



**2nd International Congress on  
Analytical and Bioanalytical Chemistry  
(2nd ICABC 2020)**

**PROCEEDING BOOK**

**Editor: Mehmet Yaman**



**11-14 March 2020  
Antalya-Turkey**

**ICABC 2020**

## **SPONSORS**



### **Preface**

The organizing committee of the **2nd ICABC 2020** would like to welcome all participants to the "**2nd International Congress on Analytical and Bioanalytical Chemistry**", held in Antalya between 11-14 March 2020. The 2nd ICABC 2020 was started one year ago and covers all areas of Analytical and Bioanalytical Chemistry as well as applications of Chemical and Biochemical Analysis.

The scientific congress program consists of 18 sessions that include **26 invited and 52 oral** presentations as well as **62 posters** to be presented in the respective sessions. In addition, researchers of Academia (**56 universities from 14 countries**) and Research Institutes will present up-to-date developments on analytical and bioanalytical 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 4 days of the meeting will make **2nd ICABC** 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 and have a pleasant stay in Antalya, hoping to meet you again during the next **ICABCs**.

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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## ICABC 2020

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### **GENERAL INFORMATION**

#### **Introduction**

The 2nd International Congress on Analytical and Bioanalytical Chemistry will be held on 11-14 March 2020 in Antalya-Turkey is a four-days scientific meeting covering all areas of Analytical and Bioanalytical Chemistry and applications of Chemical and Biochemical Analysis. The international congresses have provided an excellent framework for the presentation of new concepts, instruments, methods, and applications in the area of modern chemical and biochemical 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 those areas. At the same time, it provides the grounds for the graduate and postgraduate students to present their projects, discuss scientific collaborations with other groups, as well as to explore employment opportunities. An exhibition of analytical and bioanalytical instruments and accessories will be also organized in the conference place whereas social events are planned to be included in the program of the. **2nd ICABC 2020.**

I strongly believe that young researchers will have chance to improve their knowledge in deep of the analytical and bioanalytical chemistry by coming together with experienced scientists including invited speakers and scientific committee members.

## ICABC 2020

### Topics

To promote collaboration among analytical and bioanalytical (including biochemists, food engineering, molecular biology and genetics and similars) scientists from different countries, “2nd ICABC 2020” will provide adequate opportunities.

The topics include all areas of analytical and bioanalytic chemistry in applications such as, but not limited to, environmental, biological and food matrices, environmental protection, biochemical studies, drug characterisation, method innovation and validation, instrumental development and applications, sensors and nanobiosensors, chromatography, spectrometry and electrochemistry.

The congress covers determination of inorganic and organic components in environmental, biological and food matrices as well as the following subjects: Food Safety: Omics analysis including GMO, all studies on interactions between metabolic disorders and foodstuffs.

The main aim and theme of the congress is to enlighten the innovations and current trends with analytical and bio analytical chemistry (including organic and food chemistry).

### Location of Conference

**2nd ICABC 2020** will be held in Kemer-Antalya in north-coast of the Mediteranean Sea. Kemer-Antalya, is around 40 km away from the Antalya airport. Every region in Antalya has a history of the ancient times and has been home to many important ancient settlements and cities. Antalya is also Turkey’s World famous tourism city as well as a holiday district.

Some Places to See and Sights in Kemer, Antalya, Turkey:

-**Phaselis** is the ancient Lycian city, archaeological site and museum located in Tekirova, about 12 km. from Kemer.

- **Olympos** is the ancient Lycian city, located nearby Cirali, about 35 km. from Kemer. With its pine forests and Beydaglari mountains at the background, wonderful nature, unique beach and sea, the historical site and ruins, Olympos is one of the most popular sights in Kemer.

- **Yanartas (Chimaera)**, known as the place of flames, is a fascinating natural rocky formation located along Cirali village, on Mount Chimaera slopes, close to Olympos Valley, about 35 km. from Kemer.

**Goynuk Canyon** in Kemer is one of most popular sights in Kemer.

**Central Kemer Moonlight Park** is a daily entertainment and recreational area housing a blue-flagged Moonlight beach, a Dolphinarium, shopping center, mini zoo, tennis and basketball courts, recreation and green areas.

### Papers presentation

Scientific program will include Invited Speakers, which will provide an up-to-date presentation of modern trends of Analytical and Bioanalytical Chemistry as well as of related subjects of chemical and biochemical analysis-interest. Oral Presentations will be presented in two halls. 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 poster time, and remove them in the evening of the corresponding day. 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 was also taken place. These certificates were given to recognize excellence in research and presentation. The winners are given below.

### Discount in next congress in total

The -	<b>1st award: Buse Tugba ZAMAN</b>	-Yildiz Tech. U	<b>%40</b>
First	<b>2nd award: M. K. SEZGINTURK</b>	-Canakkale 18 Mart U	<b>%30</b>
Day	<b>3rd award: Gülay Bayramoğlu</b>	- Gazi U	<b>%20</b>

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The -	<b>1st award: Yalım Tosçalı</b>	- Ege U	<b>%40</b>
Second	<b>2nd award: Ömer IŞILDAK</b>	- Gaziosmanpaşa U	<b>%30</b>
day	<b>3rd award: Merve Çınar</b>	- Ege U	<b>%20</b>

## Social events

Welcome reception-11 March, 2020: The Welcome Reception will be held on March 11, 2020. at the Akka Antedon hotel adjacent-sea. Event will close with local traditional dances and a folklore party.



Conference Gala dinner- 12 March, 2020: The Conference Gala Dