular regions was additionally investigated. It was found that the highest pollution is present in the areas with abounded or active mines (Pb, Zn, Cu, As, Sb, Ni, Cr), metallurgical plants (Pb, Zn, Cd, Fe-Ni, Fe-Cr, Fe-Si, Fe, steel) or thermoelectric power plants. High content of some heavy metals were also found in the areas where their contents usually vary gradually across the geochemical landscape and depend on the geochemistry of the underlying lithology. The distributions of such elements reflect natural processes indicated by the elements that are either rarely or never involved in the industrial processes. The obtained data are statistically processed and spatial distribution maps for each specific element are prepared to give a proper interpretation of the obtained results.

Key word: heavy metals, pollution, air, soil, water, sediments, Republic of Macedonia

IS5- Fabrication of the temperature and pH-responsive grafted polymer brushes for stimuli-modulated bioseparations and orientations

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Modern analytical chemistry includes not only numerous fields of experimental and theoretical studies in term of applications and measuring methodologies of different specificity and requirements but also fabrication and application of the novel nanomaterials for biomedical engineering. Nowadays, many bioactive compounds, proteins, and therapeutic cells are used in biomedical engineering and medical practice. Materials and methods for the effective purification, orientation and analysis of such compounds and cells that retain their activities are greatly needed.

In last years, stimuli-responsive grafted polymer brushes are widely used for this purpose. Presented study describes various types of materials based on thermo- and pH-responsive polymers brushes for bioanalysis, bioseparations or orientations, e.g., thermo- or pH-responsive protein adsorption. Poly(N-isopropylacrylamide), poly(oligo(ethylene glycol)ethyl ether methacrylate, poly(butyl methacrylate) as well as its derivatives show stimuli-dependent transitions and conformational changes of the chains in response to external temperature or pH changes. Properties of the grafted brush coatings were determined using time of flight-secondary ion mass spectrometry, X-ray photoelectron spectroscopy, ellipsometry, contact angle measurements, atomic force microscopy and fluorescent microscopy.

Analyzed coatings may be potentially useful for various applications in biotechnology and biomedicine, for separation and orientation of the biomacromolecules or cells which can be achieved by simply changing the external temperature or pH, without using hard reagents that damage and deactivate biological compounds, proteins, and cells.

IS6- Determination of Site Specific Protein-PEGylation by MALDI-MS and IM-MS

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Biocompatible and non-toxic at high molecular weight Poly(ethylene glycol) (PEG) is a highly investigated polymer for the modification of biological macromolecules in many pharmaceutical and biotechnical applications. After the PEGylation of peptides and proteins, recognition and degradation of PEGylated biomolecules by proteolytic enzymes could be prevented. PEGylation changes the size of the biomolecules and decreases its renal filtration and altering biodistribution. Most important issue in the PEGylation is the different molecular weight distribution of PEG and the incorporation of modified PEG having different functional end group with different site of the protein without selectively. In this way, PEGylated peptide and proteins retain their activity depending on the steric effect of PEG chains close to active center longer. The typical problems still are available such as short circulating half-life, immunogenicity, proteolytic degradation, and low solubility of active peptide and proteins. To overcome these problems, PEGylation plays very important role. In the PEGylation, the number of the polymer chain attached to same biomolecule, molecular weight and the structure of PEG chains, the location of the PEG sites on the polypeptide and also the chemistry of the PEG attachment to the polypeptide are played very crucial role. Furthermore, for the elimination of the cross linking effect for PEG in the polypeptide modifications, recently mPEG have been used very widely. In the PEGylation studies, in order to solve the main problems, site specific starting reactivated PEG moieties could be used to be directed the PEG chains to the desired functional groups onto the polypeptide to retain the original activity of it. For this purpose, many different functionalized PEG chains have been used widely in recent years. In order to follow the site specificity of different functional group modified PEG and mPEG at different molecular weight ranges were used in this study and the site specificity of these PEG and mPEG moieties were examined in the case of Angiotensin II peptide and insulin hormone. To characterize the attachment site of PEG and mPEG on the polypeptides, Matrix-assisted Laser Desorption/Ionization-Mass spectrometry (MALDI-MS) and also Ion Mobility-Mass Spectrometry (IM-MS) techniques were used.

Keywords: Protein PEGylation, site specific, MALDI-MS, IM-MS

Acknowledgements

This work was supported partially by The Scientific and Technical Research Council of Turkey (Project Number: 115Z241) and Ministry of Development-Republic of Turkey (Project Number: 2016 K 121230).

References:

1. M.J. Roberts, M.D. Bentley, J.M. Harris. Chemistry for peptide and protein PEGylation, *Adv. Drug Deliv. Rev.*, 54 (2002) 459 –476.

IS7- Geography and Analytical Chemistry: Trace elements Analysis for Geographic Origin

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Analytical chemistry as information science will serve very important role for understanding many topics in both physical, natural, and social sciences in order to understand how each influences the other. Among most of the subdivisions of analytical chemistry geography is likely to be one of the most important discipline that helps us to understand the physical, environmental, and cultural interactions with an emphasis in interconnections and spatial dynamics between these environments. Geography is also an important spatial science and uses geospatial technology to understand the timer dependent behaviors of various environments. This could be achieved by using technologies such as satellite imagery, aerial photography, global positioning systems and geographic information systems as well analytical technologies.

There is an increasing interest by consumers for high quality food products with a clear geographical origin. These products are encouraged and suitable analytical techniques are needed for the quality control.

This overview concerns an investigation of the current analytical techniques that are being used for the determination of the geographical origin of food products. The analytical approaches have been subdivided into four groups; mass spectrometry techniques, spectroscopic techniques, separation techniques, and other techniques. The principles of the techniques together with their advantages and drawbacks, and reported applications concerning geographical authenticity are discussed. A combination of methods analyzing different types of food compounds seems to be the most promising approach to establish the geographical origin. Chemometric analysis of the data provided by the analytical instruments is needed for such a multifactorial approach. This study reviews some chemometric approaches for determining the geographical origin of some food and marble samples. As an example of environmental materials.

Keywords: Analytical Chemistry, Geographic Origin; Trace elements Analysis; Chemometric analysis

1.<http://www.opengeography.org/ch-1-intro-to-geographic-science.html>

IS8- Surface-enhanced Raman Scattering for Living Single Cell Analysis

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Single cell analysis aims to extract molecular level information from single eukaryotic cell other than a cell population. There are several techniques such as mass spectroscopy and capillary electrophoresis investigated in single cell analysis for last two decades. Although these techniques are very powerful due to their sensitivity, they require the destruction of the cells before analysis. Surface-enhanced Raman scattering (SERS) can be an alternative technique with its suitability to study real-time dynamics of molecular processes taking place in living cells, especially upon external stimulation, in a contactless, noninvasive, and nondestructive way[1]. The molecular information obtained such as a way can open up new venues to not only understand the cellular dynamics of a single cell but also novel ways of diagnosing diseases challenging humanity. The process we develop in our laboratory is presented in the figure below.

In this presentation, the focus will be on obtaining a reproducible SERS spectrum and the origin of the spectra obtained from living cells, which can be related to external stimuli[2]. Once the relationship between the spectral changes and the stimuli is established, molecular changes related to the stimuli can be monitored for diagnosis and prognosis.

The authors acknowledge financial support from TUBITAK (Project No:113Z554) and Yeditepe University.

Keywords: SERS, Single-Cell, Gold Nanoparticles, Detection

References:

- 1- Gamze Kuku, Mine Altunbek, and Mustafa Culha “Surface-Enhanced Raman Scattering for Label-Free Living Single Cell Analysis” *Anal. Chem.* (2017) 89 (21), 11160–11166.
- 2- Gamze Kuku, Melike Saricam, Farida Akhatova, Anna Danilushkina, Rawil F. Fakhrullin, and Mustafa Culha “Surface-Enhanced Raman Scattering to evaluate nanomaterial cytotoxicity on living cells” *Anal. Chem.* (2016), 88 (19), 9813–982.

IS9- Dark-Field Microscopy and Hyperspectral Imaging for Detection and Identification of Nanoscale and Microscale Particles in Cells, Tissues and Live Organisms

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Nanoscale and microscale particles, such as engineered nanoparticles, soot microparticles and plastic microparticles, may pose significant threat to organisms and environment. Detection and identification of nanosized materials in cells, tissues and organisms is still challenging, a number of conventional approaches exist, however, novel methods are being developed. The need in a fast and direct approach to visualise and characterise nanoparticles uptake and distribution is further stimulated by such emerging technologies as nanotoxicology, cell surface engineering, drug delivery and tissue engineering. Conventional approaches for nanoparticles imaging and characterisation both inside the cytoplasm and/or on the cell or tissue outer surfaces, such as transmission and scanning electron microscopies, are unquestionably potent tools, having excellent resolution and supplemented with chemical analysis capabilities. However, imaging and detection of nanomaterials in situ, in wet unfixed and even live samples, such as living isolated cells, microorganisms, protozoans and miniature invertebrates using electron or atomic force microscopy-based is practically impossible, because of the elaborate sample preparation requiring chemical fixation, contrast staining, matrix embedding and exposure into vacuum. Atomic force microscopy, in several cases, can be used for imaging and mechanical analysis of live cells and organisms under ambient conditions, however this technique allows for investigation of surfaces only. Therefore, a different approach allowing for imaging and differentiation of nanoscale particles in wet samples is required.

Dark field microscopy as an optical contrasting technique has been popular among researchers, however until recently the commercially available instrumentation was not well suitable for imaging of nanosized particles. Currently, several companies supply equipment for the so-called enhanced dark field microscopy (EDF) based on using higher numerical aperture light condensers and variable numerical aperture objectives, which allows for imaging of nanoscale particles (up to 20 nm) using almost conventional optical microscopy methodology. In addition, hyperspectral imaging can also be realised using the dark-field microscopy instrumentation via spectrometer\CCD camera interfaced with the optical microscope. Here we report our results on application CytoViva dark-field and hyperspectral microscopy instrument assembled on Olympus BX51 upright microscope equipped with fluorite 100x objective and DAGE CCD camera.

In our studies, we have successfully employed enhanced dark-field microscopy to visualise a number of nanoscale particles and materials. This allowed us to better characterise the materials and their impact on cells and organisms under ambient conditions. In particular, silver nanoparticles deposited on the cell walls of microorganisms were imaged in aqueous media [1,2]. EDF microscopy was essential for imaging of cellular uptake and intercellular distribution of various nanoscale particles (Ag, Au, ZnO, TiO, carbon nanotubes, halloysite nanotubes and halloysite-based composite materials) by a number of human cell lines [2-6]. We also applied EDF along with hyperspectral mapping based on preliminary collected spectral libraries to detect magnetic nanoparticles and clay nanotubes in tissue engineering scaffolds and multicellular clusters [7,8]. We also used EDF to visualise nanoparticles delivery and spatial distribution of various nanoparticles into *Caenorhabditis elegans* nematodes [9] and *Paramecium caudatum* protozoans [10] in nanotoxicity studies.

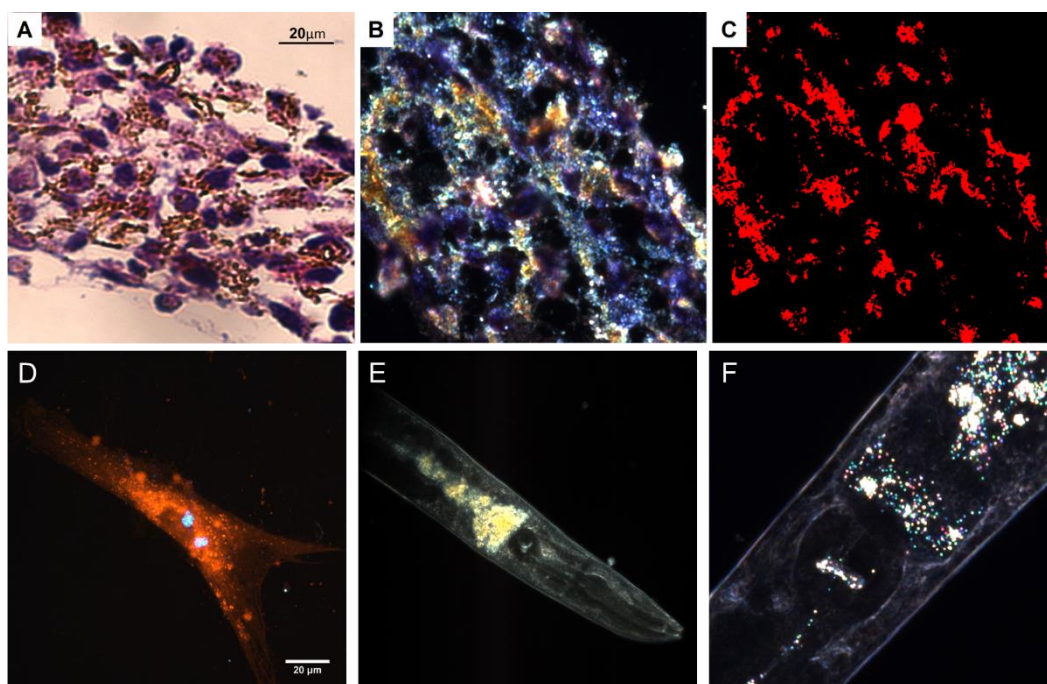


Figure 1. Optical microscopy images of thin-sectioned haematoxylin-eosin stained multicellular cluster fabricated using magnetically-functionalised A549 cells and fibroblasts (a); hyperspectral microscopy image of the multicellular cluster (b); corresponding hyperspectral map (matching the image in b) illustrating the distribution of magnetic nanoparticles (red) [8]; dark-field and epifluorescence image of halloysite\quantum dots composite in fibroblasts [6]; distribution of magnetic (e) and silver nanoparticles in *C. elegans* nematodes imaged using dark-field microscopy.

We have demonstrated the potential of EDF in imaging of nanoscale materials and qualitative and semi-quantitative detection and spatial mapping of nanomaterials in several popular biological models (Figure 1). Unlike many other techniques, EDF is easy in sample preparation and handling, allowing for working even with live cells and organisms. Imaging of distribution and colloid behaviour of nanoscale particles in nematode intestines or real-time imaging of cellular uptake of nanotubes by human cells can be easily realised using EDF. Hyperspectral imaging within Vis-near IR and corresponding chemical mapping of nanomaterials distribution is also promising.

Keywords: dark-field microscopy, hyperspectral microscopy, nanotoxicity, biomaterials

Acknowledgement: This study was performed according to the Russian Government Program of Competitive Growth of Kazan Federal University and supported by Russian presidential grants for young scientists (MD-6655.2018.4.)

References:

1. S.A. Konnova, A.A. Danilushkina, G.I. Fakhrullina, F.S. Akhatova, A.R. Badrutdinov, R.F. Fakhrullin Silver nanoparticles-coated "cyborg" microorganisms: rapid assembly of polymer-stabilised nanoparticles on microbial cells, *RSC Adv.* (2015) 5 13530-13537.
2. S.A. Konnova, Y.M. Lvov, R.F. Fakhrullin Nanoshell Assembly for Magnet-Responsive Oil-Degrading Bacteria, *Langmuir* (2016) 32 12552-12558.
3. M.R. Dзамukova, E.A. Naumenko, Y.M. Lvov, R.F. Fakhrullin Enzyme-activated intracellular drug delivery with tubule clay nanoformulation, *Sci. Rep.* (2015) 5 10560.
4. R. Yendluri, Y. Lvov, M. DeVilliers, V. Vinokurov, E. Naumenko, E. Tarasova, R. Fakhrullin Paclitaxel Encapsulated in Halloysite Clay Nanotubes for Intestinal and Intracellular Delivery, *J. Pharm. Sci.* (2017) 106 3131-3139.
5. G. Kuku, M. Saricam, F. Akhatova, A. Danilushkina, R. Fakhrullin, M. Culha Surface-Enhanced Raman Scattering to evaluate nanomaterial cytotoxicity on living cells, *Anal. Chem.* (2016) 88 9813-9820.
6. B. Micó-Vicent, F.M. Martínez-Verdú, A. Novikov, A. Stavitskaya, V. Vinokurov, E. Rozhina, R. Fakhrullin, R. Yendluri, Y. Lvov Stabilized Dye-Pigment Formulations with Platy and Tubular Nanoclays, *Adv Func Mater* (2017) 1703553.

7. E.A. Naumenko, I.D. Guryanov, R. Yendluri, Y.M. Lvov, R. F. Fakhrullin Clay nanotube–biopolymer composite scaffolds for tissue engineering, *Nanoscale* (2016) 8 7257-7271.
8. M.R. Dзамukova, E.A. Naumenko, E.A. Rozhina, A.A. Trifonov, R.F. Fakhrullin Cell surface engineering with polyelectrolyte-stabilised magnetic nanoparticles: a facile approach for fabrication of artificial multicellular tissue-mimicking clusters, *Nano Res.* (2015) 8 2515-2532.
9. G.I. Fakhrullina, F.S. Akhatova, Y.M. Lvov, R.F. Fakhrullin Toxicity of halloysite clay nanotubes *in vivo*: a *Caenorhabditis elegans* study, *Environ Sci: Nano* (2015) 2 54-59.
10. M. Kryuchkova, A. Danilushkina, Y. Lvov, R. Fakhrullin Evaluation of toxicity of nanoclays and graphene oxide *in vivo*: a *Paramecium caudatum* study, *Environ. Sci.: Nano* (2016) 3 442-452.

IS10- The Analytical Chemistry for Industrial Needs and Scientific Laboratory Accreditation

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Decisions on such issues as human and animal health, consumer and environmental protection, international trade and other kind of activities are all strong dependent on the validity of analytical measurements and the interpretation placed upon them by professional analytical chemists. In this regard, the University faculties are now subject to “quality audits” of their teaching and it no doubt that proper attention to quality is vital for analytical chemistry. At the last time for educators of many countries, the critical question is: “Did the modern Universities and analytical chemistry education is sufficient for practical activity, training and makes use of the opportunities for reform or did they stumble on the obstacles?”

This report are devoted to comparing requirements for personals according to International Standard ISO 17025-2006 “General requirements for the competence of testing and calibration laboratories” and degree of descriptions to teaching in some of Turkey and Ukraine Universities that are formulated for general qualifications (i.e., Bachelor’s, and Master’s) and professional qualifications as objectives under three headings: knowledge and understanding, skills and abilities, and judgment and approach.

We think, that in many cases the sufficient of Bachelor’s and Master’s students for future activity as personnel for modern industrial and scientific analytical chemistry laboratories are not meet requirement of education, training, technical knowledge and experience for their assigned functions.

It seems evidently that forming of “National Register of Chemist-Analytics” and its joint activity with “European Association for Quality Assurance Agency in Higher Education” will be step towards improving of educational area, standard, competence and professional skill personnel of industrial analytical chemistry laboratories including in their professional testing activity, as well as quality and safety control of substance and materials.

IS11- Pine Tree as Environmental Pollution Indicator: X-Ray Fluorescence Studies

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Scots pine (*Pinus Silvestris*) is widely distributed around Europe, especially northern part and is used as a suitable tool for diagnostics of anthropogenic pollution and nutritional status of forests. Pine trees are widely used in an air and soil pollution studies. Various parts of the pine, such as fine roots, tree core, pine needles, have been widely used as environmental indicators. Tree needles are often sampled for such studies. It is known that certain elements in the pine needles changes with time. These changes show dynamics of uptake, transportation, storage and retranslocation processes of different nutrients and pollutants. Most common analytical methods, used in the analysis of biological samples, gives us the mean concentration of studied elements. There is limited information about the elemental distribution along the length of pine needle.

The practise used e.g. in northern Europe is to sample needles during the dormant period from either the current or in some cases several needle age classes from a fixed position of a number of trees in a stand. The sampled needles are then dried and ground for nutrient analysis. The sampling aims to minimise the spatial and temporal variation in needle nutrient concentrations for estimating the availability of nutrients in soil through needle analysis. For the current studies, the needles were sampled at the certain time interval during the whole life of pine needles. Needles were sampled at one or two week intervals (current needles) and at monthly intervals (C+ needles) from mature Scots pine (*Pinus sylvestris* L.) trees at Jugla (56°56' N, 24°26' E) and other a relatively unpolluted sites in Latvia. For the scanning experiments, six 25-year-old trees were randomly chosen for sample trees. The branch chosen for sampling from each tree was situated at the height of five meters from the ground and on south side of the canopy. At every sampling time, one needle from the middle of the three branch was chosen for analysis, and transported to the laboratory in a plastic bag. Altogether more 200 needles were analysed by scanning Energy Dispersive X-Ray fluorescence [1] and electrothermal AAS methods.

The aim of this study was to determine the distribution of different elements along the length of single Scots pine needles of different age. The scanning EDXRF measurements were made continuously at only a few mm distance along the needle length.

In this study, Ca, Mn, Fe, Sr and Br that increased with needle age, were considered as poorly mobile elements; K, Ni and Rb that decreased with needle age were considered clearly mobile; and Cu and Zn were relatively mobile elements. Similar classification for Ca, Mn, Fe, K and Zn was presented by Nieminen and Helmisaari [2]. Wytenbach and Tobler (1988) presented similar classification with the exception of Fe and Br, which were classified in the intermediate group, based on their pattern of seasonal variation.

Keywords: Pine needles, Energy dispersive X-Ray Fluorescence, nutrient concentration, mobility

References:

- 1 Viksna, A., Selin Lindgren, E. and Standzenieks, P. Analysis of pine needles by XRF scanning techniques. X-Ray Spectrom. (2001)30, 260-266.
2. Nieminen, T. and Helmisaari, H-S. Nutrient retranslocation in *Pinus sylvestris* L. growing along a heavy metal pollution gradient. Tree Physiology (1996) 16, 825-831.

IS12- Design Principles of Optical and Electrochemical Sensors for The Determination of Antioxidant Ability and Reactive Species

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Antioxidant activity (AOA)/capacity (TAC) levels are usually measured so as to classify food, to diagnose oxidative stress conditions and identify/quantify the corresponding antioxidative defenses in human sera and tissues. Compared with classical instrumental methods, optical sensors for AOA measurement have many advantages such as low cost, flexibility, speed, remote control, miniaturization and on site/*in situ* analysis. Optical sensors evaluate analytical information by using optical transduction techniques (absorbance, reflectance, luminescence, etc.) and may also be useful in colored and turbid media. On the other hand, bioelectrochemical sensors using modified electrodes, often with the aid of nanotechnology, better catalyze the bioelectrochemical reactions leading to the quantification of oxidant/antioxidant species. The electrochemical sensing strategy is based on the measurement of oxidative damage produced on lipids, proteins, and DNA, and restoration of their original electrochemical signals in the presence of protective antioxidants. Most electrochemical sensing approaches focus on cytochrome *c*, superoxide dismutase and DNA probes.

The working principle of optical nanoprobess using metal nanoparticles for AOA estimation may be broadly classified under four categories: (i) formation and growth of noble metal nanoparticles, by chemical reduction of Au(III) or Ag(I) salts with antioxidants, resulting in surface plasmon resonance (SPR) absorbance enhancement; (ii) aggregation/agglomeration of the preformed noble metal nanoparticles giving rise to shifts in SPR band maxima; (iii) disintegration of existing nanoparticles by oxidant species causing an attenuation in band intensities; (iv) specific interaction of nanoparticles, under the influence of antioxidant analytes (*e.g.*, in ceria nanoparticles, causing oxide (O-2p) and Ce(IV) (Ce-4f) electronic transitions), by possible magnification with indicator redox-active dyes (such as tetramethyl benzidine) to increase assay sensitivity. Naturally, these mechanisms can coincide in various aspects.

The widely-used CUPRAC (Cupric Reducing Antioxidant Capacity) total antioxidant capacity (TAC) assay was introduced by our research group to world literature in 2004^[1]. Antioxidants react with the CUPRAC reagent (cupric neocuproine) to produce the Cu(I)-neocuproine (Nc) chromophore measured spectrophotometrically at 450 nm. A low-cost CUPRAC antioxidant sensor was developed by immobilizing the Cu(II)-Nc reagent onto a perfluorosulfonate Nafion® membrane, on which the color developed with antioxidant addition^[2]. A similar optical sensor based on ferric-phenanthroline immobilization on a Nafion membrane was recently used for TAC determination^[3]. For assaying hydroxyl radicals, a salicylate probe could be converted to CUPRAC-reactive dihydroxybenzoic acids (DHBAs) in a Fenton system, which in turn could be measured with the CUPRAC sensor; the activity of •OH scavengers could be measured by the bleaching of color on the Nafion membrane sensor. This sensor can be indirectly used for measuring hydrogen peroxide and superoxide anion levels as well as for the assay of their scavenger antioxidants. A colorimetric sensor capable of simultaneously measuring oxidative status (in terms of the hazard produced by ROS) and antioxidant activity (in regard to ROS-scavenging ability of antioxidant compounds) was developed. The colored semi-quinone cations, formed with ROS oxidation of N,N-dimethyl-*p*-phenylene diamine (DMPD), were formed in solution and immobilized on a Nafion membrane^[4]. By exploiting the localized surface plasmon resonance absorption of noble metal (usually gold and silver) nanoparticles, polyphenols and vitamins (C,E) could be determined^[5], along with biothiols^[6] and nitrite^[7] after proper modification of gold nanoparticles.

Some of the future challenges regarding the design of new sensors and nanoprobe for antioxidant research should address specificity, sensitivity, stability and surface inactivation, response time and extended use, repeatability/reproducibility, and filling the gap between science, technology and commercialization. As oxidative stress gives rise to a number of diseases, such sensors are expected to be used more often in the future as a part of point-of-care diagnostic devices for determining wellness parameters within the context of personal nutrition and wellness advisor systems.

Acknowledgement: The authors wish to thank the 'Research & Application Centre for the Determination of Food Antioxidants' of Istanbul University (IU-GAAM) for the research facilities provided.

REFERENCES

- [1] Apak, R., Güçlü, K., Özyürek, M., Karademir, S. E. *J. Agric. Food Chem.* **2004**, 52, 7970-7981.
- [2] Bener, M., Ozyurek, M., Guclu, K., Apak, R. *Anal. Chem.* **2010**, 82, 4252-4258.
- [3] Bener, M., Apak, R. *Sens. Actuators B* **2017**, 247, 155-162.
- [4] Cekic, S. D., Avan, A. N., Uzunboy, S., Apak, R. *Anal. Chim. Acta*, **2015**, 865, 60-70.
- [5] Ozyurek, M., Gungor, N., Baki, S., Guclu, K., Apak, R. *Anal. Chem.* **2012**, 84, 8052-8059.
- [6] Guclu, K., Gungor, N., Ozyurek, M., Baki, S., Apak, R. *Anal. Chim. Acta* **2013**, 794, 90-98
- [7] Uzer, A., Can, Z., Akın, I., Erçağ, E., Apak, R. *Anal. Chem.* **2014**, 86, 351-356.

IS13- Challenges and Strategies for the Removal of Toxicants from Water

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Water is the vital source of life. It is essential to life because our body contains 55% to 78% of water. It is recommended that the human body requires at least 1 to 2 litre of water per day for proper functioning. Contamination of water bodies by the different sources created a large number of troubles in the entire globe. Water is contaminated through a number of toxicants including organic pollutants (pesticides, dyes, pharmaceuticals, and organic solvents), inorganic pollutants (salts and metals) and also with thermal and radioactive pollutants. Pollution due to metal ions has got considerable importance and extensive attention; because these pollutants are incapable of undergoing biological degradation and cause many adverse effects to all living organisms by entering into the food chain and their accumulation in various tissues. Many strategies have been employed for the purification of polluted water including chemical and physical approaches. Among them adsorption technique (solid phase extraction) is most widely employed by various groups. Subsequently, the calixarenes are a remarkable class of synthetic cyclic compounds, generally applied as sorbents in various fields such as separation of unwanted ionic as well as neutral species from aqueous environment, nanotechnology, chromatography, catalysis, sensor technology and etc. [1-2]. Herein, different designing approaches of various calixarene based materials and their applications in separation of different toxicants from aqueous environment and in the field of sensor technology will be discussed.

Keywords: Calixarene, Supramolecular Chemistry, Molecular Machines, Separation Science, Membrane Technology, Sensor Technology.

References

- [1] CIA – The world fact book". Central Intelligence Agency Retrieved 20 December 2008.
- [2] C.D Gutsche, in: J.F. Stoddart. (Ed.), Calixarenes Revisited; Royal Society of Chemistry, Cambridge, England, 1998.

IS14-From Graphite to Graphene: A Novel Electrochemical Approach

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Graphene is a material which has formed by two dimensional planar sheets of sp^2 -bonded carbon atoms and was discovered in 2004 [1]. It has a significant interest because of its amazing physical, mechanical and electrical properties [2,3]. Application areas of graphene such as energy storage systems, bio-electrochemistry, anti-corrosion coating, sensors, opto-electronics and etc. are growing up day by day since it has brought a new approach to carbon based systems [2-4]. Since importance of graphene is increasing rapidly in materials research, production of it by different methods is having attention by researchers. The production of graphene could be done by four main methods: epitaxial growth on silicon carbide (SiC), chemical vapor deposition (CVD), chemical derivation and electrochemical exfoliation [5]. The prepared graphene in these methods have many preparation steps such as separation, purification, drying, modification, and etc. [5]. In the preparation of graphene based electrode, there is an extra step: immobilization [5]. Preparation of graphene based electrode in one step by electrochemical methods has great potential for further application of these materials in many areas.

Cyclic voltammetry is one of the most used electrochemical methods, can bring a novel approach in the production of graphene based electrode in one step [2,4,6]. Graphite based electrode can be converted directly to graphene based electrode in one step by using of cyclic voltammetry. The method can allow the controlled formation of graphene layers from the graphite by changing some parameters such as cycle number, scanning rate, electrolyte composition and etc.. The method also allows the functional production of hetero atom doped graphene [2,4,6]. The functional groups such as hydroxyl, carbonyl and epoxy can be saved or converted to another group by the arranged of scanned potential range in cyclic voltammetry [2,4,6]. This novel, fast, low cost, and environmentally friendly method can be used in the production of graphene based electrodes for many application areas such as sensors, optics, energy storage, electronic devices, and etc..

Keywords: Graphene, Hetero atom doped graphene, Cyclic voltammetry, Graphene electrode

References:

1. K.S. Novoselov, A.K. Geim, S. V Morozov, D. Jiang, Y. Zhang, S. V Dubonos, I. V Grigorieva, A.A. Firsov, Electric field effect in atomically thin carbon films, *Science*. 306 (2004) 666–669.
2. H. Gürsu, M. Gençten, Y. Şahin, One-step electrochemical preparation of graphene-coated pencil graphite electrodes by cyclic voltammetry and their application in vanadium redox batteries, *Electrochim. Acta*. 243 (2017) 239–249.
3. D.A.C. Brownson, D.K. Kampouris, C.E. Banks, Graphene electrochemistry: fundamental concepts through to prominent applications, *Chem. Soc. Rev.*, 41 (2012) 6944-6976.
4. H. Gürsu, M. Gençten, Y. Şahin, Preparation of Sulphur-Doped Graphene-Based Electrodes by Cyclic Voltammetry: A Potential Application for Vanadium Redox Flow Battery, *Int. J. Electrochem. Sc.* 13 (2018) 875-885.
5. V .Skakalova, A. Kaiser. Graphene: Properties, Preparation, Characterisation and Devices, 2014, Woodhead Publishing Limited, Amsterdam
6. Y. Şahin, H. Gürsu, M. Gençten.'A method in order to generate graphene based electrode' PCT/TR2017/050121

IS15- Misnomers and oxymorons

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Using correct terminology is essential in dissemination of knowledge, whether this is in a scientific article, a presentation or even a simple conversation. Scientists should be able to present their opinions, scientific findings and research results efficiently and accurately. In this talk, the meaning and examples for misnomers and oxymorons will be presented. A *misnomer* is a wrong or inaccurate name or designation. It is a term or a name which is not proper or not reflecting the actual content. A widespread example is the use of “tin can” instead of a metal can as a container for a soft drink or food today. On the other hand, the term *oxymoron* refers to two single words that do not logically go together. The examples are “Research University”, “Sustainable Growth” and “Living Organism”. Sometimes some terms become misnomers through time; such as “Organic Chemistry” since today it does not deal only with organic compounds. Other terms will also be handled and discussed regarding their ability to reflect truth.

Keywords: Misnomer, oxymoron, terminology

References:

1. <http://below2c.org/2015/03/is-sustainable-growth-an-oxymoron/>
2. <https://www.grammarly.com/blog/misnomer/>
3. <https://en.m.wikipedia.org/wiki/Oxymoron>

IS15- Developments and applications in enzyme based photoelectrochemical biosensors

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Photoelectrochemistry is the science of measuring and evaluating the electrical current generated by illumination of an electrode or metal in an electrolyte. Photoelectrochemical studies are usually carried out to provide a better understanding of the nature of the electrode-solution interface. Photoelectrochemistry has often been used in electrochemical analysis in recent years in addition to being widely-used in industrial applications, including mechanisms for illumination (spectroelectrochemical research) and use of photovoltaic processes¹. One of important application of photoelectrochemistry is the development of enzymatic biosensor and their applications area in analytical science and technology. Because, photoelectrochemical measurements possessing several significant advantages such as reduced undesired background signal, high sensitivity, fast response, simple, cheap and portable sensor system design, easy integration and miniaturization, and extension to light addressable sensors²⁻⁴.

A light source, electrochemical cell and electrochemical workstation with the traditional three electrodes are the main components of the photoelectrochemical instrument system. The choice of the electrode material or modification of the electrode surface is especially important in terms of sensitivity and selectivity in photoelectrocatalytic systems and construction of an enzymatic photoelectrochemical biosensor. For this purpose, the following photoactive materials have been used in the modification of electrode surfaces: i) semiconductors and quantum dots, ii) complexes and derivatives iii) organic materials, iv) conductive polymers such, and v) composite materials formed by the combination of organic or inorganic material or two inorganic materials having different band gaps²⁻⁴. Composite materials have been used more often in photoelectrochemical analysis systems since they have higher photoelectric conversion efficiency than single materials. In this study, we describe the principles, mechanisms of and applications of enzyme based photoelectrochemical biosensors and the new trends in this area.

Keywords: Photoelectrochemical biosensor; enzyme; voltammetry; amperometry

References:

1. A.J. Bard, L.R. Faulkner, *Electrochemical Methods, Fundamentals and Applications* (2001) Second Edition, John-Wiley
2. Z.X. Zhang, C.Z. Zhao, Progress of photoelectrochemical analysis and sensors, *Chin. J. Anal. Chem.* (2013) 41 436-444.
3. W.W. Zhao, J.J. Xu, H.Y. Chen, Photoelectrochemical enzymatic biosensor, *Biosens. Bioelectron.* (2017) 92 294-304.
4. Z. Yue, F. Lisdat, W.J. Parak, S.G. Hickey, L. Tu, N. Sabir, D. Dorfs, N.C. Bigall, Quantum-dot-based photoelectrochemical sensors for chemical and biological detection. *ACS Applied Materials & Interfaces* (2013) 5 2800-2814.

ORAL PRESENTATIONS (OP)

OP1- Development of a Noninvasive Method for Biomarker Determination in Human Breath for Early Diagnosis of Lung Cancer

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Lung cancer is the most common kind of cancer in the world and it is a leading cause of cancer death by late diagnosis. Early diagnosis is one of the most important strategies to reduce lung cancer morbidity rate and improve the survival rate. To improve the overall survival, many screening methods including tomography and radiography have been used. However, current screening techniques for lung cancer are always expensive and not comfortable, even harmful for patients. The detection of molecular bio markers specific for lung cancer in human breath is a relatively new strategy for early cancer screening and detection. This technique is non-invasive, painless, easy to perform and no risk to patients. Exhaled breath contains 3500 different kinds of volatile organic compounds (VOCs) [1,2]. Breath analysis has taken a long time to be a useful tool because of the lack of standardized breath collecting methods and the low concentrations of VOCs which are present in nano or picomolar level. On the other hand, the direct sampling does not provide the necessary detection limits therefore; there is a need for development of reliable sampling techniques for breath analysis.

In this study, we aimed to develop a new sensitive and reliable method for the determination of biomarkers of lung cancer in human breath by solid phase microextraction (SPME) sampling. For this purpose, the thin film of polymers was being coated electrochemically on a stainless steel wire by using cyclic voltammetry and following the surface characterization by using several techniques, the fibers were placed in breath sampling device to enrich the analytes in human breath.

For this purpose, breath samples of the cancerous and asthmatic control groups were taken in cooperation with the Faculty of Medicine of the University of Ege, and these compounds collected on the SPME fiber were subjected to GC-MS with thermal desorption to perform the desired sensitivity analysis. Following the optimization studies, the method was validated for a number of volatile organics, including hydrocarbons, alcohols, ketones and aldehydes. Breath VOCs were assessed using a chemometric approach using principle component analysis (PCA). A good classification of cancer cases and healthy volunteers for lung cancer diagnosis has been obtained. It has been shown for the first time in our country that SPME fibers prepared in our research laboratory can be used for early detection of lung cancer.

This study was supported by Tübitak (113Z672).

Keywords: Breath analysis, SPME, Chromatography, Lung Cancer

References:

- 1 B. Buszewski, M. Kêsy, T. Ligor, A. Amann, Biomed. Chromatogr. **2007**, 21, 553-566..
- 2.T.H. Risby, Ed. A. Amann, D. Smith, World Scientific, **2005**.

OP2-Mass Spectrometry Based Targeted and Untargeted Metabolomic Analysis for Early Diagnosis of Breast Cancer Using LC-ESI-qTOF-MS and LC-ESI-MS/MS

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Metabolomics analysis has a great potential in the elucidation of the mechanism and the early diagnosis of breast cancer as well as other diseases (1-2). Today's innovative technologies permit comprehensive screening of the genome, transcriptome, proteome, and metabolome holds an immense potential for understanding mechanism of diseases, for their early diagnostics, choosing personalized therapeutic strategy and assessing its effectiveness. In metabolomics analysis based on mass spectrometry analytical techniques are compared to the others come to the forefront. According to the analysis conducted in metabolomics studies are divided into two groups as targeted and untargeted metabolomics studies. Untargeted studies were searching for new biomarkers, targeted studies carried out for the determination of the amount of known metabolites can easily be applied in routine analysis.

In this study, comparative metabolomic analysis of plasma samples obtained from patients with breast cancer was carried out based on untargeted with LC-ESI-qTOF-MS. Three different chromatographic columns were tested for LC-ESI-qTOF-MS based metabolomics profiling. The optimal separation was achieved on ZIC-HILIC column (100x4.6mm I.D., 5 µm) at a column temperature of 40°C using a mobile phase of 0.1% formic acid in acetonitrile and 0.1% formic acid in water (80:20, v/v) by gradient elution at a flow rate of 0.3 mL/min. The plasma samples were compared with control group in order to identified potential biomarkers for diagnosis. 174 plasma samples from early stage cancer patients, 97 plasma samples from metastatic cancer patient and 105 plasma samples from healthy women were used for LC-ESI-qTOF-MS based metabolomic profiling.

Targeted analysis for selected three metabolites (citrulline, tyrosine and choline) that found significantly changed in breast cancer patients were performed using LC-ESI-MS/MS. The specific transition and collision energy employed in the quantification of the metabolites are m/z 176→113 for citrulline, m/z 182→136 for tyrosine and m/z 104 → 60 for choline. The optimal separation was achieved on ZIC-HILIC column (150x4.6mm I.D., 5 µm) using a mobile phase of 0.1% formic acid in acetonitrile and 0.1% formic acid in water (70:30, v/v) at a flow rate of 0.3 mL/min. The LC-ESI-MS/MS method has been developed and validated according to FDA Bioanalytical Method Validation guideline. In this study, the advantages and disadvantages of targeted and untargeted analysis to each other were also discussed.

References:

- [1] Anthony, L., Angelina, N., Tony, K., Kristina, C.O., Tina, M. C., J. Chromatog. B (2014) 944 166–174.
- [2] Nemutlu E, Zhang S, Gupta A, Juranic NO, Macura SI, Terzic A, et al. Physiol Genomics (2012) 44 386-402.

This work was supported by The Scientific and Technological Research Council of Turkey (TÜBİTAK) (Project no: 114Z390).

OP3- Speciation of chromium in waters using phosphomethylated polystyrene-divinylbenzene hairy brushes prior to inductively coupled plasma mass spectrometric determination

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Chromium is found in two oxidation states in waters, as Cr(III) and Cr(VI). The former is essential to mammals for the maintenance of glucose, protein and lipid metabolism whereas Cr(VI) is considered to be toxic for both plants and animals causing several diseases like liver, kidney, lung and gastrointestinal cancers. Thus, its determination is very important in aquatic environments.

The present work describes the synthesis and application of a novel sorbent, phosphomethylated triethylene tetramine (TETA) modified poly-oligomeric (ethylene glycol) methyl ether methacrylate block poly-glycidyl methacrylate (POEGMA-b-PGMA) grafted polystyrene-divinylbenzene (PS-DVB), for the determination of Cr (III) and Cr(VI) in water samples using inductively coupled plasma mass spectrometry. Batch type adsorption experiments were performed and the optimum experimental conditions viz., pH, sorbent amount, contact time and temperature have been established. The sorption process was investigated both from a kinetic perspective (intraparticle diffusion, pseudo-first order, pseudo-second order models) and also in terms of Freundlich, Dubinin-Radushkevich and Langmuir isotherm models to characterize the uptake of chromium species by the sorbent. The proposed method was applied for the determination of chromium species in ultrapure, tap, bottled drinking and waste water samples. The overall results have demonstrated that the sorbent is a good candidate for the removal of Cr(III) and Cr(VI) from aqueous solutions.

Keywords: Chromium, Speciation, Inductively coupled plasma mass spectrometry, Waters

References:

1. J.L Manzoori, H. Mohammed, F. Shemirani, Chromium speciation by a surfactant—coated alumina microcolumn using electrothermal atomic absorption spectrometry, *Talanta* (1994) 42 1151–1155.
2. S. Chen, S. Zhu, Y. He, D. Lu, Speciation of chromium and its distribution in tea leaves and tea infusion using titanium dioxide nanotubes packed micro-column coupled with inductively coupled plasma mass spectrometry, *Food Chem* (2014) 150 254–259.

OP4 “Cryogel-cemented” Humic Acid as an Alternative Separation Medium

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A new functional monolithic material (PHEMA-HA) was prepared by cryogellation of 2-hydroxyethylmethacrylate (HEMA) in presence of humic acid (HA) through a straightforward procedure [1, 2]. The material was characterized by means of various methods and techniques (such as FTIR, SEM, mercury intrusion porosimetry, surface charge measurements (e.g. pH_{pzc}), solubility tests, water permeability) to show successful immobilization of HA into monolithic cryogel matrix. It was evidenced that the proposed procedure led preparation of a material with a high hydrolytic stability which is untypical for HA itself.

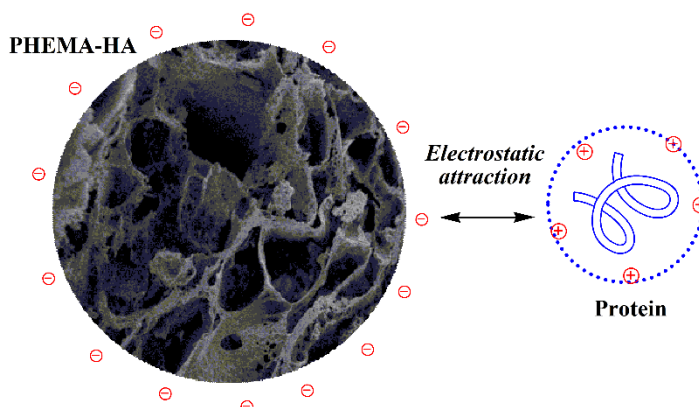


Figure 1. A representative view for interaction of PHEMA-HA with proteins

The functionality of PHEMA-HA was studied by utilizing it as a protein ion-exchange chromatography stationary phase (Figure 1). The experiments were performed by using some model proteins, and the effect of mobile phase pH and the gradient slope (i.e. change in modifier, NaCl, concentration with time) on protein retention was studied. In the study, predominance of ion-exchange mechanism in protein retention on PHEMA-HA was evidenced. Finally, baseline separation was achieved for some of the studied model proteins from their aqueous mixtures. Hence, the functionality of HA has been successfully adapted to the chemistry of monolithic cryogels, and PHEMA-HA is proposed as an efficient monolithic separation medium.

Keywords: Chromatography, Cryogel, HPLC, Humic acid

References:

1. K. Şenlik, O. Gezici, I. Guven, A.I. Pekacar, Adsorption of nitroaniline positional isomers on humic acid-incorporated monolithic cryogel discs: Application of ligand-exchange concept, *J. Environ. Chem. Eng.* 5 (2017) 2836-2844.
2. A.E. Özkan, İ. Güven, O. Gezici, Protein ion-exchange chromatography on a biomacromolecule-immobilized monolithic cryogel, *Turk. J. Chem.*, in press, doi:10.3906/kim-1612-65.

Acknowledgement: Authors wish to thank Niğde Ömer Halisdemir University for financial support (Project no: FEB 2015/17 BAGEP).

OP5- Selective Determination of Cr(VI) by a Novel FI-SPE-FAAS Method Using Tris(2-aminoethyl)amine-Functionalized Silica Gel

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Chromium (Cr) is widely used for metal smelting, electroplating, leather tanning and other industrial products. Cr can mainly be found in the environmental samples to be Cr(III) and Cr(VI). While Cr(III) is an essential trace element for mammals, Cr (VI) is very harmful to living beings even at the trace level. If chromium is ingested excessively in the human diet, the toxicity of Cr(VI) can stimulate and corrode the skin and Cr(VI) is more easily absorbed by the body, which has been recognized as a kind of carcinogen [1].

To eliminate matrix ions from a sample solution and to increase the metal concentration before the measurement step, a preconcentration and separation method is required such as solid-phase extraction (SPE), solvent extraction, cloud point extraction, and precipitation. Recently, among these preconcentration methods, solid-phase extraction has been frequently used in online and offline modes for the separation and preconcentration of trace metal ions of various inorganic and organic materials. The online SPE has several advantages as it reduces the contamination of the sample, provides a high preconcentration factor and high sampling frequency, consumes a small volume of the sample and/or organic solvents, and can simply be operated and be coupled with many modern instruments.

In this study, an online preconcentration procedure using silica gel functionalized with tris(2-aminoethyl)amine was developed for the selective determination Cr(VI) ions in the presence of Cr(III) ions in environmental water samples. The effects of parameters such as pH, sample and eluent flow rates, matrix ions, and type, concentration and volume of eluent on the online preconcentration of these ions were examined. The proposed method was successfully applied for the selective determination of Cr(VI) concentrations in water samples.

Keywords: Chromium, online SPE, preconcentration

References:

1. Wu, H., Wang, J., Xu, J., Jiang, Y., Zhang, T., Yang, D., and Qiu, F. Environmentally friendly cleaner water-soluble fluorescent carbon dots coated with chitosan: synthesis and its application for sensitivity determination of Cr (VI) ions. *J. Iran. Chem. Soc.* (2018) 15(1) 23-33.
2. Sivrikaya, S., Imamoglu, M. Online Solid-Phase Extraction of Cd (II), Cu (II), and Co (II) Using Covalently Attached Bis (salicylaldimine) to Silica Gel for Determination in Food and Water by Flame Atomic Absorption Spectrometry. *Anal Lett* (2017) 1-19.

OP6- Determination of Trace Elements in Different Tissues Of Green Tiger Shrimp (*Penaeus semisulcatus*) from Eastern Mediterranean, Turkey

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Fish is a significant food for a healthy life due to containing high-quality protein, low saturated fat, vitamins, and minerals. On the other hand, depending on the spread of technological and industrial productions, the increase in domestic waste in parallel with the increase in population, and the mixing of industrial wastes with waters by various ways cause the waters to be polluted. As a result, marine animals to be exposed to many toxic matters, and they can absorb many most of the metals in high amount into their bodies.

In this study, different organs of green tiger shrimp (*Penaeus semisulcatus* De Haan, 1844) taken from the seas of İskenderun Bay, Eastern Mediterranean, Turkey, in January–February, 2016, were analyzed by ICP-MS after digesting by microwave radiations. The obtained results were evaluated in terms of risk for human health. Mean metal concentrations in different tissues were in the following ranges: Se: 0.641–0.868, Cu: 3.489–14.578, Pb: 0.100–0.148, Ni: 0.110–0.434, Al: 1.855–11.321, Zn: 14.197–30.319 mg kg⁻¹ based on wet weight. Metal concentrations in the muscles of the examined species were generally lower than those in skin and liver. The described method was validated by analysis of certified reference material. Limits of detection and quantitation were found to be (as µg L⁻¹) 0.035 and 0.1 for Se, 0.2 and 0.5 for Cu, 0.07 and 0.2 for Pb, 0.35 and 0.95 for Ni, 1.0 and 3.0 for Al, 1.5 and 5.0 for Zn, respectively.

Keywords: ICP-MS; Risk assessment; Metals; Shrimp.

OP7- Nanoporous Polymer Membranes by Blockcopolymer Technique and Their Analytical Applications

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Polymer membranes are of very significant interest as research materials and a number of applications such as health sector, food industry, sustainable water treatment and energy conversion and storage. However, the widespread use of membrane technology has been impeded some factors including cost, performance, durability and etc. These limitations can be directly related to the membrane used. In particular, advances in the design and fabrication of nanoporous materials are expected to open up new opportunities for the development of membrane technology particularly for selective separations of the substances. The bulk of membranes used in industry for filtration, separation and bio-separation etc. are made by simple phase inversion in polymer-solvent-non-solvent solutions [2]. However, this technique generates a 3D, sponge-like network of pores in the um size range and for many applications, including ultra-filtration, this pore size is too large for size selective separation of small molecules. Block copolymer (BCP) self-assembly is a promising to generate nanopatterns, nanopore, nanoarrays on the surfaces with a high density of evenly sized nanoscale pores. Block copolymers consist of macromolecules composed of two or more chemically different blocks which can self-organize into different morphologies with characteristic sizes in the nanometer scale. Block copolymers with immiscible blocks microphase-separate into domains in which the equilibrium domain size (typically 3–100 nm) and morphology are governed by the Flory-Huggins interaction parameter between the blocks and the degrees of polymerization of each block.

In this presentation, a new approach to the development of novel membranes, and their applicability for trace analysis, that takes advantage of well - controlled molecular transport through the uniform cylindrical nanopores, will be presented.

OP8- Simultaneous Determination of Levodopa, Dopamine, Homovanilic Acid and 3,4-Dihydroxyphenylacetic Acid by using HPLC-UV system

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A wide range of neural pathway originated catecholamine employ biogenic amines as neurotransmitters. The neurotransmitters in cerebrospinal fluid can be an indicator of neuronal functioning in nearby diencephalon structures. Hence, it is very crucial to quantitate neurotransmitters and their metabolites level in the extracellular fluid for monitoring neurotransmission process [1]. Numerous methods such as; spectrophotometry, gas chromatography (GC) with mass detection, high performance liquid chromatography (HPLC) with various detection system, chemiluminescence, amperometric and voltammetric determination, potentiometry, radioimmunoassay and flow injection analysis (FIA) have been reported for the determination of catecholamines in various biological samples and pharmaceutical preparations [2]. Among them HPLC is very practical tool for sensitive, selective and accurate method for the quantification of dopamine precursor and metabolites.

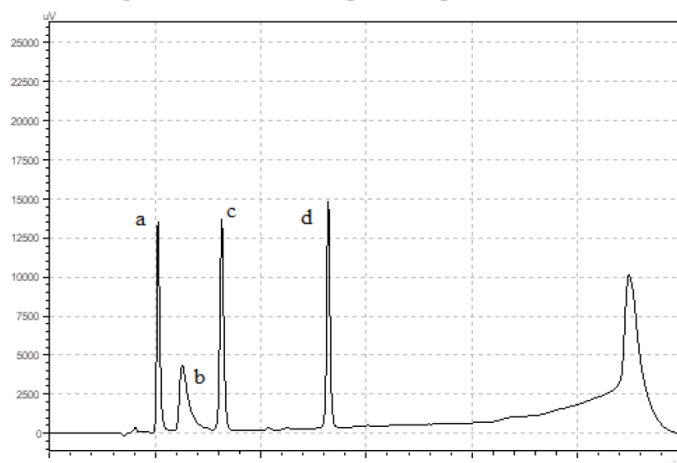


Figure 1 HPLC–UV chromatograms recorded for 5 mg L-1 a) L-Dopa, b) DA, c) DOPAC and d) HVA by using C-18 column with a mobile phase solution of 0.05% TFA.

In the present study, simultaneous determination of levodopa, dopamine, DOPAC and HVA was performed by using HPLC-UV system. Effect of analytical column type (C-18, C-8 and HILIC), the pH and composition of mobile phase and composition of dilution solvent was investigated (Fig.1). After the optimization, method developed was validated by using artificial cerebrospinal brain fluid.

Keywords: HPLC, Catecholamines, Artificial Brain Fluid

References:

1. Bugamelli F, Marcheselli C, Barba E, Raggi MA, Determination of L-dopa, carbidopa, 3-O-methyldopa and entacapone in human plasma by HPLC–ED, J. of Pharm. and Biomed. Anal. 54:562–567
2. Goldstein, D.S., Kopin, I.J., Sharabi, Y., Catecholamine autotoxicity. Implications for pharmacology and therapeutics of Parkinson disease and related disorders, Pharmacology & Therapeutics 144:268–282

OP9- Fast, easy and green method employing magnetic dispersive solid phase extraction for the determination of pesticide residues

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Pesticides are widely used in agricultural production nevertheless, their residues in food and environmental samples are harmful to human health because of their potential mutagenicity and carcinogenic properties. Their extensive use in world-wide agricultural practice in addition to industrial emission during their production has led to substantial occurrence of pesticide residues. For these reasons, the evaluation of pesticide residues is nowadays a priority objective to ensure safety and quality.

Standard methods for pesticide residue analysis is generally carried out in a sequence of several steps. As one of the standard method solid phase extraction (SPE), displays some drawbacks such as multistage operation, large consumption of reagent, low enrichment factor and time consuming. These problems accomplished by using micro-SPE (μ -SPE) extraction technique developed in 2006. Magnetic SPE (MSPE) is one of the new procedure of μ -SPE based on the use of magnetically modified adsorbents. This technique has gained much attention due to easy separation under an applied external magnetic field, minimum solvent consumption and the extraction efficiency. In this novel sample preparation method, a series of magnetic particles have been used for the extraction of organic pollutants including, nano Fe_3O_4 , Fe_3O_4 -grafted graphene, carbon nanotubes, graphene, Fe_3O_4 -CNT [1,2] and so on.

In this work, the information about novel green extraction methodological approach including MSPE is presented. A $\text{Fe}_3\text{O}_4/\text{Ni}/\text{Ni}_x\text{B}$ nanocomposite magnetic adsorbent was synthesised and used for the determination of pesticides by MSPE method. Characterisation of the adsorbents was performed using scanning electron microscopy combined with energy dispersive X-ray spectroscopy (SEM-EDX). The parameters related to the extraction efficiency were screened. The performance of extraction of Bromopropylate (BRP) and Lambda-Cyhalothrine (LMD) pesticides were compared by using gas chromatography with electron capture detector. Gas chromatography-MSPE method was applied for pesticide determination in well water, wine and grape juice samples. The regression coefficients relating to linearity were at least 0.99. Under optimized conditions the linear range was found between $0.1 - 5 \text{ ng mL}^{-1}$, and the detection limits for BRP and LMD were calculated as 0.02 and 0.03 ng mL^{-1} , respectively. Recoveries from spiked samples range from 80 to 97% and RSDs were no higher than 15%. Hence, this developed micro extraction method shares the advantages of the classical extraction methods such as excellent enrichment performance, easy operation and ability to employ a wide range of “green” extraction way.

Keywords: Magnetic dispersive, green chemistry, Pesticide, Chromatography

References:

1. X. Cao, J. Chen, X. Ye, F. Zhang, L. Shen, W. Mo, **J. Sep. Sci.** (2013) 36, 3579–3585.
2. Z. Du, M. Liu, G. Li, **J. Sep. Sci.** (2013) 36, 3387–3394.

OP10- A Simple and Green Deep Eutectic Solvent Based Air Assisted Emulsification Liquid-Liquid Microextraction of Palladium from Water and Environmental Samples

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Palladium is known as precious and toxic element for industry and human health. Direct determination of palladium ions in water and environmental samples by using flame atomic absorption spectrometry is difficult because of low detection limit of instrument and matrix effects of some cations, anions and transition metal ions (1). A simple and green deep eutectic solvent based air-assisted emulsification liquid-liquid microextraction (DES-AA-ELLME) method was optimized for the determination of palladium ions. In the developed procedure, the palladium extraction was attained using 2-hydroxy-3-methoxybenzaldehyde thiosemicarbazone as a complexing agent, tetrahydrofuran as emulsifier solvent and choline chloride-phenol as extracting solvent. Concentration of palladium was determined by using flame atomic absorption spectrometry. Various analytical parameters including type and molar ratio of deep eutectic solvent, pH, amount of ligand, pulling and pushing of syringe were studied and optimized. The limit of detection, relative standard deviation and preconcentration factor were found to be 1.2 $\mu\text{g L}^{-1}$, 3.6 %, and 70, respectively. The accuracy of the method was confirmed with certified reference material. The developed method was applied to real water samples with standard addition method. Method was also applied to the digested environmental samples.

Key words: Palladium; Deep eutectic solvent; Air assisted liquid phase microextraction.

References:

1. P. Liang, E. Zhao, F. Li, Dispersive liquid-liquid microextraction preconcentration of palladium in water samples and determination by graphite furnace atomic absorption spectrometry, *Talanta* (2009) 5, 1854-1857.

OP11- Determination of Cu(II) Ions at Trace Level in Some Vegetable Samples by a Novel SPE-FI-FAAS Method

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On-line solid phase extraction is highly efficient and simple preconcentration method for the determination of trace level of metal ions in environmental samples (1).

In this work, N-[[3-[(3-aminopropyl)amino]propyl]-2,3-dihydroxy-benzaldimine bonded silica gel was synthesized and characterized by C,H,N elemental analysis and FTIR spectroscopy and then, used for on-line solid phase extraction of Cu(II) ions coupled with flame atomic absorption spectrometer. Optimum FI-SPE conditions for Cu(II) ions were studied by examining the effects of parameters such as sample pH, eluent concentration and types, flow rates and volumes of sample and eluent, and matrix ions. Optimum pH was found to be pH 8.5 for the best absorbance of Cu(II) ions. Sample and eluent flow rate was optimized to be 7.5 mL/min. 250 μL of 0.5 M HNO_3 was found to be efficient to elute adsorbed Cu(II) ions from the column. LOD of Cu(II) ions based on the 3 times the SD (3s) of the blank signal and preconcentration factor were calculated to be 0.47 $\mu\text{g/L}$ and 25.6, respectively. The developed method was validated by analyzing Cu content of certified reference materials such as Virginia tobacco leaves (CTA-VTL-2), water-trace elements (NWTM-15.2) and water samples (LPCS-01-5). The method was successfully used for the determination Cu(II) ions in several vegetables including potato, red cabbage, green pepper, cucumber, lettuce, scallion, tomato, fresh squash and eggplant.

Keywords: FAAS, trace analysis, solid phase extraction, modified silica gel

References:

1. S. Sivrikaya, M. Imamoglu. Online solid-phase extraction of Cd (II), Cu (II), and Co (II) using covalently attached bis (salicylaldimine) to silica gel for determination in food and water by flame atomic absorption spectrometry. *Anal. Lett.* (2017) 51(5) 773-791.

OP12- Development of PPy-CNT-SPME Fiber Development and Their Use in Pesticide Determination in Apple Juice by GC-MS

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Pesticides are widely used in agricultural production and due to their harmful effects for the environment and human health; accurate and reliable analytical methods are required for their analysis.

Present study describes a direct immersion solid-phase microextraction (DI-SPME) method for the determination of selected pesticides used in apple production namely bifentrin, propargite, tebuconazole and tebufenpyrad prior to the GC-MS analysis. The SPME fiber was fabricated by using a stainless steel wire dipped into the voltammetric cell containing pyrrole monomer, tartrate solution and carbon nanotube mixture where it was subjected to electropolymerization by cycling the potential in a range of -0.5 - 2.0 V. The multiwall carbon nanotubes (MWCNT), amino-functionalized multiwall carbon nanotubes (NH₂-MWCNT), carboxylated multiwall carbon nanotubes (COOH-MWCNT) and fullerenes have been used for this purpose. Fiber surfaces have been characterized by SEM measurements and the analyses have been performed with GC-MS system in SIM mode.

Operational parameters effecting the extraction including the medium pH, adsorption time, desorption time and temperature have been investigated by using central composite design (CCD). Analytical characteristics of the method have been investigated and it was revealed that these pesticides can be determined in ppb level with high recovery percentages well below the MRL values, without any solvent consumption or labor intensive pre-treatment processes.

Keywords: Pesticide residue analysis, SPME, GC-MS, polypyrrole, carbon nanotube.

Acknowledgement: Authors would like to thank Ege University Research Project (2015 FEN 032) for financial support.

References:

1. J. Chen, J. Zou, Preparation and evaluation of graphene-coated solid-phase microextraction fiber, *Anal Chim Acta* (2010) 678 44-49.
2. A. M. Filho, F. N. Santos, Development, validation and application of a method based on DI-SPME and GC-MS for determination of pesticides of different chemical groups in surface and groundwater samples (2010), *Microchem J.*, 96 139-145.

OP13- Quadrupole Isotope Dilution Mass Spectrometry-Dispersive Liquid-Liquid Microextraction Using Multivariate Optimization: A Novel Analytical Approach for the Determination of Parathion Methyl in Water

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Dispersive liquid-liquid microextraction (DLLME) is a very useful method which results in high analyte preconcentration [1] and quadrupole isotope dilution mass spectrometry (ID⁴MS) is a technique known for its high accuracy and precise measurements [2]. These two techniques were combined in this study for the first time in the determination of parathion methyl in water. DLLME was optimized using an experimental design to determine the effects of main parameters and their interactions. Analysis of variance was used to evaluate experimental results and the results fitted the design model. Sample and calibration blends (three point) were prepared gravimetrically by spiking each blend with deuterated parathion methyl. The blends were allowed to equilibrate for three hours before performing DLLME under optimum conditions. The amount of parathion methyl determined corresponded to a percent recovery of 99.9%, which signified high accuracy, and the 0.32% relative standard deviation obtained also signified high precision. ID⁴MS was performed at 40 ng/mL due to its coupling with DLLME. The two methods complemented each other, with DLLME offering determination of parathion methyl at trace level while ID⁴MS kept the precision and accuracy of the method at a very high level.

Keywords: Parathion methyl, DLLME, isotope dilution-mass spectrometry, multivariate optimization.

References:

1. D.S. Chormey, C. Buyukpinar, F. Turak, O.T. Komesli, S. Bakirdere, Simultaneous determination of selected hormones, endocrine disruptor compounds, and pesticides in water medium at trace levels by GC-MS after dispersive liquid-liquid microextraction, *Environ. Monit. Assess.* (2017) 189 277.
2. E. Pagliano, J. Meija, Z. Mester, High-precision quadrupole isotope dilution method for simultaneous determination of nitrite and nitrate in seawater by GCMS after derivatization with triethyloxonium tetrafluoroborate, *Anal. Chim. Acta* (2014) 824 36-41.

OP14- Monitoring Conformational Features of Protein-Polyelectrolyte Complexes Using Ion Mobility-Mass Spectrometry

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The conformations of proteins in biological systems have direct effect on the functional features of these macromolecules. In studies where the functioning mechanisms of proteins are examined, they should be analyzed in their native forms without any degradation. By monitoring the interactions between molecules in analytical studies in which the native structures of the proteins can be maintained, it is possible to elucidate the process of formation of many diseases as well as to develop targeted disease treatment methods. In mass spectrometry based-protein characterization studies, it is aimed to determine the native states of structures using various analytical strategies. Ion mobility-mass spectrometry (IM-MS) provides conformational information about the analyzed molecules.

In this study, it is aimed to preserve the natural forms of the structures as a result of using the polyelectrolyte-protein interactions to prevent impulses between the charge centers on protein structures. Interactions and conformational changes in structures have been examined in detail using various mass spectrometers having ion mobility capabilities.

Keywords: IM-Mass Spectrometry, Protein Conformation, Polyelectrolyte, Noncovalent Interactions

OP15- Hepatoprotective extracts of *Cuscuta campestris* Y. and Their Best Known Anticancer Flavonols Isolated by Preparative HPLC

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Plant derived antioxidants have a potential to prevent cell damage caused by free radicals. *Cuscuta campestris* is being used as herbal tea and well known in traditional medicine for being remedy of liver injury and cancer.

The purpose of this study is to identify the anticancer associated content of *Cuscuta campestris* by spectroscopic and chromatographic methods. The plant was collected from Talas district of Kayseri in Turkey and extracted with diethyl ether, ethyl acetate, methanol, *n*-butanol and water at 37°C for 2.5 h in a shaker. Anticancer activities of the extracts were evaluated on SNU-398 hepatocellular carcinoma cell lines and controlled on normal adult human dermal fibroblasts (hDFs) cells. Methanol and ethyl acetate extracts showed cytotoxicity on the SNU-398 with the CC₅₀ values of 18.7 and 19.6 µg/mL, respectively. Only butanol extract was found cytotoxic on the hDFs cells among the ethyl acetate, methanol and butanol extracts. Main fractions of ethyl acetate extract, collected with preparative HPLC, were purified and identified as quercetin, kaempferol and isorhamnetin by using UV, MS-MS and FT-IR spectra.

The best known flavonols, quercetin, kaempferol and isorhamnetin, may have caused this hepatoprotective effect due to their abundance in the extracts. In conclusion, ethyl acetate and methanol extracts containing quercetin, kaempferol and isorhamnetin from *Cuscuta campestris* are worth of further study to develop anticancer drugs.

Key Words: *Cuscuta campestris*, quercetin, anticancer, preparative-HPLC

OP16- Analysis of Human Plasma Proteome by Using UPLC/MS Method

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Proteomics is the study of proteome that included all gene-encoded proteins in highly complex biological specimen. Concept of proteomics involves determining of protein abundance, structure, interactions and modifications. It gives information about the state of organism at a given time. Human blood is the most preferred and used human body fluid in clinical practice for proteomics. Human blood contains thousands of proteins, which are secreted or leaked into the blood by normal cell, tissue processes or cell damages. Therefore, blood composition is a relevant indicator of the overall physiology of an individual (1). Presence or change in expression of proteins might indicate the onset and progression of most disease states. However, systematic analysis of the plasma proteome is extremely challenging task due to its complexity. It contains thousands of proteins differing by extraordinary dynamic range of at least 9-10 orders of magnitude. Mass spectroscopy (MS) based methods, which give high sensitivity and resolution, have been used as a choice for analysis of complex protein samples like plasma. Today Bottom-up or shotgun proteomics is the most common MS-based method for studying proteins. In bottom-up proteomics studies, a mixture of proteins from biological sample is isolated and cleaved into peptides. The resultant peptide mixture is fractionated using liquid chromatography (LC) and other method before MS analysis. Nano LC that is miniaturizing form of regular HPLC has been used in protein analysis as a main tool for 20 years (2). Main advantage of nano LC is to provide high sensitivity and analyze less abundant proteins in complex protein mixtures like cell, tissue and body fluids. Although nano LC offers advantages, system has some drawbacks like leaking, high backpressure. As an alternative to nano LC system UPLC based proteomics analysis has been studied several times.

In this study first time we developed UPLC/MS method to analyze plasma proteome. We used acetonitrile and water as mobile phase and gradient time was 150 min. Peptides were separated in C18 column. We used Maxquant software to analyze MS-MS data of peptides and 91 proteins were identified with three replicate. We also used label free quantification method to show whether UPLC/MS method could be used for semi quantitative proteomics analysis. Our result showed that UPLC/MS method could be used for identification and semi quantification of plasma proteins.

Keywords: UPLC, Chromatography, Plasma, Proteomics

References

1. Anderson, N. L., & Anderson, N. G. The human plasma proteome: history, character, and diagnostic prospects. *Mol Cell Proteomics*, (2002)1(11), 845-867.
2. Valdes, A., Artemenko, K. A., Bergquist, J., Garcia-Canas, V., & Cifuentes, A. Comprehensive Proteomic Study of the Antiproliferative Activity of a Polyphenol-Enriched Rosemary Extract on Colon Cancer Cells Using Nanoliquid Chromatography-Orbitrap MS/MS. (2016 *Journal of Proteome Research*, 15(6), 1971-1985.

OP17- The Phenolic Compounds of Some Cornelian Cherry (*Cornus mas L.*) Genotypes Grown in Turkey

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Cornelian cherry (*Cornus mas L.*) is a plant belonging to the *Cornaceae* family of *Umbelliferae* kind, falling its leaves in winter, getting tall up to 7-8 m and being 2.5-4.5 cm the trunk diameter. Cornelian cherry which the motherland is Anatolia, Caucasus and European have a wide spreading area in our country. It grows naturally in single or several trees or in forest areas, usually on the edges of fields and gardens. The fruit which mature at the end of the summer season may be red, dark red, pink or yellow depending on the its species. Cornelian cherry juice is evaluated as jam, marmelade, syrup, jelly, compote, pulp, fruit juice, fresh and dried fruit (1). In cultivated regions, fruit is used in folk medicine, skin diseases, metabolic disorders and diarrhea. In addition, it is utilized from the antiseptic properties of flowers, leaves, seeds, shells and roots (2).

In this study, phenolic compounds of 10 Cornelian cherry genotypes collected in the surveys conducted in different cities of our country were determined. It was used for extraction the solvent mixture of methanol: water: HCl, and the phenolic compounds in the extracts were determined on HPLC-DAD. The detection and quantification limit values with HPLC were found for all phenolic compounds. The relative standard deviation (RSD%, n=10) average was in the ranged from 2 to 4,06% for phenolic compounds and the correlation coefficient was in the ranged 0.9996-0.9989.

The gallic acid in the fruit samples was found within the range of 10.35 – 176.66 ppm, chlorogenic acid 26.83 – 72.45 ppm, epicatechin 6.60 – 78.82 ppm, rutin 172.73 – 1142.82 ppm, kaempferol 9.90 – 79.03 ppm and quercetin 0.13 – 0.61 ppm.

Keywords: Cornelian cherry, extraction, phenolics component, HPLC

References:

1. Didin, M., Kızılaslan, A. ve Fenercioğlu, H., Malatya’da Yetiştirilen Bazı Kızılcık Çeşitlerinin Nektera İşlenmeye Uygunluklarının Belirlenmesi Üzerine Bir Araştırma, Gıda, (2000) 25, 435-441.
2. Türk, R., Erkan, S. ve Yalçınkaya, E., Bazı önemli kıızılcık (*Cornus mas L.*) tiplerinin morfolojik ve fenolojik özellikleri, Türkiye IV. Ulusal Bahçe Bitkileri Kongresi, Antalya, (2003) 289-291.

OP18- Simple, Fast and Reliable Method for Determination of H_2O_2 in Aqueous Samples

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H_2O_2 is a reactive oxygen species, which has a great importance in chemistry, biochemistry, and in the field of life sciences. H_2O_2 , which is an unstable compound cause radical formation in some cases and may have carcinogenic effects to human [1]. Because of the rapid degradation of H_2O_2 , monitoring and identification of it with a fast technique is so important analytical problem. In the literature different methods were used for the determination of H_2O_2 [2]. However, most of them do not have access to adequate sensitivity and these are time consuming methods.

In this study a simple, rapid and sensitive spectrophotometric method is described for the determination of hydrogen peroxide using iron(III) and EDTA as a reagent. The method is based on the complexation of hydrogen peroxide with the Fe(III)-EDTA mixture in the alkaline medium. A purple color of low stable peroxo-iron(III)-EDTA complex was formed in the method and it has a maximum absorbance peak at 525nm. (Fig 1).



Figure 1. The color of a) Fe(III)-EDTA- NH_3 and b) Fe(III)-EDTA- NH_3 - H_2O_2 complex

Limit of detection and limit of quantification was found as 2.5×10^{-6} and 8.5×10^{-6} mol/L. Intraday and interday relative standard deviation of the proposed method for 2.0×10^{-4} mol/L of H_2O_2 were found as 1.5% and 6.1% respectively. The proposed method was successfully applied to real water samples namely drinking water, tap water and seawater with acceptable recovery values between 90% and 118%. Nearly none of common ions except Fe(II) showed interfering effect to proposed method so, the method can be easily acceptable to real water samples without any sample preparation step.

Keywords: Hydrogen peroxide, Spectrophotometry, Peroxo-iron complex

References:

1. J.M. Desesso, A.L. Lavin, et al., Food and Chem. Toxicol. (2000) 38,1021-1041.
2. M.E. Abbas, W. Luo et al. Food Chem. (2010) 120, 327-331.

OP19- Molecularly Imprinted Nanoparticles for Selective Tetracycline Adsorption

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Tetracycline (TC) and its analogues such as doxycycline (DC), chlortetracycline (CTC) and oxytetracycline (OTC) are antibiotic drugs that are still commonly used in veterinary medicine. In recent years, concerns have been raised regarding the public health impact of the occurrence of these antibiotics in the aquatic environment [1]. For the determination of target chemicals in routine analytical tasks, a thorough pretreatment procedure is generally required, and solid-phase extraction (SPE) is widely used for sample preparation. Nowadays, molecularly imprinted solid-phase extraction (MISPE) is ubiquitously utilized for sample pretreatment, using molecularly imprinted polymers (MIPs) as adsorption materials.

In this study, a novel solid phase extraction (SPE) method was developed for determination of tetracyclines (TCs) in water samples by using molecularly imprinted nanoparticles. Poly(hydroxyethyl methacrylate-N-methacryloyl-L-glutamic acid methyl ester [poly(HEMA-MAGA)] nanoparticles were prepared by using tetracycline (TC) as a template. The poly(HEMA-MAGA) nanoparticles were characterized by Fourier transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS), scanning electron microscope (SEM) and transmission electron microscope (TEM) analysis. The maximum TC adsorption capacity of the poly(HEMA-MAGA) nanoparticles was determined as 28.6 mg/g at pH 5.0, 25 °C.

Keywords: Tetracycline, molecularly imprinted nanoparticles, solid phase extraction

References:

1. R.Suedee, T. Srichana, T. Chuchome, U. Kongmark, Use of molecularly imprinted polymers from a mixture of tetracycline and its degradation products to produce affinity membranes for the removal of tetracycline from water, *Journal of Chromatography B* (2004) 811191–200.

OP20- A New Off-line Solid Phase Extraction Method for Antimony using Silica Functionalized with N,N'-bis(4-methoxysalicylidene)-1,3-propanediamine

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A new solid phase extraction method for preconcentration and determination of Sb was developed using silica functionalized with N,N'-bis(4-methoxysalicylidene)-1,3-propanediamine (Si-MSPA) [1] and inductively coupled plasma optic emission spectrometry [2]. The off-line preconcentration method was carried out at following optimal conditions: sample pH= 4.0; flow rate 6.0 mL min⁻¹; sample volume 25.0 mL. The retained Sb ions were desorbed with 3.0 mol L⁻¹ HCl in 1.0 % thiourea at 6.0 mL min⁻¹ flow rate. The interfering effects of some anions and cations were also studied. The accuracy of the suggested method was tested with Sb spiked real samples and certified reference materials. The successful applications and satisfactory results showed that the method may be used for preconcentration and determination of Sb in water samples.

Keywords: Solid phase extraction, antimony, N,N'-bis(4-methoxysalicylidene)-1,3-propanediamine, ICP-OES

References:

- 1-Koluman, M., Tokay, F., Bağdat, S. Schiff base immobilized silica gel framework as an efficient sorbent for preconcentration of Pb and Zn ions in aqueous media. *Turk J Chemistry* (2016) 40 953-964.
- 2-Tokay, F., Bağdat, S. Preconcentration and Determination of Metal Ions Using Fluorescein-Modified Silica Gel and Inductively Coupled Plasma-Optical Emission Spectrometry. *Anal Chem* (2018) 51 119-132.

OP21- Fractionation and Bioaccessibility Studies: Are they Meet the Demand for the Risk Assessments of Some Elements in Baby Foods?

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Despite the importance of the complementary foods and strict rules for manufacturing processes, infants consume more food relative to their body weight¹, thus, essential elements may be even toxic depending on consumption levels². When this factor is taken into account, appropriate sample preparation strategies will be important for accurate results and final evaluations in terms of risk assessment of metals in baby complementary foods originating from consumption.

Risk assessment studies depend on the bioaccessible levels as well as the total levels of elements. But, determining the total levels for the elements that may form insoluble species in enzymatic studies as well as volatile forms in digestion procedures will be problematic for determining these levels. For example, total levels either in fractions or digested samples may be much smaller in terms of solubility and higher percent bioaccessibilities could be achieved although these tendency is meaningful depending on the matrix effects.

In this study this observation will be discussed by evaluating some elements from fruit based complementary foods to provide more precise results and evaluate the place of Turkish markets for baby feeding. Risk assessment studies will be based on the adequate or tolerable intake values of elements and the contribution of the fractionation studies and chemometric approaches in sample preparation strategies will be discussed.

Keywords: Bioaccessibility, Risk assessment, Baby food, Complementary feeding.

References:

1. Pandelova M, Lopez WL, Michalke B, Schramm KW, Ca, Cd, Cu, Fe, Hg, Mn, Ni, Pb, Se, and Zn contents in baby foods from the EU market: Comparison of assessed infant intakes with the present safety limits for minerals and trace elements, J Food Compos Anal. (2012) 27 120-127.
2. Tuzen M, Soy lak M, Evaluation of trace element contents in canned foods marketed from Turkey, Food Chem. (2007) 102 1089-1095.

Acknowledgments: This study was supported by The Scientific and Technological Research Council of Turkey (TUBITAK) (Project number: 115Z128).

OP22- The importance of Ion pairing and polarity effects Electrochemical Double Layer in Ionic Liquids: Experimental and Modeling Study

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Room temperature ionic liquids (RTILs) are a class of electrolytes that are based on low melting point ionic solids. They have decent conductivity levels, low vapor pressures and very large electrochemical potential windows, which are very attractive qualities as electrolytes for numerous applications [1]. Theoretical and experimental understanding of electrolyte solutions have historically relied on models based on dilute solutions assuming no interaction among the solute species. This type of understanding, though very useful in numerous systems, fails to capture the details of concentrated solutions and further, RTILs. Though there are studies in the literature that are aimed at understanding the details of RTILs, they are too detailed and computationally intensive in order to be employed for practical purposes.

In this contribution, we will be presenting a straightforward approach that models not only the electrostatic interactions between the anions and the cations of the ionic liquid, but also the ion pairing constant. This approach is computationally simple, yet accurately describes the outcome of the experiments. This new modeling approach paves the way for understanding the behavior of the electrochemical double layer in greater detail under conditions where the assumptions of dilute electrolytes fail.

This modeling approach will be presented along with in-situ X-Ray Photoelectron Spectroscopy data, which provides the potential across the length of the electrolyte in a position and time resolved fashion (Figure 1) [2].

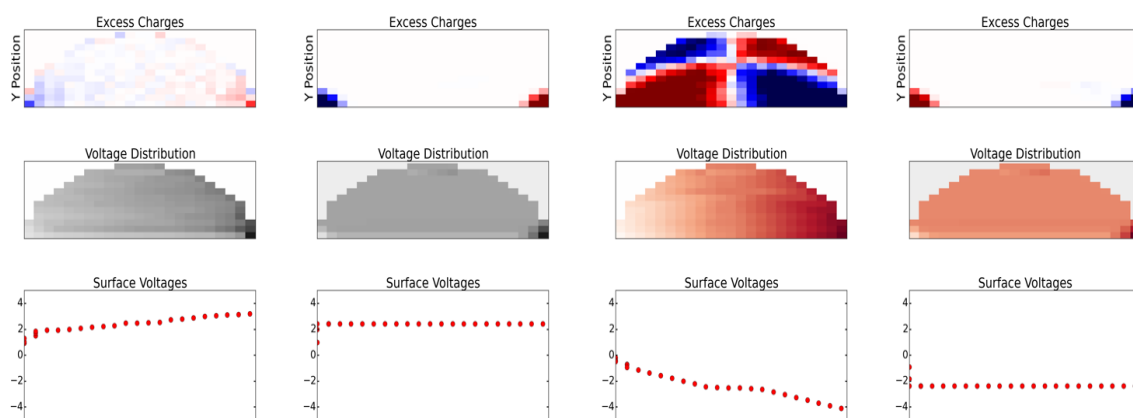


Figure 1: X-Ray Photoelectron Spectroscopy data

Keywords: Ionic Liquids, Modeling, Electrochemical Double Layer, Ion-pairing

References:

1. Freemantle, M (2009) An Introcutuon to Ionic Liquids, Royal Society of Chemistry, London, UK.
2. Camci MT; Aydogan P; Ulgut B; Kocabas C; Suzer S; "XPS enables visualization of electrode potential screening in an ionic liquid medium with temporal- and lateral-resolution" Phys. Chem. Chem. Phys. (2016),18, 28434

OP23- Cocrystallization of Energetic Materials with Enhanced Sensitivity

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Insensitive explosives have attracted significant interest in the past three decades owing to their potential application in numerous energetic materials. Research of the explosive of low impact sensitivity and high explosive performance is the vital problem in the energetic materials, which has not been solved yet (1-4). Efforts have been made to diminish this through synthesis of new molecules, however this is usually a complex, need to time consuming process cost-effective production methods, extensive validation, and requirements to meet increasing challenging performance target (5). Co-crystallisation is new an effective technique to improve the solubility, bioavailability physical and chemical stability properties of drugs without altering their chemical structure, and it is extensively used for the pharmaceutical industry (6-9). Therefore, co-crystallization may supply a new method to possibility of tuning the performance and sensitivity of an energetic material without requiring complex preparation or synthesis of novel compounds (10-13). To modify and improve the properties of energetic materials, via cocrystallization between TNT, Picric Acid with nonenergetic materials were studied. Cocrystallization studies including characterization (single crystal X-ray data, Powder X Ray diffraction), thermal behavior (Differential Scanning Calorimetry), impact sensitivity (BAM Fall Hammer), and intermolecular interactions were carried out in detail. And also we analysed the crystal packing and show how this may influence sensitivity to impact.

Key words: Trinitrotoluene, Picric Acid, Sensitivity, Cocrystal, Insensitive energetic material

References:

1. Becuwe, A.; Delclos, A. *Propellants, Explosives, Pyrotechnics*. (1993)18, 1–10.
2. Van der Heijden, A. E. D. M.; Bouma, R. H. B.; Van der Steen, A. C. *Propellants, Explosives, Pyrotechnics*. (2004) 29, 304–313.
3. Millar, R. W.; Philbin, S. P.; Claridge, R. P.; Hamid, J. *Propellants, Explosives, Pyrotechnics*. (2004) 29, 81–92.
4. Zhurova, E. A.; Zhurov, V. V.; Pinkerton, A. A. *J. Am. Chem. Soc.* (2007) 129, 13887–13893.
5. Sikder, A.K.; Sikder, N. *J. Haz. Mat.* (2004) 112, 15.
6. Shen, J. P.; Duan, X. H.; Luo, Q. P.; Zhou, Y.; Bao, Q.; Ma, Y. J.; Pei, C. H. *Cryst. Growth Des.* (2011), 11, 1759–1765.
7. Remenar, J. F.; Morissette, S. L.; Peterson, M. L.; Moulton, B.; MacPhee, J. M.; Guzman, H. R.; Almarsson, O. *J. Am. Chem. Soc.* (2003) 125, 8456–8457.
8. Weyna, D. R.; Shattock, T.; Vishweshwar, P.; Zaworotko, M. J. *Cryst. Growth Des.* (2009), 9, 1106–1123.
9. Almarsson, Ö.; Zaworotko, M. J. *Chem. Commun.* (2004) , 1889–1896.
10. Zhu, W.; Xiao, J.; Zhao, F.; Ji, G.; Ma, X.; Xiao, H. *Acta Chim. Sin.* (2007), 65, 1223.
11. Wei, C.; Huang, H.; Duan, X.; Pei, C. *Propellants, Explosives, Pyrotechnics*. (2004), 29, 81–92.
12. Landenberger, K. B.; Matzger, A. J. *Cryst. Growth Des.* (2012), 12, 3603–3609.
13. Sun, T.; Xiao, J. J.; Liu, Q.; Zhao, F.; Xiao, H. M. *J. Mater. Chem. A.* (2014), 2, 13898–13904.

OP24- SERS Based *E. coli* Enumeration in Urine With Using Casein Bound Magnetic Nanoparticles

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Paper based microfluidic systems are one of the most commonly used commercial test kits for the determination of drugs of abuse and pregnancy etc. because of its low cost, simplicity and user-friendliness advantages. Most of these commercial paper based systems has been used for qualitative detection, but various new paper based biosensor systems have been developed by using fluorescence, electrochemistry and SERS etc. for the quantitative investigations [1].

In this study, a new paper based biosensor system is developed for the quantitative determination of *E. coli*. Gold coated spherical magnetic nanoparticles were used for preconcentration of analyte. Target bacteria separated from sample matrix by using modified magnetic nanoparticles which were covalently bonded to casein as a substrate and *E.coli* antibody to capture specific target bacteria. Yeast enzyme is added to cleave the magnetic nanoparticle and to release *E.coli* because of transportation difficulties of magnetic nanoparticles on nitrocellulose membrane. The Gold nanoparticles were labeled with a Raman-active compound 5,5'-Dithiobis(2-nitrobenzoic acid) (DTNB) which has strong Raman peak at 1330 cm⁻¹ due to symmetric stretching of NO₂ group. DTNB is covalently bonded to gold nanoparticle surface via self-assembled monolayer (SAM). The free preconcentrated bacteria solutions were first interacted with DTNB labelled gold nanoparticles at conjugation pad then captured from flowing sample solution by test line which was drawn with *E. coli* antibody solution. SERS measurements were taken from test lines of paper strips. The amounts of modified magnetic nanoparticles, DTNB labelled gold nanoparticles, casein concentration and yeast enzyme concentration were optimized to get best SERS response.

The SERS signals were acquired and calibration curve was plotted by using Raman peaks area at 1330 cm⁻¹ versus logarithmic initial bacteria concentrations. This method was applied to *E.coli* spiked urine samples with different concentrations. The selectivity of this developed biosensor system was investigated by applying the same procedure to *Salmonella enteritidis* and *Bacillus subtilis* solutions [2].

Keywords: SERS, DTNB, *E.coli*, Nanoparticle

References:

- [1] B. Guven, N. Akgul, E. Temur, U. Tamer, I. H. Boyaci, Analyst, (2011), 136, 740-748
- [2] E. Temur, I. H. Boyaci, U. Tamer, H. Unsal, and N. Aydogan Anal Bioanal Chem (2010) 397, 1595-1604

Acknowledgments: TUBITAK Cost-CA15114 – 114Z783

OP25- pH in Unconventional Electrolytes: Acid Containing Lyotropic Liquid Crystalline Mesophases

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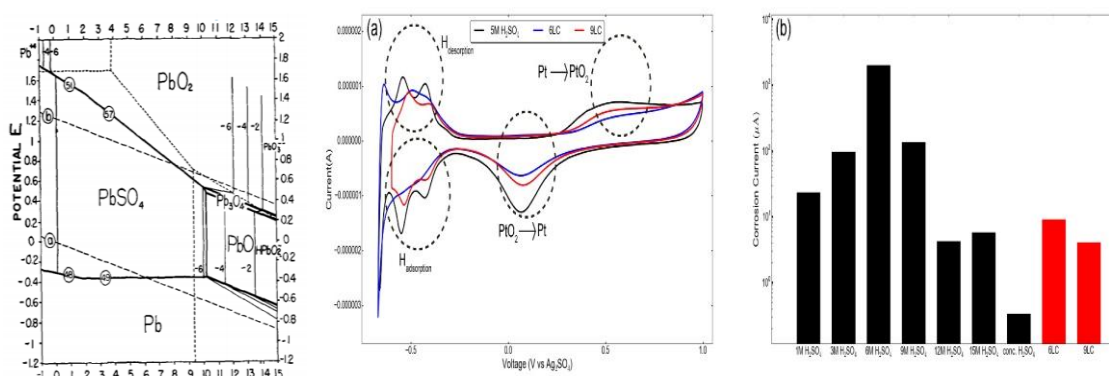
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Lyotropic Liquid Crystalline (LLC) Mesophases formed through the mixture of a non-ionic surfactant and a hygroscopic acid, in particular, H_2SO_4 , was shown to be a promising electrolyte substitute for lead acid batteries (LABs), where the industrially exciting PbO is formed over the thermodynamically stable PbSO_4 upon oxidation inside the LLC Mesophase [1,2]. The stability of the PbSO_4 is demonstrated in the below Pourbaix diagram where at the pH levels relevant to the operation of LABs (around pH 1), only PbSO_4 is stable. In the LLC, however, PbO is also stable as evidenced by X-Ray Diffraction (XRD) and Cyclic Voltammetry (CV) [1].

In conjunction with corrosion studies in aqueous solutions and CVs, a rough estimate of the effective pH inside the liquid crystal phase was achieved [1]. Exact quantification of the pH value inside this LLC mesophase, however, was never reported and it is expected to be vastly different from the value suggested simply by the mass ratio as evidenced by the curious activity shown [1]. Therefore, a careful examination of the pH of these LLC mesophases is required to elucidate the underlying mechanism for this unexpected activity.

In this contribution, we will be showing methods to quantify the effective pH levels inside several acid containing LLC Mesophases via Pt in acid CV's and CV's of other redox couples.



Keywords: Liquid Crystals, pH, Batteries

References:

1. Olutaş E. B., Balcı F. M., Dağ Ö., Strong Acid–Nonionic Surfactant Lyotropic Liquid-Crystalline Mesophases as Media for the Synthesis of Carbon Quantum Dots and Highly Proton Conducting Mesostructured Silica Thin Films and Monoliths, *Langmuir* (2015), 31, 10265–10271.
2. Uzundal C. B., Balcı F. M., Ulgut B., Dağ Ö., Lyotropic Liquid Crystalline Mesophase of Sulfuric Acid–Nonionic Surfactant Stabilizes Lead(II) Oxide in Sulfuric Acid Concentrations Relevant to Lead Acid Batteries, *ACS Omega* (2017), 2, 3785–3791.

OP26- The Usage of N,N'-bis(4-methoxysalicylidene)-1,3-propanediamine Loaded Silica Gel for Simultaneous Preconcentration of Ba, Cd, Co, Cu, Mn and Ni Ions

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In this study, a simple and rapid silica gel based solid phase extraction/preconcentration method was developed for determination of Ba(II), Cd(II), Co(III), Cu(II), Mn(II) and Ni(II) by inductively coupled plasma optic emission spectrometry (ICP-OES) detection. Separation and preconcentration of the analytes were achieved using N,N'-bis(4-methoxysalicylidene)-1,3-propanediamine impregnated silica gel [1-2]. Several factors affecting sorption and elution of the analytes such as sample pH, flow rates, sample and eluent volume, eluent type and concentration were investigated. The optimized operating conditions were found as: sample pH 5.0, sample flow rate 3 mL min⁻¹, sample volume 25 mL, eluent flow rate 3 mL min⁻¹ using 0.5 mol L⁻¹ HNO₃. Additionally, selectivity of the method was examined using various ions. The validity of the method was checked through the recovery experiments besides the analysis of certified reference materials, ERM-CA022a (soft drinking water) and TMDA-53.3 (Lake Ontario water). The improved method was applied to several aqueous samples from different origins.

Keywords: Solid phase extraction, trace elements, N,N'-bis(4-methoxysalicylidene)-1,3-propanediamine, ICP-OES

References:

- 1-Koluman, M., Tokay, F., Bağdat, S. Schiff base immobilized silica gel framework as an efficient sorbent for preconcentration of Pb and Zn ions in aqueous media. Turk J Chemistry (2016) 40, 953-964.
- 2-Tokay, F., Bağdat, S. Simultaneous Preconcentration of Cd(II) and Pb(II) with N,N'-bis(4-methoxysalicylidene) Ethylenediamine Coated Silica Gel Prior to Determination by Flame Atomic Absorption Spectrometry. Water Air Soil Pol. (2015) 226:48.

OP27- A New Analytical Strategy for the Determination of Indium

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Determination of trace indium is important due to both industrial and medical concerns. Indium is an important component of the semi-conductors, also important for the nuclear studies and the production of high purity materials ¹. Atomic absorption spectrometry (AAS) comes into prominence with its being widespread even though it is not as sensitive as advance instrumentation such as ICP-MS. This feature of AAS originates from easy operation, rapid response time and low maintenance costs. It should be underlined that this instrument is much cheaper than ICP-MS and ICP-OES ². Solid phase micro extraction by oleic acid coated magnetic nanoparticles combined with slotted quartz tube-flame atomic absorption spectrometry was suggested as a new and sensitive method for the determination of indium.

In this study, the synthesis and characterization of magnetite nanoparticles (MNPs) were done, and selective pre-concentration studies were performed. Magnetite nanoparticles were synthesized and coated with oleic acid. This coating process were carried out under nitrogen atmosphere at 80 °C for 2h. The resulting MNP were grinded to obtain homogen particles. Resulting MNP were characterized by SEM and FTIR measurements. MNPs were used to extract the analyte from aqueous solutions. Synthesized oleic acid coated magnetite nanoparticles (OAMNP) were used to preconcentrate indium atoms in aqueous samples. The experimental parameters such as pH of the analyte solution, amount of eluent, amount of MNPs were optimized using FAAS (flame atomic absorption spectroscopy). SQT-FAAS (slotted quartz tube-flame atomic absorption spectrometry) was used to further increase the the detection power of the indium. Recovery tests were also studied in order to demonstrate the accuracy of the developed method and satisfactory results were obtained.

Keywords: Indium, SQT, FAAS, nanoparticles

References:

1. M. Tuzen and M. Soylak, A solid phase extraction procedure for Indium prior to its graphite furnace atomic absorption spectrometric determination, J. Hazard. Mater., (2006) 129, 179-185.
2. K. Inagaki and H. Haraguchi, Determination of rare earth elements in human blood serum by inductively coupled plasma mass spectrometry after chelating resin preconcentration, Analyst, (2000) 125, 191-196.

OP28- Adsorption of VOCs onto Activated Carbon and Biochar Obtained from Textile Wastes for Their Determination by GC-MS

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Volatile organic compounds (VOCs) belong to a big family of carbon-based chemicals having the general characteristics of low boiling point and high vapor pressure under normal conditions. BTEX refers to the chemicals benzene, toluene, ethylbenzene and o-, p-, and m-xylenes, all severely threaten human health and the environment due to their toxic, mutagenic, and carcinogenic effects [1]. Monitoring of their levels in workplace atmosphere is of great importance in terms of occupational health and safety. A great deal of research has been conducted to investigate the adsorption properties of VOCs onto a variety of adsorbents [2]. Present study deals with the use of novel carbon materials such as activated carbon and biochar obtained from textile (cotton/polyester and acrylic) wastes. Activated carbon and nano cellulose materials obtained commercially have also been used for comparison purposes.

Targeted analytes, namely toluene, ethylbenzene and xylene have been directly injected into a GC-MS system in gas phase under optimized conditions for constructing the calibration graphs. The adsorption properties of activated carbon and biochar samples produced from textile wastes have been investigated by exposing the adsorbents to the mixed gas standards in a vial at 25°C for 15 min., the adsorbents have been, then, heated up to 60°C for 5 min in the autosampler for thermal desorption of the VOC content. Following the head space sampling, the VOC mixture was injected into the GC-MS system and separated in a wax column. From the chromatograms recorded, it was evident that cotton/polyester biochar has given the best response for the majority of analytes according to their peak areas. Further studies include the calibration construction for all of the targeted analytes and it was revealed that very sensitive analysis can be performed well below the short term exposure level (STEL) values determined for workplaces including the paint sector.

In conclusion, carbon-based adsorbents obtained from textile wastes can serve as an economic and renewable material for sensitive and selective analysis as well as an effective absorber for passive sampler systems for monitoring purposes.

Keywords: Textile wastes, Activated carbon and Biochar, headspace sampling, GC-MS

Acknowledgement: Authors would like to thank Ege University Scientific Research Project (2016 FBE 013) for financial support.

References:

1. EPA, 2017. <https://www.epa.gov/indoor-air-quality-iaq>, Indoor Air Quality (IAQ), Volatile Organic Compounds' Impact on Indoor Air Quality Access date: 19 Jan 2017.
2. X. Zhang, B. Gao, A.E. Creamer, C. Cao, Y. Li, Adsorption of VOCs onto engineered carbon materials: A review, J. Hazard. Mater. (2017) 338 102–123

OP29- Development of an Accurate and Sensitive Analytical Method for the Determination of Cadmium Using Hydrogen Assisted T-Shape Slotted Quartz Tube-Atom Trap-Flame Atomic Absorption Spectrophotometry

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Cadmium occurs naturally in the environment but human activities have led to the contamination of environmental samples such as water, soil and air. Tobacco smoking is a direct source of cadmium entry into the body of smokers but other routes of exposure include contaminated drinking water and food, polluted air inhalation and dermal contact with particulate matter [1]. Cadmium has the tendency to accumulate in tissues and organs such as the liver, kidney and bones, leading several health problems and the risk of cancer [2]. Hence, it is very crucial to determine cadmium in different environmental samples in order to protect human health from the negative effects. In this study, an inexpensive but sensitive and accurate analytical method was developed in order to be able to determine cadmium at trace levels. A T-Shape Slotted Quartz Tube (T-SQT) was attached to a regular flame atomic absorption spectrophotometer (FAAS) for the *trap and release* of cadmium atoms to obtain high analytical signals. All parameters of the system under study including sample flow rate, acetylene flow rate (flame type), T-SQT height, trapping period and hydrogen period were carefully optimized to obtain lower detection limits. Analytical figures of merit for the T-Shape Slotted Quartz Tube-Atom Trap-Flame Atomic Absorption Spectrophotometry (T-SQT-AT-FAAS) method were determined using a calibration plot developed between 0.50 and 20 µg/L. In comparison to the conventional FAAS, about 685 times enhancement in detection power was obtained by the optimum method and this correlated to a detection limit of 0.10 µg/L. Spiked recovery tests were performed on tap water to determine the method's applicability to a real sample matrix. The percent recovery results obtained for the sample spiked at 2.0, 3.0, 7.0 and 20 µg/L concentration fell between 99 and 103% and this showed that the sample matrix had no significant effect on the analyte. A coal fly ash standard reference material (1633c) was used to validate the method's accuracy and the experimental result obtained agreed with the certified value.

Keywords: Cadmium, slotted quartz tube, atom trap, hydrogen, atomic absorption

References:

1. E. Akkaya, D.S. Chormey, S. Bakirdere, Sensitive determination of cadmium using solidified floating organic drop microextraction-slotted quartz tube-flame atomic absorption spectroscopy, *Environmental Monitoring and Assessment*, (2017).
2. R. A. Bernhoft, Cadmium Toxicity and Treatment, *Sci. World J.* (2013) 2013 394652: 7 pages.

OP30- Purification of olive mill wastewater by subcritical water oxidation using hydrogen peroxide: Application of response surface methodology

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Olive mill wastewater (OMWW) pollution is a major problem that awaits to be solved by researchers. This wastewater, which releases from olive oil factories, pollutes to the aquatic environment and the soil and threatens the ecosystem [1]. The pollution will be better understood when 30 million tons of olive mill wastewater, annual production in the Mediterranean countries, are considered [2]. Thus, highly efficient methods must be applied for purification of the OMWW. Although several chemical and biological approach have been done, these methods are far from being a solution, due to their low effectiveness, the recalcitrant structure of the compounds and high concentration of polyphenolic content of the OMWW. Subcritical water oxidation is a good alternative to traditional methods by offering many advantages such as short treatment time and high efficiency as well as being an eco-friendly method [2]. Hazardous compounds can be degraded by the synergistic effect of subcritical water and oxidizing agent, H_2O_2 . Chemometric tools have been used to minimize the labor and cost in the scientific works. Herein, the popularity response surface method, one of the several types of statistical design methods, increases day by day.

The purification of OMWW was performed by subcritical water degradation in the presence of H_2O_2 . Effect of three major parameters for the process; temperature, treatment time and oxidant concentration and the interactions between them were evaluated by using central composite design. The achieved yield was determined by the means of COD removal, dephenolisation and dearomatisation efficiencies. The maximum yields were obtained as 85.74%, 96.13%, and 95.94% respectively, for COD removal, dephenolisation and dearomatisation, respectively. In addition, the reliability of the method was assessed by ANOVA test.

Keywords: Olive mill wastewater, degradation, response surface method, subcritical water.

Acknowledgements: This work was funded by Mersin University Research Fund (Project No: BAP 2017-1-AP3-2243).

References:

1. A. Mekki, A. Dhoubi, S. Sayadi, Review: Effects of olive mill wastewater application on soil properties and plants growth, *Int. J. Recycl Org. Waste Agricult.* (2013) 2 15
2. E. Yabalak, Ö. Görmez, B. G. Sönmez, Degradation, dephenolisation and dearomatisation of olive mill wastewater by subcritical water oxidation method using hydrogen peroxide: Application of multi-response central composite design, *J. Serb. Chem. Soc.* (2017). <https://doi.org/10.2298/JSC170909113Y>

OP31- Stability constants of cobalt (II) complexes of octahydropyrrolo[3,4-c]pyrrole N-benzoylthiourea derivatives

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It is very important to determine the stability constants of complexes, because metals are an integral part of living systems and complexes of many transition metals such as cobalt, platinum, nickel, zinc have an important place in pharmaceutical chemistry. Stability constants of complexes provide information about properties of metals and ligands, such as complex formation requests, coordination and solubility of complex molecules. It is known that aroylthiourea derivatives have been intensively studied in coordination chemistry as bidentate ligands, in analytical chemistry as sensor and in medicinal chemistry as pharmacophore group or intermediate compounds for synthesis of pharmacologically important compounds [1-3].

As a continuation of our previous work [4, in this study, we determined stability constants of the complexes formed by the five ligands, octahydropyrrolo[3,4-c]pyrrole N-benzoylthiourea derivatives, with cobalt using HYPERQUAD program from data which were potentiometrically obtained at 25.0 ± 0.1 °C in a 25% (v/v) acetonitrile:water mixture. A number of species formulated as CoL_2 , CoHL_2 , CoH_2L_2 , CoH_3L_2 , CoH_4L_2 , $\text{CoH}_{-1}\text{L}_2$ and $\text{CoH}_{-2}\text{L}_2$ of the Co(II)-octahydropyrrolo[3,4-c]pyrrole N-benzoylthioureas complex occurred in acetonitrile-water solution at the different ranges of the pH. As a result, it can be said that the highest stability constant was obtained for CoH_4L_2 type complexes.

Keywords: Stability constants, Cobalt (II) complex, Thiourea, Pyrrolidine

References:

1. Martell AE, Motekaitisand RJ, Smith RM. Structure-stability relationships of metal complexes and metal speciation in environmental aqueous solutions. *Environ Toxicol Chem* (1998) 7(6) 417-434.
2. Koch KR. New chemistry with old ligands: N-alkyl-and N, N-dialkyl-N'-acyl (aroyl) thioureas in co-ordination, analytical and process chemistry of the platinum group metals. *Coordination Chem Rew*, (2001) 216 473-488.
3. Saeed A, Flörke U, Erben MF. A review on the chemistry, coordination, structure and biological properties of 1-(acyl/aroyl)-3-(substituted) thioureas. *J Sulfur Chem* (2014) 35 318-355.
4. Gemili M, Sari H, Ulger M, Sahin E, Nural Y. Pt (II) and Ni (II) complexes of octahydropyrrolo [3, 4-c] pyrrole N-benzoylthiourea derivatives: Synthesis, characterization, physical parameters and biological activity. *Inorg Chim Acta* (2017) 463 88-96.

OP32- Change of Some Physical Parameters Alter the Antioxidant Capacity of Pine Honey

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Honey is a health promoting natural food due to its bioactive compounds such as phenolic acids and flavonoids. Antioxidant, antimicrobial, antitumoral, antiinflammatory and wound healing activities reveal its medicinal value (1-3). However, the quality of honey is the primary factor effects these health benefits. On the other hand, the quality of honey depends on various parameters such as floral origin, environmental conditions, industrial processing and storage.

Crystallization is one of the main problem during industrial processing and storage. Heat treatment is useful way to solve this problem. Ultrasonication is an alternative technique in order to prevent and delay crystallization (4). Exposing to sunlight is an important environmental condition during the period on the shelf.

In the present study, it is aimed to evaluate the effects of thermal treatment, ultrasonication and sunlight on antioxidant activity of pine honey, which is one of the quality parameters of honey. In brief, results indicated that thermal treatment at 60 and 80 °C, exposure to sunlight for 10 days negatively affected antioxidant properties of pine honey. However, ultrasonication for 60 min. promoted to the values.

Keywords: Pine Honey, Antioxidant, Quality, Heat treatment

References:

1. Md. S. Hossen, Md. Y. Ali, M. H. A. Jahurul, M. M. Abdel-Daim, S. H. Gan, Md. I. Khalil (2017). Beneficial roles of honey polyphenols against some human degenerative diseases: A review, *Pharmacological Reports* 69: 1194–1205.
2. M. Badolato, G. Carullo, E. Cione, F. Aiello, M. C. Caroleo (2017). From the hive: Honey, a novel weapon against cancer, *European Journal of Medicinal Chemistry* 142: 290-299.
3. F. M. Bueno-Costa, R. C. Zambiasi, B. W. Bohmer, F. C. Chaves, W. P. da Silva, J. T. Zanusso, I. Dutra (2016). Antibacterial and antioxidant activity of honeys from the state of Rio Grande do Sul, Brazil, *LWT - Food Science and Technology* 65: 333-340.
4. R. Subramanian, H. U. Hebbar, N. K. Rastogi (2007). Processing of honey: A review, *International Journal of Food Properties*, 10: 127–143.

OP33- Spectrophotometric Determination of Allura Red (E129) in Foodstuffs after Cloud Point Extraction

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Allura red (AR) is a synthetic azo dye which has been widely used in foodstuffs to improve and upgrade their features such as color, taste and stability. Structure of AR is given in Fig. 1. Most of synthetic dyes are produced from aromatic hydrocarbons contained in petroleum [1]. Use of AR in foodstuffs is regulated with a certain limited concentration. Some health problems can be observed in humans when consumed excessive amount of AR via foodstuffs. Although AR is not acute toxic, it can lead to asthma, skin irritation, rash and eczema in humans. In addition, hyperactive behavior disorders have been observed in children [2]. Therefore, developing reliable determination method is of great importance to control AR contents of foodstuffs and ensure food safety.

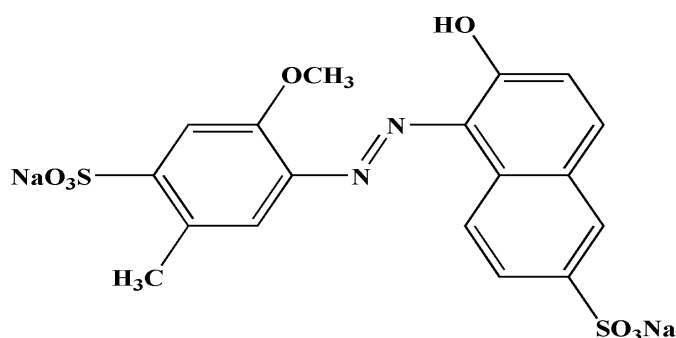


Figure 1. Chemical structure of AR dye

Cloud point extraction combined UV-Vis spectrophotometric determination method for AR dye was developed. Extraction parameters (H_2SO_4 concentration, TNP-7 surfactant amount, salt concentration, equilibrium temperature and time) affecting to quantitative extraction of AR was investigated and optimized. Determination of AR was performed at 506 nm. At the optimum conditions limit of detection was determined as 3.0 ng/mL of AR. Calibration graph was plotted as linear in a concentration range between 0 and 6.0 $\mu\text{g/mL}$. Preconcentration factor of the method was obtained as 25. Finally, method was applied to drink powder, energy drink, candy, syrup and jelly samples to determine AR contents of them. 47-231 $\mu\text{g/mL}$ and 9-499 $\mu\text{g/g}$ of AR concentrations were determined for liquid and solid foodstuff samples, respectively. In order to prove reliability and precision of the method analyte addition technique was also applied to real samples. Quantitative recovery results were obtained between 98 % and 100 %.

Keywords: Allura red, cloud point extraction, foodstuffs, UV-Vis spectrophotometry

References:

1. Soylak M, Unsal YE, Tuzen M, Spectrophotometric determination of trace levels of allura red in water samples after separation and preconcentration, *Food. Chem. Toxicol.* (2011) 49 1183-1187.
2. Bişgin AT, Uçan M, Narin I, Comparison of Column Solid-Phase Extraction Procedures for Spectrophotometric Determination of E129 (Allura Red) in Foodstuff, Pharmaceutical, and Energy Drink Samples, *J. AOAC Int.* (2015) 98 946-952.

OP34- Solvent and Molecular Structure Effects on Acidity Strength in Non-Aqueous Medium

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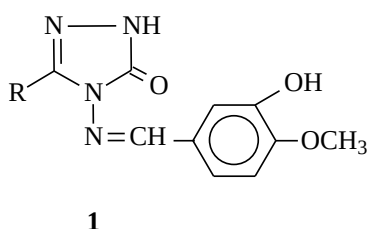
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There are two major factors influencing the acidity or basicity of a molecule is well known. These factors are solvent and structural effects. Unfortunately, in the majority of molecules there are two or more structural effects and it is usually difficult to assess how much each effect contributes to the acidity or basicity of the molecule [1]. This paper reports on the acidic properties of ten 3-alkyl(aryl)-4-[3-hydroxy-4-methoxy-benzylidenamino]-4,5-dihydro-1*H*-1,2,4-triazol-5-ones (**1**) were investigated. Due to very low solubility of 4,5-dihydro-1*H*-1,2,4-triazol-5-one derivatives in water, non-aqueous media were used. The compounds were synthesized according to the literature [2]. In non-aqueous medium, isopropyl alcohol and *tert*-butyl alcohol among the amphiprotic type, and acetone and *N,N*-dimethylformamide among the dipolar aprotic type were preferred as solvent. The potentiometric method used in determining the end-points in titrimetric analyses was utilized for the determination of acidity. The acidity strength of **1** type compounds with varying R groups in different solvents, dielectric constant of the solvents, autoprotolysis constant and leveling-differentiation effects were investigated.



R	1
a	CH ₃
b	CH ₂ CH ₃
c	CH ₂ CH ₂ CH ₃
d	CH ₂ C ₆ H ₅
e	CH ₂ C ₆ H ₄ CH ₃ (<i>p</i> -)
f	CH ₂ C ₆ H ₄ OCH ₃ (<i>p</i> -)
g	CH ₂ C ₆ H ₄ Cl(<i>p</i> -)
h	CH ₂ C ₆ H ₄ Cl(<i>m</i> -)
i	C ₆ H ₅
j	

This study was supported by a grant (Project Number: 2008.116.006.1) from Scientific Research Projects Coordination Unit of Karadeniz Technical University.

Key Words: 1,2,4-Triazoles, non-aqueous media, acidity strength

References

- Gündüz T, Kılıç E, Ertüzün V., G, Part V. Acid-Base Behaviour of Substituted 2-Amino-3-cyanofurans, 2-Amino-3-cyanothiophenes and 2-Amino-3-cyanopyrrole and Their Schiff Bases with Salicylaldehyde and 2-Hydroxy-1-naphthaldehyde, *Analyst*, 111 (1986) 1439-1442.
- Bahceci S, Yıldırım N, Gursoy-Kol O, Manap S, Beytur M, Yüksek H, Synthesis, Characterization and Antioxidant Properties of New 3-Alkyl(aryl)-4-(3-hydroxy-4-methoxy-benzylidenamino)-4,5-dihydro-1*H*-1,2,4-triazol-5-ones, *Rasayan J. Chem* 9 (2016) 494-501.

OP35- Optimization of Poly (Acrylic Acid-co-N-vinyl 2-pyrrolidone) / SiO₂ Nanocomposite Hydrogels

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Copolymeric hydrogels have better mechanical stability and durability than homopolymeric hydrogels. In this study, the monomers (acrylic acid and N-vinyl 2-pyrrolidone) were used to optimizing the monomer/monomer ratio and reinforcement material which shows the best strength property selected from those hydrogels obtained in this study. Nano-sized SiO₂ (20-30 nm) was commercially purchased and used as a reinforcing material for synthesis of nanocomposite hydrogels.

Amount of monomer/monomer in volume ratios for acrylic acid (AA)-vinyl pyrrolidone (VP) 3-1; 2-2 and 1-3 were used. In the presence of optimized 1% cross-linker (molar ratio) and radical initiator (APS), by mass of free radical polymerization method 0.05%; 0.5% and 1% nano SiO₂ added AA-co-VP nanocomposite hydrogels were synthesized. % Conversion (%C), % crosslinking (%CL) and % swelling (%S) values of the obtained hydrogels were calculated. The chemical bonds of the hydrogels were clarified by FTIR analysis. The surface morphologies of the hydrogels were shown by SEM analysis. These hydrogels, which also showed superadsorbent properties, were optimized for the removal of cationic dyes under this study.

Keywords: nano SiO₂, nanocomposite, hydrogel, acrylic acid.

OP36- Spectrophotometric determination of cefpodoxime proxetil in Pure and Pharmaceutical Preparations

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Cefpodoxime proxetil is a broad-spectrum oral cephalosporin. Cefpodoxime proxetil is a prodrug which is thought to be deesterified to its active metabolite (cefpodoxime) by intestinal wall esterases. The drug acts by binding to penicillin-binding proteins and affects bacterial wall synthesis (1). Its chemical name is (RS)-1(isopropoxycarbonyloxy)ethyl(+)-(6R,7R)-7-[2-(2-amino-4-thiazolyl)-2-[(Z)methoxy-imino]acetamido]-3-methoxymethyl-8-oxo-5-thia-1-azabicyclo [4.2.0]oct-2-ene-2-carboxylate(2).

In this study, a novel, sensitive and selective spectrophotometric method was developed for the determination of cefpodoxime in pharmaceutical preparations. The proposed method is based on the reaction of cefpodoxime proxetil with 7,7,8,8-Tetracyanoquinodimethane (TCNQ) as a new derivatization reagent in alkaline solutions. Maximum absorbance of derivative in acetonitrile is measured at 601 nm. The described method was validated and the analytical parameters of linearity, limit of detection, limit of quantification, accuracy, precision, recovery and robustness were evaluated. A linear relationship existed between absorbance and cefpodoxime proxetil concentration over 10-60 µg/mL range. The proposed method was successfully applied for the determination of cefpodoxime proxetil in pharmaceutical preparations and the obtained results were in good agreement with those obtained by the reference method.

Keywords: cefpodoxime proxetil, spectrophotometry, TCNQ (7,7,8,8 Tetracyanoquinodimethane)

References:

1. M. T. Borin, G. S. Hughes, C. R. Spillers, and R. K. Patel, Pharmacokinetics of Cefpodoxime in Plasma and Skin Blister Fluid following Oral Dosing of Cefpodoxime Proxetil, *Antimicrob. Agents Chemometer.* (1990) 1094-1099.
2. Subbayama, A.B, Rambabu C. Spectrophotometric Determination of Cefpodoxime Proxetil in Tablets. *Asian J. Chem.* (2010) 3345-3348.

OP37- Voltammetric Determination of Boron in Dried Fruits and Nuts

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This study describes the application of two different voltammetric methods for determination of boron in dried fruits and nuts. In the first method, the oxidation current of Alizarin Red S (ARS) in the Boric acid-ARS complex was monitored as a signal at the copper or gold nanoparticles and multiwalled carbon nanotubes modified glassy carbon electrode (CuNP/CNT/GCE and AuNP/CNT/GCE). In the second method, the oxidation current of Tiron in the Boric acid-Tiron complex at the poly xylenol orange modified and oxidized pencil graphite electrode (poly-XO/ox-PGE) was measured. Both of the methods were applied to hazelnut, peanut, almond, raisin, prune and date samples after microwave digestion procedure for voltammetric determination of boron. The results were compared with ICP-MS technique and there is no significant difference between the methods for 95% confidence level. In addition, the accuracy of these methods was proved with UME CRM 1202 (Elements in Hazelnut).

Keywords: Boron, Alizarin Red S, Tiron, Dried Fruits and Nuts

References:

1. Liv, L., Nakiboglu, N., Simple and rapid voltammetric determination of boron in water and steel samples using a pencil graphite electrode, Turk. J. Chem. (2016) 40 412-421.
2. Liv, L., Nakiboglu, N., Voltammetric determination of boron using a poly xylenol orange modified pencil graphite electrode, Anal. Lett. (2018) 51 170-185.

OP38- Spectrophotometric Determination of Bioactive Compounds in *Spirulina platensis*

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Algae, a group of marine or freshwater organisms, are traditionally used for direct consumption in Asian countries, and recently, their consumption as functional foods have also spread to Western countries. Apart from high-quality proteins containing essential amino acids, dietary fiber, essential fatty acids, minerals, and vitamins, algae could also be a good source of phenolic compounds. Microalgae have been used as sources of important biochemicals, which may be rare in plant and animal. In this regard, the blue-green alga *Arthrospira* (*Spirulina*) *platensis* is one of the most promising microalgae, which may be used as a food and pharmaceutical source. Due to several studies have shown that *Spirulina* and its extracts could prevent or inhibit cancer in humans and animals and have immuno-promoting effects, antimicrobial activity, antioxidant activity, antihypertensive activity, there is an increasing interest in natural antioxidants from microalgae. Among bioactive compounds, phenolics and flavonoids are receiving attention mainly because of their wide range of potential applications and serve as natural antioxidants.

In this study total phenolics, flavonoid content, antioxidant activity and reducing power activity were determined for *Spirulina platensis*. Biomass production and harvesting conditions, and extraction procedures used for bioactive compounds were shown in figure.

Keywords: *Spirulina platensis*, bioactive, phenolic, flavonoid, antioxidant

OP39- Electrochemical Determination of Bisphenol-A Based on Copper Oxide-Zinc Oxide/Graphene Oxide Modified Electrode

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Bisphenol A (BPA), one of the key monomers in the production of epoxy resins and polycarbonate, is well-known as an important endocrine disruptor [1,2]. Therefore, it is very important and interesting to determine its presence in various sample. Although chromatographic and spectroscopic methods have been widely used for highly sensitive and determination of BPA, electroanalytical techniques, with cheap instrumentation, simple procedure, time saving, high sensitivity and selectivity are alternative to monitor lower concentration of BPA [3,4].

Herein, a simply and selectively electroanalytical method for determination of BPA was developed using CuO-NiO and graphene oxide (GO) modified glassy carbon electrode (GCE). The composite electrodes surface morphology and chemistry were identified using cyclic voltammetry (CV), scanning electron microscopy (SEM), X-Ray photoelectron spectroscopy (XPS) and electrochemical impedance spectroscopy (EIS). The optimization of some parameters related electrode preparation such as electrodeposition potential and time of CuO and ZnO deposition on GO, volume of GO on the GCE surface etc. was found by cyclic voltammetry in pH 7.0 Britton-Robinson buffer solution containing 0.1 mM BPA. The validation parameters were evaluated with square wave voltammetry. The electrochemical sensor responds linearly to BPA in the 3.0 nM to 6.0 μ M concentration range, and the detection limit is 1.1 nM (at an S/N of 3). The proposed CuO-ZnO/GO/GC modified electrode was applied successfully for the analysis of BPA in various plastic samples such as baby feeding bottles, baby nipple, drinking water bottle and food container samples. The studies indicated that the CuO-ZnO/GO/GCE exhibited remarkable electrocatalytic activity for the electrooxidation of BPA, which lead to the sensitive, easy and cheap method was established to analyze of BPA in various plastic samples.

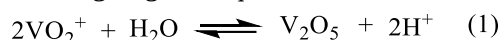
Keywords: Bisphenol A, graphene oxide, composite electrode.

References:

1. Y. Li, Y. Gao, Y. Cao, H. Li, Electrochemical sensor for bisphenol A determination based on MWCNT/melamine complex modified GCE, *Sens. Act. B* (2012) 171-172 726-733.
2. A. Ghanam, A.A. Lahcen, A. Amine, Electroanalytical determination of Bisphenol A: Investigation of electrode surface fouling using various carbon material, *J. Electroanal. Chem.* (2017) 789 58-66.
3. P. Deng, Z. Xu, Y. Kuang, Electrochemical determination of bisphenol A in plastic bottled drinking water and canned beverages using a molecularly imprinted chitosan-graphene composite film modified electrode, *Food Chem.* (2014) 157 490-497.
4. Y.T. Yaman, S. Abacı, Sensitive adsorptive voltammetric method for determination of bisphenol a by gold nanoparticle/polyvinylpyrrolidone-modified pencil graphite electrode, *Sensors* (2016) 16 756-768.

OP40- Organic Based Additives for Vanadium Redox Battery Systems**Metin Gençten¹, Hurmus GURSU², Yucel Sahin²**¹Yildiz Technical University, Faculty of Chemical and Metallurgical Engineering, Department of Metallurgy-Material Engineering, 34210, Istanbul, Turkey;*E-mail: mgençten27@gmail.com²Yildiz Technical University, Faculty of Arts&Science, Department of Chemistry, 34220, Istanbul

Electrochemical energy storage devices have great potential to storage of electric energy obtained from renewable energy sources in chemical form [1]. Although traditional energy storage devices such as lithium-ion and lead acid battery have been used in storage of this energy, they are not sustainable for the future due to their limitations. Since lithium metal is a critical element and will soon be consumed, this battery type has an important limitation. Lead based batteries has also many negative effects on the environment [2]. Here, vanadium redox flow battery (VRB) emerges an alternative energy storage to the conventional systems. A VRB consists of vanadium ion at different oxidation states in sulfuric acid in a divided cell by nafion based membrane [4, 5]. Thermal precipitation of V(5) ions as V₂O₅ form at high temperature is a crucial problem for VRB system according to given equation in 1 [3, 4].



In this study, l-histidine and p-aniline sulfonic acid were used for the anti-precipitant agents for positive electrolyte of VRB. The optimum amount of additives were determined by using of electrochemical methods such as cyclic voltammetry and electrochemical impedance spectroscopy. Thermal stability of V(5) ions at high temperature was determined with precipitation test at -20, 25 and 60 °C. The additives showed good anti-precipitant properties for V(5) ions. The effects of these additives on discharge capacity of VRB were also investigated by cyclic charge-discharge tests. The used additive played an important role on the improving performance of VRB (Fig. 1).

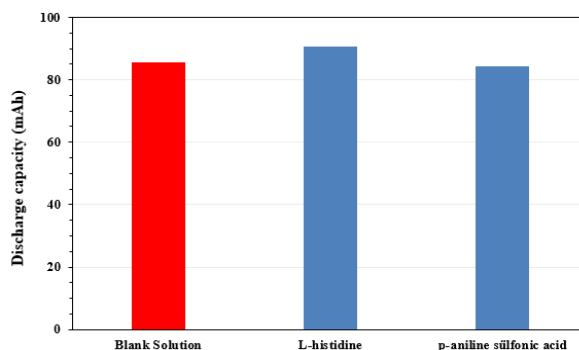


Fig. 1 Discharge capacity of V(5) solutions, blank and consisting additives

Keywords: Cyclic voltammetry, charge-discharge tests, l-histidine, p-aniline sulfonic acid

*This work was supported by TUBİTAK with the 114Z774 project code. M. Gençten and H. Gürsu thank to TUBİTAK-BİDEB.

References:

1. M. Gençten, H. Gürsu, Y. Şahin, Electrochemical investigation of the effects of V(V) and sulfuric acid concentrations on positive electrolyte for vanadium redox flow battery. *Int. J. Hyd. Energ.* 41(2016) 9868-9875.
2. Enos, D.G. (2015). Lead-acid batteries for medium and large-scale energy storage. C. Menictas, M. Skyllas-Kazacos, T.M. Lim (Eds.), *Advances in batteries for medium and large-scale energy storage: types and applications* (s. 57-71). United Kingdom: Elsevier Ltd.
3. M. Gençten, H. Gürsu, Y. Şahin, Anti-precipitation effects of TiO₂ and TiOSO₄ on positive electrolyte of vanadium redox battery. *Int. J. Hyd. Energ.* 42 (2017) 25608-25618.
4. M. Gençten, H. Gürsu, Y. Şahin, Effect of α- and γ-alumina on the precipitation of positive electrolyte in vanadium redox battery. *Int. J. Hyd. Energ.* 42 (2017) 25598-25607.

OP41-Electrochemical Impedance Spectroscopy Based Modelling Approach for Primary LiSOCl₂ Battery

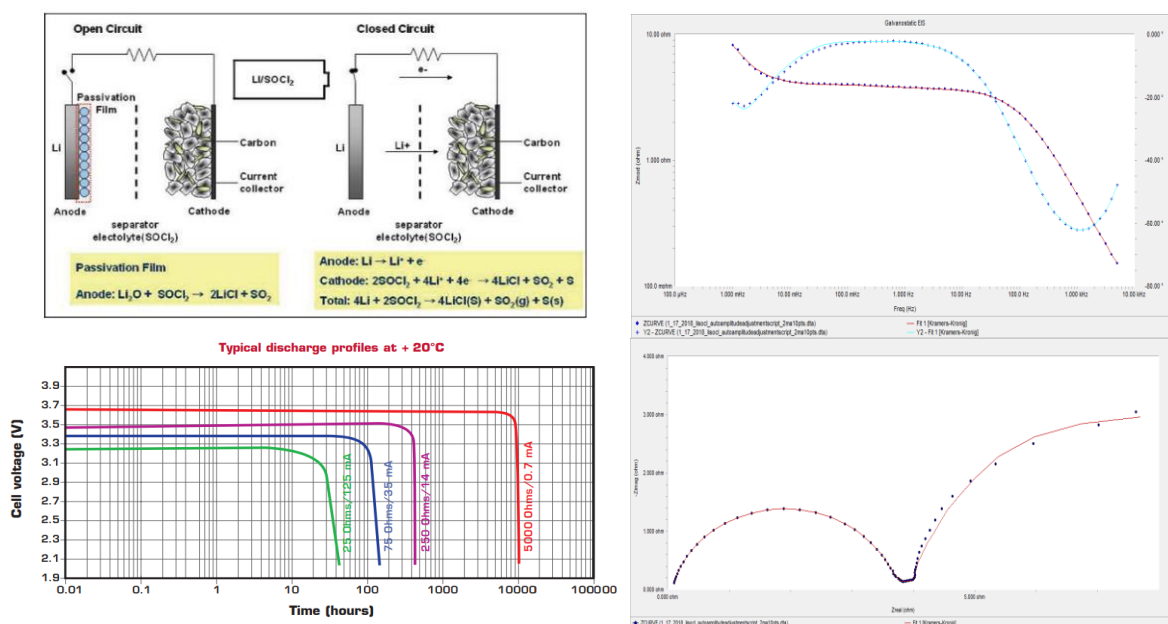
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Lithium Thionyl Chloride (LiSOCl₂) primary battery has received considerable attention as a primary energy source due to its high energy density, high operating cell voltage, voltage stability over 95% of the discharge, large operating temperature range (-55 to 70°C), long storage life, and low cost of materials¹. These outstanding properties found valuable applications in significant fields mainly in military and defense systems. Understanding the behavior of the battery is important for finding suitable application conditions, extending their lifetime and increasing the safety of use.

Electrochemical impedance spectroscopy (EIS) is a powerful technique for investigating the properties of various electrochemical systems. It is based on measuring the response of the electrochemical system to alternating current (AC) signals with various frequencies. In the literature, not many studies have been done to characterize LiSOCl₂ using EIS. The difficulty of utilizing EIS for LiSOCl₂ battery characterization lays on the nonlinear behavior of the battery at various frequencies. In this work, we will demonstrate a successful method that apply EIS to determine the electrochemical properties of LiSOCl₂. The method is based on the analysis of the AC response signal harmonics, which allows us to establish measurement parameters. The new parameters are suitable for obtaining acceptable impedance spectrum and eliminate the nonlinearity behavior.



Keywords: Electrochemical impedance spectroscopy, Primary battery, Lithium Thionyl Chloride

References:

1. M. Jain, G. Nagasubramanian, R. G. Jungst, J. W. Weidner, Analysis of a Lithium/Thionyl Chloride Battery under Moderate-Rate Discharge, Journal of The Electrochemical Society (1999) 146 (11) 4023-4030.
2. Thomas B. Reddy (2011), Linden's Handbook of Batteries, McGraw-Hill Education, New York.

OP42- Development of Cobalt Oxide Modified Electrodes and Their Investigation in Electrocatalytic Applications

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Transition metal oxides are gaining special interest in the last two decades due to their unique electronic and electrocatalytic characteristics. Cobalt oxides attract significant attention mainly due to their excellent electrocatalytic activity toward various compounds. Several methods have been developed for the preparation of cobalt oxide including but, electrochemical method offers a practical way for tuning its catalytic properties [1].

This study concerns with electrochemical preparation of cobalt oxide and platinum nanoparticle composite electrodes on a GCE surface in the pursuit of its catalytic applications. Electrochemical deposition of the metal-metal oxide film structure has been carried out by pulsed deposition and cyclic voltammetry. The parameters affecting the deposition conditions have been evaluated by monitoring oxygen reduction reaction (ORR) in pH 5.0 acetate buffer. Solution parameters such as medium pH, buffer type and Co(II) concentration have been optimized along with the operational parameters such as scan rate and pulse duration. In case of cyclic voltammetry, the repetitive cycle number has been optimized. Under optimal conditions, the electrodes developed by both electrochemical modes have been compared with bare and Pt nanoparticle and CoOx modified electrodes. In both methods, the Pt/CoOx/GCE has given the best results for ORR in terms of peak current and peak potential. This electrode was used as a platform for a biosensor. Polyphenol oxidase enzyme was immobilized onto the electrode surface and used for phenol detection. Medium pH and the enzyme amount have been optimized as pH 7.0 and 0.4 mg, respectively. Chronoamperograms were recorded for phenol detection in micromolar levels.

Keywords: Metal–metal oxide electrodes, Pulsed deposition, oxygen reduction reaction, biosensor

References:

- I. G. Casella, M. Contursi, J Solid State Electrochem (2012) 16:3739–3746
Z. Meng, B. Liu, J. Zheng, Q. Sheng, H. Zhang, Microchim Acta (2011) 175:251–257

Acknowledgement: Authors would like to thank E.U. Scientific Research Project (2017 FEN 061) for financial support.

OP43- Determination of Ultratrace Cadmium by a New Combination: Solid Phase Microextraction by Stearic Acid Coated Magnetic Nanoparticles Prior To Batch Type Hydride Generation Atomic Absorption Spectrometry

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One of the greatest challenges facing mankind is heavy metal toxicity and this arises from anthropogenic activities such as mining and industrial processes [1]. Cadmium is one of the heavy metals which is said to be nonessential for humans and enters the body through contaminated water, food, particulate matter and smoking of cigarette. Long term exposure to cadmium can lead to health disorders such as bone diseases, hypertension, kidney disease and liver disease [2]. The World Health Organization therefore set an allowable limit of cadmium in drinking water as 3.0 ng/mL.

In this study, magnetite nanoparticles were used to preconcentrate cadmium from aqueous solution for determination by a hydride generation atomic absorption spectrometry system. The extraction capacity of the nanoparticles was enhanced by coating them with stearic acid which increased the surface area of extraction. The magnetic property of the nanoparticles also facilitated their separation from the aqueous solution after extraction. Experimental parameters influencing the extraction efficiency for cadmium and instrumental parameters influencing the generation, transport and atomization of cadmium hydride were thoroughly optimized to lower the detection limit of the system. The limit of detection (LOD) and limit of quantification (LOQ) of this solid-phase microextraction (SPME) method based on stearic acid coated magnetic nanoparticles (SACMNP) coupled to batch type-hydride generation atomic absorption spectrometry (BT-HGAAS) were 81.7 ng/L and 270.8 ng/L. Other figures of merit including linearity, precision, dynamic range and regression coefficient were satisfactory. Applicability of the developed method to tap water was tested by performing spiked recovery studies. Matrix matched standards were used to overcome possible matrix interferences to improve analyte recovery. The percent recoveries obtained for 6.0 and 8.0 ng/mL spiked concentrations were 88.56±8.92 and 97.43±9.76, respectively. These results validated that the method can be used to determine cadmium in tap water with good accuracy and precision.

Keywords: Cadmium, Magnetic nanoparticles, Stearic acid, Solid phase microextraction

References:

1. Y. Yang, X. Q. Ye, B.Y. He, J. Liu, Cadmium potentiates toxicity of cypermethrin in zebrafish, *Environ. Toxicol. Chem.* 35 (2016) 435-445.
2. N. A. Kasa, D. S. Chormey, Ç. Büyükpınar, F. Turak, T.B . Budak, S. Bakirdere, Determination of cadmium at ultratrace levels by dispersive liquid-liquid microextraction and batch type hydride generation atomic absorption spectrometry, *Microchem. J.* 133 (2017) 144-148.

OP44- Temporal Changes in Trace Element Concentrations in Spring, Shallow and Deep Well Waters from the Uluova Plain, Elazig

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Assessment of trace, particularly toxic trace, elements has high popularity due to widespread occurrence of pollution resulting from industrial and agricultural activities. In the past several decades, our understanding of water quality has expanded with our increased understanding of the importance of the hydrologic cycle for groundwater quality conditions. Additionally, other natural factors, including geology, tectonic structure and geomorphology, also control the timing and amount of surface and groundwater flow and the transport of waterborne constituents, including contaminants. Furthermore, natural complexity is increasing because of changes in climate, resulting in new patterns of seasonal precipitation, runoff, and spatial and temporal distribution of snow versus rain^{1,2}.

In this study, our aim was to examine temporal changes in trace element concentrations at three different locations, including spring, shallow and deep well waters, in a monthly frequency between November 2012 and October 2013. The water sampling sites are in the middle of Uluova plain near Elazig city. There are industrial factories and Elazig airport near the study area. Furthermore, agricultural activities are intense in the region where the aquifer beneath the plain provides drinking water of Elazig city with a population of 600 000. The results obtained suggest that arsenic levels in most of the studied water samples are higher than the maximum concentration allowed in drinking water. However, the concentrations of other elements were lower than the permissible levels. Climatic conditions change the trace element concentrations, especially in water samples taken from spring and shallow well waters.

Keywords: Trace Elements, Arsenic, Water, ICP-MS.

References:

1. Matthew, C., Larsen, Pixie, A. Hamilton, William H. Werkheiser, Water Quality Status and Trends in the United States, Monitoring Water Quality (2013), 19-57.
2. Larsen M.C. Water Resources Impact. American Water Resources Association (2012), 14(5), 3–7.

OP45- Separation and Determination of Rosmarinic and Carnosic Acid Contents of Anatolian *Salvia* Species by Capillary Electrophoresis: Correlation Between Ingredient Contents and Plant Antioxidant Activities

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Salvia genus which belongs to Lamiaceae family, is widely distributed in several regions of the world particularly in the Mediterranean and Asia with more than 900 species (1). Anatolia is one of the major centre for *Salvia* where almost the half of these species are endemic. It is important to reveal the chemical and biological profiles of *Salvia* species. Rosmarinic acid is one of the most well-known diterpenoid found in the members of the genus *Salvia* (1). It has many biological activities against tumor, HIV-1, hepatitis and inflammation. Together with rosmarinic acid, carnosic acid is another diterpenoid generally accepted as the most antioxidant compound in *Salvia*.

In this study, rosmarinic acid and carnosic acid contents of 29 Anatolian *Salvia* species were investigated by a simple and well-validated capillary zone electrophoresis method. The running buffer composed of 20 mM sodium tetraborate adjusted to pH 9.0. Rosmarinic acid was detected as the major compound in capillary electrophoresis profiles of all the *Salvia* species. Carnosic acid was found in only 3 species. Furthermore, the antioxidant activities and total phenolic contents of *Salvia* extracts were researched. A strong positive correlation was observed between the rosmarinic acid contents and antioxidant activities of *Salvia* species, as expected. In consideration of these data, commercial use of these species by the food industry or alternative use as medicinal drug throughout the world can be provided.

Keywords: Antioxidant activity, Capillary electrophoresis, Rosmarinic acid, *Salvia*

References

- 1- Lopresti A, *Salvia* (Sage): A Review of its Potential Cognitive-Enhancing and Protective Effects, *DRUGS IN R&D*, (2017) 17:53–64.

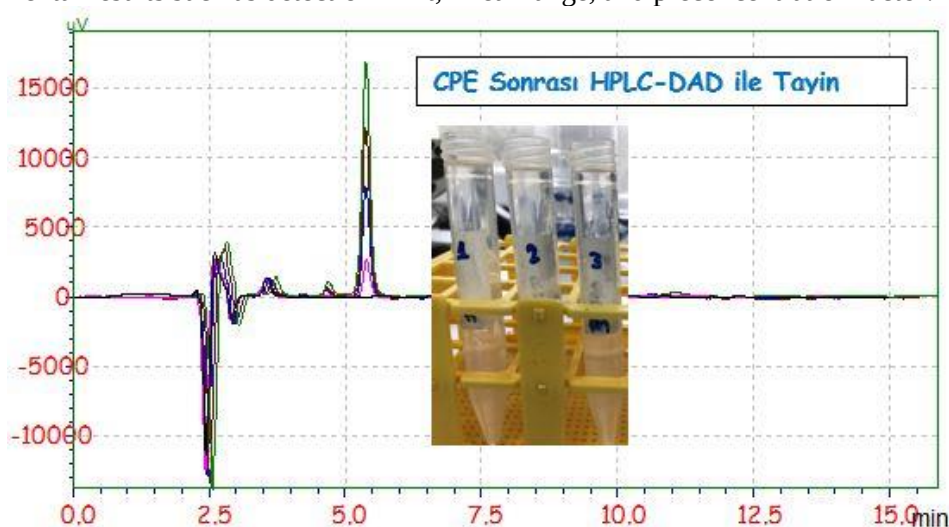
OP46-Development of an Analytical Method for Determination of Tetracycline Residues in Food Samples

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In this study, a preconcentration and determination method for tetracycline residues in food samples was developed based on cloud point extraction and HPLC-DAD analysis. Tetracycline molecules was extracted to surfactant phases of poly ethylene glycol (PEG-6000) in the presence of pH:5.0 buffer and high electrolyte concentration. The samples were centrifuged in order to increase phase separation and then diluted and filtered by 0.45 μm membrane filter prior to HPLC determinations. Experimental variables were optimized and examined such as concentration of electrolyte and surfactant, pH, time effect. All analytical merits were determined by using experimental results such as detection limit, linear range, and preconcentration factor.



In development method, determination of tetracycline molecules in the surfactant rich phase was carried out by isocratic elution of pH:4 oxalate buffer;methanol;acetonitrile solvents and absorbance measurements of DAD detector at 276 nm and 358 nm. By optimization all experimental parameters, linear ranges for both wavelengths were calculated as 10-1000 ng mL^{-1} and 25-800 ng mL^{-1} , respectively. Detection limits were 2.98 and 7.46 ng mL^{-1} while relative standard deviations (RSD%) were lower than 4.20 % for 100 ng mL^{-1} tetracycline. Finally, the developed method was applied to food samples including milk, liver and breast tissues of chicken.

Acknowledge: This study has been supported by Cumhuriyet University Scientific Research Projects Commission as the research project with the ECZ-052 code.

Keywords: Tetracycline, PEG-6000, HPLC, Cloud point extraction, Food samples

References:

5. David Moreno-González, Ana M.García-Campaña, Salting-out assisted liquid-liquid extraction coupled to ultra-high performance liquid chromatography-tandem mass spectrometry for the determination of tetracycline residues in infant foods, Food Chemistry (2017) 221 (1763-1769).
6. Michael Pérez-Rodríguez, Roberto Gerardo and et al. An overview of the main foodstuff sample preparation technologies for tetracycline residue determination, Talanta (2018) 182 (1-21).

OP47- Dynamic Phosphometabolomic Profiling of Caco-2 cell lines by ¹⁸O-assisted GC-MS

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Today's innovative technologies permit comprehensive screening of the entire genome and transcriptome, proteome, and metabolome. Metabolomic profiling of disease states mandates comprehensive and simultaneous systematic fingerprinting of multiple metabolites. Many of the diseases are complex and multifactorial diseases and can be understood by analyzing the phenotypic determination of the disease and the changes in the level of cells, tissues and organisms at the level of more than one gene, pathway, protein and metabolite. Characterization of metabolic phenotypes requires not only the metabolite levels, but also the speed of the turnover rates. In particular, there may be no change in the concentration of many metabolites with low concentrations and high turnover rates. However, there are significant increases or decreases in turnover rates. These types of analyzes, which are based on the calculation of the metabolite levels as well as the turnover rates, are called pathway analysis (fluxomic). Stable isotope (¹³C, ¹⁵N and ¹⁸O) tracer-based metabolomic technologies allow simultaneous determinations of metabolite levels and their turnover rates with subsequent evaluation of metabolic network dynamics. ¹⁸O stable isotope traces, which have many advantages than the others [1].

In this study, cancer -dependent shift of cellular energetics and phosphotransfer kinetics of CaCo-2 cell lines and FHC (control cell line) were determined using ¹⁸O-labeling phosphometabolomic methodology and mass spectrometry. A 400 µL of the each phosphometabolites was evaporated to dryness in a vacuum dryer concentrator and methoxyaminated and derivatized with MSTFA with 1% TMCS. After derivatization, the samples were transferred into GC-MS vials. Metabolomic profiling was performed using GC-MS (Shimadzu GCMS-QP2010 Ultra) with DB-5MS stationary phase column (30 m +10 m duraguard × 0.25 mm i.d. and 0.25-µm film thickness). Once analysis completed, the complex chromatograms were deconvoluted using AMDIS and the retention time correction and data matrixes creation were done using XCMS software. To get a broader picture of rearrangements in the energetic system and insights into mechanisms, turnover rates of inorganic phosphate (an indicator of ATP utilization), γ-ATP (an indicator of ATP synthesis), β-ATP and β-ADP (indicators of AK flux), glucose-6-phosphate (an indicator of glycolytic flux), glucose-1-phosphate (an indicator of glycogenolytic flux), and glycerol 3-phosphate (an indicator of substrate shuttle activity) were determined using ¹⁸O -assisted mass spectrometry. Combination of metabolome and fluxomic data allows us to better stratification ($R^2 > 0.80$ and $Q^2 > 0.8$) between CaCo-2 and FHC cell lines.

Keywords: ¹⁸O-labeling, fluxomic, phosphometabolomic, GC-MS

Reference:

[1] E. Nemutlu, S. Zhang, N.O. Juranic, A. Terzic, S. Macura, P. Dzeja, (18)O-assisted dynamic metabolomics for individualized diagnostics and treatment of human diseases, Croatian Medical Journal, 53 (2012) 529-534.

This work was supported by The Scientific and Technological Research Council of Turkey (TBİTAK) (Project no: 116Z292).

OP48- Determination of Khellin and Visnagin Contents of *Amni Visnaga* and *Amni Majus* by Capillary Electrophoresis with UV Detection

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Plants are valuable sources of various bioactive compounds which can be used in many pharmaceutical formulations. Their curative abilities strongly related with these bioactive constituents. *Amni visnaga* also known as *bisnaga*, *toothpick weed* or *khella* and *Amni Majus* are belongs to the *Apiaceae* (*Umbelliferae*) family which has about 300 to 455 diverse genera and thus over 3000 species. Among these species *Amni Visnaga* is well known for using as herbal medicine to heal coronary diseases and bronchial asthma. *Amni Majus* is indigenous species compared to *Amni Visnaga* and grown in Egypt; however *Amni Visnaga* grown in warm climates such as Mediterranean areas including Turkey. Khellin and Visnagin active compounds which shows positive effects on inhibiting the spasms and involvement of a calcium channel-blocking mode of action and many other activities and *Amni* species are consider as a source for them. Capillary electrophoresis (CE) is a fast and reliable method with high separation power, as a result, it is a valuable technique for the analysis and separation of valuable compounds in highly complicated natural matrices such as plants [1, 2].

In this study, a simple and reliable capillary electrophoretic method for determination of khellin and visnagin contents of *Amni Visnaga* and *Amni Majus* species was developed. The khellin and visnagin contents are evaluated in five different parts of each *Amni* species, namely: leaf, root, flower, seed and peduncle. Complete capillary electrophoretic separation of khellin and visnagin was achieved by using 20 mmol/L sodium tetraborate, 20 mmol/L sodium dodecylsulfate and 5 (v/v)% methanol as separation electrolyte at pH 9.6. The injections were carried out hydrodynamically under 50 mbar pressure for 6 seconds and the separation voltage was selected as 25 kV. It was found that *Amni Visnaga* seeds, flowers and leaves are good sources of khellin and visnagin both, despite *Amni Majus* only contains khellin in its seeds and flowers.

Key Words: Khellin, Visnagin, *Amni Visnaga*, *Amni Majus*, Capillary electrophoresis

References:

- 1- Gunaydin K, Erim F.B., Determination of khellin and visnagin in *Amni visnaga* fruits by capillary electrophoresis, J Chromatogr A, 954 (2002) 291–294.
- 2- Saima, H., Jan, A., Marwat, K.B., Khani M.A., Phytochemistry and medicinal properties of *Amni visnaga* (*apiaceae*), Pak. J. Bot., 46(3) (2014) 861-867.

OP49- Optimization Of Solid-Phase Extraction By Experimental Design Methodology For Separation Of Sterol And Tocopherol In Sunflower Oil Deodorizer Distillate Using Zeolite As A Novel Sorbent

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Zeolites are crystalline solid structures made of silicon, aluminum and oxygen that form a framework with cavities and channels inside where cations, water and/or small molecules may reside. They are often also referred to as molecular sieves. The properties of the porous materials depend both on the pore structures and the chemistry of the framework. The continuously increasing demands for materials with highly specific chemical and physical properties as zeolites have inspired scientists to make new porous materials with unique structures [1]. Solid-phase extraction (SPE) is a sample preparation process by which compounds that are dissolved or suspended in a liquid mixture are separated from other compounds in the mixture according to their physical and chemical properties [2].

In this study, a novel sorbent was synthesized and characterized by SEM, XRD, FTIR and DLS. The application of this sorbent was made in separation of sterol and tocopherol in SuDOD. Some parameters including eluent concentration, eluent volume, flow rate and amount of sorbent which are important factors in desorption process were optimized by using a three-level three variables Box-Behnken experimental design.

The main novelty of this work is designing SPE cartridges with dealuminated zeolites (ZSM-5 and A type) which have three different Si/Al ratios ranging from 0.8 to 1650. Effects of dealumination conditions on Si/Al ratios were investigated using different parameters such as type of catalyst (HCl, and KOH), temperature (80, 110, and 150°C) concentration of the catalysts (0.5 M, 1.0 M, 2.0 M, and 4.0 M) and treatment time (6, 15, and 24 hours). Particle size of materials were measured between 59 and 119 nm by DLS. After SPE packing optimization, cartridges were applied to SuDOD for separation and isolation of sterols and tocopherols.

This study has been supported by TUBITAK grants ARDEB-215M279.

Keywords: Solid phase extraction, zeolite, experimental design, sterol and tocopherol

References:

1. <http://asdn.net/asdn/chemistry/zeolites>
2. Plotka-Wasyłka, Justyna, et al. "Modern trends in solid phase extraction: new sorbent media." TrAC Trends in Analytical Chemistry 77 (2016): 23-43.

POSTER PRESENTATION (PP)

PP1- Quantitative Determination of Di Hydroxy Benzoic Acid Isomers Formed by Attack of Hydroxyl Radicals onto Salicylate Probe in the Presence of Humic Acid

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Photodegradation of Natural Organic Matters (NOMs) results in formation of hydroxyl radicals. To determine the concentration of hydroxyl radicals, many probes such as benzene, nitrobenzene, 4-nitrophenol, benzoic acid, salicylate, and dimethylsulfoxide are used. It has been known that salicylate probe has advantage because of having high second order rate constant, sensitivity, and having stable hydroxylated products. DHBA isomers (2,3-dihydroxybenzoate, 2,4- dihydroxybenzoate and 2,5- dihydroxybenzoate isomers) and/or catechol occur as a result of attack of hydroxyl radicals onto salicylate probe (1).

In this study, the sample of humic acid solution (as NOM) was exposed to UV radiation in the presence of salicylate probe, and the species occurred as a result of hydroxylation were determined by using HPLC Method. No peak was observed at 275 nm which means catechol species did not occur under the attack of hydroxyl radicals onto salicylate probe. On the other hand, two peaks appeared at 303 nm. The spectra recorded by the UV–Vis detector of the HPLC instrument showed that the extracted samples consist of two components: One of them is 2,3-DHBA ($\lambda_{\text{max}}=317$ nm) and other is 2,5-DHBA ($\lambda_{\text{max}}=330$ nm).

Keywords: radical, photohydroxylation, HPLC, DHBA isomers

References:

1. Ghiselli A. (1998) Methods in Molecular Biology, in: Free Radical and Antioxidant Protocols, vol. 108, Humana Press.

PP2- Filter-Furnace Atomizer with Carbon Thread Collector at The Direct Electrothermal Atomic Absorption Spectrometry Determination Of Copper In Mineral Drinking and Medical Waters

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Electrothermal atomic absorption spectrophotometry (ET AAS) is one of the most effective method, which advised by International Standards for controlling the grade of concentration of varying microelements including Cu in the mineral drinking and medical waters for sanatorium-and-health resort and commercial bottling. However, at the direct ET AAS determination $\leq 0,001$ mg/L of Cu in mineral waters it must be taken into account on the analysis result the interference effect from their main components, such as chlorides and sulfates. At the same time, International Standard ISO 15586:2012 "Water quality. Determination of trace elements using atomic absorption spectrometry with graphite furnace" at the direct ET AAS analysis of mineral waters with middle and high mineralization have proposition to additional researches for established of suitable operation parameters of atomizer heating.

This work presents the results on investigations of possibility and analytical characteristic for longitude heating electrothermal graphite furnace with graphite-filter (FF), carbon thread collector (CTC) and chemical modifier (CM) - ammonium dihydrophosphate ($\text{NH}_4\text{H}_2\text{PO}_4$) for the direct ET AAS determination Cu in some of mineral drinking and medical waters. At these the subject of researches were temperature effect of FF with CTC on the charring and atomization step, as well as drawback effects from main components of listed mineral waters: chlorides, sulfates, carbonates, sodium, calcium and magnesium, on Cu analytical signal value and non-spectral absorption of light.

There was established that at evaporation of Cu from a furnace wall without of CM lead to decreasing up to 2-3 times according to the results of ET AAS analysis of mineral, drinking and medical waters. The proposed technique of ET AAS with filter-furnace, carbon thread collector and $\text{NH}_4\text{H}_2\text{PO}_4$ as CM allowed to increase atomizer furnace temperature on charring step to 1000°C without significant Cu losses, to eliminate matrix effect 3,0-4,0 g/dm³ and up to 2 times to increase sensitivity of Cu determination compare with the International Standard ISO 15586:2012 method.

The subjects of analysis were natural and packed-up mineral waters with various cationic and anion composition and mineralization (M) from 0.66 to 10.89 mg/L. Limit of Cu determination (LOD) in mineral waters with proposed method -0.00075 mg/L, relative standard deviation (Sr) does not exceed 7% and time of element determination 5-7 minutes. The accuracy of analysis results checked with accordance of basic of mathematical statistic rule.

Keywords: GFAAS; Copper; interferences; mineral water.

PP3- Urea and thiourea substitute piperazine compounds for the colorimetric and spectroscopic determination of cyanide anion

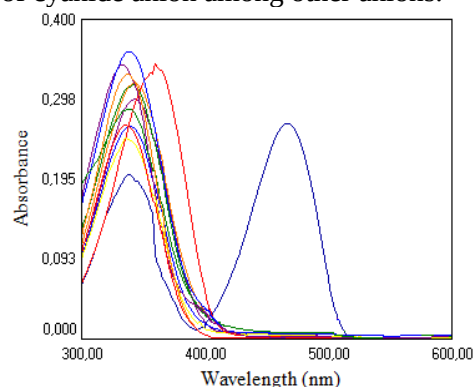
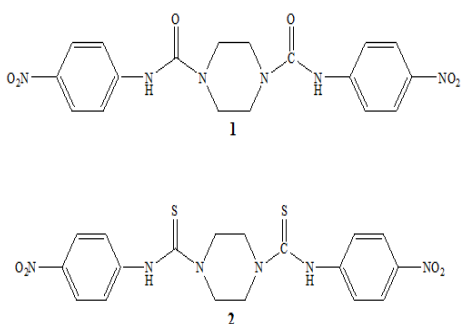
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Recently, the synthesis of functional materials for ion recognition has been intensely attractive. The ions have the important roles in biological and environmental processes [1]. Therefore, researchers have focused on the design and synthesis of host molecules having optical sensor properties for the determination of anions. Among these molecules, urea and thiourea derivatives are most frequently encountered [2]. In this study, we synthesized and characterized novel urea and thiourea compounds **1** and **2**. In addition, we used to compounds **1** and **2** for the selective naked-eye and spectrophotometric detection of cyanide anion among other anions.



Keywords: Sensor, urea, cyanide, colorimetric.

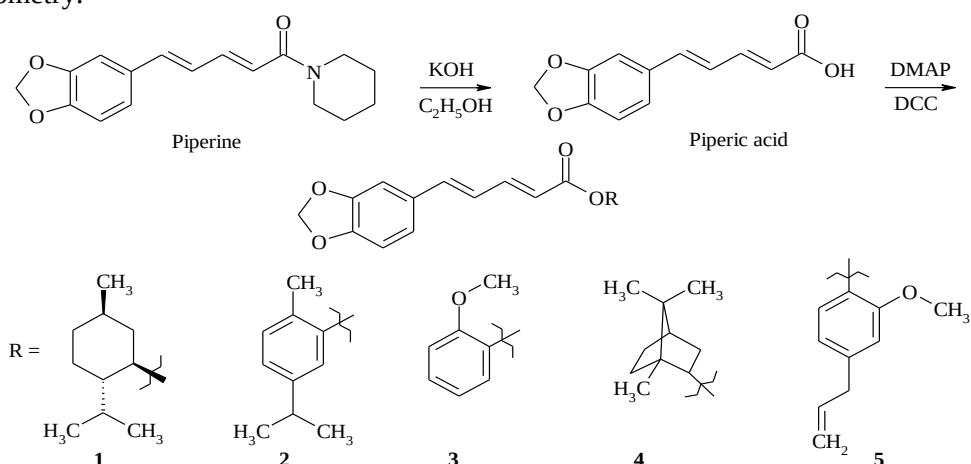
Acknowledgment: This study was produced from a part of the Master thesis of Hatice İnegöl.

References:

1. H. Ozay, M. Yildirim, O. Ozay, Synthesis and characterization of novel urea and thiourea substitute cyclotriphosphazene compounds as naked-eye sensors for F⁻ and CN⁻ anions, Turk. J. Chem. (2015) 39 777-788.
2. M. G. Murali, K. A. Vishnumurthy, Sindhu Seethamraju, Praveen C. Ramamurthy, Colorimetric anion sensor based on receptor having indole- and thiourea-binding sites, RSC Adv. (2014) 4 20567–20572.

PP4- Novel piperine derivatives as analgesic agents for transdermal delivery**Mariia Nesterkina^{A, B, *}, Iryna Kravchenko^{A, B}**^A Odessa National Polytechnic University, 65044, Ukraine, Boulevard of Shevchenko, 1^B I.I. Mechnikov Odessa National University, 65026, Ukraine, Dvorjanskaya, 2*E-mail: mashaneutron@gmail.com

The aim of the present study is synthesis of novel esters based on piperic acid and monoterpenoids with further investigation of their analgesic activity. For this purpose piperine was isolated from black pepper, converted to piperic acid via alkaline hydrolysis followed by ester synthesis using DCC/DMAP coupling method. All the compounds were fully characterized by the following analytical methods: ¹H-NMR, ¹³C NMR, IR-spectroscopy and FAB-, ESI-mass spectrometry.



In the current research, piperic acid esters were applied topically in an ointment (5% w/w) using mixture of polyethylene glycol (PEG 1500), polyethylene oxide (PEO 400) and 1,2-propylene glycol in the ratio of 4:2:3 as a base. Antinociceptive activity was evaluated by model of chemical stimulus with the use of the TRPV1 agonist capsaicin. Following the adaptation to the experimental conditions, capsaicin (6 µg/paw) solution was injected subcutaneously under the skin of the dorsal surface of the right hindpaw. The ointments were applied to the plantar surface of the right paw by gently rubbing 10 min before antinociceptive activity evaluation. Control animals received only a corresponding amount of the ointment base. Animals were observed individually for 5 min, after capsaicin administration. The amount of time that mice spent licking their injected paws (reaction time in seconds) was recorded and considered as an index of pain. As an example, below we present the results corresponding to analgesic action of menthyl ester of piperic acid (compound **1**) in comparison with the initial acid and piperine.

The application of ointment base followed by the intraplantar injection of capsaicin elicited a nociceptive paw licking response in experimental animals with duration of 86 ± 2.9 s, while for piperic acid this value was 51 ± 7.8 s. The maximum analgesia after transdermal delivery was defined for piperine (30 ± 0.3 s) and compound **1** (25 ± 5.2 s). However, the potential utility of esters **1–5** as analgesics with prolonged action is also a subject of our current research since these derivatives undergo hydrolysis with gradual release of initial terpenoids and piperic acid. Herein, we propose competitive binding between terpenoid esters of piperic acid and TRPV1 agonist capsaicin as an explanation of the significant analgesic effect of piperine derivatives.

Keywords: piperic acid esters, analgesic activity, transdermal delivery

PP5- Determination of Pb and Cd in Beauty Blush Creams

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Beauty blush (BB) creams are one of the most popular cosmetic products, nowadays. They are not only lighter than foundation and colorful than skin creams but also they offer to neutralize skin tone, correct imperfections, moisturize, protect your skin from UV radiation, and correct your skin. BB creams are basically a tinted moisturizer, anti-aging and skin care cream in one product. Various undesirable components like heavy metals end up in the final product through the raw materials used in the preparation procedure. Acceptable heavy metal levels in cosmetic products according to the cosmetic regulations are declared as 5 ppm As, 5 ppm Cd, 1 ppm Hg, 20 ppm Pb, 10 ppm Sb (1). An exposure to these metals above the limits can cause health problems including allergic reactions, cancer, and multiple system diseases (2).

In this study the levels of Pb and Cd in different brands of BB creams marketed in Turkey were investigated. Samples were prepared by wet digestion using some acids and/or acid combinations, and lyophilization (freeze drying), then analyzed by using Flame Atomic Absorption Spectroscopy (FAAS) via standard addition calibration. The obtained results under optimum experimental conditions will be compared with the cosmetic regulation and their statistical evaluation will be given.

Keywords: BB Creams, heavy metals, cosmetic, AAS

References:

1. Turkish Medicines And Medical Devices Agency. "Turkish Medicines And Medical Devices Agency Guideline on Heavy Metal Impurities in Cosmetics." [ONLINE] Available at: <https://www.titck.gov.tr/PortalAdmin/Uploads/UnitPageAttachment/AOZpWpNn.pdf>. [Last updated 10 May 2016].
2. Çağlar, A. B., Saral, S., "Kozmetolojide Toksikite Sorunu" Turk J Dermatol, (2014) 4:248-51.

PP6- Use of Chemometric Approach for Analysis of Binary Mixture of Diesel Biodiesel and Mineral Oils with FTIR-ATR

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Because of the great variation in charges, increased fuel prices and regular tax payments, liquids fuels are probably one of the most counterfeited products. It is clear that consumption of fraud mineral oil for fuel leads to vehicle fires, explosions, accidents and fatality due to mineral oil being flammable. According to the data published in 2010, in Turkey 44.873 tone residual oil was gathered and the rest 205.127 tone was not recorded (1). As far as Petroleum Industry Association 2015 Annual Report is concerned there is a big gap noticeable between the quantity of mineral oil in Turkey supplied to the market and amount consumed. This reflects that large amount of mineral oil could have been converted to adulterated fuel.

Present study deals with the chemometric approach for binary mixture of diesel/biodiesel and mineral oil and partial least square (PLS) calibration method was used as a tool for developing calibration model using FTIR spectra data. Initial studies have been conducted to construct a set of calibration and validation sets and then, the sets have been analyzed by using FTIR-ATR. Hence, the obtained calibration model was validated with spectra data of validation sets. In order to assess the performance of the models was established, Root Mean Square Error of Cross Validation (RMSCV), calculation of predictive error of the complete data system, was carried out. To assess the complete data set, Minitab software was employed.

Keywords: Mineral Oil, Diesel, Chemometrics, Partial Least Square (PLS)

References: 1. Petrol Sanayi Derneği (2011) Atık Yağların Yönetimi Projesi 2011 Yılı Faaliyet Raporu

PP7- Computurized System Validation in Accredited Laboratory

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Computerized system such as HPLC are widely used during development and manufacturing of drugs and medical devices. Proper functioning and performance of software and computer systems play a major role in obtaining consistency, reliability and accuracy of data. Therefore, computer system validation (CSV) should be part of regulated laboratory practice.

A computerized system consist of the hardware, software, and network components, together with the controlled function and associated with documentation. Computerized system validation plan consist of; what activities are required, who is responsible how they will be performed and, what their output will be, what the requirements are for acceptance, how compliance will be maintained for the lifetime of the system. Computer system validation is the process of documenting that a computer system meets a set of defined system requirements.

In this study type of validation documentation of computerized systems will be discussed.

Keywords: Computerized system, validation, laboratory

References:

1. GAMP 5, A risk-based approach to compliant GxP Computerized Systems (2008) 21
2. Guidance for industry: Computerised system validation, drug office department of health (2013) 8

PP8- Operational Qualification of HPLC-UV system regulated by Good Laboratory Practice

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High Quality and reliable analytical data is grounded on instrument qualification, method validation and training of analyst. Operational qualification is the process that an instrument will function according to its operational qualification. This qualification verifies that the HPLC system complies with requirements as specified. In HPLC system it is recommended to perform system tests including: leak test, baseline test, temperature test and detector test. Wavelength accuracy is performed by using holmium oxide and caffeine. Benzene and anthracene may be used for performance qualification of diode array detector.

In this study, HPLC systems operational qualification parameters, acceptance criteria and actual results will be discussed and reported in detail.

Keywords: operational qualification, HPLC-UV, reliable data

References:

1. Qualification of Equipment, Annex 1: Qualification of HPLC equipment. PA/PH/OMCL (11) 04 CORR, 2015

PP9- Simple and sensitive determination of Dapagliflozin by first order derivative spectrophotometric method in pharmaceutical preparations

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Dapagliflozin(DAP), a selective inhibitor of renal sodium glucose co-transporter 2, is under development for the treatment of type 2 diabetes mellitus. [1,2]. A simple method for determination of DAP in pharmaceutical dosage forms was proposed and validated using UV spectrophotometry. Firstly, Zero-Order spectrophotometric method, the solutions of the active material were prepared at varying concentrations in the appropriate solvent and the maximum absorbed wavelength of these solutions was selected to be 224.0 nm for the estimation. The determinations were performed in methanol. Also, the second method, the first order derivative spectrophotometric method was used to measure the first derivative amplitude of the first derivative spectra. The first derivative amplitude at 231.7 nm was used to show the analytical performances. The analytical working range of proposed method was calculated in range of 5.0-25.0 $\mu\text{g mL}^{-1}$ and the related correlation coefficient was calculated as 0.9998. The described method was successfully applied to pharmaceutical dosage forms containing DAP. The obtained recovery value was 99.99%. The analytical performance of the proposed spectrophotometric method is compared with the results of the fully-validated high performance liquid chromatographic (HPLC) method. The proposed simple, rapid, selective and sensitive spectrophotometric approach could be a promising analytical tool for the quantitative determination of DAP in pharmaceutical or clinical samples.

Keywords: Dapagliflozin; UV spectrophotometry; HPLC;

References

- [1] Sreeneeranj Kasichayanula, Xiaoni Liu, Weijiang Zhang, Marc Pfister, Frank P. LaCreta, David W. Boulton and Sreeneeranj Kasichayanula and Sreeneeranj Kasichayanula, *Clinical Therapeutics*, 33(11), 1798–1808,2011.
- [2] Osaki, Aya; Okada, Shuichi; Saito, Tsugumichi; Yamada, Eijiro; Ono, Kumeo; Nijima, Yawara; Hoshi, Hiroto; Yamada, Masanobu, *Journal of diabetes investigation* 7(5),751-754,2016
- [3] A. Dhir, *DrugToday* 49 (2013) 781–790.
- [4] L.Liu, N. Cao, X.Ma, K. Xiong, L. Sun, Q.Zou ve L. Yao, *Journal of Pharmaceutical and Biomedical Analysis*, 117 (2016) 325–332.

PP10- A Novel Microplate-Based Method for The Estimation Glutathione Peroxidase Activity in Tissue Homogenats

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Glutathione peroxidase (GSH-Px) enzymes belong to a family of selenoproteins whose function is to catalyze the reduction of H₂O₂, organic hydroperoxides of polyunsaturated fatty acids, and cumene hydroperoxides to water and the corresponding alcohols using the reducing power of GSH [1]. The major cellular GSH-Px is expressed in all tissues and contributes to most of the GSH-Px activity present in kidney, liver, and erythrocytes. GSH-Px enzyme is one of the major enzyme in the antioxidative defense mechanism present in the cells. GSH-Px enzyme with its main biological role is to protect the organism from oxidative damage by free radicals is a key indicator of oxidative stress.

In this study a novel and simple microplate-based method was developed, using the cupric neocuproine complex (Cu(Nc)₂²⁺) as a chromogenic oxidizing reagent, for the assessment of GSH-Px activity of biological samples for the first time [2]. The proposed assay was standardized by adapting to the microplate reader in terms of consistency of the measurements and reaction rate in comparison with a manuel spectrophotometric cupric ion reducing antioxidant capacity (CUPRAC) method. The GSH-Px enzyme activity measurement, the GSH-Px catalyzed oxidation of reduced GSH gives rise to oxidized form of GSH (GSSG). The recommended method was based on the reduction of Cu(Nc)₂²⁺ to highly colored Cu(I)-neocuproine complex (Cu(Nc)₂⁺) by the unconsumed GSH, and measurement spectrophotometrically at 450 nm, the difference being correlated to GSH-Px activity of the analytes. Under the optimum conditions, a linear calibration graph was obtained in the range of 18.5-92.5 µM of GSH with limit of detection (LOD) of 1.03 µM. Application of developed method to tissue homogenates (n = 9) provided GSH-Px activity values in agreement with those of the reference GSH-Px-5,5'-dithio-bis(2-nitrobenzoic acid) (DTNB) method. The CUPRAC method was implemented in a microformat (96 well plates) and the reaction time of original CUPRAC method was significantly shortened from 30 to 4 min. The developed method is a useful tool for investigating the role of GSH-Px enzyme in clinical samples and is alternative to limited number of microplate methods in literature.

Keywords: GSH-Px activity, microplate based method, modified CUPRAC method

References:

1. Knapen, M.F.C.M., Zusterzeel, P.L.M., Peters, W.H.M., Steegers, E.A.P., Glutathione and glutathione-related enzymes in reproduction A review, Eur. J. Obstet. Gynecol. Reprod. Biol. (1999) 82 171-184.
2. Melek Ugar, Ayşe Nur Tufan, Mehmet Altun, Kubilay Güçlü, Mustafa Özyürek, Glutathione Peroxidase Activity of Biological Samples Using A Novel Microplate-Based Method, Curr. Anal. Chem. (2018) 14 in pressed.

PP11- Determination the Radical Scavenging and Antioxidant Activity of β -Carotene within β -Cyclodextrin Complexes in Aqueous Medium

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The encapsulation with oligosaccharides such as cyclodextrins (CDs) can be applied as a convenient technique to enhance the solubility, bioavailability and stability of hydrophobic guest molecules such as β -Carotene (BC). Due to the polarity of hydrophilic exterior walls, CDs are able to generate inclusion complexes with hydrophobic guest molecules incorporated into the inner cavity by displacing water in aqueous medium.

Carotenoids are an important class of bioactive compounds mostly abundant in yellow, orange, and red-colored fruits and vegetables, such as apricot, carrot, pepper, tomato and dark-green leafy vegetables, such as spinach and kale. In this class, β -carotene is a naturally occurring orange-colored carbon- hydrogen carotenoid in our diet, which has antioxidant, antimutagenic, anticardiovascular, antiinflammatory, and anticancer activities [1]. The hydrophobic property of BC limits its effective use as an antioxidant in many applications. In order to evaluate the antioxidant capacity/activity of BC in aqueous media, we investigated the inclusion complexes of BC with methyl- β -cyclodextrin (Me- β -CD), 2-hydroxypropyl- β -cyclodextrin (HP- β -CD) and 2-hydroxyethyl- β -cyclodextrin (HE- β -CD) that enhance water solubility and chemical stability [2]. For antioxidant activity assessment, cupric ion-reducing antioxidant capacity (CUPRAC), ABTS-persulfate, peroxy and hydroxyl radical scavenging assays were applied. CD-complexed BC exhibited less effective antioxidant and radical scavenging activity than free BC dissolved in acetone. β -Carotene showed the highest trolox equivalent antioxidant capacity (TEAC) in the presence of HE- β -CD (1.49), and the lowest with Me- β -CD (1.02), probably due to the deeper and more hydrophobic cavity of the latter. Fourier-transform infrared spectroscopy (FT-IR) technique was applied to characterize solid inclusion complexes. The total antioxidant capacities (mg trolox g⁻¹ sample) of the complexed carrot extract and apricot extract with Me- β -CD were evaluated using the CUPRAC and ABTS methods. The present study also demonstrates the possibility for BC to be protected from oxidative degradation by complexation or encapsulation to the Me- β -CD cavity; this finding may be important to estimate the shelf-life of BC-containing foodstuffs.

Keywords: β -Carotene, β -cyclodextrin, inclusion complexes, antioxidant capacity

References:

1. S. T. Mayne, D. R. Janerich, P. Greenwald, S. Chorost, C. Tucci, M. B. Zaman, M. R. Melamed, M. Kiely, and M. F. McKneally, Dietary Beta Carotene and Lung Cancer Risk in U.S. Nonsmokers, J. Natl. Cancer Inst. (1994) 86 33-38.
2. S. E. Çelik, B. Bekdeşer, A. N. Tufan and R. Apak, Modified Radical Scavenging and Antioxidant Activity Measurement of β -Carotene with β -Cyclodextrins Complexation in Aqueous Medium, Anal. Sci. (2017) 33 299-305.

PP12- Seasonal Changes in concentrations of Heavy Metals in Tap Water from the City Center of Canakkale, Turkey

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Environmental problems are very important because of they affect large areas and communities from all over the world. Industry establishments which discharge heavy metals are the most important establishments which pollute the environment [1]. Heavy metals which have various toxic effects even if they are a low concentration in water, cause to disease by damaging to human health and may even lead to death. So, it is very important to determine these heavy metals.

In this study, tap water samples were seasonally collected from five different sampling regions between May 2012 and February 2013 in Canakkale, Turkey. Cd, Cr, Cu, Fe, Mn, Ni, Pb and Zn heavy metals were analyzed by an Inductively Coupled Plasma and Optical Emission Spectrophotometer (ICP-OES). The results of heavy metals concentrations were discussed comparatively with the drinking water standards of the European Union (EU), World Health Organization (WHO), Environmental Protection Agency (EPA), and Turkish Standards Institute (TSE 266) [2]. While concentrations of biotoxic metals Cr, Cd, Ni, and Pb were under undetectable limits, levels of bioactive metals Cu, Fe, Mn, and Zn varied between 0.000 and 0.003 ppm, 0.000 and 0.173 ppm, 0.000 and 0.009 ppm, 0.000 and 2.343 ppm, respectively. As a result, the public tap waters of Canakkale (Turkey) were found healthy in terms of heavy metals at studied period. Two-way variance analysis (ANOVA) was used to test the significance of differences between sampling points and seasons (two factors) at 0.01 and 0.05 levels in view of heavy metal concentrations.

Keywords: Turkey, Canakkale, Tap water, Heavy metals.

References:

1. Kaçar H. (2013). Çanakkale şehir merkezi şebeke sularındaki bazı ağır metal ve anyon derişimlerinin fiziko-kimyasal parametrelerle birlikte mevsimsel olarak belirlenmesi. Çanakkale Onsekiz Mart Üniversitesi, Fen Bilimleri Enstitüsü, Yüksek Lisans Tezi Çanakkale.
2. Tonguc Yayintas, O., Yilmaz, S., Turkoglu, M. & Dilgin, Y. (2007). Determination of heavy metal pollution with environmental physicochemical parameters in waste water of Kocabas Stream (Biga, Canakkale, TURKEY) by ICP-AES. Environ. Monit. Assess., 127: 389-397.

PP13- Seasonal Variations of Tap Water Quality Parameters in the City Center of Canakkale, Turkey

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Water pollution is so important environmental problems that affect living organism (people, animal and plant etc.) [1].

Therefore, in this study, in order to understand whether the tap waters in the city center of Canakkale (Turkey) are healthy or not, physico-chemicals such as temperature, pH, and conductivity and dissolved oxygen (DO), and anions such as sulfate, nitrate, nitrite, cyanide, and bio-chemicals such as chemical oxygen demand (COD) and biochemical oxygen demand (BOD) were determined [2]. Tap water samples were seasonally collected from five different sampling regions between May 2012 and February 2013 in city center of Canakkale (Turkey) and analyzed suitable techniques. The results of physico-chemicals and biochemical were discussed comparatively with the drinking water standards of the European Union (EU), World Health Organization (WHO), Environmental Protection Agency (EPA), and Turkish Standards Institute (TSE 266) [2]. As a result, the public tap waters of Canakkale (Turkey) were found healthy in terms of physico-chemicals and biochemical at studied period. However, the average pH value in the tap waters was 6.85 ± 0.12 indicate that Canakkale tap waters can be considered a little acidic. Two-way variance analysis (ANOVA) was used to test the significance of differences between sampling points and seasons (two factors) at 0.01 and 0.05 levels in view of relevant tap water quality parameters.

Keywords: Turkey, Canakkale, tap water, physico-chemicals, bio-chemicals.

References:

1. Kaçar H. (2013). Çanakkale şehir merkezi şebeke sularındaki bazı ağır metal ve anyon derişimlerinin fiziko-kimyasal parametrelerle birlikte mevsimsel olarak belirlenmesi. Çanakkale Onsekiz Mart Üniversitesi, Fen Bilimleri Enstitüsü, Yüksek Lisans Tezi Çanakkale.
2. Anonymous (2012). Standard Methods for the Examination of Water and Wastewater (22nd Edition). APHA, AWWA, WEF. 5220 B. 5.06-5.21.

PP14- Investigation of Correlation between Nickel and Imazamox in Soil Samples in Kırklareli and Kavaklı Regions

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Pesticides are chemical substances that are meant to control pests, including weeds with especially herbicide, insecticides nematocide, molluscicide, piscicide, avicide, rodenticide, bactericide, insect repellent, animal repellent, antimicrobial, etc. The unconsciously used herbicides are problematic due to the chemical properties of agricultural soils and their contaminations with the metals found in the soil.

This study was aimed to investigate the correlation between the residue level amounts of nickel and Imazamox, which was investigated in soils with two different pH characteristics in Kırklareli and Kavaklı regions. The analyses were completed by flame atomic absorption spectrometer (FAAS) for Ni and by gas chromatography-mass spectrometer (GC-MS) for imazamox. According to the Pearson's Correlation Coefficient evaluation (PCCE), there is a high level correlation between Imazamox and Ni in soil samples. These correlations of $r_{\text{imz-Ni}}$ were found to be 0.95 and 0.93 at soil samples in Kırklareli and Kavaklı regions, respectively.

Keywords: Soil, Nickel, Imazamox, GC-MS.

Acknowledgements: The authors thank to the Kırklareli University Research Foundation for its financial supports (KLUBAP/121).

PP15- Optimization of Endosulfan Pesticide in Water samples by Gas Chromatography-Mass Spectroscopy

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Contamination of water resources by pesticide residues is one of the major challenges for the preservation and sustainability of the environment [1]. Pollution has been very damaging to aquatic ecosystems, and may consist of agricultural, urban, and industrial wastes containing contaminants that have proven to be very damaging to aquatic habitats and species [2]. Endosulfan is a substance found in the insecticide group from pesticides used in agricultural applications.

The aim of this study was to investigate the level of Endosulfan in water samples from the Ergene and Meriç Rivers as well as Yenikarpuzlu pond. The concentration of Endosulfan pesticide was determined by using gas chromatography-mass spectrometry (GC-MS) after solid phase extraction. The LOD, LOQ and RSD% were $0.79 \mu\text{g L}^{-1}$, $2.64 \mu\text{g L}^{-1}$, $7.39 \mu\text{g L}^{-1}$ and $< 10\%$, respectively. Recoveries of the spiked Endosulfan in water samples ranged from 87.0% to 107%, respectively. Using the optimized method to water samples, Endosulfan content was found in range from ND to $9.35 \mu\text{g L}^{-1}$.

Keywords: Endosulfan, GC-MS, Water, Pesticide.

References

1. S. H. G. Brondi, S.H.G., de Macedo, A.N., Vicente, G.H.L., Nogueira, A.R.A., Evaluation of the QuEChERS Method and Gas Chromatography–Mass Spectrometry for the Analysis Pesticide Residues in Water and Sediment, Bull Environ Contam Toxicol (2011) 86:18–22.
2. EPA – Environmental Protection Agency (2009) Aquatic biodiversity. Available <http://www.epa.gov/bioiweb1/aquatic/pollution.html>. Accessed 10 July 2010.

Acknowledgements: The authors thank to the Kırklareli University Research Foundation for its financial supports (KLUBAP/120).

PP16- Assessment of trace element levels in wastewater of two industrial zones; Ergene Basin and Gaziantep

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Due to rapid industrialization and increase in agricultural and industrial activities, soil and water resources are dangerously polluted, particularly in recent years. Further, household-industrial wastes, emissions of industrial gases and uncontrolled use of agrochemicals damage also to agricultural areas and water resources, which threatens the ecological environment.

The aim of this study is to assessment of trace element and heavy metal pollution in wastewater of two industrial zones in Turkey; Ergene Basin and Gaziantep Plain. The surface wastewater samples collected from Ergene River and Çorlu Stream in Ergene Basin and Nizip and Bağirsak Streams in Gaziantep Plain, in two periods; (1) May-June 2016, (2) August-October 2016. Microwave digestion method was carried out to preparation of wastewater samples. For this purpose, a portion of 10 ml of wastewater sample was digested using 5 ml of nitric and hydrochloric acids mixture (3 ml +2 ml). After the mixture of concentrated acid was evaporated and the appropriate dilutions were made, the quantitative analysis of the elements was carried out by ICP-MS.

The accuracy of the method was verified with wastewater SRM samples. The results were compared with the maximum trace element concentration values established by different countries and standard organizations for the wastewater used for irrigation purposes. In the Gaziantep plain, the concentrations of elements in wastewater was determined to be Fe (10,7 ppm)>Al (8,53 ppm)>Zn (0,64 ppm)> B (0,50 ppm)>Mn (0,21 ppm)> Ni (0,113 ppm)> Cr (69,3 ppb)>V (49,6 ppb)>Pb (43,2 ppb)> Cu (36,8 ppb)> Sb (11,4 ppb)> Co (10,6 ppb) >Se (7,68 ppb)> As (5,66 ppb)>Ag (3,11 ppb)>Cd (1,93 ppb), respectively. In the Ergene Basin, the concentrations of elements in wastewater samples were determined as Fe (8,02 ppm)>Al (5,48 ppm)>Zn (0,956 ppm)> B (0,519 ppm)>Cr (324 ppb)>Mn (0,246 ppm)>Cu (79,3 ppb)> Ni (34,5 ppb)> Pb (22,1 ppb)>V (19,3 ppb)> Se (13,0 ppb)> Co (7,08 ppb) >As (5,17 ppb)> Sb (4,19 ppb)> Ag (1,90 ppb)>Cd (0,913 ppb), respectively.

Keywords: Contamination, heavy metal pollution, industrial wastewater, trace element

Acknowledgements: The authors thank the TUBITAK Research Foundation for their support: TUBITAK 1001, No: 215O538

PP17- Evaluation of metal accumulation in agricultural soils and wheat grown in two regions under the effect of industrial wastewaters

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Gaziantep Plain and Ergene Basin are located in high industrial area of Turkey. Further, these regions include the important productive agricultural land of Turkey. Wheat, which is one of the main foodstuffs, is grown widely in these fields. For this reason, heavy metal analyzes in the soil and wheat grown in these areas are also of great importance.

This study was undertaken to assessment of trace element and heavy metal pollution in agricultural soils and wheat grown under the effect of industrial wastewaters of two zones of Turkey: Ergene Basin and Gaziantep Plain. The soil and plant samples were simultaneous collected in May and June 2016. Microwave digestion method was carried out to preparation of soils and wheat samples. For this purpose, a portion of 0.500 g of wheat flour sample was digested using 8 ml of nitric and 2 ml hydrogen peroxide mixtures. A portion of 0.500 g of soil sample was digested in the mixture of 9 ml of nitric and 3 ml hydrogen chloride acids. After evaporation of this mixture and the appropriate dilutions, the quantitative analysis of elements (Zn, Ni, Cd, Pb, Cu, Cr, Fe and Mn) were carried out by ICP-MS. Some analyses were also carried out by AAS.

The accuracy of the results was confirmed using SRM. In the Gaziantep Plain, the concentrations of metals in soils of wheat samples grown in were found as Fe> Mn> Ni> Zn> Cr> Cu> Co> Pb> Cd, respectively. In the Ergene River Basin, the concentrations of metals in soils of wheat samples grown in were determined as Fe> Mn> Cr> Zn> Ni> Pb> Cu> Co> Cd, respectively. The results were interpreted based on reference values such as the Turkey Soil Pollution Regulation, Indian Standards, NEPAC, ECC, SEPA. The obtained all results were found lower than the acceptable limit values according to the reference values.

Keywords: Agricultural soils, heavy metal pollution, wastewaters irrigation, wheat

Acknowledgements: The authors thank the TUBITAK Research Foundation for their support: TUBITAK 1001, No: 215O538

PP18- Drainage Geochemistry of the Uluova Basin, Elazig, Turkey

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Sediments have been recognized as both the main source and sink for heavy metals in aquatic ecosystems, where sediment quality is an important indicator of water pollution^{1,2}.

The purpose of this study is to characterize the geochemistry and the levels of heavy metals in sediment samples collected from the Uluova drainage basin. Besides, outcomes of this work are expected to improve the current understanding related to the content of the heavy metals in groundwater of the Uluova Basin aquifer system. Stream sediment samples ($n = 28$) taken from the Uluova drainage basin were analyzed by AAS. In addition, the sediment samples taken from six different locations were analyzed by XRD technique to yield information about the minerals. For the same sediment samples, scanning electron microscopy (SEM) coupled with energy dispersive spectroscopy (EDS) was used for identification of trace elements. The median values for As, Ni, and Cd in the stream sediment samples were higher than the world average crustal composition, while the median values for Fe, Mn, Al, Zn, Co, Cu, and Cr were less than the world average crustal composition. The most dominant minerals in Uluova drainage basin are quartz, calcite, cristobalite, cordierite, and feldspar group minerals, as well as clay and mica group minerals.

Keywords: Uluova, XRD, SEM, Heavy metal

References:

1. Larsen, B.; Jensen, A. Evaluation of the sensitivity of sediment monitoring stationary in pollution monitoring. Mar. Pollut. Bull., (1989) 20: 556–560.
2. Lima, M.C.; Giacomelli, M.B.O.; Stüpp, V.; Roberge, F.D.; Barrera, P.B. Especiação de cobre e chumbo em sedimento do Rio Tubarao (SC) pelo Método Tessier. Química Nova, (2001) 24(6): 734–742.

PP19- Preconcentrations of Al(III), Ga(III) and In(III) after separation on dimethyl chlorosilan eaerosil

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One of the tasks of analytical control of industrial wastewater is the determination of elements with similar properties in their mixture. Extraction and sorption procedures are generally used for determination of the analog elements. For the fine separation of analog elements, the idea of combining the advantages of extraction and sorption processes in solid-phase and membrane extraction, reversed-phase and extraction chromatography, which are special cases of the distribution of matter between the hydrophilic and hydrophobic phases, is promising. In these methods, organosilica impregnated with organic solvents is used as the hydrophobic phase. For the separation and concentrating of the analogue elements Al^{3+} , Ga^{3+} , In^{3+} , in their joint presence, it is proposed to use an organized system (OS) of dimethyl chlorosilan eaerosil (DMCSA) - polar organic solvent (PS) – {OS (DMCSA – PS)}.

In this paper, the advantages of {OS (DMCSA – PS)} in the separation, concentrating and solid-phase-spectrophotometric determination of microquantities of the analog elements Al^{3+} , Ga^{3+} , In^{3+} are shown. It is found that a hydrophobically fixed organic solvent layer participates in the sorption-extraction processes on the surface of the organized system. This layer selectively extracts from the aqueous solution at optimal pH only neutral hydroxo forms of easily hydrolyzable elements, followed by their mass transfer to the silanol groups of the silica matrix DMCSA, where they are sorption fixed due to the formation of bridging chemical bonds. This makes it possible to predict and direct the extraction and separation of elements, taking into account their ability to hydrolyze and the difference in the acid-base properties of the ion-molecular forms of the elements, depending on the pH of the medium, and opens up the possibility of controlling the selectivity of separation of analog elements when the nature of the heterogeneous system components and the mass transfer conditions are varied. The possibility of using the OS for separating the microquantities of Ga^{3+} ($S = 96-99\%$) from Al^{3+} and In^{3+} as its neutral hydroxocomplexes at pH = 2.7 using OS (DMCSA – acetone) is shown; further separation of In^{3+} ($S = 88-99\%$) and Al^{3+} at pH = 5 using OS (DMCSA – ethanol). Combined methods for determining the microquantities of Ga^{3+} , In^{3+} after their preliminary sorption concentrating and separation were developed, followed by determination with xylenol orange in diffuse reflection spectroscopy and visual colorimetric test determination on the OS surface. Conducting a heterophase analytical reaction of complex formation of xylenol orange with fixed Ga^{3+} and In^{3+} on the OS surface makes it possible to increase its sensitivity due to the increase in the stability of surface complexes and to expand the boundaries of the determined concentrations in comparison with aqueous solutions.

Keywords: sorption, organized system, diffuse reflection spectroscopy, test-analysis.

PP20- Sorption of fluoroquinolones on aluminum oxides with different pH characteristics and silica SG 5/40

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Among antimicrobial drugs, occupying one of the main places, are fluoroquinolones - broad-spectrum antibiotics. They are used not only to treat people, but also for animals, birds, bees and plants. Some of these antibiotics are used as preservatives of perishable products (fish, meat, cheese, vegetables) and as growth stimulants. Representatives of the class of fluoroquinolones - ofloxacin and ciprofloxacin - antibacterial agents used for infectious and inflammatory diseases that included in the list of vital and essential drugs. Fluoroquinolones are almost not metabolized in the body and can be accumulated, resulting in food, primarily in meat and milk. Therefore, the issue of preliminary extraction with the subsequent determination of micro quantities of these antibiotics is topical. In recent years, sorption methods with the use of sorbents of various classes have become increasingly important for the extraction and concentration of biologically active substances.

In this paper, the sorptions of ofloxacin and ciprofloxacin on silica (SG 5/40) as oxide sorbent were studied. The adsorption features of SG 5/40 is due to the presence of silanol and siloxane groups and Al_2O_3 of various acid-base modifications on its surface: acid Al_2O_3 , neutral Al_2O_3 , basic Al_2O_3 with isoelectric points 5.0; 8.0; 9.2 respectively.

Extractions of ofloxacin and ciprofloxacin were investigated at pH range 2 - 9 in batch mode ($C_{\text{ofloxacin}} = 1 \cdot 10^{-5}$ mol/L, $C_{\text{ciprofloxacin}} = 3 \cdot 10^{-5}$ mol/L, $m(\text{SG 5/40}) = 0.1$ g, $m(\text{Al}_2\text{O}_3) = 0.2$ g, $\tau_{\text{sorption}} = 30$ min). The degree of sorption (S, %) was determined from the residual concentrations of the sorbates after equilibration by the UV/VIS at $\lambda_{\text{ofloxacin}} = 291$ nm and $\lambda_{\text{ciprofloxacin}} = 279$ nm. The dependences of $S=f(\text{pH})$ of ofloxacin and ciprofloxacin with silica and aluminum oxides are domed and with a maximum in the pH range of 6-7. In acidic and alkaline media, the reduction of extraction is due to the fact that functional groups of antibiotics and sorbents are protonated at $\text{pH} < 4$, and at $\text{pH} > 8$ they dissociate, which leads to the growth of like charges. In optimized conditions, the degree of extraction of ofloxacin by all investigated sorbents was more than 85%. Sorption extraction of ciprofloxacin using SG 5/40, acid Al_2O_3 and basic Al_2O_3 is less than 50%, and neutral Al_2O_3 - more than 85%. With the maximum extraction of ofloxacin and ciprofloxacin, the isotherm sorption form indicates the sufficient affinity of the antibiotics to the surface of these sorbents and it is of L-type. The corresponding static exchange capacities have been calculated from the sorption isotherms as.

Keywords: sorption, fluoroquinolones, silica, aluminum oxides.

PP21-Development of colorimetric test method for Cr(VI) based on sorption method

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One of the modern tasks of analytical chemistry is the development of portable analytical devices and tools for chemical analysis that can be used in off-laboratory conditions in the determination of eco-toxicants. Special interest in the field of combined sorption and colorimetric test methods are the indicator tubes (IT), which are miniaturized sorption columns, through which the analysis of environmental objects, as well as industrial process solutions and production emissions is carried out. The application of diffuse reflection and colorimetric spectroscopy methods that detect the analytical signal directly in the solid phase allows the development of highly sensitive, simple and sufficiently selective test methods.

The aim of this work is to optimize the experimental parameters of IT based on the cationite KU-2-8 with the dynamic recovery of the complex obtained. Spectrophotometric and colorimetric parameters (CP) for the development of a test procedure for the Cr(VI) determination is based on the Cr(VI) redox reaction with 1,5-diphenylcarbazide (pH 2, $\lambda_{\max} = 540$ nm) from model aqueous solutions.

The formation of the adsorption layers of the complex on the surface of the cationite was studied by varying the initial concentration of Cr(VI) ($c(\text{Cr(VI)}) = 0.02\text{-}5.00$ $\mu\text{g/mL}$), the volumetric flow rate of the sorbate solution through the sorbent layer (V , mL/min) sorbent weights (m , g), sorption column diameter (d_c , mm) and sorbent grains (d_s , mm), composition of sorbate solutions. The experimental parameters of the IT have been optimized ($V = 6$ mL/min, $m = 0.5$ g, $d_s = 0.43\text{-}0.50$ mm, $d_c = 10$ mm, $c_{\min} = 0.02$ $\mu\text{g/mL}$) and it is shown that the fractional composition of the sorbent has a lesser effect on the concentration efficiency and sorption mechanism than d_c . The optimized conditions were applied using extraction the cationite KU-2-8 in the dynamic regime of the DPC-Cr(III) complex. The concentrate was analyzed by the method of colorimetry using a spectrophotometer or desktop scanner as an analytical instrument for measuring the CP and estimating the sensitivity of the determination of Cr(VI). In the development of colorimetric scales constructed in the coordinates of the "CP of RGB channels (Cr(VI)) from $c(\text{Cr(VI)})$ ". For each of the color scales studied both the least and the brightest channels are distinguished, which vary depending on the intensity of the color of the carrier. With an increase in the concentration of the detected component accompanied by a deepening of the color, for all systems a decrease in the lightness of the color image of the R, G, B channels was observed. By mathematical processing of the corresponding curves, a number of approximating dependencies are obtained that allow: determining the metal content in the analyzed sample by registering the CP or using one of the color channels which numerical values correlate with the determined metal concentration; assess the sensitivity of the test system for rapid detection in water.

Keywords: Sorption, diffuse reflection spectroscopy, test-analysis, Chromium(VI).

PP22- Comparison of the artificial neural network and response surface methodology in the optimization efficiency of oxacillin degradation

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Nowadays, drug consumption has reached very serious extends. These drugs pollute the aquatic environment through wastewater from municipal, hospitals or industries. Water that polluted by antibiotics negatively affects the treatment of the disease and cause the formation of antibiotic resistance in bacteria. Actually, the presence of antibiotics in wastewater causes the return of them to the human body at the end of a certain cycle and increase the danger for human health. Antibiotics, especially β -lactams, has been detected in the various water sources in critical levels. Further, oxacillin, which is a narrow-spectrum β -lactam antibiotic, was also detected in the wastewaters as well as natural waters [1].

We aimed to compare the efficiency artificial neural network (ANN) and response surface methodology efficiency in the optimization of oxacillin degradation. Previously reported results of degradation experiments [1,2] were statistically compared using central composite design (CCD) and Box-Behnken design (BBD), which are two design methods of response surface methodology. The efficiency of all three methods in the estimation of the degradation rates, namely TOC removal percentages, were compared over coefficient of determination, root mean square error, and absolute average deviation values of each one. The prediction capabilities of ANN, CCD and BBD were statistically evaluated. The results showed that predicted values of ANN model were compatible with the experimental ones in both case of CCD and BBD. Mean square error values were obtained as 1.19 and 1.83 CCD and ANN, respectively, in the case of CCD, where these values were found as 1.01 and 1,65 for BBD and ANN, respectively, in the case of BBD. In addition, regression coefficient and absolute average deviation values were compared.

Keywords: Oxacillin, degradation, response surface method, artificial neural network

Acknowledgements: This work was supported by Mersin University Research Fund (Project No.: 2017-1-AP1-2207).

References:

1. E. Yabalak, S. Könen adigüzel, A. Adigüzel, R. S. Ergene, M. Tunçer, A. Gizir, Application of response surface methodology for the optimization of oxacillin degradation by subcritical water oxidation using H_2O_2 : genotoxicity and antimicrobial activity analysis of treated samplesDesalin. Water Treat. (2017) 81 186-198
2. E. Yabalak, A. Gizir, Optimisation of oxacillin degradation by subcritical water oxidation using RSM, 2. International Water and Health Congress, Congress Book, 481-482, 2017, Antalya/Turkey

PP23- Determination of Free Oleic acid by High Pressure Liquid Chromatography-Mass Spectrometer (HPLC-MS)

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Oleic acid is a well-known pharmaceutical excipient that has been widely used in several different drug forms. It avails as a potency multiplier in transdermal formulations, an emulsion agent in topical formulations, a solubility propagator for gastrointestinal tract delivery formulations, a surfactant in metered dose inhaler formulations. There are a lot of various analytical methods for determination of this widely used excipient such as high pressure liquid chromatography and gas chromatography. Although high pressure liquid chromatography with a UV detector is the most commonly used system for the determination of active pharmaceutical ingredients and excipients it is not easily applicable for fatty acids because of lacking of chromophore groups in fatty acids [1]. As a result of this absence derivatization process must be made. These derivatization processes must be made very carefully due to high temperature, harmful reactivities and also takes so much time. Moreover generally fatty acids must be in triglyceride form so that derivatization processes can be made. Another commonly used method for determination of fatty acids is gas chromatography. This technique also requires methylation, a derivatization process, prior to analysis because of the high boiling points of fatty acids. The European and United States Pharmacopeia represent alike GC methods, methylation before analysis, for identifying and quantifying oleic acid [2]. In the direction of these informations all of the methods have drawbacks, due to derivatization processes, such as low sensitivity, repeatability problems, poor resolution and long analysis time. When considered common use of oleic acid in pharmaceutical industry, it is important to develop a method to determine oleic acid easily.

The aim of this study is to develop a simple, robust, and HPLC-MS method that is suitable for routine analysis of oleic acid with in the presence of metered dose inhaler active pharmaceutical ingredients and placebo.

Keywords: oleic acid, mass spectrometer (MS), high pressure liquid chromatography (HPLC)

References:

1. Y. Tsuyama, T. Uchiro, T. Goto, Analysis of un-derivatized C12–C18 fatty acids by reversed-phase ion-pair high-performance liquid chromatography with conductivity detection, *J Chromatogr. A* (1992) 596181–184.
2. United States Pharmacopeia/National Formulary, in: *Proceedings of 36th United States Pharmacopeia Commission*, Rockville, USA, 2013, pp. 2111–2112.

PP24- Determination of phenolic acids and flavonoids in the leaf, sepal and seed parts of *Lallemantia iberica* (Bieb) Fisch. & Mey. from Şanlıurfa

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Phytochemical composition of plants from Labiate family has been attracted interest of researchers for many years. Because of their aromatherapy properties, they are consumed as spice and herbal tea [1].

In this study, *Lallemantia iberica* (Bieb) Fisch. & Mey. was picked from Şanlıurfa in Turkey and dried as herbarium sample. The leaf, seeds and sepals of the plant were pulverized and defatted with hexane. The residue of filtered hexane extract was subjected to methanol extraction using ultrasound water bath and vortex for effective extraction. Antioxidant compounds of the methanol extracts were evaluated with chromatographic and spectroscopic methods. Phenolic acids and flavonoids of the extracts were determined by HPLC-DAD analysis and total phenolic and total flavonoids of the extracts were measured using spectrophotometry.

It was determined that the leaf extract had the highest amount of flavonoids such as luteolin and apigenin and their derivatives according to HPLC-DAD analyses. The sepal extract was found rich in luteolin and apigenin derivatives while the seed extract was rich in *p*-coumaric acid and rosmarinic acid and their derivatives. It was revealed that the leaf extract had the highest total flavonoids as 62.47 mg quercetin equivalent/g extract, and sepal extract had the highest phenolic compounds with the value of 58.96 mg gallic acid equivalent/g extract by the results of spectrophotometric assays which were highly correlated with the results of HPLC-DAD analysis.

Keywords: *Lallementia iberica*, rosmarinic acid, luteolin, apigenin.

References:

1. F.A. Özdemir, M.U. Yıldırım, M.P. Kahriz, Efficient Micropropagation of Highly Economic, Medicinal and Ornamental Plant *Lallemantia iberica* (Bieb.) Fisch. and C. A. Mey. BioM Res Int. (2014) 476346-476351.

PP25- Simultaneous Column Solid Phase Extraction of Au(III) and Pd(II) by Silica Gel functionalized with [6-(2-thienyl)-2-pyridin-2-yl]methylamine for Their Flame Atomic Absorption Spectrometric Determination

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Preconcentration methods such as solid phase extraction, co-precipitation and solvent extraction are used for the determination several analytes at trace level by increasing their concentration and eliminating interferences (1,2).

In this study, a novel solid phase extractant was prepared by covalently attaching of [6-(2-thienyl)-2-pyridin-2-yl]methylamine group to silica gel surface. The prepared modified silica gel was characterized by C,H,N elemental analysis and FTIR spectroscopy and then, used for simultaneous solid phase extraction of Au(III) and Pd(II) ions for their flame atomic absorption spectrometric determination. In the column solid phase extraction experiments, various parameters such as acid concentration in the sample, sample volume and flow rate, type and volume of eluent, effect of matrix ions were examined. Then the analytical performance of the developed method was calculated. The developed method was used to determine the concentrations of Au (III) and Pd (II) ions in environmental water samples. The Au(III) and Pd(II) adsorption capacity of the synthesized adsorbent was also calculated. The findings will be presented in the congress.

Keywords: Solid phase extraction, silica gel, flame atomic absorption spectrometry

References:

1. S. Sivrikaya, B. Karşı, M. Imamoglu. On-line preconcentration of Pd (II) using polyamine silica gel filled mini column for flame atomic absorption spectrometric determination. Int. J. Environ. Res., 11(5-6), 579-590.
2. M. Sayın, M.Can, M. Imamoglu, M. Arslan, Highly Efficient Adsorption of Rh (III) from Chloride Containing Solutions by Triazine Polyamine Polymer. Water, Air, Soil Pollut. (2017) 228(3), 100.

PP26-Switchable Solvent Based Liquid Phase Microextraction Method for Fe (III) in Environmental Samples Prior to Furnace Atomic Absorption Spectrometric Determination

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A novel method was developed for determination of trace amounts of Fe (III) in environmental samples. Switchable solvent based liquid phase microextraction (SS-LPME) was used to preconcentrate the Fe (III) ion. 2-(5-bromo-2 pyridylazo)-5-(diethylamino) phenol (5-Br-PADAP) were complexed with Fe (III) ions at pH 3 in various samples and then extracted with switchable solvent. N, N dimethyl-n-octylamine was used as a switchable solvent which a non-ionic liquid converts to an ionic liquid under the presence of carbon dioxide. After the extraction, phases' separation is provided by the addition of sodium hydroxide which produces a change on the ionisation state of the N, N dimethyl-n-octylamine [1-2]. The main factors influencing the extraction method, including pH, volume of switchable solvent, concentration of ligand, amount of sodium hydroxide and matrix effect were studied. Under the optimum conditions, the detection limit of Fe (III) and an enrichment factor was determined. Accuracy of the developed procedure was evaluated by the analysis of certified reference materials with satisfactory results. The developed procedure was then successfully applied to environmental samples.

Keywords: Preconcentration, switchable solvent, microextraction

References:

1. Erkan Yilmaz, Mustafa Soylak, Switchable Polarity Solvent for Liquid Phase Microextraction of Cd(II) as Pyrrolidinedithiocarbamate Chelates from Environmental Samples, Anal. Chim. Acta (2015) 886 75-82.
2. Guillermo Lasarte-Aragones, Rafael Lucena, Soledad Cardenas, Miguel Valcárcel, Use of Switchable Solvents in the Microextraction Context, Talanta (2015) 131 645–649.

PP27- Determination of Capsaicine from pain patch by Gas Chromatography-Mass Spectrometry

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Capsaicine is an alkaloid, C₁₈H₂₇NO₃, derived from hot chilli pepper plants, certain capsicums that is a strong irritant to skin and mucous membranes and is used as pain relievers.

In this study, gas chromatography mass spectrometric method has been developed for determination of capsaicin in pain patch. Method has developed in %5 diphenyl %95 dimethyl polysiloxane column with high purity helium gas and 2 ml/min flow rate by using mass detector. For capsaicin analysis inlet temperature is 300 °C, transfer line temperature is 300 °C and column temperature is 250 °C. The linear range was found in between 5-50 µg/mL. For capsaicin the limit of detection was found 3.46 and the limit of quantification was found 5 µg/mL respectively. The developed method has validated and successfully applied to plaster analysis. This method is simple, reproducible and can be used safely routine analysis of capsaicin in painpatch.

Keywords: Capsicum, Capsaicine, Pain patch, Gas Chromatography-Mass Spectrometry

References:

1. Ha J, Han K, Kim K, Jeong S, Gas chromatographic analysis of capsaicin in Gochujang. Journal of AOAC International (2008) 91(2) 387-391.
2. Pena-Alvarez A, Ramirez-Maya E, Alvarado-Suarez L A. Analysis of capsaicin and dihydrocapsaicin in peppers and pepper sauces by solid phase microextraction gas chromatography-mass spectrometry. J. Chromatogr. A (2009) 1216(14): 2843-2847.

PP28- Removal of Congo Red from Aqueous Solutions Using Pomegranate Kernel as an Agricultural Waste: RSM Approach

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The biggest environmental problem is wastewaters containing organic and inorganic pollutants caused by industrial activities such as textiles, paper, leather and food sector¹. Adsorption treatment is a low cost and easy method for effective treatment of wastewater. The choice of adsorbent, which can be found in cheap and abundant quantities, is important in the adsorption process. In recent years, researchers are investigating whether the byproducts of agricultural and industrial activities can be used as low-cost adsorbents. Congo red (CR) is a dye with carcinogenic and mutagenic effect which is widely used in the industry². For this reason, removal of Congo red (CR) from wastewater with suitable procedures is of great importance.

This study introduces a novel and highly efficient biosorbent called pomegranate kernel to remove CR from aqueous solution. A Central Composite design (CCD) including three main factors (initial concentration (C_0 , mg.L⁻¹), sorbent amount (m, g) and pH) based on response surface methodology (RSM) approach was chosen to determine optimum conditions for CR removal. The experimental results shown that the CR removal efficiency ($R(\%) \pm s$) can reach to $94.67\% \pm 0.30$ (N=3) at the optimum conditions of initial CR concentration 80 mg.L⁻¹, sorbent amount 1.0909 g and pH 2. The experimental values were in reasonable agreement with the estimated values $97.58\% \pm 0.77$ (N=3) from model. As a result, it is observed that pomegranate kernel can be used as a low cost adsorbent to remove anionic dyes as Congo Red from aqueous solutions.

Keywords: Adsorption, Congo red, Pomegranate kernel, Response surface methodology

References:

1. Salleh M.A.M., Mahmoud D.K., Abdul Karim W.A.W., Idris A., Cationic and anionic dye adsorption by agricultural solid wastes: A comprehensive review, *Desalination* (2011) 280 1-13.
2. Liu S., Ding Y., Li P., Diao K., Tan X., Lei F., Zhan Y., Li Q., Huang B., Huang Z., Adsorption of the anionic dye Congo red from aqueous solution onto natural zeolites modified with *N,N*-dimethyl dehydroabietylamine oxide, *Chem. Eng. J.* (2014) 248 135-144.

PP29- Multivariate Optimization for Removal of Cu(II) and Pb(II) from Aqueous Solutions Using Synthesized Hybrid and Calcined Materials

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Hybrid materials with organic–inorganic character have been receiving attention most especially due to their unique properties and offer probability for many new applications in variable fields and industrial applications. In general, the organic polymer components of such materials have good elasticity, toughness, formability and low density, while the inorganic ceramic components are hard, stiff and thermally stable. Together, these components can produce hybrid materials which adhere well to both metallic and polymeric substrates, are chemically stable, and have good abrasion resistance¹. One of the application areas of these materials is heavy metal removal from aqueous solutions. Especially in developing countries, industrial applications of toxic heavy metals in the last few decades have increased at fields as chemical manufacturing, painting and coating, mining, extractive metallurgy, nuclear and other industries. Because toxic heavy metal pollution has resulted in serious environmental problems for soils, water and living organisms as plants, animals and humans, removal of heavy metals has become a more important and wide working subject².

Due to heavy metal removal process's characteristics are very complex, detecting the optimum values of removal conditions become significant to get the optimum removal efficiency. By way of this base, it is thought that process optimization is vital to determine the values of design parameters at which the response reaches its optimum. Response Surface Methodology (RSM) involves the multiple parameters and appertaining to reduced number of experimental trials needed to evaluate multiple parameters and their interactions; and Central Composite Design (CCD) is the most frequently used method of RSM¹.

In this study, organic–inorganic hybrid material on the basis of metal alkoxide $\text{Al}(\text{O}^i\text{Bu})_3$ -GPTS-KO ^iBu -Hydrolyzate (Al -GPTS-KO ^iBu -H) and its calcined form (KAlSiO_4) were synthesized, characterized and applied to remove Cu(II) and Pb(II) ions from aqueous solutions by adsorption process. Different parameters such as contact time, initial pH, adsorbent amount, and initial metal ion concentration were studied, and CCD was applied for each metal ion and each material to optimize metal ions removal. According to results of some optimum adsorption conditions; with a determination coefficient (R^2) value greater than 97%; for Cu(II) and Pb(II) respectively, with hybrid material 91.82%-93.59%; and with calcined material 85.95%-86.36% metal removal values have been reached.

Keywords: Organic-inorganic hybrid material, Heavy metal removal, Central composite design

References:

1. Kayan, A., Preparation, characterization and application of hybrid materials having multifunctional properties, *JIOMP* (2015) 25 1345-1352.
2. Bingöl, D., Karayünlü Bozbaş, S., Removal of lead (II) from aqueous solution on multiwalled carbon nanotube by using response surface methodology, *Spectrosc Lett* (2011) 45 324-329.

PP30- The Role of High-Energy Milling Applications on Leaching Performance

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Typical metallurgical methods used for processing laterite ores cause chemical consumption in large quantities in addition to challenging reaction conditions and environmental pollution¹. Therefore, new and more economical processing technologies should be developed for nickel recovery. Mechanochemical processing, chemical reactions and phase transformations take place because of mechanical energy applied in high-energy milling. Mechanical activation generated by high-energy milling may break down crystals. Thus, fresh and active surfaces expose, which both enhance mass transfer to initiate chemical reactions and provide the required contact for solid reaction partners².

In this study, it is aimed to overcome the disadvantages of the conventional metallurgical methods to achieve high-efficiency nickel recovery from lateritic ore at lower temperature and lower acid concentration. Atmospheric acid leaching (AL) was performed for nickel recovery from raw and mechanically treated ore to show the effect of mechanical activation (MA) on leaching. Both MA and AL processes were performed according to the experimental plan generated by the Central Composite Design (CCD) as an experimental design method. Response Surface Method (RSM) approach has been used in evaluating the experimental results to determine the optimum processes conditions. In this way, these processes were expressed with mathematical model equations. Surface plots were generated from the model to show the effects and interactions of factors on nickel recovery. According to high determination coefficients ($\text{Adj-R}^2 > 95\%$) for all processes, estimated results by models showed good agreement with the experimental results.

Optimum activation conditions were found as milling time: 135 min, B/C ratio: 15 and rotational speed: 425 rpm. AL was applied to mechanically activated ore samples in these conditions. Optimum AL conditions were found as 1.30 M H_2SO_4 concentration, 100 mL/g L/S ratio and 85 °C at 30 min, respectively. Estimated and experimentally nickel recoveries ($R (\%) \pm s$) ($N=2$) for mechanically activated ore were determined as $98.76\% \pm 0.10$ and $97.92\% \pm 1.83$, respectively.

In addition, optimum AL conditions for raw ore were found as 3.08 M H_2SO_4 concentration, 27.9 mL/g L/S ratio and 95 °C at 240 min. Estimated and experimental nickel recoveries were determined as $99.96\% \pm 1.18$ and $97.99\% \pm 1.83$, respectively. The results showed that the changes in ore structure during mechanical activation had a positive effect on acid leaching. High nickel recovery could be achieved in a shorter leaching time and lower acid concentration due to this effect.

Keywords: Mechanical activation, Laterite ore, Leaching, RSM approach

References:

1. Gao J., Yan Z., Liu J., Zhang M., Guo M., A novel hydrometallurgical approach to recover valuable metals from laterite ore, *Hydrometallurgy* (2014) 150 161-166.
2. Baláz P., Baláz M., Bujňáková Z., Mechanochemistry in technology: from minerals to manomaterials and drugs, *Chem. Eng. Technol.* (2014) 37 747-756.

Acknowledgement: We are grateful to TUBITAK (Project Number:116M076) for financial support.

PP31- Cinnamon Shell as Low-Cost Adsorbent to Remove Indigo Carmine from Aqueous Solutions

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Environmental pollution is increasing day by day with industrial development. Textile sector is one of the leading and developing sectors in the world and our country. Therefore, wastewater containing high amounts of dyes is serious problems. The main source of color in such wastewaters is the used dyes, which may be dissolved or colloidal. Indigo carmine (IC) is one of the most widely used dyes in many industrial areas such as food, cosmetic industries, medical diagnostics and textiles. IC is a highly toxic dye and is known to cause damage to skin and permanent eye damage¹.

This study was conducted to investigate the utility of cinnamon shell as an agricultural waste for removal of IC from aqueous solutions with batch experiments. The effects of experimental factors such as pH, adsorbent dose, initial dye concentration, time and temperature on IC removal were investigated. The results showed that the adsorption of IC onto cinnamon shell was strongly depended on solution pH. The amount of adsorbed IC decreased with increasing pH due to electrostatic repulsive interactions². The high efficiency dye removal was achieved at pH 2. IC adsorption on cinnamon shell was investigated according to Langmuir, Freundlich, Halsey Temkin and Dubinin-Radushkevich isotherm models. Temkin isotherm was suitable for IC adsorption on cinnamon shell with the highest correlation value ($R^2 > 95\%$) for all temperatures. IC adsorption kinetic was well fitted with Pseudo-second-order model. In addition, thermodynamic parameters such as enthalpy, entropy, free energy and activation energy were determined. Moreover, the thermodynamics studies shown that IC adsorption onto the surface of cinnamon shell was found to be endothermic.

Keywords: Adsorption, Cinnamon shell, Indigo carmine, Kinetic

References:

1. Singh H., Chauhan G., Jain A.K., Sharma S.K., Adsorptive potential of agricultural wastes for removal of dyes from aqueous solutions, J. Environ. Chem. Eng. (2017) 5 122-135.
2. Tan I. , Hameed B., Ahmad A., Equilibrium and kinetic studies on basic dye adsorption by oil palm fibre activated carbon, Chem. Eng. J. (2007) 127 111-119.

PP32- Removal of Phthalic Acid from Water by Anion Exchange Resin

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Phthalic acid diesters (PAEs), which are widely used as plasticizers in plastics, are suspected to be carcinogenic and have been shown to interfere with the reproductive systems and development of animals and humans. As endocrine-disrupting chemicals (EDCs), PAEs may cause the hormone system and the reproductive system. Even worse, PAEs is a persistent and easily bio-accumulated material, which can readily be transported over long distances; as a consequence, and it has been found in indoor dust, foods, air, water and soil.

In this work, the removal of phthalic acid from water was studied. The influences of pH, contact time, initial concentration, and temperature of phthalic acid solution on the removal behavior were investigated. The Langmuir adsorption isotherm was adequate to represent the experimental data and the sorption kinetic was well represented by a pseudo-first order kinetic model. The thermodynamics analysis indicated negative free energy and enthalpy changes suggesting that the adsorption of phthalic acid onto ion exchange resin is spontaneous and exothermic.

Keywords: Ion exchange resin, Phthalic acid, Water treatment,

Acknowledgements: This study was supported by The Scientific and Technological Research Council of Turkey (TUBITAK-Program code: 2209). We would like to acknowledge Finex Company for providing us the resin sample.

References:

1. Julinová, Markéta, and Roman Slavík. "Removal of phthalates from aqueous solution by different adsorbents: a short review J. Environ. Manage. (2012) 94 13-24.
2. Zhengwen Xu, Ling Cheng, Jing Shi, Jiangang Lu, Weiming Zhang, Yunlong Zhao, Fengying Li, Mindong Chen, Kinetic study of the removal of dimethyl phthalate from an aqueous solution using an anion exchange resin, Environ Sci Pollut Res (2014) 21 6571–6577

PP33- Removal of Copper (II) from Water by sulfonated biodegradable polymer

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Copper is an important trace element required by humans for its role in enzyme synthesis, tissues and bones development. However, the divalent copper is toxic and carcinogenic when consumed in excess through ingestion. Various technologies exist for the removal of Cu(II). They include filtration, chemical precipitation, ion exchange, adsorption using activated carbon, electrode-position and membrane process. Biosorption or sorption to material of biological origin is recognized as an emerging technique for the treatment of wastewater containing heavy metals.

In this study, we prepared water-insoluble sulfonated cellulose through a simple two-step reaction of the cellulose with sodium periodate then followed by reaction with sodium metabisulfite to introduce sulfonic acid groups into the cellulose. The modified cellulose was then used as an sorbent in a series of batch sorption experiments to investigate the behaviors and mechanisms of the modified cellulose in removing Cu (II) ions from aqueous solutions. The effects of sorbent dose, initial solution pH and temperature on the removal performance of sulfonated cellulose were investigated. Isothermal data were modeled with the Langmuir and Freundlich isotherm models. Thermodynamic study revealed an exothermic sorption process.

Keywords: Cellulose, Copper, Ion exchange, water treatment

Acknowledgements: We thank Assoc. Prof. Dr. Emrah Çakmakçı for his valuable comments on this work. The authors thank Denkim Kimya A.Ş. for providing the cellulose sample

References:

1. Haluk Aydın, Yasemin Bulut, Çiğdem Yerlikaya, Removal of copper (II) from aqueous solution by adsorption onto low-cost adsorbents, J. Environ. Manage. (2008) 87 37–45
2. Y. Nuhoglu , E. Oguz, Removal of copper(II) from aqueous solutions by biosorption on the cone biomass of Thuja orientalis, Process Biochem. (2003) 38 1627/1631

PP34- Acid dissociation constants of 4-aryl-2-(pyrrolidin-1-yl)thiazole derivatives in dimethyl sulfoxide-water hydroorganic solvent

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Acid dissociation constants (pK_a) are among the important parameters having critical significance in the field of pharmacy, chemistry, biology, medicine etc. due to providing information related to acidity, solubility, lipophilicity, bonding to receptors, transport behavior properties of pharmaceutically important molecules, determining the optimum conditions in the development of analytical methods, and understanding the interactions between ligands and metal ions in complexes [1-3]. The thiazole and pyrrolidine scaffolds are intensively studied in drug research, and both of these pharmacophore groups exhibited a wide range of pharmacological activities such as antimicrobial, anticancer activities [3-6].

As a continuation of our previous work [3], in this study, three acid dissociation constants were calculated for the compounds using the HYPERQUAD computer program from data which were obtained by potentiometric titration method in 50% (v/v) dimethyl sulfoxide-water hydroorganic solvent at 25 ± 0.1 °C, at an ionic background of 0.1 mol/L of NaCl. The obtained three acid dissociation constants might be associated with SH, OH and NH, which was formed by the protonation of the thiazole ring, groups.

Keywords: Acid dissociation constants, Potentiometric titration, Thiazole, Pyrrolidine

Acknowledgements: This work was funded by Mersin University Research Fund (Project No: BAP 2016-2-AP3-1800).

References:

1. Manallack D.T. The acid–base profile of a contemporary set of drugs: implications for drug discovery. *SAR and QSAR in Environmental Research*, (2009) 20 611-655.
2. Narin I, Sarioglan S, Anilanmert B, Sari H. (2010). pK_a determinations for montelukast sodium and levodropropizine. *J Solution Chem* (2010) 39 1582-1588.
3. Gemili M, Sari H, Ulger M, Sahin E, Nural Y. Pt (II) and Ni (II) complexes of octahydropyrrolo [3, 4-c] pyrrole N-benzoylthiourea derivatives: Synthesis, characterization, physical parameters and biological activity. *Inorg Chim Acta* (2017) 463 88-96.
4. Shankar B, Jalapathi P, Nagamani M, Gandu B, Kudle KR. Synthesis, anti-microbial activity, and cytotoxicity of novel 1-[5-[6-[(2-benzoylbenzofuran-5-yl) methyl]-2-oxo-2H-chromen-3-yl] thiazol-2-yl] urea derivatives. *Monatsh Chem* (2017) 148 999-1009.
5. Şahin Ö, Özdemir ÜÖ, Seferoğlu N, Genc ZK., Kaya K., Aydiner B., Tekin S, Seferoğlu, Z. New platinum (II) and palladium (II) complexes of coumarin-thiazole Schiff base with a fluorescent chemosensor properties: Synthesis, spectroscopic characterization, X-ray structure determination, in vitro anticancer activity on various human carcinoma cell lines and computational studies. *J Photoch Photobio B* (2018) 178 428-439.
6. Lotfy G, Said MM, El Sayed H, El Sayed H, Al-Dhfyar A, Aziz YMA, Barakat A. Synthesis of new spirooxindole-pyrrolthiazole derivatives: Anti-cancer activity and molecular docking. *Bioorg Med Chem* 2017 25 1514-1523.

PP35- Graphene and Molecularly Imprinted Polymer Based Sensor Production with One Step Electrochemical Procedure for Trinitrotoluene Detection

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Molecularly imprinted polymers are very suitable for fabricating chemical/biological sensors with enhanced selectivity. Graphene, which is a single layer of carbon atoms organized in a closely packed honeycomb two-dimensional lattice, has attracted great attention¹. The advantages of using polymer and graphene composites as sensing material can be expressed with respect to increased surface area, higher numbers of analytical recognition sites, low resistance and improved environmental stability, due to the unique properties of both components. Graphene and/or reduced graphene oxide are able to be prepared by an electrochemical method which is effective and green route². Graphene oxide reduction and electropolymerization of a monomer in the presence of template molecule are usually performed by separate procedures.

Here in this work electropolymerization of aniline and electrochemical reduction of graphene oxide were taken place simultaneously.

It is aimed to detect trinitrotoluene (TNT) using an electrochemical sensor based on graphene and molecular imprinted polymer (MIP). Picric acid served as a model molecule for TNT. The reduction of the synthesized graphene oxide and polymer formation were carried out electrochemically using cyclic voltammetry by scanning 15 cycles in the potential range of -1.0 to + 1.2V (vs Ag / AgCl) as one step process. The methanol-acetic acid mixture was used to remove the repressed picric acid from the sensor and the experimental conditions were optimized. After the sensor was developed, square wave voltammetry, SEM and electrochemical impedance measurements were used for characterization. The prepared new composite film possessed the advantages of both polyaniline and reduced graphene oxide with a synergistic effect, which has excellent electrocatalytic activity.

Keywords: Molecularly imprinted polymer, graphene, trinitrotoluene.

References:

1. M.J. Allen, V.C. Tung, R.B. Kaner, Honeycomb carbon: a review of graphene, Chem. Rev. 110 (2010) 132–145.
2. H.L. Guo, X.F. Wang, Q.Y. Qian, F.B. Wang, X.H. Xia, A green approach to the synthesis of graphene nanosheets, ACS Nano 3 (2009) 2653–2659.

PP36- Voltammetric Determination of Chlorite Using Carbon Paste Electrode Modified with N-Cetylpyridinium-Smectite

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The addition of the solid-phase component into electrode-solution interface enhances properties of the modified electrode that differ from those of the bare surface in many regards. The ability to promote electron transfer and the stability during electrolytic applications are often enhanced. The promoted electron transfer is very crucial for electroanalysis while the latter is primarily accrued by altering the course of electrolysis mechanisms in a manner that eliminates adsorbed intermediates and/or products, but in some cases an improved stability results from the modifier occupying active adsorption sites. [1]

Because of the great concern have been focused on the determination of disinfectant by-products, many methods, such as; liquid chromatography, flow injection, and capillary electrophoresis, electrochemical methods, were developed for the quantification of chlorite ions. Among these methods electrochemical methods are effective, low-cost, non-laborious and environment friendly processes [2].

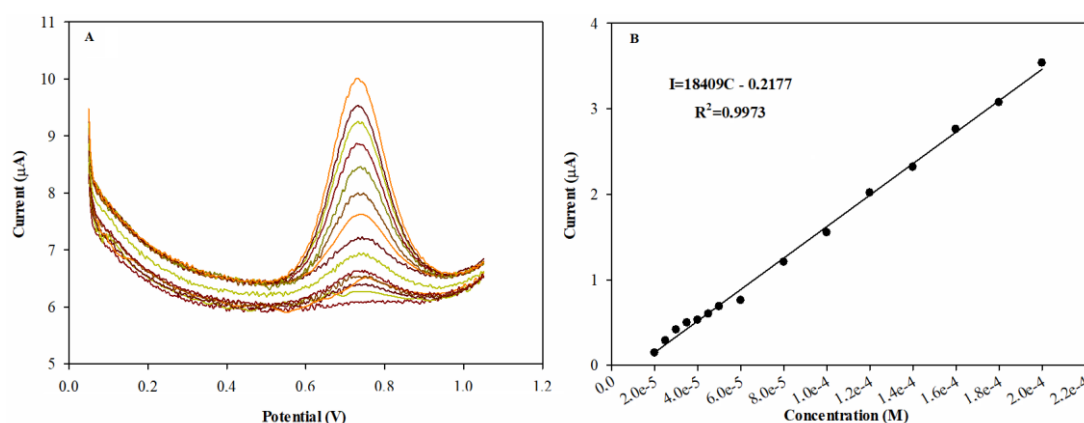


Figure 2. A) Calibration differential pulse voltammograms of 2.10^{-5} to 2.10^{-4} M chlorite ions in pH 5.0 BR buffer and **B)** calibration curve for these data

In this study, N-Cetylpyridinium modified smectite was synthesized and characterized by various techniques and this composite exhibited catalytic effect towards chlorite oxidation. After the optimization studies, the calibration curve was plotted (Fig. 1) and the modified electrode, was utilized for the determination of chlorite in bottled water.

Keywords: N-Cetylpyridinium, Smectite, Voltammetry, Chlorite determination.

References:

1. J. A. Cox, R. J. Pawel, J Kulesza Electroanalysis with Electrodes Modified by Inorganic Films electroanalysis 3 (1991) 869-877.
2. E. Al-Zahrani, M. T. Soomro et al., Fabrication and performance of magnetite (Fe₃O₄) modified carbon paste electrode for the electrochemical detection of chlorite ions in aqueous medium, Journal of Environmental Chemical Engineering 4 (2016) 4330-4341.

PP37- Characterization of Magnetite Decorated Bentonite and Its Voltammetric Application for Chlorite Determination

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The modification of the electrode surface with inorganic materials such as zeolites, silica-based hybrid materials, and clays plays significant role for achieving sensitive, selective and accurate signals in electro analysis [1]. Determination of disinfectant by-products have been gained high concern to ensure the safety and quality of the drinking water due to ever-increasing use of disinfectant agents in water treatment. Being cost effective, efficient, and easy to handle, chlorine and its dioxide are the disinfectant of choice to control taste and odor of the drinking water. However, the reduction of chlorine dioxide in water leads to the formation of chlorite ion (ClO_2^-) in drinking water that are hazardous to living beings. Although, many methods were devoted to determine chlorite ions such as; chromatographic, flow injection, and capillary electrophoresis, electrochemical methods are probably most promising [2].

In this study, magnetite nanoparticles decorated onto bentonite surface and this nanocomposite was characterized by various techniques. Further, this nanocomposite material was used as a modifier in carbon paste electrode (CPE) and this electrode exhibited catalytic effect towards chlorite oxidation. After the optimization studies, the electrode, developed, was applied to the determination of chlorite in bottled water.

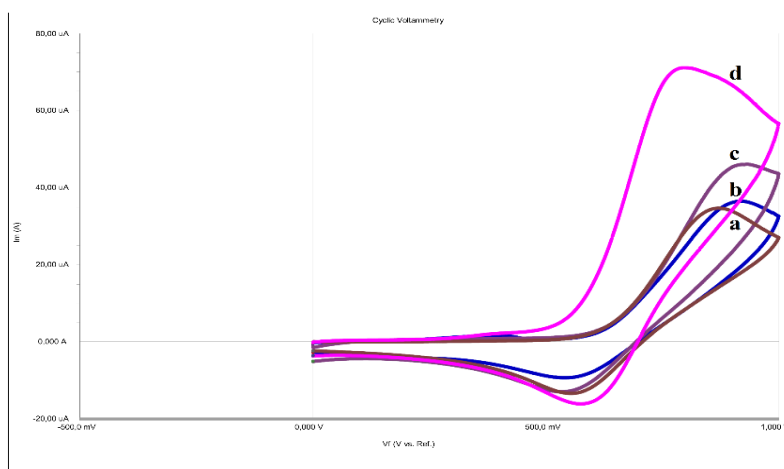


Figure 3 Cyclic voltammograms of 5×10^{-3} M ClO_2^- recorded for **a)** Bare CPE, **b)** Bentonite modified CPE, **c)** Magnetite modified CPE **d)** Magnetite decorated bentonite modified CPE in pH 8.00 BR buffer with scan rate of 100 mVs^{-1}

Keywords: Magnetite decorated bentonite, Voltammetry, Chlorite determination.

References:

1. C. Mousty, Sensors and biosensors based on clay-modified electrodes-new trends, Applied Clay Science 27 (2004) 159-177.
2. E. Al-Zahrani, M. T. Soomro et al., Fabrication and performance of magnetite (Fe_3O_4) modified carbon paste electrode for the electrochemical detection of chlorite ions in aqueous medium, Journal of Environmental Chemical Engineering 4 (2016) 4330-4341.

PP38- Carbon-paste electrode modified with silica and cetylpyridinium chloride for voltammetric determination of food dyes

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Synthetic dyes are a class of food supplements. They are used to give food an attractive appearance as well as to compensate for the loss of the natural colour of food after processing and storing. Food supplements, such as Tartazine, Sunset Yellow FCF, Allura Red AC, Carmoisine and Ponceau 4R are synthetic azo dyes which are used to colour drinks, food, cosmetics and pharmaceuticals. A significant range of research has investigated the toxicological properties of these dyes and their metabolites, and it has been established that they are potentially dangerous for human health. Therefore, the development of highly sensitive and selective analytical methods for food dyes determination is an urgent task.

The redox behavior of Sunset Yellow FCF on modified carbon-paste electrode has been studied. It has been established that the oxidation current of Sunset Yellow FCF has an adsorption character. The optimum conditions for the square wave voltammetric determination of the dye were established. Using the SWVs technique under optimal conditions (adsorption accumulation was carried out at 400 mV during 180 seconds from Britton-Robinson buffer solution with pH 2 and sweep rate of 100 mV/s, amplitude 40 mV, frequency 25 Hz), the calibration curve (Figure) for the determination of Sunset Yellow FCF is linear in the concentration range 4.5×10^{-4} – $0.45 \mu\text{g} \cdot \text{mL}^{-1}$ and is described by the equation: $i[\mu\text{A}] = 9,3651[\mu\text{A}/\mu\text{g} \cdot \text{mL}^{-1}] \cdot C[\mu\text{g} \cdot \text{mL}^{-1}] + 0,0123[\mu\text{A}]$ ($R^2=0.98$). With an increase in the accumulation time up to 10 min, a determination of 1×10^{-9} M Sunset Yellow FCF is possible.

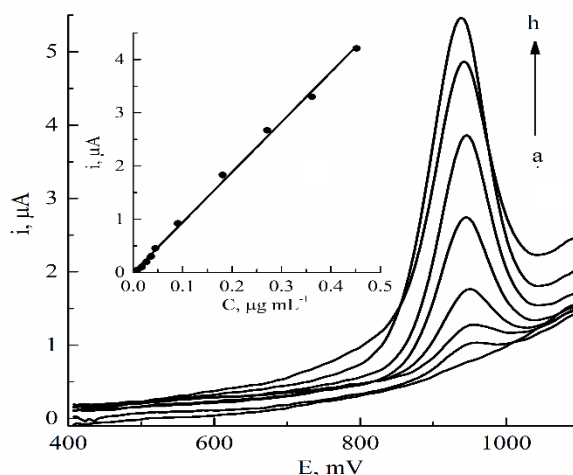


Figure. SWVs of Sunset Yellow FCF at different concentrations (from a to h): 0.0, 0.05, 0.1, 0.2, 0.3, 0.35, 4.5, 5.5 $\mu\text{g} \cdot \text{mL}^{-1}$. Inserts - plot of the peak currents as a function of dye concentration.

The developed technique was satisfactorily applied to the analysis of Sunset Yellow FCF in non-alcoholic drinks with RSD of 4.5%. The dye concentration in commercial drink ($96 \mu\text{g} \cdot \text{mL}^{-1}$) after 1000 fold dilution was determined from the calibration curve and the standard addition technique.

Keywords: carbon-paste electrode, voltammetry, food dyes.

PP39- Boehmite as Inorganic Additive for Positive Electrolyte of Vanadium Redox Battery

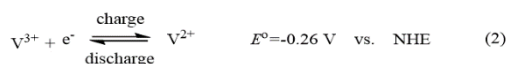
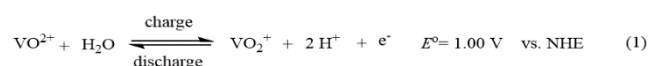
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Renewable energy sources such wind, solar and etc. have great potential for sustainable energy in the future [1]. Since these sources are discontinues, storage of this energy in chemical form is important. Vanadium redox battery (VRB) system is a kind of new generation energy storage system consisting of vanadium ions at different oxidation states and sulfuric acid solution inside of both cell and it was discovered and employed for the first time in University of New South Wales (UNSW) [1]. The following reaction occur between V(4)/V(5) (Eq.1) and V(3)/ V(2) (Eq.2) in positive and negative electrolyte solutions on electrode surface, respectively [2]. The capacity of a VRB is mainly related with the concentration of sulfuric acid and active vanadium ions [2-5]. Thermal precipitation of V(5) ion at high temperature (> 40 °C) effects negatively of the performance of the battery (Eq. 3). The stability of V(5) ions at high temperature can be improved by using of additive [2-5].



In this study, an inorganic additive boehmite was used as anti-precipitating agent for V(5) ions. The optimum additive amount was determined by cyclic voltammetric and electrochemical impedance spectroscopic analysis. The interactions of additives and V(5) ions were also investigated by UV analysis. Scanning electron microscopic analysis and XPS analysis were carried out to determine structural and chemical change of the surface used carbon based working electrodes, respectively after voltammetric analysis. The additive showed a good anti-precipitating properties for V(5) ions according to the result of precipitating test performed at 40 and 60 °C. The effects of additive on the battery performance was also investigated by cyclic charge-discharge tests.

*This work was supported by TUBİTAK with the 114Z774 project code. M. Gençten and H. Gürsu thank to TUBİTAK-BİDEB.

Keywords: Cyclic voltammetry, charge-discharge tests, inorganic additive, boehmite

References:

1. V. Khare, S. Nema, P. Baredar, Renewable and Sustainable Energy Reviews. 58 (2016) 23-33
2. M. Skyllas-Kazacos, M. Rychcik, R.G. Robbins, A.G. Fane, M. Green, J. Electrochem. Soc. 113 (1986) 1057-1058.
3. M. Gençten, H. Gürsu, Y. Şahin, Electrochemical investigation of the effects of V(V) and sulfuric acid concentrations on positive electrolyte for vanadium redox flow battery. Int. J. Hyd. Energ. 41(2016) 9868-9875.
4. M. Gençten, H. Gürsu, Y. Şahin, Anti-precipitation effects of TiO₂ and TiOSO₄ on positive electrolyte of vanadium redox battery. Int. J. Hyd. Energ. 42 (2017) 25608-25618.
5. M. Gençten, H. Gürsu, Y. Şahin, Effect of α- and γ-alumina on the precipitation of positive electrolyte in vanadium redox battery. Int. J. Hyd. Energ. 42 (2017) 25598-25607.

PP40- Metal Oxide based Ternary Nanocomposites and Free-standing Li-ion Anode Applications

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Lithium ion batteries (LIBs) having high energy and power density are in demand for electric vehicle (EV) applications. The conventional anode of LIBs is graphite, and it can only intercalate one Li atom per six carbon atoms (LiC_6), so it provides low energy and power density with a theoretical capacity of $372 \text{ mA}\cdot\text{h g}^{-1}$. Whereas, SnO_2 and Fe_2O_3 have a great potential for commercial applications due to their high capacities of 1490 and $1005 \text{ mA}\cdot\text{h g}^{-1}$, respectively [1]. However, these metal oxides suffer from huge volume expansion during Li^+ alloying-dealloying process, which cause capacity decrease and mechanical disintegration. To prevent this undesired expansion, graphene (G) based structures can be used as buffer material. Graphene has special structure with a one-atom-thick 2D sheet of sp^2 -bonded carbon atoms having thermal transport properties and exceptional mechanical. It is widely used to modify electrode materials for of Li-ion battery owing to its properties. Therefore, it can not only act as framework to support the metal and wrap the metal oxides to accommodate their volume changes but also enhance conductivity and short the Li-ion diffusion path [2].

In this study, to benefit from synergistic effect of two different metal oxide structure and graphene, $\text{SnO}_2\text{-Fe}_2\text{O}_3\text{-rGO}$ (reduced graphene oxide) ternary nanocomposite structure was produced as a free-standing, elastic and binder-free anode material. For this aim, firstly, metal oxide powders were synthesized via sol-gel method. Secondly, graphene oxide was synthesized by modified Hummer's method from graphite powder. Free-standing papers were produced by vacuum filtration of the mixture of this ternary structure after 2 hours ultrasonication. $\text{SnO}_2\text{-Fe}_2\text{O}_3\text{-rGO}$ nanocomposite anode was obtained after reduction of these papers with hydrazine. This composite structure was characterized by Field Emission Gun Scanning Electron Microscopy (FEG-SEM), and X-ray diffraction (XRD) analyses. Electrochemical performance of the anodes was determined via a BST8-MA MTI model battery tester at a constant current. As a result, high capacity metal oxide / rGO ternary nanocomposite anodes were improved as flexible, binderless and free-standing disc anodes.

Keywords: Ternary metal oxide-rGO free-standing anodes, Li-ion batteries

References:

1. Xia, G., Li, N., Li, D., et.al., Graphene/ Fe_2O_3 / SnO_2 Ternary Nanocomposites as a High-Performance Anode for Lithium Ion Batteries, *ACS Appl. Mater. Interfaces* (2013) 5 8607–8614.
2. Lin, F., Wang, H., $\text{Fe}_2\text{O}_3\text{-SnO}_2$ -graphene films as flexible and binder-free anode materials for lithium-ion batteries, *J. Mater. Res.* (2015) 30(18) 2736-2746.

PP41- SiO₂-based Free-standing Anodes for Li-ion Batteries

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Lithium-ion batteries (LIBs) have become the most promising renewable power source since they are one of the most important energy storage devices for portable electronics and also are considered as storage systems for electric vehicles [1]. SiO₂ has been considered as one of the promising materials thanks to its low discharge potentials, abundance and low cost [2]. Gao et al. claimed that commercial SiO₂ nanoparticles, 7 nm in diameter, could react with Li with a reversible capacity of 400 mA·h g⁻¹ between 0 and 1.0 V (vs. Li/Li⁺). However, the low electronic conductivity of SiO₂ obstructs its application as an anode material [1]. Graphene, 2D carbon material, is expected to be an improved anode material in Li-ion batteries because of its advanced features such as high surface-to-volume ratio, superior electrical conductivity, structural flexibility, ultrathin thickness, and chemical stability. The graphene layers could provide a support for SiO₂ nanoparticles and play role as highly conductive matrix and also could effectively prevent the aggregation of nanoparticles and volume expansion during Li alloying-dealloying process [1].

In this work, SiO₂-reduced graphene oxide (rGO) nanocomposite was produced as flexible free-standing paper anode for LIBs. Firstly, graphite oxide was produced via Hummers method from graphite. After that, SiO₂ nanoparticles synthesized by sol-gel method were added to aqueous graphite oxide suspension and ultrasonicated for 1 hour. Afterwards, this suspension was filtrated via vacuum filtration method and free-standing anode paper was obtained after reduction process in hydrazine solution.

Produced SiO₂/rGO nanocomposites were characterized by Scanning Electron Microscopy (SEM), and X-ray diffraction (XRD) analyses. Optical properties of SiO₂/rGO nanocomposites were investigated via Fourier transform infrared spectroscopy (FT-IR).

Keywords: SiO₂/rGO nanocomposites, reduced graphene oxide, structural properties, Li-ion battery anodes

References:

1. Ren, Y., Wei, H., Xiaobing, H., Ding, J., A Facile Synthesis of SiO₂@C/graphene Composites as Anode Material for Lithium Ion Batteries, *Int J Electrochem Sci* (2014) 9 7784-7794.
2. Gong, H., Li, N., Qian, Y., Synthesis of SiO₂/C Nanocomposites and Their Electrochemical Properties, *Int J Electrochem Sci* (2013) 8 9811-9817.

PP42- A PVC-Membrane Potentiometric Copper(II)-Selective Electrode Based on a Schiff Base Compound as Ion Carrier

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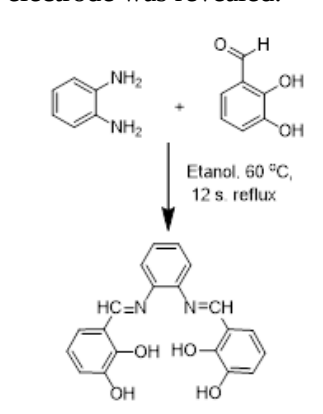
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Potentiometric ion-selective electrodes (ISEs) for determining the metal ion have been routinely used well-established analytical tools due to simplicity, adequate selectivity, relatively low detection limit, nondestructive analysis, low-cost sample preparation, applicability to colored sample and turbid solution. One of the most important component of a sensor membrane is ionophore which plays a key role in the selectivity and sensitivity of an ion-selective electrode (ISE). Potentiometric response of the PVC-membrane electrode is generated by the selective complexation of the target ion by the ion carrier (ionophore) that is impregnated in a poly(vinyl chloride) (PVC) matrix [2]. For that purpose, large numbers of ionophores exhibiting high selectivity to specific metal ions in the structure of potentiometric sensors for the determination of the target metal ions have been studied up to present. The demand for ionophores with high selectivities has been still ongoing in the field of ionselective electrodes (ISEs). One of the most important classes of compounds used as ionophore is Schiff bases.

In the current study we have synthesized a new Schiff base from the starting compounds o-phenylenediamine and o-phenylenediamine according to the similar procedure and reaction given Schema 1 [3]. The resultant Schiff base was utilized as ionophore in the structure of PVC-membrane electrode. The constructed electrode exhibited fairly selective potentiometric responses to Cu(II) ions (Fig. 1). The potentiometric performance characteristics of the proposed electrode was revealed.



Schema 1. The synthesis reaction of the current Schiff base

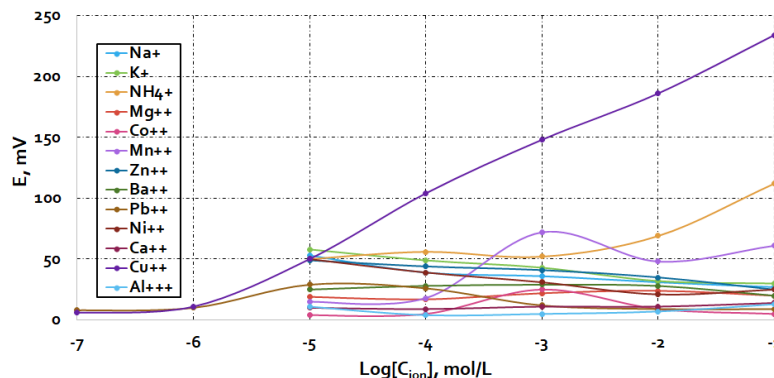


Figure 1. The potentiometric response of the proposed electrode towards various cationic solutions at different concentrations.

Keywords: Schiff base, potentiometry, ion selective electrode, copper(II) determination.

References:

1. T.M. Fasina, O. Ogundele, F.N. Ejiah, C.U. Dueke-Eze, Biological Activity of Copper (II), Cobalt (II) and Nickel (II) Complexes of Schiff Base Derived from O-phenylenediamine and 5-bromosalicylaldehyde, International Journal of Biological Chemistry, 6 (2012) 24–30.
2. A. Homafar, F. Maleki, Z. Abbasi, Lead (II)-Selective Polymeric Electrode Using PVC Membrane Based on a Schiff Base Complex of 1,2- Bis(Salicylidin Aminoxy) Ethane As an Ionophore, Energy and Environmental Engineering, 1 (2013) 99–104.

PP43- Voltammetric Determination of Copper(II) Using a Carbon Paste Electrode Modified with a 4-(2-pyridylazo)resorcinol Doped and Cross-linked Polymer

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Copper is one of the most widely distributed elements in the environment. It exists in all organisms, land and water. Trace amount of this metal is essential for the enzymatic reactions, but higher amounts of it may provoke severe toxicity to living organisms due to inhibition of metabolic reactions [1]. Therefore, application of methods to monitor copper amounts in environment is indispensable. Electrochemical detection is an alternative attention drawing method for the determination of electroactive species, since its intrinsic advantages of simplicity, ease of miniaturization, high sensitivity and relatively low-cost. Carbon paste electrode (CPE) is one of the most convenient choices due to advantages such as renewable surface, high stability, long lifetime, good signal repeatability and accuracy. The chemically modified carbon paste electrodes (CMCPEs) are composed of a modifier and carbon paste and the modifier, which provides a specific function to the electrode, plays a significant role in achieving sensitive, selective and accurate signals in electro analysis. Upto now, some organic polymers, ligands, as well as inorganic ion exchangers and chemically modified nano structures have been used as modifiers depending on the analyte of interest [2].

In the present study, we synthesized a cross-linked and 4-(2-pyridylazo)resorcinol doped polymer based on 1-vinylimidazole and 4-vinylpyridine monomers. The synthesized polymer was utilized as a modifier in the structure of carbon paste electrode (CPE) prepared for the purpose of Cu(II) determination. The modification of the electrode resulted in a two-fold enhancement in copper(II) peak height (Figure 1). The proposed electrode was successfully employed for the Cu (II) determination in spiked water samples by using Differential Pulse Voltammetry (DPV) technique.

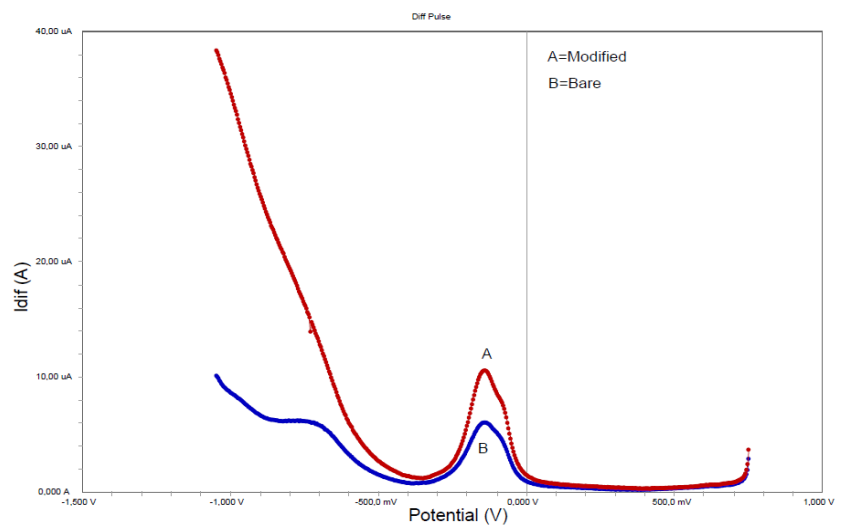


Figure 1. DP voltamograms of bare and modified electrode in 5×10^{-4} M Cu(II) containing pH=5 buffer solution.

Keywords: Polymer, 4-(2-pyridylazo)resorcinol, Voltammetry, Copper(II) determination.

References:

1. E.E. Tyralla, N.L. Brodsky, V.H. Auerbach, Urinary copper losses in infants receiving free amino acid solutions, *Am. J. Clin. Nutr.* 35 (1982) 542–545.
2. V.S. Somers, L.H. Hernandez, E.I. Iwuoha, Stripping voltammetric measurement of trace metal ions using screen-printed carbon and modified carbon paste electrodes on river water from the Eerste-Kuils River System, *Journal of Environmental Science and Health A46* (2011) 17–32.

PP44- Chromatographic Determination Methods for Haloacetic Acids in Water Samples

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Chlorination of drinking water leads the formation of disinfection by-products through the reaction between natural organic matter and free available chlorine species. The dominant absorbable organic halogens in surface waters originated from this reaction are haloacetic acids (HAA) and trihalomethanes (THM) [1]. Some of the HAAs have been classified as human carcinogens and therefore, the US EPA has set a maximum concentration level (MCL) of 0.060 mg/L for the total concentrations of five HAAs (HAA5); including Dichloroacetic acid (DCAA), trichloroacetic acid (TCAA), monochloroacetic acid (MCAA), monobromoacetic acid (MBAA) and dibromoacetic acid (DBAA).

In the present study, a number of methods have been tested including an international standard method where the sample is dissolved in mobile phase including acetonitrile: water 10:90 mixture added into 2 mL of phosphoric acid [2]. The sample is injected into HPLC-UV system involving a C8 column and detected by an UV detector set at 214 nm. In the other method the mobile phase ratio has been changed and isocratic elution program was applied with 1.2 mL/min flow rate. However, the sensitivity of the method was not satisfactory to attain the MCL of the HAAs in water samples. Therefore, a solid phase extraction (SPE) cartridge including strong anion exchanger was used for preconcentration for subsequent LC analysis. Alternatively, supported liquid membrane microextraction technique was utilized for HAA analysis and a significant enrichment factor was obtained. The overall results have been evaluated and the SPE method was applied to the determination of HAAs in water samples obtained from swimming pool.

Keywords: Haloacetic acid, HPLC, UV detection, SPE, SLLME

Acknowledgement: Authors would like to thank Ege University Scientific Research Project (2015 FEN 034) for financial support.

References:

1. USEPA, 2009 "Basic Information About Drinking Water Disinfection" 2/24/2009.
2. ISO, 2014, www.iso.org, "Surface active agents-Determination of chloroacetic acid (chloroacetate) in surfactants" Part 1: HPLC method, ISO 17 293-1.

PP45- DNA-decorated multi-walled carbon nanotube(MWCNT)-modified biosensor for the detection of DNA sequence related to the Influenza B virus DNA

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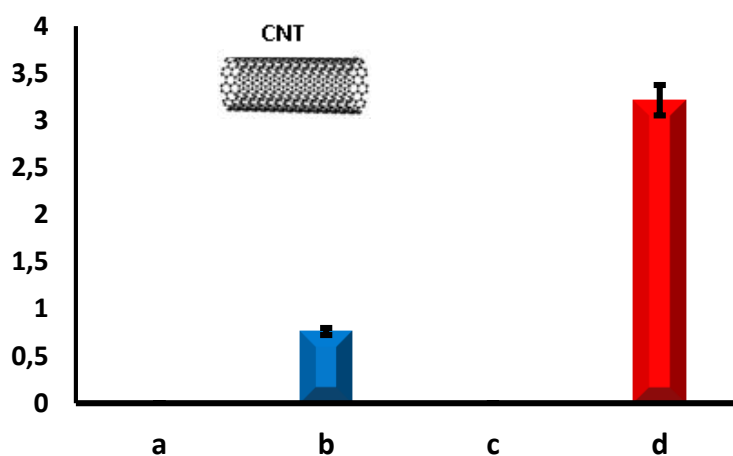
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Carbon nanotube-contained biosensor systems have still received considerable attention due to the advantages such as their large surface areas and many of electrochemical biosensors have been published up to now (1-2).

This study shows DNA-wrapped multi-walled carbon nanotube(MWCNT)-modified biosensor for the detection and discrimination of DNA sequence related to the Influenza B virus DNA while Influenza A virus sequence was used to control of the biosensor selectivity. There is no report about Influenza B virus detection by using DNA-wrapped MWCNTs, pencil graphite electrode(PGE) and YES/NO detection system in the literature.

In the presented study, the Influenza B-specific probe was interacted with MWCNTs and the capture probe-wrapped MWCNTs “nanohybrid” was immobilized onto pretreated PGE for the detection of DNA hybridisation. Electrochemical behaviors of the nanohybrid modified PGEs were investigated using differential pulse voltammetry(DPV) based on the guanine peak at about 1.0V. A significant enhancement at the guanine signal related to the hybrid DNA was observed in the presence of the nanohybrid modification on graphite transducer. Preliminary results were presented here. The discrimination of probe DNA, hybrid DNA and non-complementary DNA was achieved and evaluated by measuring changes in the voltammetric oxidation signal of guanine.



a: Probe cnt-free PGE b: Hybrid -cnt-free PGE c: Probe -cnt modified PGE d:Hybrid -cnt modified PGE

Keywords: MWCNT, Electrochemical DNA Biosensors, guanine, Influenza B virus

References:

1. Ozkan-Ariksoysal, D., Kayran, Y.U., Yilmaz, F.F., et al. Talanta, 166, 27-35, (2017).
2. Subak H., Ozkan-Ariksoysal D., Sens Actuators B Chem. 2018, in press.

PP46- Development of A Novel Protein-Based Solid-Biosensor for Determining Cu(II)-Induced Prooxidant Activity of Phenolic Compounds

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Phenolic compounds exhibit a wide range of physiological properties such as antiallergenic, anti-inflammatory, antimicrobial, and antioxidant effects. Although most of the health-beneficial effects of these compounds are assumed to originate from their antioxidant activity, there is growing evidence that natural or synthetic antioxidants can act as prooxidants. These prooxidant effects usually depend on the concentration of compounds, free radical source, and other molecules in the medium. In the presence of O₂, transition metal ions such as copper(II) and iron(III) catalyze the redox cycling of these compounds leading to the formation of reactive oxygen species (ROS) and phenoxyl radicals (Ar-O[•]) that can damage biomacromolecules such as DNA, lipids, and proteins¹.

In this work², we aimed to develop a protein-based solid prooxidant biosensor for determining the Cu(II)-induced prooxidant activity of selected antioxidant compounds including gallic acid (GA), catechin (CAT), epicatechin (ECAT), quercetin (QUE), chlorogenic acid (CLA), myricetin (MYR), and ascorbic acid (AA). In this context, a solid material consisting of egg white proteins was precipitated with calcium chloride. This assay involved the reduction of Cu(II) ions to Cu(I) by antioxidant compounds (simultaneously giving rise to reactive oxygen species) and binding of the formed Cu(I) to the solid biosensor. The protein-bound Cu(I), an indicator of prooxidant activity of antioxidants on proteins, was colorimetrically determined at 450 nm with neocuproine (Nc). The method was applied to synthetic binary antioxidant mixtures and herbal (sage, green tea, mint, and marjoram) infusions.

LOD and LOQ values of the proposed method for the chosen standard compound ECAT were 1.2 and 4.0 µM, respectively. Within- and between-run precision values were 2.84 and 3.18%. The order of molar absorption coefficients of test compounds with respect to the biosensor based Cu(II)–Nc assay was as follows: QUE > ECAT > CAT > GA > CLA > MYR > AA. According to modified Cu(II)–Nc assay findings, ECAT, CAT, GA, CLA, QUE, and MYR had prooxidant activity above 2.5 µM while AA had prooxidant activity above 0.25 mM concentration. This low-cost biosensor can be prepared in large quantities and used for a long time. Our findings showed that this protein-based solid biosensor can sensitively and reproducibly measure Cu(II)-induced prooxidant activity of polyphenols and some herbs.

Keywords: Prooxidant biosensor, protein precipitation, phenolics, cupric neocuproine.

References:

1. Galati, G., O'Brien, P.J., Potential toxicity of flavonoids and other dietary phenolics: Significance for their chemopreventive and anticancer properties, *Free Radic. Biol. Med.* (2004) 37(3), 287-303.
2. Akyüz, E., Sözgen Başkan, K., Tütem, E., Apak, R., Novel protein-based solid-biosensor for determining pro-oxidant activity of phenolic compounds, *J. Agric. Food Chem.* (2017) 65, 5821–5830.

PP47- Determining Prooxidant Activity of Phenolic Compounds with Carbonyl Detection Assay Using A Novel Protein-Based Solid-Biosensor

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Proteins are covalently modified by the direct action of ROS or indirectly by reaction with secondary by-products of oxidative stress. The oxidation of amino acid residues in proteins causes several modifications such as deamination, carbonyl group formation and peptide bond cleavage. Protein carbonyl formation *in vitro* and *in vivo* is induced by different ways, including metal catalyzed oxidation, ozone, HOCl, singlet oxygen, and ionizing radiation, and is used as a biomarker of oxidative stress. Colorimetric detection of carbonyl groups, emerging as the most widely used measurement of protein oxidation, involve their derivatization with 2,4-dinitrophenylhydrazine (DNPH), leading to the formation of a stable dinitrophenyl (DNP) hydrazone product¹.

In this study², the carbonyl detection assay was applied to a protein-based solid prooxidant biosensor for determining the Cu(II)-induced prooxidant activity of gallic acid (GA), catechin (CAT), epicatechin (ECAT), quercetin (QUE), chlorogenic acid (CLA), myricetin (MYR), and ascorbic acid (AA). In this context, to develop a protein-based biosensor, a solid material consisting of egg white proteins was precipitated with calcium chloride. Because the solid protein-based sensor was used in our study, the carbonyl assay was modified. This assay is based on the measurement of the absorbance of dinitrophenylhydrazones formed from the reaction of 2,4-DNPH with the carbonyl groups emerging from protein oxidation at 370 nm. The assay was applied to synthetic binary antioxidant mixtures and herbal (sage, green tea, mint, and marjoram) infusions.

Considering the results of the modified carbonyl detection assay, the absorbance values were almost constant within a wide concentration range and a significant increase could only be observed at very high concentrations. LOD and LOQ values of the assay for the chosen standard compound ECAT were 3.0 and 10.0 µM, respectively. Within- and between-run precision values were 4.26 and 5.05%. The order of molar absorption coefficients of test compounds with respect to the modified carbonyl detection assay was as follows: QUE > CLA > CAT > GA > ECAT > AA > MYR. According to the modified carbonyl detection assay results, total prooxidant activity (TPA) values of herbal extracts exhibited a decreasing trend with increasing dilution ratios, and practically did not change above a given ratio depending on the type of herb.

Keywords: Prooxidant biosensor, protein oxidation, phenolics, carbonyl detection.

References:

1. Levine, R.L., Williams, J.A., Stadtman, E.R., Shacter, E., Carbonyl assays for determination of oxidatively modified proteins, *Methods Enzymol.* (1994) 233, 346-357.
2. Akyüz, E., Sözgen Başkan, K., Tütem, E., Apak, R., Novel protein-based solid-biosensor for determining pro-oxidant activity of phenolic compounds, *J. Agric. Food Chem.* (2017) 65, 5821–5830.

PP48- A Novel Gold Nanoparticles–Based Colorimetric Sensor for Antioxidant Capacity Measurement

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Reactive oxygen species (ROS) attack various biological macromolecules (proteins, enzymes, DNA, etc.) under ‘oxidative stress’ conditions to give rise to certain diseases such as cardiovascular disease and some types of cancer. The best way to combat such health risks is to balance these ROS through diet by the consumption of foods rich in antioxidants. In this work, we describe the development of a new gold nanoparticle–based colorimetric sensor for antioxidant capacity measurement.

In the present work, a novel heparin-stabilized gold nanoparticles–based CUPRAC (cupric reducing antioxidant capacity) colorimetric sensor for the measurement of total antioxidant capacity (TAC) was proposed. The electron transfer (ET)-based CUPRAC method developed in our laboratory uses Cu(II)-Cu(I) reduction in the presence of antioxidants.¹ We used a green method for the synthesis of negatively-charged gold nanoparticles solution by using heparin as a reducing and stabilizing agent. In the proposed sensor, the cationic Cu(I)-Nc chelate formed by the reaction between the CUPRAC reagent and antioxidants is electrostatically adsorbed on the surface of the oppositely-charged AuNPs. The absorbance of the resulting Cu(I)-Nc-AuNP (Cu(I)-Nc adsorbed gold nanoparticles) was measured at a wavelength of 455 nm against a reference blank without antioxidant.

A number of antioxidants were tested by the proposed colorimetric sensor so as to compare their trolox-equivalent antioxidant capacities with those found by the conventional CUPRAC assay. The proposed sensor was validated to measure the TAC values of synthetic mixtures, fruit juices and medicinal plant extracts.

Keywords: Gold nanoparticles, Heparin, CUPRAC, Colorimetric sensor, Antioxidants

References:

1. Apak, R., Güçlü, K., Özyürek, M., Karademir, S. E., Novel total antioxidant capacity index for dietary polyphenols and vitamins C and E, using their cupric ion reducing capability in the presence of neocuproine: CUPRAC method. J. Agric. Food Chem. (2004) 52 7970-7981.

PP49- Molecularly imprinted electrochemical sensor for determination of lipopeptide antibiotic drug Daptomycin

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Daptomycin (DAP) was the first approved drug from a new class of antimicrobials, the cyclic lipopeptides, and presents a broad spectrum of activity against a wide range of gram-positive bacteria. The DAP structure is composed of a peptide moiety containing 13 D- and L- amino acids, only three of these being with D-stereochemistry. Molecularly imprinted polymers (MIP) contain selective recognition sites for a target molecule in a polymer matrix: They are synthesized by polymerizing functional monomers in the presence of the template molecule which is subsequently removed. This process leaves behind cavities in the polymer which are to some extent complementary in size, shape and functionality to the template. Therefore, it is preferentially rebound even from complex mixtures [1, 2].

An electrochemical sensor based on MIP film for DAP detection at the surface of a glassy carbon electrode (GCE) was proposed in this study. The sensor was prepared by electropolymerization of o-phenylenediamine in the presence of DAP molecules using cyclic voltammetry. Several parameters affecting the performance of the imprinted (MIP) and non-imprinted films (NIP) were optimized by various factors such as monomer and template concentration, number of cycles of electropolymerization process, extraction solvent and incubation time. Under optimized operational conditions, the proposed MIP/GCE sensor exhibits linear response ranging from 5×10^{-9} M to 5×10^{-6} M with a correlation coefficient of 0.990. The stepwise preparation of the MIP and NIP was characterized electrochemically by means of cyclic and differential pulse voltammetry employing ferrocyanide/ferricyanide as a redox probe. The analytical performance of the MIP and the imprinted effect to the template molecule was compared to the NIP.

The selectivity of the MIP/GCE sensor against DAP was investigated in the presence of various interfering molecules. The proposed sensor was successfully applied for the detection of DAP in pharmaceutical forms and human serum.

Keywords: Daptomycin, Electrochemistry, Molecularly Imprinted Polymer, o-phenylenediamine

Reference:

1. Xiaochu Ding, Patricia A. Heide, Recent developments in molecularly imprinted nanoparticles by surface imprinting techniques, *Macromol. Mater. Eng.* (2014) 299 268–282
2. Aysu Yarman, Sevinc Kurbanoglu, Katharina J. Jetzschmann, Sibel A. Ozkan, Ulla Wollenberger, Frieder W. Scheller, Electrochemical MIP-Sensors for Drugs, *Current Medicinal Chemistry*. (2018), 25 1-13

PP50- Electrochemical DNA Biosensor for The Interaction of Doxorubicin with DNA

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Doxorubicin, (7S,9S)-7-[(2R,4S,5S,6S)-4-amino-5-hydroxy-6-methyloxan-2-yl]oxy-6,9,11-trihydroxy-9-(2-hydroxyacetyl)-4-methoxy-8,10-dihydro-7H-tetracene-5,12-dione, which is an antineoplastic in the anthracycline class. Doxorubicin is a topoisomerase inhibitor and an anthracycline antibiotic with antineoplastic activity. Doxorubicin intercalates between base pairs in the DNA helix, thereby preventing DNA replication and ultimately inhibiting protein synthesis. Additionally, doxorubicin inhibits topoisomerase II which results in an increased and stabilized cleavable enzyme-DNA linked complex during DNA replication and subsequently prevents the ligation of the nucleotide strand after double-strand breakage. Doxorubicin also forms oxygen free radicals resulting in cytotoxicity secondary to lipid peroxidation of cell membrane lipids; the formation of oxygen free radicals also contributes to the toxicity of the anthracycline antibiotics, namely the cardiac and cutaneous vascular effects. Drug-DNA interaction studies can give us brief information about the mechanism of the drug in the human body.

Electroanalytical methods are widely used in drug analysis, due to high sensitivity and selectivity, their low cost, reagents consumption and relatively short analysis time. They are used to measure electrical quantities such as current, potential, or charge and their relationship to chemical parameters. Subsequently the use of chemically modified electrodes as electrochemical sensors have been highly recommended, as they since the modification enhances electron transfer rate and the sensitivity of working electrode by decreasing the redox potential of analyte. Nowadays, nanomaterials are generally used in designing nanobiosensors due to their high surface area, good mechanical properties, inertness etc. Especially, silver nanoparticles (AgNPs) and platinum nanoparticles (PtNPs) are reason of choice depending on their biocompatibilities.

In this study, an electrochemical DNA-based biosensor was developed for the detection of DNA-doxorubicin interaction through the electroactive properties of guanine and adenine nucleotides. Interaction time, nanoparticle amount (AgNPs, PtNPs), modification strategy and concentration of drug in response to guanine were optimized. Layer-by-layer modification of the screen-printed electrode-based DNA biosensor gave the highest guanine response and resulted in a well-followed decrease in guanine signal after interaction with 0.5 ppm doxorubicin. With 5 min interaction time, the SPE/PtNPs/AgNPs/AgNPs DNA biosensor gave a linear response between 0.1 and 1.5 ppm doxorubicin.

Keywords: Doxorubicin, DNA biosensor, voltammetry, nanoparticles

References:

1. S.A., Ozkan, *Electroanalytical Methods in Pharmaceutical Analysis and Their Validation*. HNB Publishing, New York, 2012.

PP51- Esmolol Determination Using A Novel Electrochemical Nanosensor

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Esmolol (ESM), methyl 3-[4-[2-hydroxy-3-(propan-2-ylamino) propoxy] phenyl] propanoate, is a cardio selective beta-adrenergic blocker in class II antiarrhythmic that used in the treatment of acute supraventricular tachycardia, arrhythmias and severe hypertension. ESM decreases the force and rate of heart contractions by blocking beta-adrenergic receptors of the sympathetic nervous system, which are found in the heart and other organs of the body. ESM prevents the action of two naturally occurring substances: epinephrine and norepinephrine. The overdose of ESM can cause both cardiac and central nervous system side effects such as bradycardia, cardiac arrest, respiratory depression, mood disturbance and coma. For this reason, it is very important to determine ESM levels in biological fluids such as human serum, urine and pharmaceutical preparations.

Electroanalytical methods emerge with the interplay between electricity and chemistry; in other words they were used to measure electrical quantities with the chemical parameters. These methods are widely used in fields like environmental monitoring, industrial quality control or biomedical analysis . Another common field in which electrochemical methods are extensively used in drug analysis and these methods have proved to be highly sensitive due to the straight forwardness, low cost and relatively short analysis time. Carbon based electrodes are preferred substrates for electroanalytical studies. Especially, glassy carbon electrode is the most common carbon-based electrode because of its excellent mechanical and electrical properties, wide potential range, chemically inert nature and impermeability to gases. They are easily mounted, polishable and compatible with all common solvents. They allow many applications in many different areas, since their performances are relatively reproducible. Nanomaterials can increase the surface of the transducing area of the substrates that in turn increases catalytic behaviors.

In this study, the electrochemical behavior of ESM was investigated in a wide pH range using square wave voltammetric technique at glassy carbon electrode. Maximum current was observed in the pH 6.0 acetate buffer by square wave voltammetry (SWV). As a result of scan rate studies, diffusion-controlled mechanism was occurred. Interference and modification effects were investigated. The optimum effect of modification was obtained by sodium dodecyl sulphate and platinum nanoparticles. A linear relationship was plotted between ESM concentrations and the current response. The proposed method has been extensively validated according to ICH Guidelines. The developed electro-chemical method is also used for the determination of ESM in spiked human serum.

Keywords: Esmolol, Nanosensor, Square Wave Voltammetry, Nanoparticles

References:

1. Alarfaj NA, El-Tohamy MF (2016) Electrochemical System for Determination of Esmolol Hydrochloride Using Square Wave Adsorptive Stripping Voltammetry. *Int. J. Electrochem. Sci*, 11, 2709-2720.
2. Ozkan S.A., *Electroanalytical Methods in Pharmaceutical Analysis and Their Validation*. HNB Publishing, New York, 2012.

PP52- Simultaneous Estimation of Olmesartan, Amlodipine, and Hydrochlorothiazide in Their Ternary Mixture by HPLC

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Hypertension is an important problem that requires chronic treatment. Angiotensin-converting enzyme (ACE) inhibitors were primarily considered as antihypertensive drugs able to reduce significantly high blood pressures in hypertensive patients. Ternary tablet formulations containing the Amlodipine (AML) belongs to the dihydropyridine class of calcium channel blockers. Angiotensin II receptor antagonist Olmesartan (OLM) and the diuretic hydrochlorothiazide (HCT) are used in the therapy to treat high blood pressure.

The aim of this research is optimization and development of a simple, rapid, economical, sufficiently precise and accurate, reproducible, and fully validated HPLC method with good detection limits for estimation ternary combination of active pharmaceutical ingredients in bulk form and pharmaceutical preparations. Validation were also performed according to International Council on Harmonization (ICH) Guidelines and The United States Pharmacopeia (USP).

In order to achieve this goal, various mobile phase systems, buffer types, and buffer concentrations were prepared to provide an appropriate separation. Different types of reverse phase columns were tested to find the best and efficient resolution with the optimum resolution of the studied compounds. In the present work, Symmetry Shield RP-18 (150 mm x 4.6 mm x 3.5 μ m) used for the separation of the ternary mixture with the 1 mL/min flow rate at 25 °C.

Finally, the proposed method provides a simple procedure for the simultaneous analysis of the compounds in their pharmaceutical dosage form by DAD detection. Developed method is fully validated from the view point of selectivity, sensitivity, precision, accuracy, linearity, limit of detection and quantification.

Keywords: HPLC, separation, validation, amlodipine, hydrochlorothiazide, olmesartan

PP53-Electrochemical Determination of Folic Acid by Using of Polypyrrole Based Modified Electrode

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Folic acid (FA) known as ((2S)-2-[(4-[(2-amino-4-hydroxypteridin-6-yl)methyl]amino}phe-nyl)formamido]pentanedioic acid) and B9 vitamin, is a curial vitamin for human. This chemical substance plays important roles in some biochemical process of human body such as division and growth of the cell, synthesize the nucleotides, prevent neural tube defects in the fetus (for pregnant), and etc. [1, 2]. When it is in the absence of the body, some diseases like cancer, megaloblastic anaemia, heart diseases and neural tube defects in developing fetuses [3]. Since it has many effects on the health of human, sensitive determination of the FA has great importance and taken attention by many studies of the researcher [3]. Electrochemical methods has many advantages such as low cost, sensitive, fast and simple detection of FA [3]. Preparation of selective, easy preparing and low cost electrodes are still necessary for electrochemical detection of FA.

In this work, we have prepared a novel polypyrrole based modified electrode for sensitive electrochemical detection of FA. The prepared novel electrodes were characterized by electrochemical methods such as cyclic voltammetry and electrochemical impedance spectroscopy. Structural features of PGE during the modification process was investigated by scanning electron microscopic analysis. Prepared electrodes were used as electrochemical sensor for FA with differential pulse voltammetric analysis. The analytical response of polypyrrole based modified electrode was higher than that of PGE (Fig. 1). This novel electrode can be used for sensitive electrochemical determination of FA.

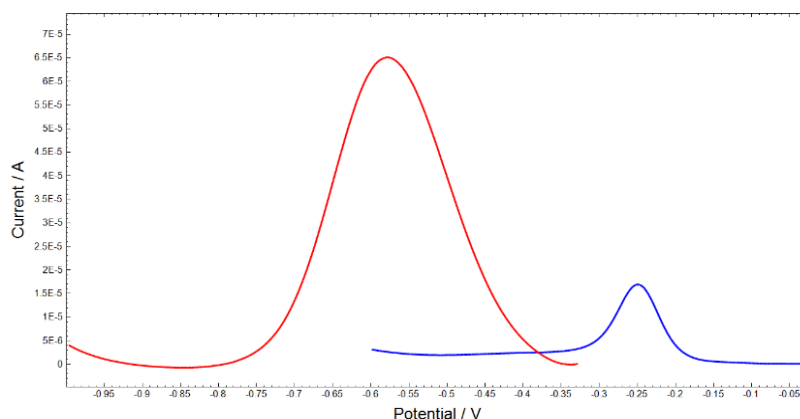


Fig.1 Differential pulse voltammograms of PGE (blue) and polypyrrole based modified PGE (red) in the 1 M H₂SO₄ solution consisting 0.001 M of Folic Acid (FA)

Keywords: Differential pulse voltammetry, polypyrrole, folic acid, sensor

References:

1. F. Wang, M. Cao, N. Wang, N. Muhammad, S. Wu, Y. Zhu. Simple coupled ultrahigh performance liquid chromatography and ion chromatography technique for simultaneous determination of folic acid and inorganic anions in folic acid tablets. *Food Chemistry* 239 (2018) 62–67.
2. H.X. Guo, Y.Q. Li, L.F. Fan, X.Q. Wu, M.D. Guo. Voltammetric behavior study of folic acid at phosphomolybdic-polypyrrole film modified electrode *Electrochimica Acta*, 51 (2006) 6230-6237.
3. S. Akbar, A. Anwar, Q. Kanwal. Electrochemical determination of folic acid: A short review. *Analytical Biochemistry* 510 (2016) 98-105.

PP54- Sensitive Voltammetric Detection of L-tyrosine by Using of Over Oxidized Polypyrrole Modified Pencil Graphite Electrode

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Tyrosine (Tyr, 4-hydroxyphenylalanine) is an aromatic structure amino acid that arranges signal transduction of proteins. Tyrosine is one of the secondary amino acids used by the body for important functions and processes [1]. It facilitates the regulation of the emotional state and provides the necessary stimulation for correct functioning of the nervous system. Tyr is forerunner of dopamine, norepinephrine, adrenaline, melanin, thyroid hormones, and catecholamine [2]. Excess amount of Tyr is reason of serious health problems such as hyperthyroidism and sister chromatid exchange. The absence of Try could cause hypochondrium, depression, and other psychological diseases. For this reason, it is very important to determine the efficient level of Tyr in the diagnosis and treatment of diseases [3]. Electrochemical methods have many advantages such as relatively low cost, fast response, and high applicability.

In this study, a tyrosine sensor was prepared using of over oxidized polypyrrole modified pencil graphite electrode (PGE). These electrodes were characterized by cyclic voltammetry and electrochemical impedance spectroscopy. Structural changes of the surface morphologies of modified and unmodified electrodes were investigated by scanning electron microscope. The sensor properties of the electrodes were investigated by differential pulse voltammetry. The analytical response of the modified electrode was higher than that of unmodified PGE electrodes.

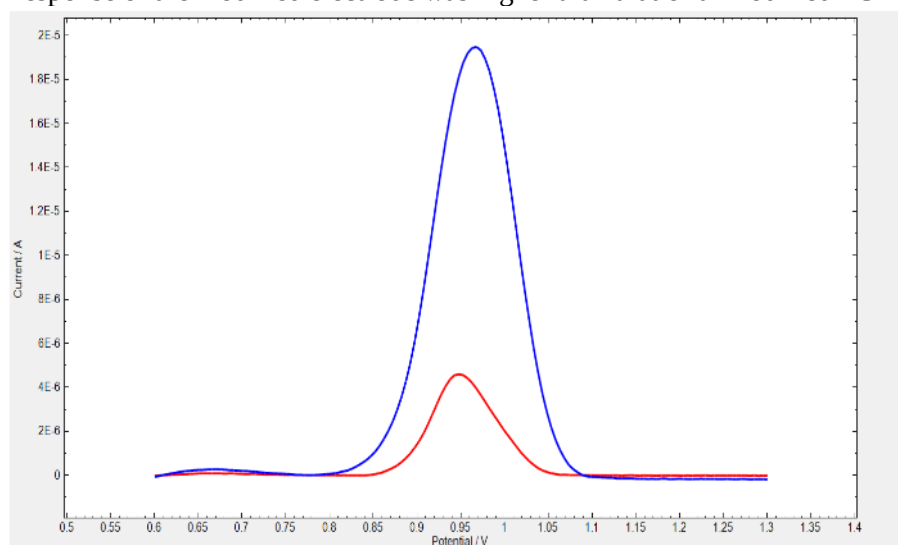


Fig.1 Differential pulse voltammograms of PGE (red) and over oxidized polypyrrole modified PGE (blue) in the 1 M H₂SO₄ solution consisting 10⁻³M of Tyrosine (Tyr)

Keywords: Polypyrrole, tyrosine, sensor, voltammetry

References:

1. JP Jin, XQ Lin, The electrochemical behavior and amperometric determination of tyrosine and tryptophan at a glassy carbon electrode modified with butyrylcholine, *Electrochem. Commun.* 2004,6,454
2. Sayed Mehdi Ghoreishi, Mohsen Behpour, Mona Delshad, Asma Khoobi Electrochemical determination of tyrosine in the presence of uric acid at a carbon paste electrode modified with multi-walled carbon nanotubes enhanced by sodium dodecyl sulfate *Central European Journal of Chemistry* 2012,10,6, pp 1824–1829
3. Junhua Weil, Jingjing Qiu, Li Li, Liqiang Ren, Xianwen Zhang, Jharna Chaudhuri and Shiren Wang A reduced graphene oxide based electrochemical biosensor for tyrosine detection *Nanotechnology* 23 (2012) 7

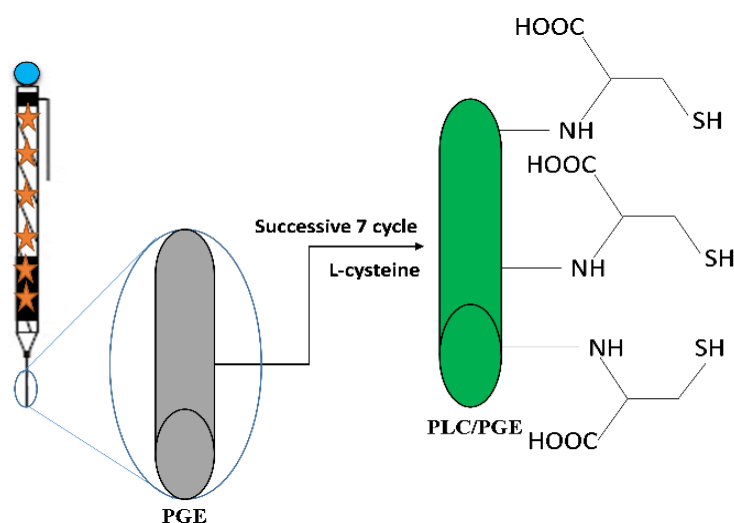
PP55- Poly(L-Cysteine) Modified Pencil Graphite Electrode for Determination of Sunset Yellow in Food and Beverage Samples by Differential Pulse Voltammetry

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Synthetic food colorant has been commonly used to replace natural food colour in food production industry because of excellent colour uniformity, high stability during preparation processes and low-cost production. Synthetic colorants widely contain aromatic ring structures and azo (N=N) functional group, which may able to adversely affect human health. Sunset Yellow (E110) (SY), is a synthetic azo dye which is widely used in beverages, foods, cosmetics, medicines and colorings, although it can cause migraines, eczema, allergies, diarrhea, anxiety and cancer when over-consumed [1].



In this study, we have focused on the use of a poly(L-cysteine) (PLC) modified pencil graphite electrode (PGE) as a sensor for determination of Sunset Yellow (SY). The performance of the modified electrode was studied using cyclic voltammetric and differential pulse voltammetric method. The modified electrode was characterized by electrochemical impedance spectroscopy, cyclic voltammetry and Fourier transform infrared spectroscopy. The surface of the modified electrode was examined by scanning electron microscope. The electrochemical behavior of Sunset Yellow in phosphate buffer solution (pH 7.0) was investigated using unmodified PGE and PLC modified PGE (PLC/PGE). The results showed that the electrochemical response of PLC/PGE to Sunset Yellow was significantly improved. The PLC/PGE showed linear responses in the electrochemical oxidation of SY between the concentration values of 1.0 μM and 1000 μM . The sensor (PLC/PGE) showed a good response for SY with a detection limit of 0.125 μM (S/N=3). Analytical application of PLC/PGE was successfully tested in the determination of SY in food and beverage samples.

Keywords: Sunset Yellow, Poly(L-cysteine), Pencil Graphite Electrode, Food Additive

References:

1. Ozge Koyun, Yucel Sahin, Poly(L-Cysteine) Modified Pencil Graphite Electrode for Determination of Sunset Yellow in Food and Beverage Samples by Differential Pulse Voltammetry, *Int. J. Electrochem. Sci.*, (2018) 13 159-174.

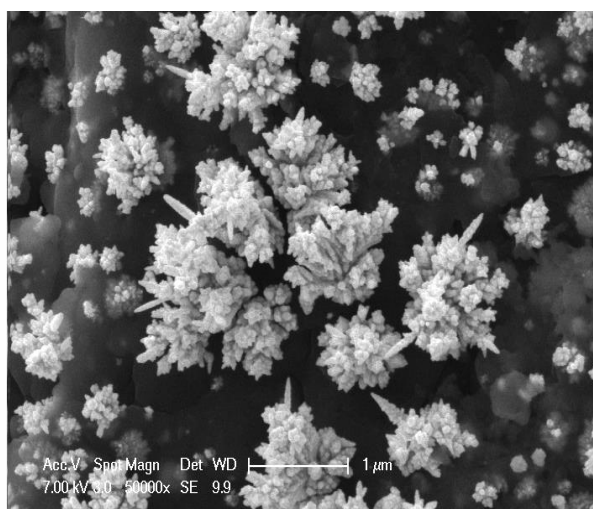
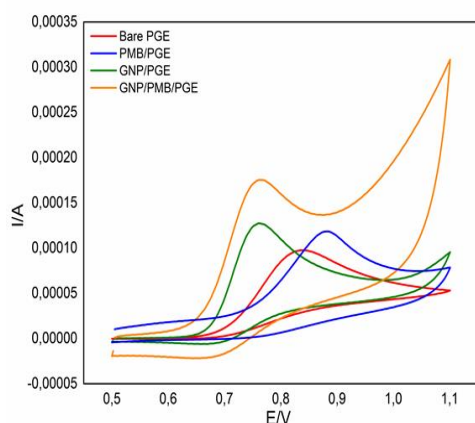
PP56- Voltammetric Determination of Nitrite with Gold Nanoparticles/Poly (Methylene Blue) Modified Pencil Graphite Electrode: Application in Food and Water Samples

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Nitrite is naturally occurring a chemical compound which is commonly used as an additive and a corrosion inhibitor in the preservation of food and environmental samples. Its application areas show range from dye manufacture to food preservation as versatile chemical agent. Nitrite is commonly used in food preservation (E249, E250) and it is possible to find some forms of nitrite in many food products as salt. However, the continuous ingestion of its ions causes to harm human and animal health [1].



This work presents the preparation and characterization studies of gold nanoparticle/poly(methylene blue) (GNP/PMB) modified pencil graphite electrode (PGE) and its use for electrochemical determination of nitrite. The working electrode (PGE) was functionalized by electropolymerization of methylene blue (MB) and modified with gold nanoparticles (GNPs) to produce the final (GNP/PMB/PGE) electrode in a two-step procedure. Characterization and morphology studies of GNP/PMB/PGE were performed by electrochemical impedance spectroscopy, cyclic voltammetry, X-ray photoelectron spectroscopy, Fourier transform Infrared spectroscopy and scanning electron microscopy techniques. The highest peak current response was obtained for nitrite at pH 5.0. A wide linear relationship between the concentration and oxidation peak currents of nitrite was observed in the concentration range of 5-5000 μM. The limit of detection (0.314 μM, S/N=3) and reproducibility (RSD=2.38% for N=10) have been identified. Finally, the electrode platform was successfully applied to commercial sausage and mineral water samples.

Keywords: Sodium Nitrite, Poly(Methylene Blue), Gold Nanoparticle, Food Additive

References:

1. Ozge Koyun, Yucel Sahin, Voltammetric determination of nitrite with gold nanoparticles/poly(methylene blue) modified pencil graphite electrode: application in food and water samples, Ionics., (2018) 1 1-11.

PP57- Supercritical fluid extraction and Determination of Phytol and Neophytadiene Content in *Nicotiana tabacum* L. cv. Samsun Leaves

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Related with medicinal value, phytol has antinociceptive and antioxidant activities, as well as anti-inflammatory and immunostimulatory effects. Similarly, neophytadiene is a good analgesic, antipyretic, anti-inflammatory, antimicrobial, and antioxidant compound.

In this study, *Nicotiana tabacum* L. cv. Samsun leaves grown on the Black Sea coast, Batumi, Adjara region, Georgia, were collected in August 2016. Supercritical fluid extraction with CO₂ was used for the phytol and neophytadiene extraction from *Nicotiana tabacum* L. cv. Samsun leaves. The extraction was carried out on a lab-made apparatus at two different pressures 15 MPa and 30 MPa, T =40°C and CO₂ flow rate 2kg h⁻¹. The extract obtained was analysed by gas chromatography (GC) and chromatography–mass spectrometry (GC–MS). In the extract, obtained at the 15MPa, the phytol content was 0.80% and neophytadiene was present in the percent of 14.33%. In the extract obtained at 30MPa pressure applied, the phytol content was 0.53% and neophytadiene was detected in 12.40%. The effect of different extraction parameters: pressure, temperature and extraction time on supercritical fluid extraction of phytol and neophytadiene from *Nicotiana tabacum* L. cv. Samsun leaves is still under investigation.

Keywords: *Nicotiana tabacum*, supercritical fluid extraction, GC-MS, phytol

PP58- The Effect of Extraction Process on Aroma Compounds in Virgin Olive Oils

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Virgin olive oil is a primary part of diet in daily life. Olive oil is produced from the fruit by means of mechanical or physical methods and includes triglycerides as the major component. Minor components are phenolic compounds, sterols, tocopherols, phospholipids, hydrocarbons and volatile compounds [1]. Among them volatile compounds are responsible for sensorial acceptability and correlated with the quality of olive oil. Volatile aroma compounds are included at variable concentrations within the extracted virgin oil depending on the maturity, variety of olive and cultivation, even the altitude of orchards of olive trees [2]. There is another parameter affecting the concentration of aroma compounds in olive oil which is the type of extracting methods employed for extracting the olive oil such as hummer mill, disc mill, stone mill, pitter mill [3] even traditional rarely experienced technique squeezed olives by foot in rural areas.

Present study describes a GC-MS method for the determination of aroma components of olive oil samples collected from Akhisar where the dominant type of olives is memecik varieties. The oil samples have been extracted with several techniques and the type and the percentages of the aroma compounds have been analyzed by using a chemometric approach. Overall results have shown that composition of the oil is immensely affected by the extraction technique used.

Keywords: Olive Oil, Aroma Compounds, Chemometrics, Principal Component Analysis (PCA)

References:

1. S. Cevik, G. Ozkan, M. Kiralan, Optimization of Malaxation Process using Major Aroma Compounds in Virgin Olive Oil, Brazilian Archives of Biology and Technology 59 (2016)
2. C. Toker, U. Aksoy, H. Ertas, The Effect Of Fruit Ripening, Altitude and Harvest Year on Volatile Compounds of Virgin Olive Oil Obtained from The Ayvalik Variety, Flavour and Fragrance Journal Vol 31, Issue 3, (2016)195-205
3. <http://cesonoma.ucanr.edu/files/27187.pdf>

PP59- Studies on retention behaviour of Sulfonylureas: Prediction of the Solute Retention in Liquid Chromatography over a Wide Range of Mobile-Phase Compositions

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Retention in reversed phase liquid chromatography (RPLC) is determined by the difference in various of interactions which a solute can undergo in the mobile and stationary phase. Moreover it is known that solute descriptors plays an active role in the separation process. Many papers investigate the influence of different mobile-phase compositions on retention behavior in RPLC from a view of the linear solvation energy relationship (LSER). LSER theory describes the retention on the basis of selective solute-solvent interactions[1]. Sulfonylurea derivatives (gliclazide, glipizide, glibenclamide, glimepiride) are commonly prescribed hypoglycemic drugs for the treatment of non-insulin-dependent diabetes mellitus when diet modification has not been sufficient to achieve an appropriate control. The sulfonylureas are described as lipophilic drugs. These compounds possess higher number of proton acceptor and proton donor groups [2]. Gliclazide, glimepiride, glipizide, glibenclamide, contain an acidic function (N-H group of the sulfonamide moiety). The pKa values of sulfonylurea moiety in the hypoglycemic drugs studied are in the range of 5.2–6.2 and are characterized, as weak organic acids.

The retention times seem thus to be a valuable tool for characterizing the structure/response relationships observed in the RPLC analysis of the compounds. The applicability of a LSER model to prediction of the retention behavior of sulfonylureas in RP-HPLC mobile phases has been studied. The effect of the mobile-phase composition was studied using THF –water mobile phases containing 40%, 45%, 50%, 55% and 60% (v/v) of organic modifier. Tetrahydrofuran (THF) is one of the most widely used dipolar aprotic solvents [1], being employed as a reaction medium in liquid chromatography. Its relatively weak solvating power of many polar solutes. Experimental studies were carried out using special THF column (YMC J'sphere ODS-H80; 4 µm, 150 mm x 4,6 mm ID) to obtain appropriate interaction between analyte and column. The classical five term LSER equation have been applied to retention data evaluation. Dependences of regression coefficients on the composition of the THF-water mobile phase were investigated. The importance of regression coefficients in the THF–water mobile phase were also evaluated. The influence of the THF content of the mobile phase on the chromatographic retention of a series of structurally similar sulfonylureas were investigated.

Keywords: Quinolones, Retention behavior, RP-HPLC, Aqueous pKa

References:

1. Remko, M., Theoretical Study of Molecular Structure, pKa, Lipophilicity, Solubility, Absorption, and Polar Surface Area of Some Hypoglycemic Agents. *Journal of Molecular Structure Theochem*, 2009, 897, 73–82.
2. Jirkal Š., Machovcová M. and Ševčík J.G.K., Application of Solvation Model to Prediction of the Solute Retention in Liquid Chromatography over a Wide Range of Mobile-Phase Compositions. *Acta Chromatographica* 2016, 28, 1, 59–78.

PP60- Structure-antioxidant activities relationship of some amino acid-benzothiazole conjugates†

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Benzothiazole belongs to the family of bicyclic heterocyclic compounds having benzene nucleus fused with five-membered 1,3-thiazole moiety.¹ Benzothiazole is an important scaffold for drug development, because it has exhibited a broad spectrum of pharmacological activities. As part of our continuing interest in the screening of antioxidant properties of heterocyclic compounds,² we wish to explore the possible link with the substituents at the benzothiazole scaffold and antioxidant properties. For this purpose, we compared the antioxidant activities of the substituents at the both amino acid and benzothiazole part of the some amino acid-benzothiazole conjugates.

Among the tested compounds seen below, the compounds bearing ethoxy group at 6 position of the benzothiazole ring was found to be the most effective antioxidant at 62.5 µg/mL and 125 µg/mL concentrations with 32% and 38%. The compounds bearing methylsulfonyl at 6 position of the benzothiazole ring was found to be next effective antioxidant at 62.5 µg/mL and 125 µg/mL concentrations with 22 % and 23 %. With respect to the amino acid part of the compounds, glycine and phenylalanine derivatives seem to be more active for the antioxidant activities.

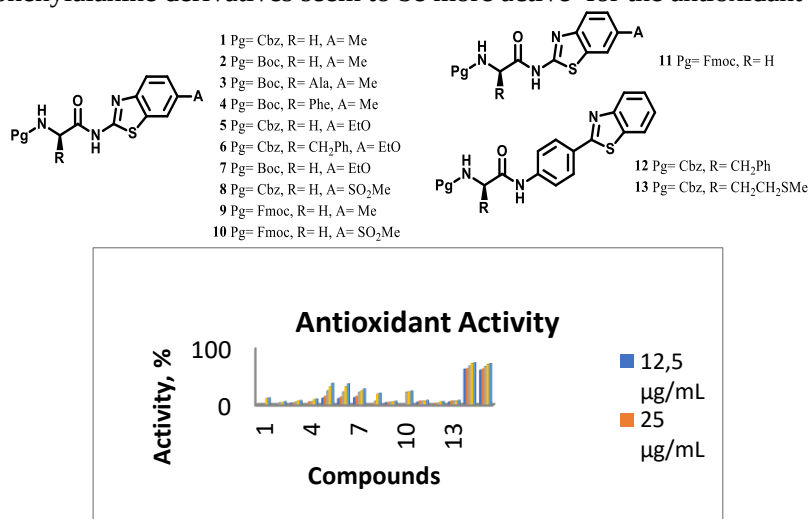


Figure. The chemical structure and antioxidant activity of different concentrations of compounds, BHA and α -tocopherol by 1,1-phenyl-2-picrylhydrazyl radicals.

Keywords: Antioxidants, benzothiazole, amino acid.

References:

- Gill, R. K., Rawal, R. K., Bariwal J. Recent advances in the chemistry and biology of benzothiazoles. Arch. Pharm. (2015) 348, 155-178.
- Buğday, N., Küçükbay, F. Z., Apohan, E., Küçükbay, H., Serindağ, A., Yeşilada, Ö. Lett. Org. Chem. (2017) 14, 198-206.

† This work was financially supported by the İnönü University Research Fund (BAPB-2016/94)

PP61- Determination of Dexmedetomidine by Ultra Performance Liquid Chromatography

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Dexmedetomidine, approved by the Food and Drug Administration (FDA) in 1999 as a sedative for use in the intensive care unit, is a potent and highly selective α_2 -adrenoceptor agonist with significant sedative, analgesic and anxiolytic effects mostly (1-2).

In this study, ultra-performance liquid chromatography (UPLC) method has been developed for the determination of dexmedetomidine and related substances in dexmedetomidine hydrochloride injection preparations, as there is no official monograph or analytical method by UPLC. The method was developed in Waters UPLC system and on Aquity UPLC BEH C18 (2.1 x 50 mm, 1.7 μ m) column with an isocratic elution at a flow rate of 0.2 mL/min. using a mobile phase of 60-40% of methanol and phosphate buffer. The detection was performed with an UV detector. The method was validated for specificity, linearity, accuracy, precision, robustness, also at the same time subjected to different stress conditions like acid, base, peroxide, thermal, photostability studies and checked for its specificity and degradation. The limit of detection and quantification for dexmedetomidine was found 0.0063 and 0.021 μ g/mL, respectively. The overall mean recoveries of Dexmedetomidine were 99.6% for Limit of Quantitation and 99.0 % for 130% (overall % RSD not more than 2.0 %). Developed method is simple, reproducible, and robust and can be used safely routine analysis of dexmedetomidine in dexmedetomidine hydrochloride injection.

Keywords: Dexmedetomidine, UPLC, pharmaceutical preparation.

References:

1. Li C, Qu J., Efficacy of dexmedetomidine for pain management in knee arthroscopy: A systematic review and meta-analysis. *Medicine* (2017) 96, 1-6.
2. Hetland B, Hayes S.M, Skaar D, Tracy M.F, Weinert C.R, Chlan L., Letting the Patient Decide: A Case Report of Self-Administered Sedation During Mechanical Ventilation. *Crit. Care Nurse*. (2018) 38, 17-23.

PP62- Omega 3 fatty acid analysis in Analytical Chemistry

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Omega 3 fatty acids; eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) have an important role in human health for preventing heart diseases (1), hypertension, depression, loss weight and even cancer. In the literature, several analytical methods have been developed for omega 3 fatty acids derivatization and chromatographic separation including statistical process by GC (2) and also determining in biological material after the lipid extraction.

In this study, details of omega 3 fatty acid analysis are given and discussed with each other.

Keywords: Omega 3 fatty acids, EPA, DHA, Gas chromatography.

References:

1. Mehta J., Lopez L.M., Wargowich T., Eicosapentaenoic acid: Its relevance in atherosclerosis and coronary artery disease, *Amer. J. Cardiol.* (1987) 59 155-159.
2. Saglik S., Imre S., Omega-3 fatty acids in some fish species from Turkey, *J. Food Sci.* (2001) 66 210-212.

PP63- Development of UHPLC-FLD method for the determination of T-2 and HT-2 toxins in rat plasma and brain microdialysate samples

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T-2 toxin and its metabolite HT-2 toxin are trichothecene mycotoxins produced by various *Fusarium* species. They have been found in several foods such as wheat, maize, oats, barley, rice, beans, soybean, corn flakes, flour, infant foods, malt, and beer. Due to adverse health effects of T-2 and HT-2 toxins, their analyses are very important. In this study, UHPLC-FLD method was developed for the determination of T-2 and HT-2 toxins in rat plasma and brain microdialysate samples. Intraperitoneal dose of 3 mg kg⁻¹ T-2 toxin was administered as a single dose to the rats. T-2 toxin and HT-2 toxin were derivatized with 4- (dimethylamino) pyridine and 1-antroylnitrile to give fluorescence properties and determined at λ_{ex} : 381 nm and λ_{em} : 470 nm. The analysis was performed on a Poroshell 120 EC-C18 column (3.00 mm x 100 mm x 2.7 μ m) using gradient elution with the mobile phase of acetonitrile: water (65:35; v:v). The flow rate and injection volume were 0.6 mL min⁻¹ and 10 μ L, respectively. The UHPLC-FLD method has been successfully applied for the detection of T-2 and HT-2 toxins in rat biological samples.

Keywords: T-2 toxin, HT-2 toxin, method development, UHPLC

References:

1. Visconti, A., Lattanzio, V.M.T., Pascale, M., Haidukowski, M., Analysis of T-2 and HT-2 toxins in cereal grains by immunoaffinity clean-up and liquid chromatography with fluorescence detection, *J. Chromatogr. A* (2005) 1075 151-158.
2. Lippolis, V., Pascale, M., Maragos, C.M., Visconti, A., Improvement of detection sensitivity of T-2 and HT-2 toxins using different fluorescent labeling reagents by high-performance liquid chromatography, *Talanta* (2008) 74 1476-1483.

PP64- Surface Enhanced Raman Spectroscopic Detection of Heroin

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Surface Enhanced Raman Spectroscopy (SERS) use in forensic area is becoming widespread especially in analysis of illicit drugs. Heroin still takes place among the most commonly abused illicit drugs. However, there are still few studies about SERS-based identification of heroin which are controversial.

This study aims to assess SERS spectra of heroin in the most accurate way. Heroin, in acetonitrile solution, in concentration of 1 mg/mL was used as sample solution. Heroin solution was dropped as 2 µL on to Raman active Au surface. After an incubation period of five minutes, 2 µL of colloidal Ag nanoparticles was dropped on sample. Raman spectra gathered by a Raman Spectroscope with 785 nm laser source. Major peaks of heroin were determined as 407, 478, 913, 1094, 1119 and 1341 cm⁻¹, respectively. Gathered spectra of heroin have been compared with those given in related literature.

The present study suggests that most of the studies in the literature have found out SERS spectra of heroin partially and in low intensities. Our method which is described in this study has more accurate peaks with higher intensities when compared to related studies in the literature.

Keywords: Forensic toxicology, SERS, drugs of abuse, heroin

PP65- Anthocyanin profile of wild grapes grown naturally in Tunceli province

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The anthocyanins are compounds in flavonoid group. They are given red, purple, orange and blue colors to many fruits, vegetables and plants. These compounds are being investigated in various food due to they are used as natural food coloring and are effective against many chronic diseases [1]. For this reason, it is significant to identification of these compounds in various foods. In this novel study, the anthocyanin profile of the wild grapes grown naturally in the Kolonkaya village bounded to the Pertek county of Tunceli province was determined. The determination of anthocyanin profiles in samples was carried out by high-performance liquid chromatography-diode-detector (HPLC-DAD) using optimum conditions determined for the analysis of these compounds. The compounds of anthocyanins investigated in samples; delphinidin, cyanidin, pelargonidin and malvidin and theirs 3-glucoside forms. Extraction parameters (solvent type, extraction time and solid:liquid ratio etc.) were examined in determining the anthocyanin profile of the samples. The concentrations of anthocyanin of wild grapes were determined in the best conditions.

Keywords: Anthocyanins, wild grape, extraction.

References

1. Navas, M.J., Jiménez-Moreno, A.M., Bueno, J.M., Sáez-Plaza, P., Asuero, A.G., Analysis and Antioxidant Capacity of Anthocyanin Pigments. Part IV: Extraction of Anthocyanins, Critic Rev Anal Chem (2012) 42 313-342.

PP66- Studies on Log $P_{o/w}$ of Quinolones: A Comparison of RP-HPLC Experimental and Predictive Approaches

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Quinolone drugs are zwitterionic compounds which are a family of antibacterial agents extensively used in human and veterinary clinics[1]. Among pharmacokinetic properties of these drugs, lipophilicity has great attention because of penetration of quinolones into bacteria, intestinal absorption and membrane permeability are related to their lipophilicity.

The logarithm of n-octanol/water partition coefficient (log $P_{o/w}$) is the most frequently used parameter for measuring as it has been shown that this partition system is a good model for many biological processes. In fact, log $P_{o/w}$ is also used as one of the standard properties identified by Lipinski in the “rule of 5” for drug-like molecules [2]. HPLC provides an easy, reliable and accurate way to determine the partition properties of compounds based on their chromatographic retention times [3,4].

In this paper, the viability of the RP-HPLC method to measure the partition/distribution coefficients of some quinolones is studied. In the present study, the retention behavior of some quinolones were investigated with reversed phase chromatography. The combined effect of the mobile-phase composition/pH was studied using acetonitrile-water mobile phases containing 10, 15, 20% (v/v) of organic modifier. The aqueous pK_a values were calculated from these experimental values with Yasuda-Shedlovsky extrapolation equation. In order to judge if the experimentally measured log t_r can be used to predict the log $P_{o/w}$ value, a calibration curve was created with the retention times of the reference compounds and a linear regression equation between the log $P_{o/w}$ and log t_r was determined. Moreover, the capability of different predictive methods is evaluated. Consequently, the results show that the determination of log $P_{o/w}$ for quinolones can be successfully carried out by measuring the retention times based on the simple RP-HPLC method.

Keywords: Quinolones, Retention behavior, RP-HPLC, Aqueous pK_a

References:

1. G. Sarközy, Quinolones: a class of antimicrobial agents, Vet. Med. Czech, 2001, 46, 257-274.
2. Christopher A. Lipinski, Franco Lombardo Beryl W. Dominy, Paul J. Feeney, Experimental and computational approaches to estimate solubility and permeability in drug discovery and development settings, Adv. Drug. Deliv. Rev. 2001,46, 3-26.
3. Salwa K. Poole, Colin F. Poole, Separation methods for estimating octanol-water partition coefficients, J. Chromatogr. B, 2003, 797, 3-19.
4. Stephen F. Donovan, Mark C. Pescatore, Method for measuring the logarithm of the octanol–water partition coefficient by using short octadecyl–poly(vinyl alcohol) high-performance liquid chromatography columns, J. Chromatogr. A, 2002, 952, 47-61.

PP67- Evaluation of Some Physicochemical Parameters for Phenytoin**Abdullah Cengiz, Hülya Ortak, İkbâl Demet Nane, Güleren Alsancak, Ebru Çubuk Demiralay**

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Phenytoin (5,5-diphenylhydantoin) is one of the oldest non-sedative antiepileptic drugs, which is employed in cases of generalized tonic-clonic seizures and focal motor seizures. The physicochemical parameters such as lipophilicity ($\log P$), solubility ($\log S$) and dissociation constant (pK_a) of an active pharmaceutical ingredient belong to crucial properties used to estimate the absorption, distribution, metabolism, and excretion (ADME) of a compound in biological systems as well as quantitative structure-activity and structure-property relationship (QSAR/QSPR) profiling [1]. The aim of the present work is to determine the influence of acetonitrile (ACN) on the retention times and pK_a value of phenytoin, and to quantify the shift in pK_a between water and ACN. In this study the relationship between retention time and pH in acetonitrile-water mixtures (35, 40 and 50% (v/v) ACN) were evaluated. The example of the dependence of the retention time (t_R) on the pH value in the mobile phase (35% v/v) is given in Fig. 1. As can be seen, there are important effect of the eluent pH on the shape of sigmoidal. The data evaluation was performed by using NLREG software. The pK_a value of this compound is graphically determined at the inflexion point of the sigmoidal curve. A change in acetonitrile concentration also effects the shape of sigmoidal.

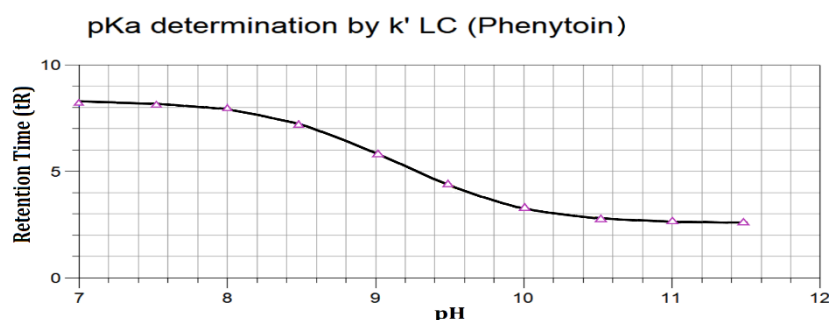


Figure 1. Plots of the retention factors versus the pH of the mobile phase for 35% v/v acetonitrile. The solid lines indicate the predicted retention time.

Lipophilicity is also key property of drugs which significantly contributes to understand and model drug–target interactions. Both pharmacokinetic and pharmacodynamic behaviours of the drug are influenced by the lipophilicity, it is clear that its proper determination plays a major role in drug discovery. The Abraham solvation parameters provide a very convenient means to predict solute transfer properties for organic compounds having known solute descriptor values. In the study, $\log P_{ow}$ and $\log S$ values of phenytoin were estimated using these descriptors.

Keywords: Epilepsy, Dissociation constant, RPLC, Solute descriptors

References:

1. Pavlina Marvanova, Tereza Padrtova, Klara Odehnalova, Ondrej Hosik, Michal Oravec, Petr Mokry, Synthesis and Determination of Physicochemical Properties of New 3-(4-Arylpiperazin-1-yl)-2-hydroxypropyl 4-Alkoxyethoxybenzoates, *Molecules* 2016, 21, 2-13.

PP68- Determination of Heavy Metals in Different Brands of Spices by ICP-MS

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Spices can be in the form of buds (cloves), bark (cinnamon), roots (ginger), berries (peppercorns), aromatic seeds (cumin), and even the stigma of a flower (saffron). They impart flavor and reduce the need for salt and fatty condiments. However, besides adding to the taste, spices have functions that include combating foodborne microorganisms and reducing food poisoning; they also have good antioxidant, antimicrobial, anti-inflammatory, antidiabetic, antihypertensive and anti-carcinogenic properties. Spices are the storehouse of several elements over a wide range of concentrations with important positive or negative health impacts. Among techniques used for the determination of trace elements in spices, inductively coupled plasma-mass spectrometry (ICP-MS), which has high sensitivity, a wide linear dynamic range, low detection limits, and quickness of measurement, is a reliable and effective method for multi-element analysis.

In the study, the concentrations of Cr, Mn, Fe, Co, Ni, As, Cd, Pb and Zn in nineteen different spices from eleven different brands (in total 69 samples) collected from Kayseri, Turkey were determined by ICP-MS after microwave digestion. In general, the mean metal concentrations in the spices were decreased in the following sequence: Fe>Mn>Zn>Ni>Cr>Co>Pb>As>Cd. The toxic element concentrations in spices were compared with their maximum tolerable concentration limits in plants. The concentrations of Pb did not exceed the permitted level in any of the samples. The concentrations of As and Cd elements in all the studied spices were found to be below their maximum permissible levels, except for two brands of ginger. Ginger is a rhizome and in general, the roots contain the highest levels of heavy metals.

Keywords: Different type and brand spices, microwave digestion, ICP-MS, trace elements.

References:

1. M Viuda-Martos, Y Ruiz-Navajas, J Fernández-López, JA Pérez-Álvarez, Spices as functional foods, Crit Rev Food Sci Nutr (2011) 51 13–28.
2. LC Tapsell, I Hemphill, L Cobiac, DR Sullivan, M Fenech, Health benefits of herbs and spices: the past, the present, the future. Med J Aust (2006) 185 S1-S24.

PP69- Activated Carbon@Fe_xMn_yO_z Composite as Adsorbent for Vortex-Assisted Dispersive Solid Phase Extraction of Copper and Lead

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Activated carbon (AC) has been widely used for removal of a wide variety of pollutants because of its high adsorption capacity, large surface area, high surface reactivity degree and rich internal microporous structure. AC is defined as a hydrophobic solid material which adsorbs non-polar or slightly polar substances in aqueous solutions and can be made highly selective and specific to any analyte of interest by surface modification with metal oxides/hydroxides. This modification can significantly increase the removal percentage, adsorption rate, and adsorption capacity due to the increase in hydrophilic character and number of reactive sites. It is lauded as an excellent nano/microscale adsorbent for organic/inorganic contaminants.

In this work, the AC@Fe_xMn_yO_z composite was synthesized and characterized by using X-ray powder diffraction, scanning electron microscopy, Brunauer, Emmett and Teller surface area and zeta potential measurements. This composite material was used for the first time as an effective adsorbent for dispersive solid phase extraction of copper and lead from real samples. The determination of analytes was made by FAAS. The effect of analytical parameters including pH, adsorption and elution contact times, eluent type, volume and concentration, sample volume, and interfering ions was investigated. Optimum pH was found to be 6. A vortex time for both adsorption and elution was selected as 2 min. The eluent was 3 mL of 2 mol L⁻¹ HNO₃. The preconcentration factor of the method was found to be 33.

Keywords: Activated carbon metal oxide composite, dispersive solid phase extraction, copper, lead.

References:

1. A.H. AbdEl-Salam, H.A. Ewais, A.S. Basaleh, Silver nanoparticles immobilised on the activated carbon as efficient adsorbent for removal of crystal violet dye from aqueous solutions. A kinetic study, J Mol Liq (2017) 248 833–841.
2. T. Huang, R. Zhou, J. Cui, J. Zhang, X. Tang, S. Chen, J. Feng, H. Liu, Fast and cost-effective preparation of antimicrobial zinc oxide embedded in activated carbon composite for water purification applications, Mater Chem Phys (2018) 206 124-129.

PP70- Spectrophotometric Determination of Trace Levels of Formaldehyde in Some Beverages after Preconcentration with Ion-Pairing Ultrasound Assisted-Cloud Point Extraction

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In foodstuffs, formaldehyde (FA) comes mainly from two ways: one is natural production during fermentation; the other is its illegal use in the food processing industry, because the adulteration of FA in some foodstuffs not only prolongs their storage lives but also changes their colors and smells to attract consumers.^{1,2}

Due to these properties of FA, a new ion-pair formation based separation method using 2-hydrazinobenzothiazole (2-HBT) as derivatisation agent, Eosin Y as ion-pairing and non-ionic surfactant, Triton X-114 as extractant for preconcentration of low levels of FA before determination by spectrophotometry has been developed. The extraction of FA from sample matrix were assisted by ultrasound energy. The method is based on the micellar extraction of ternary complex, the product of the condensation and then ion-pairing reaction of FA with 2-HBT and Eosin Y, at pH 5.0. The ternary complex was pre-concentrated in surfactant rich phase, and then improved and determined spectrophotometrically at 526 nm. The variables affecting ion-pair formation and extraction efficiency were studied, and the analytical characteristics of the method were obtained. Good linearity was obtained in the range of 8–200 $\mu\text{g L}^{-1}$ and the limits of detection and quantification of the method was 2.41 and 8.02 $\mu\text{g L}^{-1}$. The recovery of the method after spiking (n:5, 25 and 100 $\mu\text{g L}^{-1}$) was in range of 97.3-101.8% with a RSD lower than 5.2%. The interference effect of possible matrix components was also tested. The method was successfully applied to the determination of FA in some beverages.

Keywords: Cloud point extraction, Eosin Y, 2-Hydrazinobenzothiazole, Spectrophotometry, Formaldehyde

References:

1. Saison D, De Schutter DP, Delvaux F, Delvaux FR (2009) Determination of carbonyl compounds in beer by derivatisation and headspace solid-phase microextraction in combination with gas chromatography and mass spectrometry. *J Chromatogr A* 1216:5061–5068.
2. Wang H, Ding J, Du X, Sun X, Chen L, Zeng Q, Xu Y, Zhang X, Zhao Q, Ding L (2012a) Determination of formaldehyde in fruit juice based on magnetic strong cation-exchange resin modified with 2,4- dinitrophenylhydrazine. *Food Chem* 131:380–385.

PP71- Extraction, Preconcentration and Determination of Low levels of Sulfite from some Beverage Matrices by Ultrasonic Assisted-Cloud Point Extraction coupled with Spectrophotometry

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Sulfite (as sulfite, bisulfite and metabisulfite) is one of legal food additives, and have been used in a large variety of foodstuffs and beverages contributing to the preservation of foods by preventing enzymatic oxidation, browning reaction, and microbial spoiling.^{1,2} Therefore, there is an increasing demand for new analytical methods that allow the reliable quantification of high-risk substances.

In this context, we describe a new ultrasonic assisted-cloud point extraction (UA-CPE) method for the preconcentration of sulfite from foodstuffs prior to analysis by spectrophotometry. The method is based on the conversion of sulfite to anionic hydroxymethane sulphonate (HMS) by formaldehyde at pH 5.0, and ion-pair formation of the subsequent selective derivatisation product, which is linearly related to sulfite concentration, with cationic phenazine dye, Safranin T. After that, the formed ion-pair complex was easily extracted into the micellar phase of nonionic surfactant, Triton X-114 by means of ultrasound energy. The FA levels in surfactant rich phase for construction of calibration curve after preconcentration by UA-CPE were detected by spectrophotometry at 520 nm. The method allows the determination of trace levels of sulfite in the range of 2–120 $\mu\text{g L}^{-1}$ with a detection limit of 0.41 $\mu\text{g L}^{-1}$. The method was validated by intra-day and inter-day accuracy and precision studies after spiking at known concentration levels. After validation, the method was successfully applied to monitoring and determination of trace amounts of FA in some beverage samples.

Keywords: Ultrasound Assisted-Cloud point extraction, Sulfite, Safranin T, Spectrophotometry

References:

1. S. I. Martins; W. M. Jongen; and M. A. Van Boekel, Trends in Food Science and Technology, 2000, 11, 364–373. 453.
2. L. Manzocco; S. Calligaris; D. Mastrocola; M. C. Nicoli; and C. R. Lerici, Trends in Food Science and Technology, 2000, 11, 340–346.

PP72- All Solid State Chromium (III) Selective Sensor Based on Newly Synthesized Schiff Bases Derivative as an Ionophore in PVC Matrix

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Although the developed modern spectrophotometric devices over the past decade can be used for determinations with high precision and accuracy, these devices are many expensive and must be used by specialists. Potentiometric determinations using ion-selective electrode are based on high selectivity, wide operating range, low detection limits, high accuracy and precision, short analysis time, simple design, low cost and no damage to the measured material. Due to these properties, the potentiometric determinations are often used in many field analyzes, as an alternative to complex and costly methods [1].

In this study, all solid state chromium (III) selective potentiometric poly(vinylchloride) (PVC) membrane sensors were constructed by using the newly synthesized ionophores called (E)-4-((4-Nitrophenylimino)methyl) phenyl acrylate, (E)-4-((4-Chlorophenylimino)methyl) phenyl acrylate and (E)-4-(4-hydroxybenzylideneimino)benzoic acid acrylate [2]. The most suitable membrane composition having the best potentiometric characteristics was examined by using membrane cocktail coated electrodes containing 1,0 % (m/m) ionophores, 67,0 % (m/m) o-nitrophenyloctyl ether (o-NPOE), bis(2-ethylhexyl) sebacate (DOS), dibutyl phthalate (DBP) and 32,0 % (m/m) PVC. In addition, the effect of KTpClPB and NaTPB in ionic compounds was investigated to determine optimum membrane composition. The optimum membrane composition was determined as 1,0% (m/m) (E)-4-((4-Chlorophenylimino)methyl) phenyl acrylate, 67% (m/m) o-NPOE and 32% (m/m) PVC.

On the condition of pH 2,5–5,0, the prepared electrode exhibited linear responses over the concentration range of $1,3 \times 10^{-7}$ – $1,0 \times 10^{-1}$ M Chromium (III) ($R^2 = 0,9961$) with a sensitivity of $24,6 \pm 0,3$ mV/decade, detection limit of $6,3 \cdot 10^{-8}$ M and short response time of ~ 10 s. The influences of some widely used cation species over electrode responses were studied. In order to investigate whether the proposed electrode could be used analytically, potentiometric determination of chromium (III) ion was carried out in city waters with chromium (III) ion added in known quantities.

Keywords: Potentiometric sensor, Ion-Selective Electrodes, Schiff bases Ionophores, All solid-state.

References:

1. Osman Cubuk, Bulent Caglar, Cihan Topcu, Fatih Coldur, Gokhan Sarp, Ahmet Tabak, Erdal Sahin, Structural characterization of hexadecyltrimethylammonium-smectite composites and their potentiometric electrode applications, Applied Surface Science (2015) 338 99-112.
2. Pakize Silku, Sevil Ozkinalı, Zeynel Oztürk, Abdurrahman Asan, D. Ali Kose, Synthesis of novel Schiff Bases containing acryloyl moiety and the investigation of spectroscopic and electrochemical properties, Journal of Molecular Structure (2016) 1116 72-83.

PP73- Deep Eutectic Solvent Extraction of Selenium and Arsenic from Edible Mushroom Samples and Their Graphite Furnace Atomic Absorption Spectrometric Determination

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Deep eutectic solvents (DESs) are normally obtained using a mixture of ammonium salts (such as choline chloride) and hydrogen bond donor (HBD, urea, carboxylic acids) (1). DES are known relatively non-toxic, nonflammable, non-volatile, renewable, and biodegradable (2).

A green, simple and cheap method based on choline chloride-oxalic acid (ChCl-Ox) as deep eutectic solvent (DES) was optimized for the extraction of selenium and arsenic from edible mushroom samples. Various analytical parameters including DES composition, DES volume, temperature, extraction time, sample amount, centrifugation, etc. were optimized. Analyte ions were determined by graphite furnace atomic absorption spectrometry (GFAAS). Ashing and atomization temperatures for GFAAS were optimized. The results were compared with conventional wet acid digestion method. The detection limit (LOD) and the limit of quantification (LOQ) were found as 0.32 $\mu\text{g L}^{-1}$ and 1.06 $\mu\text{g L}^{-1}$ for As and 0.50 $\mu\text{g L}^{-1}$ and 1.65 $\mu\text{g L}^{-1}$ for Se, respectively. The accuracy of the developed method was confirmed with analyzing of certified reference material CS-M-3 of mushroom powder (*Boletus edulis*). Relative standard deviation (RSD) was found to be 4.2%. The developed method was applied to various edible mushroom samples for the determination of arsenic and selenium.

References

- 1.A.P. Abbott, D. Boothby, G. Capper, D.L. Davies, R.K. Rasheed, Deep eutectic solvents formed between choline chloride and carboxylic acids: versatile alternatives to ionic liquids, *J. Am. Chem.* (2004), 126, 9142-9147.
- 2.Y.H. Choi, J. van Spronsen, Y. Dai, M. Verberne, F. Hollmann, I.W. Arends, R. Verpoorte, Are natural deep eutectic solvents the missing link in understanding cellular metabolism and physiology?, *Plant Physiol.* (2011), 156, 1701-1705.

Acknowledgements

Rizwan Ali Zounr is grateful to the TUBITAK (Scientific and Technological Research Council of Turkey) for being awarded Research Fellowship Program of 2216 and providing financial support.

PP74- Simultaneous Voltammetric Determination of Carbidopa and Levodopa in Pharmaceutical Products

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Parkinson's disease is a neurodegenerative disorder due to loss of dopamine neurotransmitter brain cells. Levodopa (L-Dopa) is the precursor of catecholamine which is the principal drug used in the treatment. For better therapeutic effect and lower toxicity, carbidopa (Car) is administered with levodopa in pharmaceutical formulation as an inhibitor on the decarboxylase activity [1]. Therefore, analytical methods allowing the simultaneous determination of L-Dopa and Car in pharmaceutical preparations are required [2].

In present study, an analytical method based on differential pulse voltammetry (DPV) on a pencil lead electrode for the simultaneous determination of levodopa and carbidopa was developed. Cyclic voltammograms (CV) have displayed reversible peaks observed in anodic and cathodic scan at 0.42 V and 0.46 V for L-Dopa. For Car, two oxidation peaks at 0.46 V and 0.86 V have been obtained and no cathodic peak was observed at slow scan rates. Since the oxidation product of Car does is not reduced in the cathodic potential scan, L-Dopa can be determined in the presence of Car by monitoring its reduction signal. The reduction peak of L-Dopa and the second oxidation peak of the carbidopa have been utilized in simultaneous determination of L-Dopa and Car and linear calibration graphs have been obtained in mikromolar levels. The method was applied to a commercial tablet and satisfactory recovery values have been obtained.

Keywords: Carbidopa, Levodopa, pencil lead electrode, voltammetry

Acknowledgement: Authors would like to thank Ege University Research Project (2014 FEN 056) for financial support.

References:

1. K. V. Özdokur, E. Engin, Ç. Yengin, H. Ertaş, F. N. Ertaş, Determination of Carbidopa, Levodopa, And Droxidopa By High-Performance Liquid Chromatography–Tandem Mass Spectrometry, *Anal Lett.*, (2018) 51 73-82.
2. M. S. M. Quintino, M. Yamashita, L. Angnes, Voltammetric Studies and Determination of Levodopa and Carbidopa in Pharmaceutical Products, *Electroanalysis*, (2005) 7 655-661

PP75- Cellulose acetate butyrate based thiol-ene/sol gel hybrid crosslinked matrix for heavy metal ions removal

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Cellulose derivatives have critical parts in industry; they speak to a principle hotspot for strands, materials, coatings, thermoplastic films, sustenance added substances, and pharmaceutical advancements. Cellulose derivatives are typically named two fundamental classes, esters and ethers, as per the reactant nature. Cellulose derivatives for the most part contain free hydroxyl bunches accessible for extra medicines to yield blended esters. The blended esters have a few enhanced properties over every single slick ester. Cellulose acetate propionate (CAP) and cellulose acetate butyrate (CAB) are the most commercially important mixed esters [1]. Mixed cellulose derivatives with both ester and ether groups could be also attained. Due to their low degree of substitution and high hydrolytic stability, carboxymethyl cellulose can be further esterified with organic acid anhydrides to add either single or mixed ester groups [2]. The presence of heavy metal ions in aqueous media is one of the greatest environmental contamination issues and in this way the removal of heavy metals is an critical technique.

In this study, cellulose acetate butyrate (CAB) reacted with 3-mercaptopropyl-trimethoxysilane and CAB-SH was obtained. Then incorporated into thiol-ene using photopolymerization. The synthesized CAB based crosslinked membrane was used for heavy metal ions removal from the aqueous sample. The structure and morphology of CAB-SH and membrane were characterized by FTIR, TGA and SEM, respectively.

Keywords: Membrane, heavy metal, thiol-ene, photopolymerization.

References:

1. Balser K., Hoppe L., Eicher T., Wandel M., Astheimer H.-J., Steinmeier H., Allen J. M. Cellulose esters. In: Bohnet M., Bellussi G., Bus J., Cornils B., Drauz K., editors. *Ullmann's Encyclopedia of Industrial Chemistry*. New York, NY, USA: John Wiley & Sons; 2004.
2. Paris J. L. *Carboxymethyl cellulose acetate butyrate water-dispersions as renewable wood adhesives [M.S. thesis]* Virginia Polytechnic Institute and State University; 2010.

PP76- Synthesis, Characterization and Swelling Behavior of Poly (Acrylic Acid-co-N-vinyl 2-pyrrolidone) Copolymeric Hydrogels**Fatma Özge Gökmen*, Elif Yaman, Sinan Temel***Bilecik Seyh Edebali University, Central Research Laboratory, Bilecik-TURKEY**E-mail: fatmaozge.gokmen@bilecik.edu.tr

Hydrogels are three dimensional hydrophilic polymer networks capable of swelling in water or biological fluids, and retaining a large amount of fluids in the swollen state. Several techniques have been reported for the synthesis of hydrogels. The first approach involves copolymerization/crosslinking of co-monomers using multifunctional co-monomer, which acts as crosslinking agent. The polymerization reaction is initiated by chemically. The polymerization reaction can be carried out in bulk, in solution, or in suspension. The second method involves crosslinking of linear polymers by irradiation, or by chemical compounds [1].

In this work, acrylic acid was preferred as monomer because of its biodegradability property. Acrylic acid hydrogels were obtained in the presence of a radicalic initiator. It is discussed in this work how cross-linked hydrogels formed by sequential arrangement of acrylic acids in a homopolymer form can show superior properties in the presence of a different monomer in the environment. Thus, acrylic acid and vinyl pyrrolidone monomers obtained under optimum conditions were synthesized by free radical polymerization method using crosslinking agent. % Conversion (%C), % crosslinking (%CL) and % swelling (%S) values of the obtained hydrogels were calculated. The chemical bonds of the hydrogels were clarified by FTIR analysis. The surface morphologies of the hydrogels are shown by SEM analysis. Copolymeric hydrogels have been identified by discussing the results obtained with superiority as a homopolymeric hydrogel.

Table 1. Swelling properties of hydrogels

Plain AA			AA-co-VP (3:1)			AA-co-VP (2:2)			AA-co-VP (1:3)		
C%	CL%	S%	C%	CL%	S%	C%	CL%	S%	C%	CL%	S%
95,42	95,34	370	95,50	81,34	326	104	74,76	117	91,83	63,16	142

Keywords: Hydrogel, Acrylic acid, N-vinyl 2-pyrrolidone, swelling behavior.

References:

1. Fatma Özge Gökmen, Nursel Pekel Bayramgil, synthesis and characterization of n-[3-(dimethyl-amino)propyl] methacrylamide/(nano-SiO₂, amine-modified nano- SiO₂ and expanded perlite) nanocomposite hydrogels, Eur. Chem. Bull. (2017) 6(11) 514-518.

PP77- The Aroma Compositon of *Hypericum armenum* and *H. orientale* by Using GC-MS/FID Head Space

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The genus *Hypericum* a member of Hypericaceae family, is represented by 100 taxa, 45 being endemic to Turkey. In Turkish folk medicine, the genus *Hypericum* is known as “sarı kantaron, kantaron, binbirdelik otu, mayasıl otu” and most of them, especially *H. perforatum*, have been used for the treatment of burns, wounds, hemorrhoids, diarrhea and ulcers (1, 2). Aqueous extracts of the flowering aerial parts of the *Hypericum* species are used in the treatment of neuralgia, anxiety, neurosis and depression. The essential oil compositions of about 50 different *Hypericum* species have been identified in the previous studies (1):

In this study, the aroma contents of *H. armenum* and *H. orientale* was analyzed by GC-MS/FID. The dried aerial parts of species were cut into small pieces and subjected to hydro-distillation with water for 4 h, using a Clevenger-type apparatus to produce essential oils which were dried over anhydrous sodium sulphate and stored at +4°C until required. The essential oils were diluted by dichloromethane (1:3, v/v) before the GC run. Identification of the compounds was based on the comparison of their retention times and mass spectra with those obtained from authentic samples and/or the NIST and Wiley spectra as well as the literature data. 30 components constituting 92.12% of the aroma composition of *H. armenum*, and 26 components constituting 91.64% of the aroma composition of *H. orientale* were determined. The major components of the aroma were identified as nonane (24.04%) and cis-beta-Ocimene (14.72%) for *H. armenum* and thujopsene (28.48%) and α -pinen (21.84%) for *H. orientale*.

Keywords: *H. armenum*, *H. orientale*, Aroma, GC-MS/FID

Acknowledgements: The research was funded by grant: DUBAP FEN-15-012 from Dicle University Scientific Research Projects Coordination Unit.

References:

- 1 Robson NKB. (1966) *Hypericum*, in *Flora of Turkey and the East Aegean Islands*, Vol II ed. by Davis PH, Edinburg University Press, Edinburgh; pp. 305-401
- 2 Baytop T. (1984) *Therapy with Medicinal Plants in Turkey*, Istanbul University Press, Istanbul, pp. 185.

PP78- The Biological Activities of Essential Oil of *Salvia rosifolia* and *Salvia syriaca* from Anatolia

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The acetylcholinesterase (AChE) enzyme predominates in a healthy brain, with the butyrylcholinesterase (BChE) enzyme considered to play a minor role in regulating brain acetylcholine (ACh) levels. This implies that any compound that enhances the cholinergic system in the brain may be useful in treating some brain malfunctions (1). Most of the studies on AChE inhibitors have focused on alkaloids that have acetylcholinesterase inhibitory activity, such as galantamine. To date, more than 35 alkaloids have been reported to have AChE inhibitory activity. In order to find new natural agents for the treatment of AD based on the cholinesterase inhibitory mechanism, many research groups in the world study different plants from various families, including the Amaryllidaceae, Fumariaceae, Papaveraceae, and Lamiaceae families (2).

In this study, the essential oils of *Salvia rosifolia* and *Salvia syriaca* species were tested for antioxidant (ABTS cation radical decolorisation method, cupric reducing antioxidant capacity assay and DPPH free radical scavenging activity) and anticholinesterase (acetyl- and butyrylcholinesterase enzymes) activities and total phenolic content. Essential oil samples were obtained by a Clevenger apparatus from the whole parts of plants which were crumbled into small pieces and soaked in distilled water for 3 h. Then, these samples were dried over anhydrous Na₂SO₄ and stored at +4°C for a sufficient period of time. The essential oil of *S. syriaca* (89,18±2.98) was found higher phenolic content than the essential oil of *S. rosifolia* (72,86±2,89). The acetyl- and butyrylcholinesterase enzyme activities of *S. rosifolia* (66,12±2,86 and 90,76±1,2 respectively) were determined higher than *S. syriaca*. The essential oil of *S. syriaca* showed moderate activity (50,66±4,58) in acetylcholinesterase enzyme and it showed good activity in butyrylcholinesterase enzyme (78,33±0,2). Additionally, essential oil of *S. syriaca* was showed good activity in ABTS cation radical decolorisation method and cupric reducing antioxidant capacity assay.

Keywords: *Salvia rosifolia*, *Salvia syriaca*, Essential Oil, Anticholinesterase, Antioxidant, Phenolic

References:

1. Howes M. J.R., Perry E., The Role of Phytochemicals in the Treatment and Prevention of Dementia. Drug & Aging 2011; 28: 439-68. [CrossRef]
2. Mukherjee, P.K., Kumar, V., Mal, M., Houghton, P. J., 2007, Acetylcholinesterase Inhibitors from Plants, Phytomedicin, 14, 289–30

PP79- The Chemical Composition of *Hypericum Perferatum* Collected from Different Localities by Using LC-MS/MS

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Several of the botanical species belonging to the genus are used in folk medicine and among them *Hypericum perforatum* L., also named St. John's Wort, is especially known as a traditional remedy for the treatment of melancholia, abdominal and urogenital pain and ulcerated bums (1). Over the last two decades, it has been demonstrated that *H. perforatum* is effective as an antidepressant, an antiviral, and an antimicrobial. The drug contains peculiar chemical constituents such as naphthodianthrones (hypericins), acylphloroglucinols (hyperforin), flavonol glycosides (quercetin derivatives) and biflavones and all the metabolites seem to contribute to its pharmacological activity. Current use of *H. perforatum* is mainly for the treatment of mild to moderate depression, and drug extracts for antidepressant applications have become increasingly popular. During the recent years, several phytochemical studies on other species of *Hypericum* have also been performed, leading to the isolation of new molecules, some having biological activity (1).

In this study, The ethanol extracts of aerial parts and root of *H. perforatum* species collected from different localities (Trabzon, Van and Bitlis) were tested for chemical composition by using LC-MS/MS. The six extracts were screened and quantitatively assayed for apigenin, apigetrin, astragalin, chlorogenic acid, hesperidin, hyperforin, hypericin hyperoside, luteolin, lutollin-7-glucoside, protocatechuic acid, pseudohypernicin, quercetin, quercitrin and routine. In the same way, the samples collected from Trabzon were found to be richer than the samples collected from Van and Bitlis.

Acknowledgements: The research was funded by grant: BAP FEN.15.012 from Dicle University

Keywords: *Hypericum perforatum*, LC-MS/MS, Phenolic

References:

1. Sadia Mansour, Nouredine Djebli, Esra Eroglu Ozkan, Afife Mat "In vivo antiinflammatory activity and chemical composition of *Hypericum scabroides*" *Asian Pac J Trop Med.*,(2014) 7(1) S514-S520.

PP80- The Essential Oil and Aroma Composition of *Hypericum retusum*

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The genus *Hypericum* a member of Hypericaceae family, is represented by 100 taxa, 45 being endemic to Turkey (1). In Turkish folk medicine, the genus *Hypericum* is known as “sarı kantaron, kantaron, binbirdelik otu, mayasıl otu” and most of them, especially *H. perforatum*, have been used for the treatment of burns, wounds, hemorrhoids, diarrhea and ulcers. Aqueous extracts of the flowering aerial parts of the *Hypericum* species are used in the treatment of neuralgia, anxiety, neurosis and depression (2). The essential oil compositions of about 50 different *Hypericum* species have been identified in the previous studies.

In this study, the essential oil contents of *H. retusum* was analyzed by GC-MS/FID. The dried aerial parts of species were cut into small pieces and subjected to hydro- distillation with water for 4 h, using a Clevenger-type apparatus to produce essential oils which were dried over anhydrous sodium sulphate and stored at +4°C until required. The essential oils were diluted by dichloromethane (1:3, v/v) before the GC run. Identification of the compounds was based on the comparison of their retention times and mass spectra with those obtained from authentic samples and/or the NIST and Wiley spectra as well as the literature data. 48 components constituting 93.58% of the essential oil composition of *H. retusum*, and 31 components constituting 97.84% of the aroma composition of this species were determined. The major components of the essential oil and aroma were identified as α -pinen (28.01% and 20.27%, respectively) and Octane, 2-methyl (13.41% and 32.97%, respectively).

Keywords: *H. retusum*, Aroma, Essential Oil, GC-MS/FID

Acknowledgements: The research was funded by grant: DUBAP FEN-15-012 from Dicle University Scientific Research Projects Coordination Unit.

References:

- 1 Robson NKB. *Hypericum*, in *Flora of Turkey and the East Aegean Islands*, Vol II ed. by Davis PH, Edinburg University Press, Edinburgh; pp. 305-401 (1966).
- 2 Baytop T. (1984) *Therapy with Medicinal Plants in Turkey*, Istanbul University Press, Istanbul, pp-185.

PP81- The Biological Activities of Essential Oil of *Salvia blepharochlaena* and *Salvia hydrangea* from Anatolia

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Salvia L. (Lamiaceae) species consists of about 900 species distributed throughout the world. In Turkey, this genus is represented by 100 species, 53 (53%) of which are endemic (1). The genus is named “*Salvia*”, derived from latin “*Salveo*”, means “save, or recover” (1). Many of *Salvia* species are named “adaçayı” in Anatolia, Turkey, and used as herbal tea due to their antiseptic, stimulant, diuretic and wound healing properties (2). *Salvia* species are generally known for their multiple pharmacological effects including their antibacterial, antiviral, antioxidative, antimalarial, anti-inflammatory, antidiabetic, cardiovascular, antitumor and anticancer (2). Also, some studies showed that a part of these activities depended on essential oil composition (2).

In this study, the essential oils of *S. blepharochlaena* and *S. hydrangea* were tested for antioxidant (ABTS cation radical decolorisation method, cupric reducing antioxidant capacity assay and DPPH free radical scavenging activity) and anticholinesterase (acetyl- and butyrylcholinesterase enzymes) activities and total phenolic content. Essential oil samples were obtained by a Clevenger apparatus from the whole parts of plants which were crumbled into small pieces and soaked in distilled water for 3 h. Then, these samples were dried over anhydrous Na₂SO₄ and stored at +4°C for a sufficient period of time. The essential oils of studied two *Salvia* species; *S. blepharochlaena* (75,92±1,44), *S. hydrangea* (78,98±0,0) were determined the similar results as total phenolic content. The acetyl- and butyrylcholinesterase enzyme activities of *S. blepharochlaena* (81,5±3,60 and 95,24±3,8 respectively) were determined as high. The essential oil of *S. hydrangea* (93,4±1,20) was showed a good activity in butyrylcholinesterase enzyme. Additionally, two essential oil were showed weak antioxidant activity.

Keywords: *Salvia blepharochlaena*, *Salvia hydrangea*, Anticholinesterase, Antioxidant,

References:

1. Celep F, Dirmenci T and Guner O. *Salvia hasankeyfense* (Lamiaceae), a new species from Hasankeyf (Batman, (South-eastern Turkey) *Phytotaxa*, (2015) **227**:3, 289-294.
2. Tepe B, Daferera D, Sokmen A, Sokmen M, and Polissou M., Antimicrobial and antioxidant activities of the essential oil and various extracts of *Salvia tomentosa* Miller (Lamiaceae) *Food Chemistry*, (2005) **90** 333–340.

PP82- A Different Spectrophotometric Method For the Determination of Carvedilol

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Carvedilol (CRV), (2RS)-1-(9H-Carbazol-4-yloxy)-3-[[2-(2-methoxyphenoxy) ethyl] amino]-2-propanol, is a non-selective beta adrenergic blocking agent. It is used for the treatment of several cardiovascular disorders such as hypertension, angina pectoris and heart failure. (1-2). Spectrophotometric analytical methods have been extensively employed to many drugs. Charge transfer reactions are one of the most commonly used spectrophotometric methods. These reactions take place between an electron donor and an electron acceptor molecule. A colored complex is formed as a result of the reaction(2).

In this work, a rapid, economical, easy and sensitive method for the determination of CRV has been developed. The method is based upon the formation of charge transfer (CT) complex between cited drug and quinalizarin producing CT-complex in methanolic medium. The optimum conditions of the reaction such as the effect of solvent, reagent concentration and reaction time was investigated. All the absorption spectral measurements were made using double beam T80+ UV/Vis Spectrometer PG Instruments.

Keywords: Carvedilol, UV-VIS, Charge transfer reactions.

References:

1. Laila E. S., Abdel F., Taghreed A. M., Elham A. T., Spectrofluorimetric Determination of Carvedilol in Dosage Form And Spiked Human Plasma Through Derivatization with 1-Dimethylaminonaphthalene-5-Sulphonyl Chloride, Chemical Ind. & Chemical Eng. Quarterly, (2010), 16 (1):31-38
2. Cardoso, S.G., Ieggli C.V.S., Pomblum, S.C.G., Spectrophotometric determination of carvedilol in pharmaceutical formulations through charge-transfer and ion-pair complexation reactions, Pharmazie. (2007), 62(1):34-7.

PP83- Determination of Cobalt, Nickel and Zinc in Some Clay Face Masks

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Clays are generally referred to as natural, ground, fine granulated (less than 2 µm diameter) particles, in their structure, contain clay minerals and non-clayish minerals(1).

It is known that clay masks have been used for cosmetic and therapeutic purposes since antiquity. Today, clay facial masks are used for many purposes such as stringency and physical exfoliation, the removal of impurities, dead cells and oil from face skin(2). Clay facial masks contain various chemical substances and heavy metals play an important role among these chemical substances. As known, heavy metals show toxic effects on the body when they are above certain levels.

In this study, 11 clay masks from Diyarbakir province, both local and different brands, were provided commercially from the market. Nickel, Cobalt and Zinc elements were determined by AA-7000 Shimadzu Atomic Absorption Spectrophotometer and the results were evaluated statistically.

Keywords: Clay mask, Nickel, Cobalt, Zinc

References:

1. Favero J.S., Parisotto-Peterle J., Weiss-Angeli V., Brandalise R. N., Gomes L. B., Bergmann C. P., Santos V., Physical and chemical characterization and method for the decontamination of clays for application in cosmetics, Applied Clay Science (2016) 124-125, 252-259
2. Beringhs A. O., Rosa J. M., Stulzer H. K., Budal R. M., Sonaglio D., Green Clay and Aloe Vera Peel-Off Facial Masks: Response Surface Methodology Applied to the Formulation Design, AAPS PharmSciTech, (2013), 14, 1

PP84- The Phenolic Content of *Hypericum scabrum* Collected from Different Localities by Using LC-MS/MS

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Hypericum (Hypericaceae) is one of the 100 largest genera that comprise an estimated 22% of angiosperm diversity with the presence of 484 species from 36 taxonomic sections. *Hypericum* species are wellknown healing agents in traditional medicine due to various medicinal properties. Despite the large number of *Hypericum* species, only *Hypericum perforatum* L. has been studied in depth as pharmaceutically important medicinal crop plant which extracts widely used in Europe as a drug for the treatment of mild and moderate depression. Turkey is an important prevalence centre of *Hypericum* species. All *Hypericum* species have been used in Turkish folk medicine under the names “kantaron, peygamber çiçeği, kılıçotu, kanotu, kuzukıran and binbirdelik otu” as sedatives, antiseptics and antispasmodics. Curative value of *Hypericum* plants have been mainly attributed to the phytochemicals, namely phloroglucinol derivatives, naphthodianthrone, different phenolic compounds and essential oils which possess a great variety of bioactivities (1).

In this study, the ethanol extracts of aerial parts and root of *H. perforatum* species collected from different localities (Nevşehir, Elazığ, Van-center, Van-Bahçesaray and Kahramanmaraş) were tested for chemical composition by using LC-MS/MS. The six extracts were screened in terms of apigenin, apigetrin, astragalin, chlorogenic acid, hesperidin, hyperforin, hypericin, hyperoside, luteolin, lutollin-7-glucoside, protocathechuic acid, pseudohypericin, quercetin and quercitrin, and they were determined quantitatively.

Acknowledgements: The research was funded by grant: BAP FEN.15.012 from Dicle University

Keywords: *Hypericum scabrum*, LC-MS/MS, Phenolic, hyperoside

References:

1. Cuneit Cirak, Jolita Radusiene, Valdas Jakstas, Liudas Ivanauskas, Fatih Seyis and Fatih Yayla “Secondary metabolites of seven *Hypericum* species growing in Turkey”, Pharm Biol. (2016) Oct;54(10):2244-53

PP85- Electrochemical Oxidation Mechanism of Cytarabine-Zn(II) Metal Based Compound on Glassy Carbon Electrode

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Cytarabine, also known as cytosine arabinoside (ara-C), is a chemotherapy medication used to treat acute myeloid leukemia (AML), acute lymphocytic leukemia (ALL), chronic myelogenous leukemia (CML), and non-Hodgkin's lymphoma.

In this study, the electrochemical behavior of the Cyt-Zn(II) metal-based compound, which has been proven to be effective in the cell line Hela, has been tested using different buffer species and different pH media (pH 1-12) using cyclic voltammetric technique. Mechanism of the electrochemical reaction of Cyt-Zn(II) metal-based compound was studied by performing density functional theory (DFT) computations and mass spectroscopic analysis (LC-MS/MS). In mass analysis, electrospray ionization mode (ESI) was used in positive and negative ion mode with nitrogen as the spray and collision gas. The capillary voltage was used 4000 V for positive and negative ions.

Key words: Cytarabine, metal complex, electrochemical behaviour, mass spectrometry, leukemia

References:

1. Gómez-Almaguer D, Cantú-Rodríguez OG, Gutiérrez-Aguirre CH, Ruiz-Argüelles GJ. Hematology. The treatment of CML at an environment with limited resources. 2016 Dec;21(10):576-582. Epub 2016 May 24. Review.

PP86- Electrochemical Investigation of DNA Binding on Carbidopa by Cyclic Voltammetry

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Carbidopa (Lodosyn) is a drug given to people with Parkinson's disease in order to inhibit peripheral metabolism of levodopa. This property is significant in that it allows a greater proportion of peripheral levodopa to cross the blood-brain barrier for central nervous system effect¹. The application of electrochemical methods to the study of organic and metallointeractions to DNA provides a useful complement to the previously used methods of investigation, such as UV-visible spectroscopy, viscosity and circular dichroism studies. Small molecules, which are not amenable to such methods, either because of weak absorption bands or because of overlap of electronic transition with those of the DNA molecules, can potentially, be studied via voltammetric techniques.

In this study, the redox behaviour between carbidopa and double strand DNA was investigated on glassy carbon electrode at physiological pH. Cyclic voltammetry based assay was developed for the assessment of the effect of the medium, substituents, potential scan rate and a number of scans on the voltammetric response of carbidopa-DNA couple.

Reference

1- The American Society of Health-System Pharmacists. Archived from the original on 2015-09-24. Retrieved Aug 21, 2015. The American Society of Health-System Pharmacists. Archived from the original on 2015-09-24. Retrieved Aug 21, 2015.

PP87- Formation of Epirubicin Metal Based Composition in Analytical Conditions

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Cis-platinum ($[\text{Pt}(\text{NH}_3)_2\text{Cl}_2]$), developed by Rosenberg in 1969 and used for cancer treatment has led many research group to synthesize metal-based drugs since those years [1]. In this field, mostly Pt (II), Cu (II), Zn(II), Ru(III) and Mn(III) are used as the metal ion. In all of those studies, the drug molecules act as ligands and form mostly monodentate or bidentate molecular structures by offering pair of electrons to metal atoms. The studies of this topic over the last forty years have been gathered under the roof of “Medicinal Chemistry” [2]. The main objective of Medicinal Chemistry is to identify new anticancer agents and put them into clinical practice procedures.

In this study, the Cu(II) metal-based compound of the chemotherapeutic drug epirubicin (3) was obtained in a methanol / water mixture with pH control. The resulting metal-based compound was characterized by LC-MS/MS and basic spectroscopic techniques.

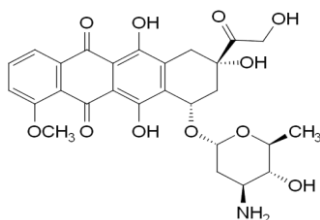


Figure 1. Epirubicin structure

Key Words: anticancer drugs, metal-based drugs, epirubicin, spectroscopy

References

- 1- Rosenberg, B. *Plat. Met. Rev.* 1971, 15, 42–51.
- 2- Kratz, F. In *Metal Complexes in Cancer Chemotherapy*; Keppler, B. K., Ed.; VCH: Weinheim, Germany, 1993; pp 391–429.
- 3- SOBOL, MM, et al. *Invitro Evidence For Direct Complexation Of ADR-529/ICRF-187[(+)-1,2-BIS-(3,5-Dioxo-Piperazin-1-Yl)Propane] Onto An Existing Ferric-Anthracycline Complex*, *Molecular Pharmacology*, 1992, 41(1) 8-16

PP88- Interaction studies of Sulpiride with Fish Sperm Double Strain DNA Using UV-Spectroscopic Technique

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Sulpiride (Fig. 1) an atypical antipsychotic drug (although some texts have referred to it as a typical antipsychotic) of the benzamide class used mainly in the treatment of psychosis associated with schizophrenia and major depressive disorder, and sometimes used in low dosage to treat anxiety and mild depression¹. Sulpiride is commonly used in Asia, Central America, Europe, South Africa and South America. So far it has not been approved in the United States, Canada and Australia.

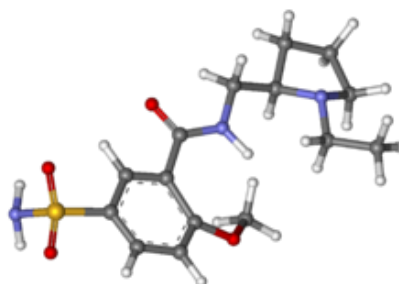


Fig.1. Chemical structure of Sulpiride

The interactions of various low-molecular-weight substances with DNA are naturally relevant mechanisms in the cellular cycle and so also used in medicinal treatment. Depending on the particular drug structure, DNA-binding modes like groove-binding, intercalating and/or stacking, give rise to supramolecular assemblies of the polynucleotides, as well as influence the DNA-protein binding.

In this study, the molecular interactions between Sulpiride and fish sperm double strain DNA have been studied in phosphate (pH 7.4) and tris-HCl (pH 7) buffer solutions using UV-Vis spectrophotometry technique.

Keywords: Sulpiride, DNA, UV-Vis spectrophotometry

Reference

1. Imondi, AR; Alam, AS; Brennan, JJ; Hagerman, LM (March 1979). "Metabolism of sulpiride in man and Rhesus monkey". *Archives Internationales de Pharmacodynamie et de Thérapie*. 232 (1): 79-91.

PP89- Effect of Frying Repeat On Composition of Fatty Acids Species In Different Oils

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Saturated fats and oils are compounds that are found as basic components of human diet. Frying of food stuffs is an intensively used method for long times. During the frying process many complex reactions such as color change in oil and oxidation, thermal polymerization, thermal oxidation, isomerization, hydrolysis may remove oils edibility also it is started that by transferring a lot of toxic substances it decreases the nutritional value of fried food. Types of fatty acids and their ratio in oil affect the physical and chemical properties. Ratios of total unsaturated fatty acids to total saturated fatty acids are signs of the sensitivity of oils to oxidation. Also during the frying process, decreases in linoleic acid-palmitic acid ratio are defined as strong indicators of oil degradation ratios.

The aim of this study, is to compare the fatty acid composition of frying commercial fats too many times. Four different oil such as (sunflower seed, riviera, olive oil, extra-virgin olive oil, corn oil) and for frying purposes, sliced and frozen potatoes are used. The acid composition of the samples taken after each frying was detected with GC-FID device.

Related with the acids composition, fatty acids of sunflower oil are found in descending order: linoleic acid (% 56,38), oleic acid (% 31,90), palmitic acid (% 6,37); Fatty acids of corn oil are linoleic acid (% 52,60), oleic acid (% 30,87), palmitic acid (% 11, 74); Fatty acids of extra-virgin olive oil are linoleic acid (% 70,01), oleic acid (% 2,76), palmitoleic acid (% 13,25), arachidic acid (% 10,59), palmitic acid (% 0,018); Fatty acids of riviera olive oil are linoleic acid (% 71,71), oleic acid (% 12,52), cis-11-eicosenoic acid (% 10,05).

Thereby, according to ratios of C18:2/C16:0 after the tenth frying, while being defined that a degradation occurs in the ratio of % 34,76 in sunflower oil, % 19,34 in corn oil, % 84,029 in riviera olive oil and % 85,22 in extra-virgin olive oil, it has been observed that extra-virgin olive oil is the one mostly occurred in degradation.

Keywords: Fried Oils, Oil Chemistry, Health, GC-FID.

References:

1. Cuesta, C., Romero, A., and Muniz, F. J., 2001. Fatty Acid Changes in High Oleic Acid Sunflower Oil during Successive Deep-Fat Fryings of Frozen Foods, Food Science and Technology International, Vol. 7, No. 4, 317-328.
2. Ersöz, M., 2005. Lipoprotein Bozuklukları ve Tedavi Yaklaşımları, Simge Yayıncılık, 1. Basım, 308, İzmit.
3. Chambers, G., September 2000. Recycled Cooking Oils; Assessment of Risks for Public Health. Working Document Page: 1-100.

PP90- Synthesis of Silver Nanoparticles Using Dimrit Grape Extract by Green Chemistry

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Silver nanoparticles are the most synthesized nanoparticles because of their improved physical and chemical properties. They are used in a wide variety of fields such as catalysis, antimicrobial agents, and sensors [1]. In recently, synthesis of them by biological systems has been drawn interested because of easily available, non-toxic, safe and fast [2].

In this study, silver nanoparticles were obtained by plant-derived nanoparticle synthesis which is fast, safer and contain non-harmful components. A suitable nanoparticle synthesis medium for green chemistry was formed for using by Dimrit Grape extract grown in Burdur Region. Some synthesis conditions influencing the silver nanoparticles in different sizes and structures were optimized such as the amount of Dimrit Grape extract, the amount of metal salt, temperature and the mixture time. The characterizations of silver nanoparticles were performed by Atomic Force Microscopy (AFM), Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) techniques.

Keywords: Dimrit Grape, green chemistry, nanoparticle, silver

References:

1. R. D. Rivera-Rangel, M. P. González-Muñoz, M. Avila-Rodriguez, T. A. Razo-Lazcano, C. Solans, Green synthesis of silver nanoparticles in oil-in-water microemulsion and nano-emulsion using geranium leaf aqueous extract as a reducing agent, *Colloids Surf. A* (2018) 536 60–67.
2. D. Kumar, G. Kumar, V. Agrawal, Green synthesis of silver nanoparticles using *Holarrhena antidysenterica* (L.) *Wall.bark* extract and their larvicidal activity against dengue and filariasis vectors, *Parasitol Res.* (2018) 117 377–389.

PP91- Preconcentration and Determination of Ni(II) using Amberlite CG-120 Resin with Flame Atomic Absorption Spectrometry in Water Samples

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Pollution of water, air and soil contaminated with metal ions has recently become an important environmental issue. The sources of heavy metal pollutants can be derived from a variety of sources such as industrial activities, fossil fuel burning, agricultural activities, mining activities, atmospheric emissions. Recently, solid phase extraction has been widely used as preconcentration and separation of elements owing to its advantages such as high enrichment factor, rapid separation and low cost[1].

In this study, Amberlite CG-120 adsorbent was used for the separation/pre-concentration of Ni(II) ions in the spring waters, drinking water and lake water before detection by flame atomic absorption spectrometry (FAAS). Various optimization parameters for Ni(II) determination such as pH, eluent type and concentration, solution and eluent flow rates, amount of adsorbent were investigated to obtain better sensitivity, accuracy, precision and quantitative recovery. Furthermore, the interference effects of some ions on the recovery efficiency of Ni(II) were also investigated. The optimum experimental parameters were obtained in the case of pH 1; 5 mL of 4 mol L⁻¹ HCl for eluent and 0.3 g for the adsorbent amount. The limit of detection was found as 1.15 µg L⁻¹ and linearity was ranged from 5 to 50 µg L⁻¹. The accuracy of the method was tested by certified reference material of TMDA-70.2 Ontario Lake Water at a 95% confidence level.

Keywords: Amberlite CG-120 resin, drinking water, Ni(II), solid phase extraction

References:

1. Ali Rehber Türker, Separation, Preconcentration and Speciation of Metal Ions by Solid Phase Extraction, Sep. Purif. Rev. (2012) 41 169–206.

PP92- Pre-concentration of Tartrazine by Zirconium Metal-Organic Framework Adsorbent Prior to Spectrophotometric Determination

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Tartrazine (E102) is a synthetic organic food dye. It can be found in common food products such as bakery products, dairy products, beverages, and candies. Recent studies show that when tartrazine is consumed in excess, it causes significant adverse effects on neurobehavioral parameters. Therefore, the presence and content of this dye must be controlled due to its potential harmfulness to human beings. Metal organic frameworks (MOFs) have attractive importance as new adsorbents in recent years due to their excellent properties such as low density, high surface area, tunable pore size and porosity. MOFs are comparable to traditional porous materials because of their large internal surface areas, extensive porosity, and high degree of crystallinity.

In this study, zirconium based-MOF was synthesized and characterized. It was used as an adsorbent for the vortex assisted-solid phase extraction of tartrazine. The determination of tartrazine in solutions was made by UV–Vis spectrophotometry. The parameters affecting the recovery values of tartrazine such as sample pH, adsorption and elution contact times, eluent type and concentration were investigated. The acid concentration of the solution for quantitative determination of tartrazine was found to be 0.02 mol L⁻¹ HCl. A vortex time for adsorption was 2 min. Elution was made by 2 mL of 0.5 M NH₃.

Keywords: Metal organic framework, tartrazine, solid phase extraction.

References:

1. R. Sahraei, A. Farmany, S.S. Mortazavi, A nanosilver-based spectrophotometry method for sensitive determination of tartrazine in food samples, *Food Chem* (2013) 138 1239–1242.
2. P. A. Kobielska, A. J. Howarth, O. K. Farha, S. Nayak, Metal–organic frameworks for heavy metal removal from water, *Coord Chem Rev* (2018) 358 92–107.

PP93- Sorption of copper using by N,N,N',N'-Tetraethyl diethylene triamine (TEDETA) modified poly styrene-divinyl benzene (PS-DVB) sorbent

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Water pollution has become an important environmental concern due to the toxic effects of heavy metals. Contamination of water is generally caused by industrial activities, acid rain, urbanization, and traffic.¹ Copper (Cu) is a widespread anthropogenic pollutant of environment, and the determination of this metal is the actual problem. It is an essential trace element which is found in a variety of cells and tissues in all living organisms. The commended daily allowance of copper ranges from 0.4 to 0.6 mg for infants, 1.5 to 2.5 mg for children, and 1.5 to 3.0 mg for adults. USEPA states that the maximum allowable contamination level of copper is 1.3 mgL⁻¹ in drinking waters. Polymers can also be manufactured in a wide range of physicochemical properties (size, porosity, hydrophilicity, etc.), and they are modifiable by inserting various ligands into the structure in order to produce specific sorbents.

In this study, N,N,N',N'-Tetraethyl diethylene triamine (TEDETA) modified poly styrene-divinyl benzene (PS-DVB) sorbent proposed for the removal of copper ions from the aqueous solutions. Cu ions were determined by an inductively coupled plasma mass spectrometer (ICP-MS). The batch method is employed, and parameters such as pH, sorption time, sorbent amount on sorption, and sorbent capacity are optimized. The sorption kinetics and equilibrium isotherms of the removal of Cu ions are also elucidated. The proposed methodology will check via spike recovery experiments with water samples.

Keywords: Copper, sorbent, ICP-MS, Solis phase extraction.

References:

1. Van der Perk, M. (2013). Soil and Water Contamination (2nd ed., p. 4). Balkema: CRC Press.
2. Dai, R., Yao, C., Liu, X., Liu, Y., Yan, Y., & Wang, Y. The effects of continuous cu(II) exposure compared with a shock dosage of cu(II) on pollutant removal in activated sludge. Desalin Water Treat, (2016) 57(13), 5842–5850.

PP94- Removal of lead using by N,N' diethyl dithiocarbamate end-group functional poly oligo ethylene glycole methacrylate (POEGMA) grafted poly styrene-divinyl benzene (PS-DVB) microbeads

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Toxic heavy metals in air, soil, and water are global problems that are a growing danger to the environment. There are hundreds of sources of heavy metal pollution, including the coal, natural gas, paper, and several industries, etc.¹ Thus, monitoring the concentration of heavy metals and their removal have become increasingly important in global systems. Lead (Pb), was metal of interest in this study due to their potential pollution impact into the environment. Moreover, toxication by Pb may lead to kidney failure, lung fibrosis and cancer; also these elements have negative effects on blood and bones. According to World Health Organisation (WHO), the permissible amounts of Pb in drinking water is 0.01, mg L⁻¹.² Therefore, its removal from the waters is a very important environmental concern. Polymers can also be manufactured in a wide range of physicochemical properties (size, porosity, hydrophilicity, etc.), and they are modifiable by inserting various ligands into the structure in order to produce specific sorbents.

In this study, N,N' diethyl dithiocarbamate end-group functional poly oligo ethylene glycole methacrylate (POEGMA) grafted poly styrene-divinyl benzene (PS-DVB) microbeads proposed for the removal of lead ions from the aqueous solutions and Pb ions determination was made by an inductively coupled plasma mass spectrometer (ICP-MS). The batch method is employed, and parameters such as pH, sorption time, sorbent amount on sorption, and sorbent capacity are optimized. The sorption kinetics and equilibrium isotherms of the removal of Pb ions are also elucidated. The proposed methodology will check via spike recovery experiments with water samples.

Keywords: Lead, sorbent, ICP-MS, Solid phase extraction.

References:

1. A. Bektashi, E. Myrtaj; Heavy Metals in the Shkodra Lake Ecosystem. J Environ Prot Ecol, (2014) 15 (3), 235.
2. F. Kaczala, M. Marques, W. Hogland; Lead and Vanadium Removal from a Real Industrial Waste Water by Gravitational Settling/Sedimentation and Sorption onto Pinus sylvestris Sawdust. Bioresource Technol, (2009) 100, 235.

PP95- Development of Thin Film in Tube Extraction Method for the Analysis of Endocrine Disruptor Pesticides

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Analysis of organic compounds such as drugs, pesticides, etc., which are of importance in terms of human health and environmental pollution, in clinical, food and environmental samples is of great importance today. These analyzes are based on the principle that the analyte can be taken in a suitable solvent environment without any breakdown or transformation and then measured at low levels in a reproducible and accurate manner. For this purpose, widely accepted methods are chromatographic methods. With these techniques, it is critical to be able to prepare analytical solutions quickly with fewer steps and less solvent consumption. The sample preparation step serves two main purposes; firstly cleaning up the sample and secondly increase the sensitivity of the method by enrichment process. Liquid-liquid extraction (LLE) and solid phase extraction (SPE) techniques from traditional sample preparation processes are time consuming techniques that consume a large amount of solvent. Therefore, there is a need for cheap and practical systems which may be alternative to existing techniques [1,2].

In this work, a very thin polymeric film coated on the inner wall of centrifuge tube was used for the extraction of endocrine disruptor pesticides. Polyacrylonitrile, Polyethylene glycol, Polydimethylsiloxane polymers and silicone were tested for the film preparation. The performance of extraction of Chlorpyrifos (CP), Permethrin (PRC), Penconazole (PNC), Bromopropylate (BRP) and Lambda-Cyhalothrin (LMD) pesticides were compared by using gas chromatography with electron capture detector. In the extraction step, 10 mL of water sample was inserted in the extraction tubes and mixed for 30 min. After the extraction the sample was removed from the tube and 500 μ L of ethanol was added and vortexed for 1 min. for the extraction and injected to the GC system. Variety of parameters affecting the signals such as pH, extraction solvent type, extraction solvent volume, vortex time, sample volume and salt effect on the extraction were optimized. Under optimized conditions the linear range was found between 2.5-50 ng mL⁻¹ and RSDs were no higher than 15%.

The developed method includes a new extraction container, which can be an alternative to the existing techniques used in sample preparation steps in pesticide analysis and shortens the analysis steps and offers a cheaper and more environmentally friendly alternative.

Keywords: Thin film in tube extraction, green chemistry, Pesticide, Chromatography

References:

- 1.F.O.Pelit, Ç. Yengin, *J. of Chrom. B.* (2014) 949-950, 109-114
- 2.F.O.Pelit, L.Pelit et al. *J. of Chrom. B.* (2012) 904, 35-41

PP96- Green Micro Solid Phase Extraction Techniques for the Determination of Endocrine Disruptor Pesticides

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Recent reports have identified chemicals capable of disrupting the endocrine system in environmental samples called as endocrine disrupting compounds (EDCs). Organophosphate EDCs, have been linked to ecological impacts at trace concentrations and detected in various samples. Therefore, there is an urgent need to develop sensitive methods to be able to detect concentrations of these chemicals at ng L⁻¹ level. For reaching this levels, residue analysis is carried out in a sequence preconcentration steps which are tedious, time consuming and labor intensive. In this study we show cheap, fast, environmentally safe sample preparation procedure which will improve the quality of analytical results in a greener way.

Solid phase extraction (SPE) have been employed for preconcentration of pesticides as a standard method. However, it displays some drawbacks such as multistage operation, large consumption of reagent, low enrichment factor and time consuming. These problems can be accomplished by using micro-SPE (μ -SPE) technique [1,2]. In this work the information about novel green extraction methodological approaches including μ -SPE is presented. Different methods including dispersive, magnetit and micropipette tipped type were used for fast analysis of EDC pesticides in water samples. Different nano particles namely, TiO₂, ZnO, SiO₂, a novel magnetic Fe₃O₄/Ni/Ni_xB nanocomposite and different clay types were utilized in fast micro extraction of pesticides. These nanofiller surfaces were used in dispersive μ -SPE, in micropipette tipped extraction and magnetic μ -SPE procedure

The parameters related to the extraction efficiency were screened. The performance of extraction of EDC pesticides were compared by using gas chromatographic systems. The regression coefficients were at least 0.99. Recoveries from spiked well waters range from 80 to 97% and RSDs were no higher than 15%.

These micro extraction methods share the advantages of the classical extraction methods such as excellent enrichment performance, easy operation and ability to employ a wide range of “green” extraction way.

Keywords: Micro-solid phase extraction, Green chemistry, Endocrine disruptors, Chromatography

References:

1. C. Aftafa, F.O. Pelit et al. **J. of Chrom A** (2014) 1361, 43-52.
2. E.E. Yalçinkaya, F.O.Pelit **J. of Porous Mater.** (2014) 21, 1151–1158.

PP97-Determination of Volatile Organic Compounds in Ambient Air by Needle Trap Device

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Volatile organic compounds (VOCs) are found as contaminants in ambient air. The presence of these compounds have resulted in various health problems and has created the need to determine and monitor VOCs in air [1-3]. The needle trap device (NTD) can be used as an extraction trap containing a sorbent within a needle [4]. Due to the fast, simple and solventless usage for chromatographically analysis, needle trap promising green tool for the field analysis. In this study needle trap packed with carbowax, carbopack X and divinylbenzene adsorbents was used for determination of several volatile organic compounds including n-pentane, dichloromethane, benzene, toluene, tetrachloroethylene, ortho-xylene, meta-xylene, para-xylene and styrene in the ambient air.

In this technique, 5 mL of air sample was passes throught the NTD device by means of sampling case at 1 mL/min flow rate. Adsorbed VOC compounds were desorped from NTD device at 290°C in the GC injection port. GC-FID analysis was performed and indoor atmosphere concentrations of targetted VOC compounds were calculated.

Various analysis parameters such as desorption time, desorption temperature, sampling flow rate were optimized. Calibration graph were drawn for each compound with good linearity and threshold limit values for targeted compounds in ambient air can be easily determined by this method.

Keywords: Needle trap device, Volatile organic compounds, Air analysis

References:

1. Elke, K.; Jermann, E et al. J. Chromatogr., A (1998) 826, 191–200.
2. Shojania, S.; Oleschuk, R. D. et al. Talanta (1999) 50, 193–205.
3. McComb, M. E.; Oleschuk et al. Talanta (1997) 44, 2137–2143.
4. Koziel, J. A.; Odziemkowski, M. et al. Anal. Chem. (2001) 73, 47-54.

PP98- Polypyrrole Solid Phase Microextraction Fiber Doped with Different Ions: Preparation, Characterization and Application to Pesticide Analysis

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In recent years, pesticides are being used more and more widely in agricultural activities. However, their hazardous effect on living organism are caused some disorders such as hormonal disorders, etc. Therefore, high sensitive sample preparation techniques are needed for indicate trace analysis. One of the most popular analytical technique is Solid-phase microextraction (SPME). This technique which an efficient, solvent-free, sample preparation method was first introduced by Arthur and Pawliszyn in 1990 [1]. Different types of commercially accessible polymers materials such as polydimethyl siloxane, polyethylene glycol, polyacrylate have been widely used as sorbent in SPME. However, they suffer from short lifetime and high cost.

In this study, we have been focused on fabricating low cost, simple, robust and long-life fiber with enhanced selectivity for the target analyte by introducing novel materials as functional coatings. For this purpose, polypyrrole (PPy) conducting polymer [2] was coated on 316 type stainless steel wire for as an alternative SPME fiber. Ions in supporting electrolyte are used as charge transfer agent (dopant) which is doped during the electro polymerization proses affects both oxidation and physical properties of the PPy polymers. In this work, different supporting electrolyte containing medium (ie. sodium dodecyl sulphate, tartrate and alkyimidazolium type ionic liquids with different alkyl chain) was used for electrochemical preparation of PPy. The thermal stability, surface and chemical properties of the fibers were investigated by thermogravimetric, XPS and FTIR methods respectively. Prepared SPME fibers were applied for the determination of pesticides in water by head space SPME method. The fibers performances were also compared with commercial ones.

Keywords: Solid Phase Microextraction, Polypyrrole Fiber, Pesticide, Chromatography

Acknowledgment: This study was supported by Tübitak (113Z672).

References:

1. C.L. Arthur, J. Pawliszyn, **Anal. Chem** (1990) 62, 2145.
2. K. Korba, L. Pelit **J. of Chrom. B** (2013) 929, 90–96.

PP99- Clinical Purpose of Solid Phase Microextraction Techniques: Biomarker Analysis for Human Cancer in Lung Cancer

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Lung cancer is the leading cause of cancer death throughout the world and its survival chance depends on the early diagnosis. Current techniques used for diagnosis are too expensive to widely used for screening purposes and also invasive and even harmful for patients. Furthermore, these techniques do not fulfill the requirements for reliable differentiation between lung cancer patients and healthy subjects. Nowadays, a vast number of researches are in the progress to develop non-invasive and reliable diagnosis [1,2].

The detection of biomarkers specific for lung cancer in human breath is a rather new strategy for screening purposes. This technique is non-invasive, painless and easy to perform. Exhaled air contains many VOCs produced during human metabolic processes including hydrocarbons along with alcohols, ketones and aldehydes. Modern techniques had enabled us to detect trace amount of commonly found VOCs in exhaled breath samples following an enrichment step.

In this project, it is aimed to develop a method for the early detection of lung cancer through analysis of commercial and lab-made SPME fibers and VOCs in the breathing air. Patients with untreated lung cancer cases and asthmatic patients with no known complaints were used. Experiments were performed with commercial PDMS / DVB / CAR SPME fiber, lab-made PTh, PPy and PANI SPME fibers. Breath samples were collected via lab-made sample collector where the VOC content was enriched on a SPME fiber and then, injected into the GC-MS system.

The best performing fiber in the classification was found as PTh SPME fiber. PLS-DA analyzes of the PTh SPME fiber were evaluated as a triple classification of a group of asthmatic and healthy subjects against a cancerous group and a dual classification of using only cancerous and healthy cases. In triple grouping, model and patient prediction success was around 70%, but when the asthma group was excluded and only the healthy and cancer group were classified, this rate reached 90%. With the established analysis method, the diagnostic success rate was found to be 73% in the experiments conducted with new lung cancer and healthy cases.

Acknowledgment: This study was supported by Tübitak (113Z672).

Keywords: Breath analysis, SPME, Chromatography, Lung Cancer

References:

- 1 B. Buszewski, M. Kêsy, T. Ligor, A. Amann, Biomed. Chromatogr. **2007**, 21, 553-566..
- 2.T.H. Risby, Ed. A. Amann, D. Smith, World Scientific, **2005**.

PP100- Determination of Chlorpyrifos by Solventless Method

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Pesticides are one of the most persistent organic pollutants present in the environment. The high toxicity, bioaccumulation in living things and non-biodegradability of these compounds represent risks to the environment in respect of human health [1]. Conventional liquid–liquid extraction (LLE) [2] and solid-phase extraction (SPE) [3,4] have been commonly used for the extraction of pesticides from aqueous matrices. LLE requires large amounts of toxic organic solvents. On the other hand, SPE is also requires an appreciable amount of toxic solvent for analyte desorption. Variety of expensive sorbent such as silica gel, C18 can be used as an adsorbent for the separation of pesticides from matrix. Therefore, there is need the development of solventless and also cheap analysis method for the determination of pesticide in different type of samples.

In this study pencil lead was used as an adsorbent for the extraction of chlorpyrifos in water samples. After extraction process pencil lead was heated by electrical current for the desorption of Chlorpyrifos in headspace vial. Then suitable volume of vapor phase chlorpyrifos directly injected into the gas chromatographic system. The “ppb” level of chlorpyrifos can be determined by this method in aqueous samples.

Keywords: Pencil Lead, Pesticide Analysis, Chromatography, Green Technique

References:

1. A Turnbull, Chlorinated Organic Micropollutants, Royal Society of Chemistry, London (1996).
2. D Barceló J. Chromatogr. A (1993) 643, 117
3. C Aguilar, F Borrull, R.M Marcé J. Chromatogr. A (1997) 771, 221
4. C Aguilar, F Borrull, R.M Marcé (1996) 14,1048

PP101- In vitro Antioxidant Activities of Different Solvent Extracts of *Crataegus monogyna* (hawthorn) from Malatya (Akçadağ), TURKEY

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Hawthorn (*Crataegus*) is widely distributed and cultivated in the East, Eastern Europe, North America and other north temperate regions for their medicinal properties. *Crataegus* is placed in a clade of genera within Rosaceae that has traditionally been referred to as subfamily Maloideae. It is one of the important forest fruits which there are different species and genotypes in Turkey. In several Turkey regions, common hawthorn, *Crataegus monogyna* is one of the species that is highly recommended in folk medicine and the ‘berries’ are compulsively consumed, because of its health benefits and abundant bioactive compounds[1]. However, very limited information is known about antioxidant activity of *Crataegus monogyna* from Turkey.

The present work aimed to determine the antioxidant activities of fruit, shell and seed parts of *Crataegus monogyna*, gathered in Malatya (Akçadağ), extracted with different solvents (water, methanol, methanol:water, 1:1, ethanol, ethanol:water, 1:1). Antioxidant properties of the extracts were determined by radical scavenging capacity, reducing power and metal chelating activity assays. The different fractions showed significant activities in all the free radical scavenging tests and these findings have also shown direct relationship between antioxidant activity and phenolic content (Figure 1, Table 1).

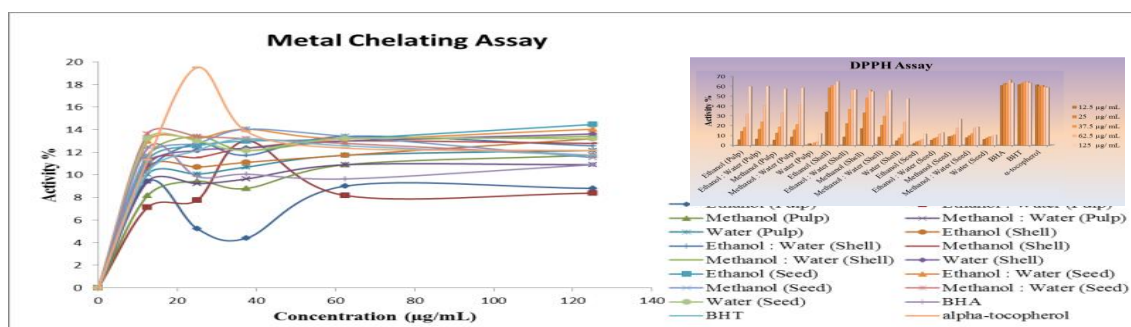


Fig.1. Metal (Fe^{2+}) chelation ability and DPPH radical activity of different extracts of *Crataegus monogyna* pulp, shell and seed

Table.1. Reducing power capacity of different extracts of *Crataegus monogyna* pulp, shell and seed

Samples	Reducing Power ($\mu\text{g/mL}$, 700 nm)			
	5,88	14,70	29,41	44,11
Ethanol (Pulp)	0.0838	0.0574	0.0941	0.1535
Ethanol : Water (Pulp)	0.0648	0.1011	0.0918	0.1535
Methanol (Pulp)	0.0804	0.0681	0.0918	0.1473
Methanol : Water (Pulp)	0.0128	0.0681	0.1168	0.1844
Water (Pulp)	0.1553	0.2179	0.1131	0.0883
Ethanol (Shell)	0.1952	0.2673	0.3056	0.4359
Ethanol : Water (Shell)	0.1145	0.1013	0.157	0.2321
Methanol (Shell)	0.1272	0.2347	0.2226	0.2756
Methanol : Water (Shell)	0.0797	0.0820	0.1410	0.2452
Water (Shell)	0.0684	0.0718	0.1019	0.1105
Ethanol (Seed)	0.0972	0.0960	0.0937	0.1032
Ethanol : Water (Seed)	0.0762	0.0866	0.1008	0.1156
Methanol (Seed)	0.0901	0.0901	0.1032	0.1308
Methanol : Water (Seed)	0.1231	0.0937	0.1255	0.1355
Water (Seed)	0.1179	0.1032	0.0786	0.0786
BHA	0.5002	1.1035	2.0633	3.3154
BHT	0.2513	0.6078	1.0467	1.5981
α -tocopherol	0.1297	0.2579	0.4399	0.5926

Keywords: DPPH, Metal chelating activity, Reducing ability, *Crataegus monogyna*

References:

1. Edward J.E., Brown P.N., Talent N., Dickinson T.A., Shipley P.R., A review of the chemistry of the genus *Crataegus*, 79 (2012) 5-26

PP102- Synthesis and antioxidant properties of new 4-acylaminomethyl) benzene sulfonamide derivatives†

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Sulfonamides are the first successfully synthesized antimicrobial drugs. The antimicrobial action mechanism of sulfonamides involves competitive inhibition of folic acid synthesis which prevents the proliferation of the bacteria [1]. Sulfonamides are considered a scaffold in medicinal chemistry to drug development with different biological activities. Due to the broad applicability of sulfonamides, it is important to find efficient methods for their synthesis. Among the broad biological properties of sulfonamides, studies on antioxidant properties are very limited [2]. Therefore, we planned to synthesize and investigate possible antioxidant properties of some novel sulfonamide derivatives bearing dipeptide moieties.

In the present study, we demonstrated the antioxidant and radical scavenging effects of the new sulfonamide derivatives using different *in vitro* bioanalytical methodologies. The antioxidant and radical scavenging activities of sulfonamide derivatives were compared to those of BHA, BHT and α -tocopherol. These comparisons were performed using a series of *in vitro* tests including DPPH radicals scavenging activities and reducing power (Fe^{3+} - Fe^{2+} transformation), and metal chelating on ferrous ion (Fe^{3+}) activities. Figure 1 shows that the most powerful metal chelating properties were found in **1** and **3**. In addition, the other the new sulfonamide derivatives (**2**, **4-7**) have marked Fe^{2+} chelating activity. As can be seen from Figure 1, all of compounds have also moderate Fe^{3+} reducing ability. However, all the new sulfonamide derivatives (**1-8**) showed negligible activity towards DPPH.

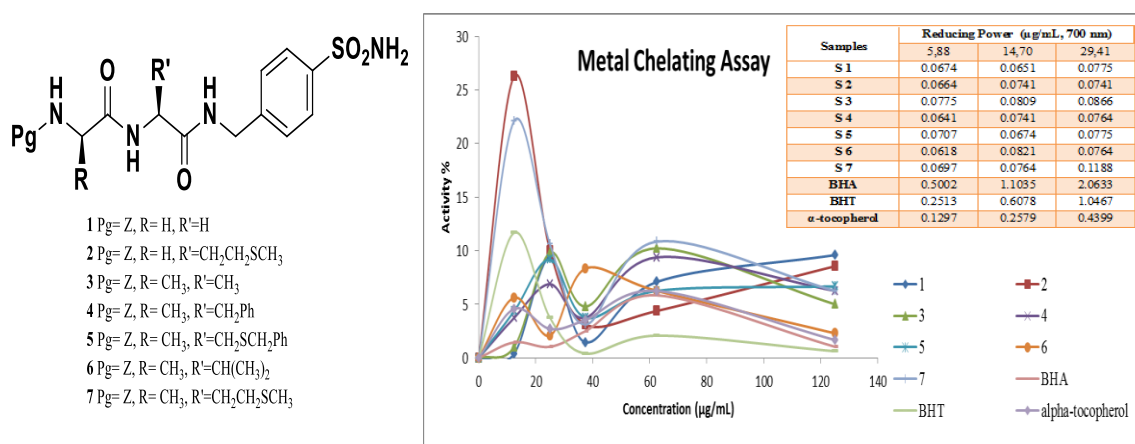


Figure 1. Total antioxidant activities of the new compounds and standard antioxidant compounds such as BHA, BHT and α -tocopherol.

Keywords: Antioxidants, sulphonamides, dipeptide.

References:

- Tacic, A., Nikolic, V., Nikolic, L., Savic, I. (2017) 6, 58-71.
- Göçer, H., Akıncioğlu, A., Öztaşkın, N., Göksu, S., Gülçin, İ. Arch. Pharm. (2013) 346, 783-792.

† We wish to thank TÜBİTAK (The Scientific and Technological Research Council of Turkey) for financial support (Grant No: 117Z293).

PP103- Extended Ratio Subtraction Spectrophotometric Method for Simultaneous Determination of Antihypertensive Drugs in Pharmaceutical Formulation

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A simple, economical and accurate method for simultaneous determination of Olmesartan medoxomil (OLM) and Amlodipine besylate (AML) in bulk powder and pharmaceutical formulations without prior separation steps.

In this method, extended ratio subtraction spectrophotometric method, the amplitudes in extended spectra at 254.84 and 359.6 nm were selected to determine of OLM and AML, respectively. The calibration curves are linear over the concentration range of 3.0 -21.0 ug/ml and 3.0-28 ug/ml for OLM and AML, with well accepted mean correlation coefficient for each drugs. This method was tested by analyzing synthetic mixtures of the above drugs and the mean recovery was $99.52 \pm 0.62 \%$ and $100.04 \pm 0.85 \%$, for each drug.

The proposed method was validated according to the ICH guidelines where accuracy, precision and repeatability was found to be within the acceptable limits. Moreover, no interference was found dosage forms excipients. This described method can be easily applied by the quality control laboratories in routine analysis of these drug in pharmaceutical dosage forms.

References

1. A. Hemdan, Spectrochim. Acta A, 2016, 164, 52-60
2. H.M. Lotfy, M.A.M. Hegazy, Spectrochim. Acta A, 2012, 96, 259-270.
3. C.V.Patel, A.P. Khandhar, A.A. Captain, K.P.Patel, Eurasian J.Anal.Chem.,2007, 2 (3), 159

PP104-Potential Use of Biowastes Modified with Citric and Sulfuric acids for Sorption of Pb and Cu

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Due to their adverse effects on human and environmental health, toxic metal pollution continues a serious issue. To determine their risk assessment, it is necessary to develop a rapid and sensitive method for accurate determination of those toxic metals. Because of the limitations in sensitivities of ICP-AES and flame AAS, preconcentration methods are commonly used [1]. If the preparation of adsorbent is non-expensive and the adsorption capacity is high enough, the preconcentration material can be applying as also a removing mediator. Modified biowastes are candidate for these purposes [2].

In this work, walnut shells were studied to be considered as sorption materials for toxic metals including Pb and Cu. The biosorption materials were chemically modified using citric and sulfuric acids, separately. The optimization of conditions was performed using parameters such as pH, contact time and initial concentration of analyte. It was found that the optimum pH was found to be 5 for both Pb and Cu using modified walnut by sulfuric acid. In case of using citric acid as modified reagent, the optimum pH is 4 for both two elements. The measurements were carried out by flame atomic absorption spectrophotometry and inductively coupled plasma-mass spectrometry (ICP-MS).

References

- [1]. O'Connell et al. Biores Technol 99 (2008) 6709–6724.
- [2] Feng N. et al. J Hazardous Materials 185 (2011) 49–54.

PP105- Spectrophotometric Studies on Ternary Metal Complexes of Naringenin and Lidocaine HCl – Mg (II) and Cu (II)

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Lidocaine Hydrochloride is an amide type local anesthetic that is absorbed quickly when applied locally. It has local anesthetic effect by preventing the ionic exchange and stabilizing the neuronal membrane during the initiation and communication of the neural response.

Naringenin has antioxidant and chelating properties due to its hydroxyl groups. These properties have an important place. Multidirectional studies on antioxidant role have been performed especially in the treatment of hyperlipidemia. Also studies on naringenin show that these flavonoids inhibit CYP3A4 enzyme inhibition as well as CYP1A2 enzyme activity.

The aim of this study is to develop a method by means of spectrophotometry for the quantitative determination of these two substances together, based on the knowledge that naringenin causes enzyme cascade and changes in lidocaine concentration.

This method, which is to be developed, is intended to be used in determining the concentration of the two substances together and in particular the change in the concentration of the lidocaine. The metal complex was determined by UV – Vis Spectrophotometric method with using mole rates method for Cu (II) – Naringenin - Lidocaine hydrochloride and Ca(II) - Naringenin – Lidocaine hydrochloride. Further, structures of ternary metals are supported by spectrophotometric method. The experimental data indicate the formation of metal 1:1 complexes Cu (II) – Naringenin - Lidocaine hydrochloride and Ca(II) - Naringenin – Lidocaine hydrochloride. Mg (II) and Cu (II) metals that were used for this study were studied as a ternary metal complexes of lidocaine hydrochloride and naringenin. An attempt was made to spectrophotometric mole rates method. By using mole ratios, the complex formation was observed in an one-to-one study.

Keywords: Lidocaine HCl, Naringenin, Spectrophotometer

References:

1. Hedek P., Trefil P., Stinborova M., Flavonoids-potent and versatile biologically active compounds interacting with cytochromes P450, Volume 139, Issue 1, 22 January 2002, Pages 1-21
2. Fuhr U., Klittich K., Staib AH., Inhibitory effect of grapefruit juice and its bitter principal, naringenin, on CYP1A2 dependent metabolism of caffeine in man, Volume 35, Issue 4 April 1993 ,Pages 431–436

PP106- Research of Bioactive Components of Some Vegetable Fats with Certain Pharmacological Properties

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Essential oil and fixed oil in leaves and fruits of some plants are benefited in the spices and food industry, in soap making and cosmetics and in the treatment of various diseases in folk medicine. These plants are important for human health as well as for new product formulations due to their phytochemical and antioxidant properties.

In this study; the plants and fruits of *Vitex agnus castus* (chasteberry), *Carthamus tinctorius* (safflower), *Urtica dioica* (dead nettle), *Aesculus hippocastanum* (horsechestnut) and *Nigella sativa* (black seed), which are widely used treatment of various diseases in the traditional therapy and which have pharmacological properties have been used. The fatty acid compositions of these samples were analyzed by GC-FID after the soxhlet extraction, and each fatty acid component was determined. As a result of GC-FID analysis of each oil extract, significant fatty acid components, such as oleic and linoleic, were found to be high.

Validation studies for method were performed by using various validation parameter, as a result of these studies, it was found that the method have high accuracy and precision for fatty acids. Oleic acid 26.16%, linoleic acid 63.34 % in safflower oil; oleic acid 62.016 % and linoleic acid 22.122 % in horsechestnut oil; oleic acid 48.559 % and linoleic acid 35.683 % in dead nettle oil; linoleic acid 60.078 % in black cumin oil and linoleic acid 55.861 % in chasteberry were found.

As a result of the analyzes, it was concluded that the studied samples had high content of unsaturated fatty acids. Thus, these oils can be considered widely in fields such as food, medicine and pharmacy.

Keywords: Vegetable fats, fatty acid composition, GC-FID

References:

1. Gunstone, F. D., (1999). Fatty Acid and Lipid Chemistry, Aspen Pub., Glasgow.
2. A. Akpınar Bayizit (2003). Doymamış Yağ Asitlerinin Beslenme ve Sağlık Açısından Önemi. *Gıda ve Yem bilimi-Teknolojisi*, 3, 28-29.

PP107- The investigation of histological alterations and accumulation in kidney, gill and muscle tissues of rainbow trout (*Oncorhynchus mykiss*) exposed to Dodine

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The application of the pesticides in agriculture has made inevitable for reduction of the proliferation of pest and to prevent increasing in food production. But it has known that the pesticides constitute one of the most hazardous groups of contaminants, posing potential risk to humans and other life forms. For example, the contamination of water bodies is a major concern for fish and other aquatic organisms which are major sources of protein. Accumulation of pesticides in these organisms has become a serious public health issue worldwide. Fish are used extensively for environmental monitoring because they concentrate to pollutants directly from water and diet, thus enabling the assessment of transfer of pollutants through the food web. Additionally, fish occupies different habitats in the ecosystem and have different feeding behaviors, thereby exhibiting different profiles of accumulation of contaminants such as pesticides (1-2).

The irrigation sites near Karakaya reservoir in the Malatya are noted for the production of large quantities of apricot and vegetables. However, to improve yield, there has been a widespread and unguided application of pesticides within the catchment by farmers.

As a result, this study was carried out to assess the level of pesticide residue in fish different tissue, and to evaluate the potential health risk posed to consumers. So, fish were exposed to different concentrations (control, 0.01, 0.1, 0.5 and 1 ppm) of Dodine for 96 hours and changes in histological and accumulation of gill, muscle and liver of rainbow trout (*Oncorhynchus mykiss*) were investigated. A validated method was applied to analyze the residue of pesticide in fish, namely Dodine; additionally, the QuEChERS method was employed to detect Dodine by LC/MS/MS.

For the QuEChERS method extraction, the recoveries obtained for the analytes (95-98 % with RSDs ≤ 6 %). The linearity of the Dodine method showed good linearity concentration ranges in the range 5–200 $\mu\text{g L}^{-1}$ and correlation coefficients (r^2) in the range 0.9993. The detection limit (LOD) and the limit of quantification (LOQ) were found as 1.02 $\mu\text{g L}^{-1}$ and 4.02 $\mu\text{g L}^{-1}$ for Dodine. It was observed that histopathological damage increased with concentration increase in liver tissues. In liver tissue samples exposed to 0.5 ppm, 0.1 ppm and 0.01 ppm dodine, it was observed that these damages decreased in a dose dependent. It were observed vacuolization, hydropic degeneration, eosinophilic picnotic nucleus cells, edema, mononuclear cell infiltration, congestion and necrosis in the liver tissues exposed to 1 ppm dodine. The methods used in the present study successfully analyzed Dodine accumulation in fish samples.

Keywords: Pesticide, rainbow trout, tissue, LC-MSMS, histopathological damage

References:

1. Essumang DK, Chokky L (2009) Pesticide residues in the water and fish (*Lagoon tilapia*) samples from Lagoons in Lagoons in Ghana. Bull Chem Soc Ethiopia 23(1):19–27
2. Jeyakumar T, Kalaarasi I, Rajavel AL, Anbu M, Kumar R (2014) Levels of organochlorine pesticide residues in water and sediment from selected agricultural sectors of Kanyakumari District, Tamil Nadu, India. Int J Environ Res 8(2):493–500

Acknowledgement: This study was supported by Inonu University BAP office with TYL-2017-640 project number.

PP108- Comparison of Different Normalization Techniques to Profile Metabolites on Cell Culture Studies

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Metabolomics is the comparison quantities of metabolites which have found in a cell or tissue and their identification and comparison of the variables determined in the study. Metabolomic studies are divided into two main categories as 'metabolite profiling' and 'targeted metabolomics' in the most general sense. Metabolite profiling is the study of the possible differences in metabolomic levels between groups identified through a defined variable (eg, a disease state). Metabolites can be found in tissues and cells a wide range of concentrations. Metabolomic studies are a reliable search for increases or decreases in metabolite concentrations so peaks are 'normalized' by interfering with statistical conversions of peak intensities over determined values. Normalization step leads to prevent increase or decrease from being caused by experimental factors such as analysis step or metabolite density and to observe these changes only by desired variable. There are many normalization methods used in metabolic studies. Normalization according to the peak average, normalization according to the peak median value, normalization according to the quality control sample, normalization according to the reference sample are frequently used in these methods (1).

In this study, samples of C group treated with cyclodextrin nanoparticles and samples of control group not treated with cyclodextrin nanoparticles were analyzed by LC/MS Q-TOF and treated with XCMS software. After that, four normalization methods which are normalization according to the peak average, normalization according to the peak median value, normalization according to the quality control sample, normalization according to the reference sample normalizing and results were compared with each other based on Principle Component Analysis (PCA) results and total matched peak numbers.

Keywords: metabolomics, cell culture, normalization, bioinformatics

References:

1. Griffiths W, Karu K, Hornshaw M, Woffendin G, Wang Y. Metabolomics and metabolite profiling: past heroes and future developments. *European Journal of Mass Spectrometry*. 2007;13(1):45-50.

PP109- Investigation of the Effect of Cyclodextrin Nanoparticles on MCF-7 Cancer Cells at Metabolome Level Using Q-TOF LC/MS

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Amphiphilic cyclodextrin (ACD) molecules have ability to spontaneously form nanoparticles. Because of these properties, they are used as nano-scale drug delivery system (1).

In this study, anticancer action mechanism of cyclodextrin nanoparticles on MCF-7 breast cancer cells has been investigated by using Q-TOF LC / MS at metabolome level. MCF7 breast cancer cell lines were treated with ACDs as drug delivery systems and studies were carried out on a series of nanoparticles (6-0) in which the cell proliferation inhibitory effect (antiproliferative effect) was observed as the highest amount. The cell lines that were handled by ACD were used as treated cell lines and named as T group. The group without any treatment was used as a control group and was named as group C. First, cytosolic fractions were obtained from T and C groups. Subsequently, metabolites in cytosolic fractions (molecules have molecular weight <3000 daltons) were purified by ultrafiltration using Amicon Ultra-0.5 mL (3k) and separated by Q-TOF LC / MS using Zorbax HILIC Plus (2.1 x 50 mm, 1.8 µm) chromatography column. For this, a gradient elution program was utilized and the analysis starting with 90% acetonitrile (containing 0.1% Formic Acid), 10% Water (containing 0.1% Formic Acid), 90% Water (containing 0.1% Formic Acid), 10% Acetonitrile (containing 0.1% Formic Acid). Thus, metabolites in different polarities can be distinguished from each other. Studies were performed 6 times and when statistical analysis of the results of the analyzes were made, it was found that between the groups T and C, amount of 35 metabolites produced or increased in metabolic pathways affected by ACD effects was at least 2 times changed (increased or decreased). MS/MS spectra of these metabolites were searched in the Human Metabolomic Data Base (HMDB) and the preventive effect of ACD on proliferation of cancer cells was investigated at metabolic level by metabolic pathway analysis. ACDs, which are used as drug delivery systems, have also been shown to be effective at the molecular level in preventing the proliferation of cancer cells.

Acknowledgment: This study is supported by TÜBİTAK 115S456 project.

Keywords: metabolomics, nanoparticles, cancer

References:

- 1) Bilensoy, E.; Gürkaynak, O.; Ertan, M.; Şen, M.; Hıncal, A.A. *J. Pharm. Sci.* **2008**, 97, 1519.

PP110- Flow Injection Amperometric Analysis of Hydrazine Using Gold Nanoparticles Modified Pencil Graphite Electrode

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Hydrazine ($\text{H}_2\text{N}-\text{NH}_2$) is generally used as textile dyes, pesticides (in agriculture), reducing agent (in nuclear fuels) and pharmaceutical applications in industrial fields. Hydrazine is a toxic compound and it can easily be taken to body by oral, dermal or inhalation. Its concentration in high levels can cause irritations in eyes, nose and throat [1]. Therefore, determination of hydrazine in real samples becomes more important for human health and environment. Among the developed methods, electrochemical methods have found a great attention due to its simple, fast and low cost determination of hydrazine. In addition, flow injection analysis (FIA) is a useful approach and offers many advantages for routine analytical determinations such as low sample consumption, short analysis time based on a transient signal measurement in a flow-through detector, and an online method for difficult operations of separation and chemical conversion of analyses into detectable species [2].

In this work, FI amperometric hydrazine sensor was described using pencil graphite electrode (PGE) which was pretreated at +1.45V for 60 s in 0.10 M phosphate buffer solution (PBS) contains 0.10 M KCl. Then, gold nanoparticles (AuNPs) were electrodeposited on pretreated PGE (p.PGE) in 0.02 M phosphate buffer solution contains 0.20 M HAuCl_4 by recording their cyclic voltammograms (CVs) with various cycle numbers. Modified electrodes were electrochemically characterized by recording their CVs and impedance curves in redox probe solution contains 0.01 M $\text{Fe}(\text{CN})_6^{3-/4-}$. Surface morphologies of the proposed electrodes were also examined by taking their Scanning Electron Microscope (SEM) images, Energy Dispersive X-Ray (EDX) spectra and X-Ray Diffraction (XRD) patterns. Cyclic voltammograms recorded at pH 10 Britton Robinson Buffer solution shows that a broadened and highly irreversible peak attributed to oxidation of hydrazine was observed at 500 mV at the bare pretreated PGE, while a well-defined irreversible oxidation peak was observed at -75 mV at the AuNPs/p.PGE. A shift in the overpotential to more negative direction (about 575 mV) and an enhancement in the peak current indicate that the AuNPs/PGE presents an efficient electrocatalytic activity toward oxidation of hydrazine. Then, flow injection analysis (FIA) of hydrazine was performed based on its electrocatalytic oxidation at the AuNPs/p.PGE using a novel home flow cell which was constructed for PGE. Interference effects on electrocatalytic peak current of hydrazine were further investigated in the presence of various biologically important molecules for selectivity of the proposed sensor. In last step, proposed electrode was used for the determination of hydrazine in a real sample.

Keywords: Hydrazine, Flow Injection Analysis (FIA), Gold Nanoparticles, Pencil Graphite Electrode.

References:

1. J.A. Oh, H.S. Shin, Simple Determination of Hydrazine in Waste Water by Headspace Solid-Phase Micro Extraction and Gas Chromatography-Tandem Mass Spectrometry after Derivatization with Trifluoro Pentandione, *Anal. Chim. Acta* (2017) 950 57-63.
2. S. Karakaya, Y. Dilgin, Flow Injection Amperometric Analysis of H_2O_2 at Platinum Nanoparticles Modified Pencil Graphite Electrode, *Electroanalysis* (2017) 29 1626-1634.

This study was conducted as part of a project and Msc thesis of İsmail TEOMAN.

PP111- Enantioseparation of novel chiral pyrazolines in HPLC by using methanol as a mobile phase

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Pyrazoline derivatives are in general well-known five-membered nitrogen-containing heterocyclic compounds. Several pyrazoline derivatives have been found to possess considerable biological activities which stimulated research activities in this field. As far as the different pyrazoline isomers are concerned, 2-pyrazoline derivatives became the most frequently studied pyrazolines [1]. Diversely substituted pyrazolines, embedded with a variety of functional groups, are important biological agents and a significant amount of research activity has been directed towards this chemical class [2].

In the frame of present work, 41 originally synthesized chiral pyrazoline derivatives were analyzed by using polysaccharide-based chiral stationary phases and methanol as a mobile phase in high performance liquid chromatography system. Seven different polysaccharide based chiral stationary phases (CSP) were used in this study [3, 4]. The columns: Amylose-1 (150 × 4.6 mm, 3 µm), Lux Amylose-2 (250 × 4.6 mm, 3 µm), Lux Cellulose-1 (250 × 4.6 mm, 5 µm), Lux Cellulose-2 (250 × 4.6 mm, 3 µm), Lux Cellulose-3 (250 × 4.6 mm, 3 µm), Lux Cellulose-4 (250 × 4.6 mm, 3 µm) and Lux i-Cellulose-5 (250 × 4.6 mm, 5 µm) used in this study were provided by Enantiosep and Phenomenex. As a result of the investigations, quite many complementary separations of enantiomers were observed on the used columns. Complementarity of the columns allows a significant increase in the combined success rate of all stationary phases. The aspects related to the structure-retention and structure-enantioselectivity relationships are also discussed.

Keywords: Pyrazoline derivatives, enantioseparation, HPLC

References:

1. Y.F. Sun, Y.P. Cui, Dyes Pigm. 81 (2009) 27.
2. N. Gökhan-Kelekçi, S. Yabanoğlu, E. Küpeli, U. Salgın, Ö. Özgen, G. Uçar et. al., (2007). A new therapeutic approach in Alzheimer disease: Some novel pyrazole derivatives as dual MAO-B inhibitors and antiinflammatory analgesics. Bioorganic and Medicinal Chemistry, 15 (17), 5775-5786.
3. Y. Okamoto, E. Yashima, Polysaccharide Derivatives for Chromatographic Separation of Enantiomers. Angewandte Chem., 37 (1998) 1020.
4. B. Chankvetadze (2012), Recent developments on polysaccharide-based chiral stationary phases for liquid phase separation of enantiomers, J. Chromatogr. A, 1269 (2012) 26

PP112- Separation of enantiomers of the novel chiral pyrazoline derivatives in HPLC via polysaccharide based chiral selectors and acetonitrile as mobile phase

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Separation of enantiomers is a hot topic for academic research, as well as in modern pharmaceutical industry. The reason for this is that more than 50 % of the small molecule drugs currently in use are chiral compounds and significant part of them are racemates consisting of an equimolar mixture of two enantiomers [1]. Chiral pyrazoline derivatives are in general well-known five-membered nitrogen-containing heterocyclic compounds.

In the frame of this study, relationships between enantioselectivity and chemical structure of different chiral stationary phases (CSPs) and chiral compounds was investigated. In order to achieve this goal, the enantiomers of 41 chiral pyrazoline derivatives were separated by using polysaccharide based chiral stationary phases (CSP) and acetonitrile as a mobile phase in high performance liquid chromatography (HPLC). Seven polysaccharide based CSPs were used in this study namely Amylose-1 (150 × 4.6 mm, 3 µm), Lux Amylose-2 (250 × 4.6 mm, 3 µm), Lux Cellulose-1 (250 × 4.6 mm, 5 µm), Lux Cellulose-2 (250 × 4.6 mm, 3 µm), Lux Cellulose-3 (250 × 4.6 mm, 3 µm), Lux Cellulose-4 (250 × 4.6 mm, 3 µm) and Lux i-Cellulose-5 (250 × 4.6 mm, 5 µm) which were provided by Enantioseph. and Phenomenex.

As this study illustrates, the affinity of enantiomers of chiral pyrazoline derivatives towards polysaccharide-based chiral stationary phases is dependent on the nature of the chiral selector [2,3]. Further studies of these phenomenon may provide useful information for understanding the chiral recognition mechanisms with polysaccharide-based chiral stationary phases.

Keywords: Pyrazoline derivatives, enantioseparation, HPLC

References:

5. Bentley, R., Chem. Soc. Rev. 34 (2005) 609.
6. West C, Enantioselective separations with supercritical fluids review. Curr Anal Chem. 10 (2014) 99
7. B. Chankvetadze (2012), Recent developments on polysaccharide-based chiral stationary phases for liquid phase separation of enantiomers, J. Chromatogr. A, 1269 (2012) 26

**PP113- Determination of Harmful Aromatic Amine Products after Azo Dyes
Reductive Cleavage by Gas Chromatography Mass Spectrometry**

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Azo dyes are dyes containing a nitrogen-nitrogen double bond and they constitute more than 50% of the dyes being used worldwide. Azo dyes have many areas of application and these include the plastics, food, cosmetics, textiles and ceramics industries [1]. Azo dyes are not harmful by themselves but upon undergoing reductive cleavage, certain aromatic amines can be released and these pose severe health risks to consumers and workers. Some countries have entirely banned the use of azo dyes while others have set maximum concentration limits in commercial products [2]. It is therefore important to analyze azo dyed products to ensure that harmful aromatic amines are not released or below the allowable limit when detected.

In this study, a simple and effective liquid-liquid extraction method was developed for the determination of selected aromatic amines in some dyed products by gas chromatography-mass spectrometry. The analytes show good linearity over a wide concentration range with low relative standard deviation. The figures of merit obtained for the analytes were satisfactory and the applicability of the method to real samples was determined by performing spiked recovery tests. The percent recovery results calculated ranged between 92-114%, showing that the method can be used to quantify the analytes with good accuracy. Some dyed products obtained from the market were analyzed with the developed method and none of the aromatic amines detected was over the 30 mg kg⁻¹ limit. The method developed was simple and very economical compared to standard methods of azo dyes determination.

Keywords: Azo dyes, LLE, aromatic amine, GC-MS

References:

1. I.A. Bhatti, S. Adeel, S. Parveen, M. Zuber, Dyeing of UV irradiated cotton and polyester fabrics with multifunctional reactive and disperse dyes, J. Saudi Chem. Soc. (2016) 20 178-184.
2. G. S Nadiger, Azo Ban, Eco-Norm and Testing, Indian J. Fibre Text. (2001) 26 55-60.

PP114- Determination of Cadmium in Environmental Samples by Slotted Quartz Tube Flame Atomic Absorption Spectrometry after Vortex Assisted Switchable Liquid-Liquid Microextraction

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Industrialization and modernization has led to an increase in anthropogenic activities such as fossil fuel consumption, mining, drilling, industrial waste disposal and chemical spillage. These activities have resulted in and continue to cause pollution of natural environmental resources. Cadmium is well known toxic element which enters the environment and enters the human body through cigarette smoking, skin contact with contaminated particulate matter, drinking contaminated water and ingesting contaminated foods [1]. The International Agency for Research on Cancer (IARC) has classified cadmium as a Group one carcinogen and cadmium has been reported to cause diseases of the heart, lungs liver, bones and kidneys [2]. Determination of cadmium in environmental samples is therefore a necessity to ensure good human health. Flame atomic absorption spectrometry (FAAS) is a well-established and commonly used technique for the determination of cadmium and other trace metals but it does not have a high enough sensitivity to detect trace amounts of most metals.

This study employed switchable liquid-liquid microextraction (SLLM) to preconcentrate cadmium from environmental samples into measurable quantities for the FAAS. The absorbance signals after preconcentration were further enhanced using slotted quartz tubes to enhance the residence time of cadmium atoms in the flame. A comprehensive optimization study was performed on extraction and instrumental parameters to obtain the highest possible signal. Under optimum extraction and instrumental conditions, the limit of detection (LOD) and limit of quantification (LOQ) were calculated as 0.70 µg/L and 2.6 µg/L, respectively. Seven replicate measurements of the lowest concentration recorded low relative standard deviations which indicated high precision. Percent recoveries between 90–103% were obtained for lake water and wastewater spiked at different concentrations within the linear calibration range. This confirmed that the method can be applied to the selected samples for an accurate and precise determination of cadmium.

Keywords: Cd, FAAS, SQT, switchable solvent extraction

References:

1. M. S. Fındıkoğlu, M. Fırat, D. S. Chormey, F. Turak, Ç. Şahin, S. Bakirdere, Determination of Cadmium in Tap, Sea and Waste Water Samples by Vortex-Assisted Dispersive Liquid-Liquid-Solidified Floating Organic Drop Microextraction and Slotted Quartz Tube FAAS After Complexation with a Imidazole Based Ligand, Water, Air, & Soil Poll. (2018) 229 37.
2. M. Fırat, S. Bakirdere, M.S. Fındıkoğlu, E.B. Kafa, E. Yazıcı, M. Yolcu, Ç. Büyükpınar, D.S. Chormey, S. Sel, F. Turak, Determination of trace amount of cadmium using dispersive liquid-liquid microextraction-slotted quartz tube-flame atomic absorption spectrometry, Spectrochim. Acta B 129 (2017) 37-41.

PP115- Switchable-Polarity Solvent-Liquid-Liquid Microextraction of Piperine from Black and White Pepper Prior to its Determination by HPLC

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A switchable-polarity solvent was synthesized from triethylamine (TEA)/water/CO₂ (1:1, excess, v/v) via proton transfer reaction and was used for the extraction of piperine from black and white pepper prior to its determination by high-performance liquid chromatography (HPLC).

The proposed method was termed as switchable-polarity solvent-liquid-liquid microextraction (SPS-LLME). Optimum extraction conditions were found as follows: Extraction solvent, TEA; extraction volume, 750 µL; and extraction time, 10s. Addition/ volume of organic modifier, centrifugation time, and ionic strength had no significant effect on the extraction efficiency. The final extract was suitable for direct injection into HPLC without any further pretreatment. Optimum HPLC conditions were found as follows: Column, Agilent Eclipse XDB C18 (4.6 mm ID × 15 cm, 5 µm); separation temperature, 20 °C; mobile phase, ACN:H₂O, 45:55 (% v/v); flow rate, 1.1 mL min⁻¹, and injection volume, 5 µL. Piperine was monitored using a diode-array detector (DAD) at 346 nm. Limits of detection (LOD) and quantitation (LOQ), calculated based on 3S_b/m and 10S_b/m, were found as 2.7 and 9.3 mg g⁻¹, respectively. Calibration graphs showed good linearity with coefficients of determination (R²) higher than 0.995. SPS-LLME-HPLC was applied for the extraction of piperine from seven black and one white pepper samples from different origins and percentage relative recoveries (%RR) were obtained within the range of 95.6% and 104.6%. The results proved that SPS-LLME can represent a simple, rapid and green alternative method for the extraction of piperine from black and white pepper prior to HPLC analysis.

Keywords: Black pepper, HPLC, Liquid-liquid microextraction, Piperine, Switchable-polarity solvent

PP116- Development of spectrofluorimetric determination of nateglinide in pharmaceutical preparations

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A sensitive spectrofluorimetric method was developed for the determination of nateglinide in pharmaceutical preparations. The method is based on reaction of nateglinide with 1-dimethylaminonaphthalene-5-sulphonyl chloride (dansyl chloride) in carbonate buffer of pH 11 to yield a highly fluorescent derivative that is measured at 517 nm after excitation at 397 nm. The described method was validated and the analytical parameters of linearity, limit of detection, limit of quantification, accuracy, precision (intra_ and inter_day), robustness and recovery were evaluated. The assay was linear over the concentration range of 25–250 ng/mL. The proposed method was applied to study of nateglinide in pure and pharmaceutical preparations. The preparation was also analyzed with an official method and statistical comparison by *t*- and *F*- tests revealed that there was no significant difference between the results of the two methods with respect to mean values and standard deviations at the 95% confidence level.

Keywords: Nateglinide, dansyl chloride, derivatization, pharmaceutical preparations

PP117- Determination of Cadmium in Aqueous Samples Using a Sensitive Closed Batch Vessel Hydride Generation Atomic Absorption Spectrometry Method

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Cadmium occurs naturally in the earth's crust and it is commonly found in the ores of copper, zinc and lead. It has a wide area of application including the manufacture of pigments, alloys, coatings, rechargeable batteries and photovoltaic devices [1]. Despite the benefits derived from its applications, cadmium is a toxic metal that accumulates in organs such as the lungs' liver' kidneys' bones, reproductive and cardiovascular systems over a period of time and eventually cause health problems and carcinogenesis [2].

This study presents a step-by-step optimization of hydride generation atomic absorption spectrometry (HGAAS) method for the determination of cadmium in water samples at trace levels. Hydride generation is a very useful technique which overcomes matrix interference and transports close to 100% of generated hydride for atomization and absorbance measurements, thus, it offers selectivity and sensitivity. Thus, parameters affecting the generation of cadmium hydride, transportation of the generated hydride to atomizer cell, and decomposition of the metal hydride were optimized. The system parameters optimized included reductant concentration and pump period, acid type and concentration, atomization temperature and inert gas flow rate. The efficiency for cadmium hydride generation was significantly increased at the end of the optimization study and the optimum parameters were used to determine the analytical performance of the method. A linear dynamic range was obtained for calibration standards between 0.10 µg/L and 2.0 µg/L. A very low detection limit of 0.029 µg/L was calculated for the method and this is comparable to other sensitive instrumental techniques such as ICP-MS and GFAAS. The method was validated with a spring water certified reference material and the results experimentally determined was compatible with the certified values. Ten river water samples and one tap water sample were analyzed under the optimum method but cadmium was not detected in any of them according to the 0.029 µg/L detection limit.

Key words: Cadmium; AAS; hydride generation; drinking water.

References:

1. L. Jarup, A. Akesson, Current status of cadmium as an environmental health problem, *Toxicol. Appl. Pharmacol.* (2009) 238 201-208.
2. M. P. Waalkes, Cadmium carcinogenesis in review, *J. Inorg. Biochem.* (2000) 79 241-244.

PP118- Study of ^{210}Po and ^{210}Pb in the marine environments of coastal Gelibolu (Marmara Sea)

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Some of the daughter products in the ^{222}Rn -decay series, such as ^{210}Po and ^{210}Pb , have been widely used as tracers in aquatic systems.

This study presents the activity concentrations of ^{210}Po and ^{210}Pb in the marine. Samples of marine surface sediments collected in Gelibolu (coast of Marmara Sea), have been examined to measure alpha radioactivity. The activity of these two radionuclides was determined by radiochemical separation of ^{210}Po and counting the activity using an alpha counter. The activity concentrations of ^{210}Pb and ^{210}Po in the sediment samples were analyzed as 22 ± 3 - 48 ± 7 Bq kg⁻¹ dry weight and 19 ± 4 - 37 ± 3 Bq kg⁻¹ dry weight, respectively.

Keywords: Sediment, Marine, Radioactivity.

PP119- Hydrothermal Synthesis and Characterization of Nanosorbent from Biowastes and Their Potential for Biosorption of Toxic Metals

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After intensive studies on sorbents based different materials for preconcentration and removing of toxic metals, it is necessary to development new adsorbents having more adsorption capacity and cheaper. The modified biowastes are candidate for these purposes [1].

In this work, the chemically-modified banana peels were studied to be synthesized as nanosorbent using hydrothermal procedure. The obtained nanosorbents were characterized using FTIR, SEM, nanoparticul sizer and BET. The characterized adsorbents were considered for potential of preconcentration and removal of toxic metals. The observed surface area was compared to the activated carbon without hydrothermal nanosorbent synthesis for the same biowaste, banana peel. The FTIR and SEM outputs were evaluated and nanoparticul sizer recovery was considered. The other results will be presented in poster section.

References

- [1] Feng N. et al. J Hazardous Materials 185 (2011) 49–54.

PP120- Synthesis and Characterization of Calcium Fructoborate and Its Bioactivity on Skin Cancer

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Skin cancer is the most common of all human cancers. It is estimated that one in seven people in the United States will develop some form of skin cancer during their lifetime. Skin cancer is a type of cancer characterized with decreased apoptosis or increased proliferation in epidermis. Early prevention or treatment of cancer at an early stage significantly reduces economic costs and mortality rates.

In this study, CaFB was synthesized by using D-fructose solution, boric acid and calcium carbonate (Figure 1).

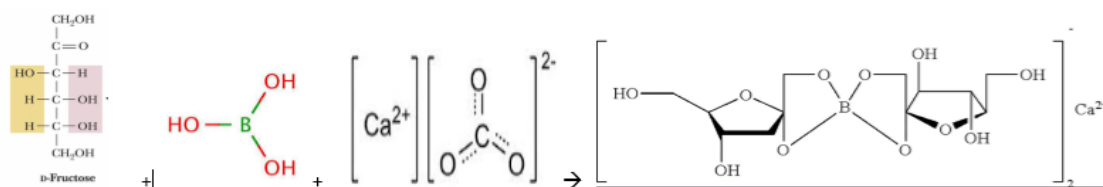


Figure 1: Synthesis of CaFB

The obtained production was characterized by DSC, TGA-DTA, Infrared (IR), Nuclear Magnetic Resonance (NMR), and ICP-MS. Different concentration of the characterized product and commercial product of CaFB were applied to Scc-25 cells. MTT test was conducted on and cytotoxic effects as well as inhibition concentration 50 (IC50) values of these two product were evaluated. It was determined that the characterized product and commercial product of CaFB inhibited cell proliferation and viability at different ratios according to the control group. the characterized product and commercial product of CaFB showed cytotoxic effects, especially at doses higher than 5 mM. IC50 doses were found to be 15.16 mM and 22.10 mM, respectively. This result indicate that these two products have an inhibitory effect on Scc-25 cell proliferation. As for IC50 doses the characterized product was observed to be even more effective.

Key words: Scc-25, Calcium Fructoborate, Skin cancer

Acknowledgment: This study was supported by BOREN (2016-31-07-15-003).

PP121- Assessment of Antimicrobial Activities of Garlic-Lemon Cure and Its Bioactive Compounds

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Thousands of natural products with the potential to act as antimicrobial agents or as a structural lead compounds still await further investigation although researches into natural products have demonstrated significant progress in the discovery of new compounds with antimicrobial activity (1). Studies exploiting the structure activity aspects of these natural compounds may provide both additional antimicrobial leads and drugs, and also significant insight into potential possibilities to overcome the antimicrobial resistance.

In this study, garlic-lemon cure used in folk medicine was subjected to work for both its antimicrobial activity and bioactive compounds. Firstly, it was tested in vitro against 5 bacterial species including subgroups of gram positives and gram negatives, and strains by the agar diffusion method. Secondly, the cure sample was analyzed for bioactive compounds including flavonoids and other phenolics by HPLC-DAD.

It was found that the cure sample showed moderate antibacterial activity (between 6-12 mm inhibition zone) against gram negative species while high antibacterial activity (up to 20 mm inhibition zone) against gram positive species. These results can compete with some drugs such as ampicillin used for this purpose. Among bioactive compounds, eriodictyol and its derivatives, luteolin derivative, chlorogenic acid, gallic acid derivative, trans-ferulic acid and apigenin derivative and p-coumaric acid were found in descending order.

Keywords: Bioactive compounds, antimicrobial activity, HPLC, traditional medicine

References:

- 1- Turumtay H, Midilli A, Turumtay EA et al., Gram (-) microorganisms DNA polymerase inhibition, antibacterial and chemical properties of fruit and leaf extracts of *Sorbus acuparia* and *Sorbus caucasica* var. *yaltirikii*, Biomedical Chromatography, (2017) 31(6) UNSP e3901.

PP122- Nitric Oxide Radical Scavenging Activity Measurement of Ascorbic Acid with Gold Nanoparticles

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Reactive nitrogen species (RNS) take part in cell signalling under various physiological conditions and also provide host defense against bacterial and fungal pathogens. While neither $\cdot\text{NO}$ nor $\text{O}_2^{\cdot-}$ are strong oxidants, ONOO^- is highly reactive and can oxidize DNA, proteins and lipids [1]. Although sodium nitroprusside is stable in a dry environment, it produces nitric oxide radicals in aqueous solution at physiological pH. Nitric oxide radicals are scavenged in the presence of antioxidant materials, and the excess of this radical is converted into nitrite and nitrate by molecular oxygen [2]. We have developed a novel nanomaterial-based colorimetric method for sensitive and selective determination of nitrite. A literature search reveals that there is no useful gold nanoparticles-based method for measuring either $\cdot\text{NO}$ or its scavengers. In the developed method, some parameters were optimized such as temperature, solvent medium, sodium nitroprusside concentration and reaction time. Before being scavenged with ascorbic acid, the nitric oxide blank absorbance (according to the proposed nano-colorimetric method) of the reference solution was stabilized at $A = 1.100$ after optimization of reaction parameters. For nitric oxide radical scavenging activity of ascorbic acid, linear calibration equation (between 3.63×10^{-5} – 1.82×10^{-4} mol L⁻¹ final concentration range) and molar absorptivity (ϵ) of the developed method were $A = 3.40 \times 10^3 C_{\text{AA}} + 3.28 \times 10^{-2}$ and $\epsilon = 3.40 \times 10^3 \text{ L mol}^{-1} \text{ cm}^{-1}$, respectively, with a correlation coefficient of $r = 0.9997$. Nitric oxide scavenging activity order of different classes of antioxidants are currently being determined with the proposed nanomaterial-based method.

Keywords: nitric oxide radical, gold nanoparticles, scavenging activity

References:

1. Apak, R., Demirci Çekiç, S., Uzer, A., Çelik, S. E., Bener, M., Bekdeşer, B., Can, Z., Sağlam, Ş., Önem, A. N., Erçağ, E., Novel Spectroscopic and Electrochemical Sensors and Nanoprobes for the Characterization of Food and Biological Antioxidants, *Sensors* (2018) 18 1-35.
2. Apak, R., Özyürek, M., Güçlü, K., Çapanoğlu, E., Antioxidant Activity/Capacity Measurement. 3. Reactive Oxygen and Nitrogen Species (ROS/RNS) Scavenging Assays, Oxidative Stress Biomarkers, and Chromatographic/Chemometric Assays, *J. Agric. Food Chem.* (2016) 64 1046-1070.

PP123- Investigation of the fatty acid compositions of *Allium tuncelium* by GC

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Tunceli garlic (*Allium tuncelianum*), which is located in Tunceli province and especially on Munzur mountain skirts, is an endemic plant species found in the Hozat county, mainly in Ovacık county. Garlic consumed as a food source has a protective effect against many health problems [1]. It decreases cholesterol and sugar level in the blood and also improves blood circulation reducing the risk of heart attack.

In this study, fatty acid chemical composition of *Allium tuncelianum* was investigated because it is considered that this composition can contribute the health effects. Fatty acids of *Allium tuncelianum* were determined by gas chromatography(GC-FID) after total fat was extracted by soxhlet extraction method. It was found that 15 of the 37 fatty acids investigated were detected in the sample extracts. Fatty acid contents were expressed in % of total fatty acids methyl esters.

The dominant fatty acids in the sample extracts were determined as polyunsaturated fatty acids (PUFAs), (linoleic, C18:2n6 (41,59 %) and monounsaturated fatty acids (MUFAs), oleic, C18:1n9 (33,30%) and a small amount of saturated fatty acids (SAFAs), mainly consisting of palmitic-C16:0 (11,74 %), henoicosanoic-C21:0, (6,95 %) and stearic-C18:0 (2,36 %) acids, respectively.

Keywords: *Allium tuncelianum*, Fatty acid, Gas chromatography.

References:

1. Shukla, Y, Karla, N., Cancer chemoprevention with garlic and its constituents, Cancer Letters (2007) 247 167-81.

PP124- Development of a Sensitive and Accurate Dispersive Liquid-Liquid Microextraction Strategy for the Determination of Harmful Azo Dyes Products by Gas Chromatography-Mass Spectrometry

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Dyes containing the azo functional group are referred to as azo dyes, and they make up a great percentage of dyes used for different applications. Some azo dyes have the tendency to undergo azo bond cleavage to produce many aromatic amines, out of which 24 have been identified to be harmful (including carcinogenesis) to humans and other living organisms [1, 2]. Determination of these harmful compounds even at low levels is therefore very important to ensure good human health.

A comprehensive optimization of dispersive liquid-liquid microextraction was performed for the determination of 24 harmful aromatic amine products of azo dyes by gas chromatography mass spectrometry. Extraction parameters such as types and amounts of extraction solvent and dispersive solvent, salt effect and amount of salt were optimized to obtain high sensitivities for all analytes. Under the optimum conditions, analytical figures of merit were determined for each analyte. In order to increase the precision of replicate measurements, deuterated bisphenol A was added to all samples and standards, and the ratio of standard/sample peak area to internal standard peak area was used for all calculations. Without the internal standard, the linear correlation coefficients (R^2) obtained for the analytes were between 0.9952 – 0.9994, but with the internal standard, improved R^2 values between 0.9993 – 0.9999 were obtained. The limits of detection calculated for the analytes ranged between 0.06 – 28 ng/mL. Accuracy and applicability of the developed method was tested by performing spiked recovery studies on tap water and two municipal wastewater samples. The percent recoveries obtained for tap water spiked at 50 ng/mL were between 91 – 106%. In order to overcome the high recovery results obtained for the waste water samples, matrix matching method was used to eliminate matrix interferences. The recovery results obtained using matrix matched standards for the samples spiked at 50 ng/mL and 100 ng/mL were found to be between 93 – 103% and 95 – 101%, respectively. The recovery results validated the accuracy and applicability of the developed method.

Keywords: DLLME, GC-MS, Aromatic Amines, Azo Dyes

References:

1. Letašiová, S., Medveďová, A., Šovčíková, A., Dušinská, M., Volkovová, K., Mosoiu, C., Bartonová, A., Bladder Cancer, A Review of the Environmental Risk Factors, *Environ. Health* (2012) 11. Suppl 1 S11.
2. Golka, K., Wiese, A., Assennato, G., Bolt, H. M., Occupational exposure and urological cancer, *World J. Urol.* (2004) 21 382-391.

PP125- Circular dichroism spectrophotometric determination of indium phthalocyanine induced destabilization of parallel and antiparallel G-quadruplex DNA structure

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G-quadruplex DNA is an important secondary structure of single-stranded DNA chain and formed by self-assembly of guanine-rich nucleic acid sequences. DNA can fold into G-quadruplex structures under conditions within the physiological range. G-quadruplexes have been reported to have biologically important regulatory roles such as mediation in gene expression, telomere maintenance, DNA replication, transcription, and translation (1, 2). From this perspective, G-quadruplex DNA is an important target for the development of chemo-therapeutic drug design due to increased and uncontrolled cell proliferation of cancer cells.

In the present study, destabilization effect of indium phthalocyanine (**InPc**) bearing (N-methyl-2-mercaptopyridine) groups at the peripheral positions of the phthalocyanine core to parallel and antiparallel G-quadruplex DNA structure was investigated. *cMYc* and *tel 21* oligomers were used as G-quadruplex forming sequences. The interaction mechanisms were elucidated by UV- visible, fluorescence and circular dichroism spectroscopic techniques. UV-visible spectroscopic data and thiazole orange based fluorescence displacement assays were used for detailed explanation of binding. The conformational deviation resulted from interaction with **InPc** was monitored by circular dichroism spectroscopic methods.

This work was supported by the Cumhuriyet University Scientific Research Foundation Council (CÜBAP), **Project Number: ECZ-043**

Keywords: Phthalocyanine, Circular dichroism, G quadruplex, cMYC, Tel 21

References:

1. João Lavrado, Stephan A. Ohnmacht, Isabel Correia, Clara Leitão, Sílvia Pisco, Mekala Gunaratnam, Rui Moreira, Stephen Neidle, Daniel J. V. A. dos Santos, Alexandra Paulo, Indolo [3, 2-c] quinoline g-quadruplex stabilizers: a structural analysis of binding to the human telomeric G-quadruplex. *ChemMedChem*, (2015) 10(5), 836-849.
2. Efkan Bağda, Esra Bağda, Ebru Yabaş, Circular dichroism spectroscopic investigation of double-decker phthalocyanine with G-Quadruplex as promising telomerase inhibitor, *J. Mol Struct.* (2017) 1127, 716-721.

PP126- The affinity determination of gallium phthalocyanine to double-stranded and G-quadruplex DNA

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The investigation of small molecules-G-quadruplex and double stranded DNA interactions is important research subject due to their potential biological and medical usages. Targeting G-quadruplex DNA structure with small molecules is a way of anti-cancer drug design. G-quadruplex DNA sequences can be found throughout the human genome. These sequences can also be found in different important parts of genome such as cancer-related regulatory regions (e.g. telomeres, oncogene promoters and RNA sequences involved in post-transcriptional regulation of cancer-associated key factors) (1)

In the present study, the interaction of water soluble quaternized gallium phthalocyanine (**GaPc**) bearing (N-methyl-2-mercaptopyridine) groups at the non-peripheral positions of the phthalocyanine core with different G-quadruplex DNA was investigated. The binding mechanism was clarified by UV-Vis., fluorescence and circular dichroism spectroscopy. The binding constants (K_b) were evaluated from data obtained by UV-Vis. spectrophotometry. The K_b constant values were found as $0.027 \mu\text{M}^{-1}$, $0.061 \mu\text{M}^{-1}$, $0.0936 \mu\text{M}^{-1}$ and $0.0124 \mu\text{M}^{-1}$ for *nucleolin*, *vegf*, *kras*, and ct-DNA, respectively. The highest K_b was found for the interaction of *kras* with **GaPc** and the lowest was found for ct-DNA. The fluorescence quenching mechanism was clarified by Stern-Volmer equation. The conformational deviations were monitored with circular dichroism spectroscopy.

Keywords: phthalocyanine, G quadruplex, nucleolin, vegf, kras, ct DNA

References:

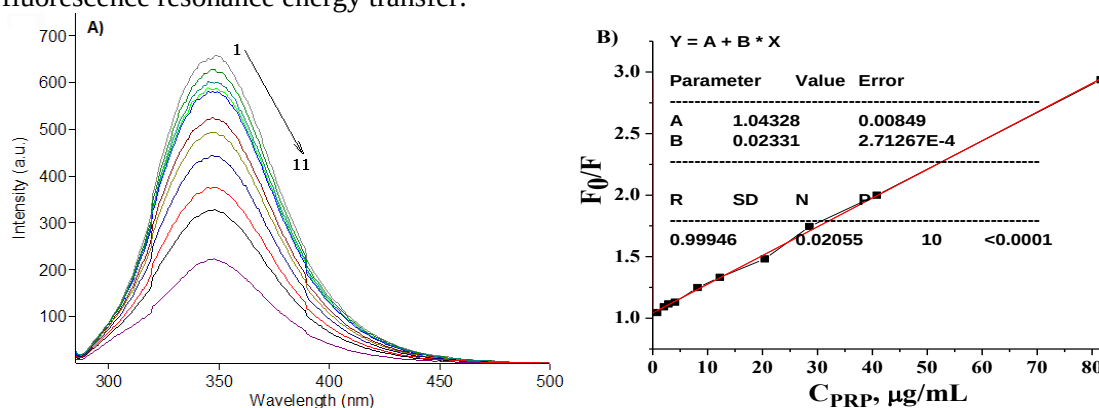
1. L. Sabater, M.L. Nicolau-Travers, A. De Rache, E. Prado, J. Dejeu, O. Bombarde, J. Lacroix, P. Calsou, E. Defrancq, J.L. Mergny, D. Gomez, G. Pratviel, The nickel(II) complex of guanidinium phenyl porphyrin, a specific G-quadruplex ligand, targets telomeres and leads to POT1 mislocalization in culture cells, J. Biol. Inorg. Chem. 20 (2015) 729–738.

PP127- Studies on the interaction HSA – propoxazepam and determination of its residues on equipment surfaces by fluorescence method
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Drug-protein binding has become an important research field in life sciences, chemistry and clinical medicine [1]. Under physiological conditions, *in vitro* interaction between the 7-bromo-3-propoxy-5-(2-chlorophenyl)-1,3-dihydrobenzo[e][1,4]diazepin-2-one (propoxazepam, PRP) and human serum albumin (HSA) was investigated at excitation wavelength 280 nm and at different temperatures (298K and 313K) by fluorescence emission spectroscopy. PRP showed a strong ability to quench the intrinsic fluorescence of HSA through a static quenching procedure (Figure (a)). The binding constant is estimated as $K_A = 1.58 \times 10^3 \text{ L} \cdot \text{mol}^{-1}$ at 298 K. The negative values of enthalpy change (ΔH°) and entropy change (ΔS°) can be attributed in part to van der Waals forces and in part to the formation of hydrogen bonds. A value of 1.32 nm for the average distance r between PRP (acceptor) and tryptophan residues of HSA (donor) was derived from the fluorescence resonance energy transfer.



Fluorescence spectra of HSA (A) in the presence of PRP and overlap (B) of the absorption spectrum of PRP (1) with the emission spectrum of HSA (2) ($C_{PRP}, \mu\text{g/mL}$), (1-11): 0, 0.8, 2, 2.8, 4, 8, 12, 20, 28, 40, 80)

Since, the pharmaceutical firms need standardized screens for protein binding in the first step of new drug design, this kind of study of interaction between HSA with PRP would be useful in pharmaceutical industry and clinical medicine.

Also in pharmaceutical industry, it is very important to remove drug residues from the equipment and areas used. This rapid, sensitive and specific luminescence method has been developed for quantitative determination of PRP (Figure (b)) in cleaning swab samples. The relative fluorescence intensity-concentration plot was linear over the range of 0.8 – 80.0 $\mu\text{g/mL}$. The proposed method was validated as per ICH guidelines. The following validation characteristics were addressed: specificity, accuracy, precision, limit of detection and quantification, linearity range and robustness. Cotton swabs, moisten with extraction solution (90% ethanol and 10% water), were used to remove residue of drug from stainless steel and glass surfaces, and give recoveries >90% at three concentration levels.

Keywords: benzodiazepin, human serum albumin, fluorescence quenching, FRET

References:

1. Gentili PL, Ortica F, Favaro G. Static and Dynamic Interaction of a Naturally Occurring Photochromic Molecule with Bovine Serum Albumin Studied by UV-Visible Absorption and Fluorescence Spectroscopy. *J. Phys. Chem. B* (2008); 112: 16793-16801.

PP128- Preparation of Au nanoparticles modified poly(cresol red) film glassy carbon electrode for differential pulse voltammetric analysis of As(III)

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Inorganic arsenic is one of the highly concerned poisons in food and environment. Its concentration in water sources has lately been increased due to increasing industrial activity related to production of pigments, insecticides, herbicides and some other related materials. The World Health Organization recommends that the As(III) level in drinking water should be below 10 µg L⁻¹. Hence, analysis of As, especially the selective and sensitive determination of higher toxicity of As(III) becomes very important for the safe application of food, drinking water and other beverages. Thus low-cost, convenient, sensitive and fast analysis for trace As(III) is an interesting and important topic. Although spectroscopic techniques have been employed for As determination, electroanalytical methods, offer advantages of ease and speed of analysis, cost effectiveness and high sensitivity [1-3].

We report here a simple and robust procedure to perform sensitive and selective differential pulse voltammetric determination of As(III) in 0.1 mol L⁻¹ HCl media on Au nanoparticles modified poly(cresol red) film glassy carbon electrode (Au/PCR/GCE). The Au/PCR/GCE /GC electrode surface was characterized using, scanning electron microscopy (SEM), X-Ray photoelectron spectroscopy (XPS) and electrochemical impedance spectroscopy (EIS). Under optimized conditions, the linear calibration graph is obtained ranged from 7.4 µg/L to 149 µg/L with a detection limit of 4.2 µg/L. The modified electrode was stored in the supporting electrolyte during the stability experiments. The current responses of electrode retained about 75 % its initial current after 7 days for 140.0 µg/L As(III). The proposed Au nanoparticles modified polymer film electrode was applied successfully for the analysis of As(III) in different spiked water samples which are mineral water, ground water and tap water.

Keywords: Arsenic(III), Au nanoparticles, poly(cresolred) film.

References:

1. L. Chao, X. Xiong, J. Liu, A. Xu, T. Huang, F. He, Q. Xie, Preparation of a porous Au electrode with a sacrificed Prussian blue analogue template for anodic stripping voltammetric analysis of trace arsenic(III), *Sens. Act. B* (2017) 253 603–611.
2. Xiao-Ping Lu, Xin-An Yang, Lin Liu, Hui-Hui Hu, Wang-Bing Zhang, Selective and sensitive determination of As(III) and tAs in Chinese herbal medicine samples using L-cysteine modified carbon paste electrode-based electrolytic hydride generation and AFS analysis, *Talanta* (2017) 165 258–266.
3. R.S. Salunke, C.K. Kasar, M.A. Bangar, P.G. Chavan, D.J. Shirale, Electrodeposition of gold nanoparticles decorated single polypyrrole nanowire for arsenic detection in potable water: a chemiresistive sensor device, *J Mater Sci: Mater Electron* (2017) 28 14672–14677.

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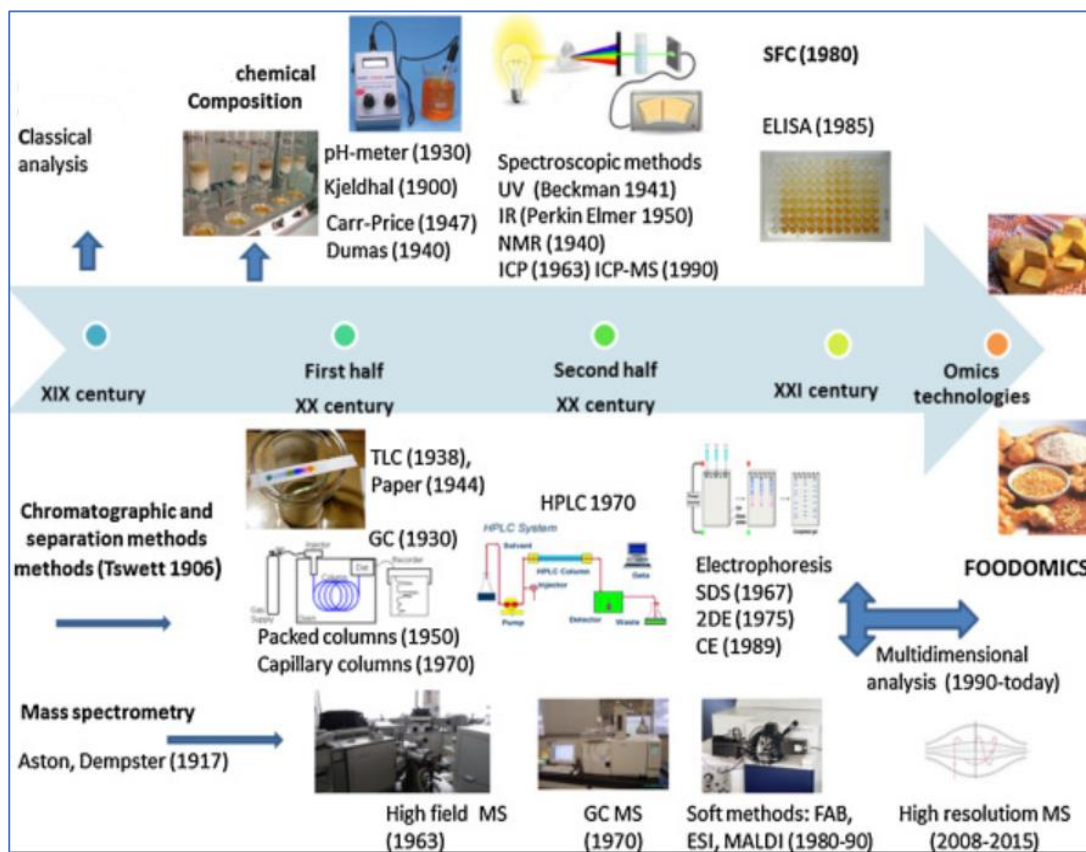
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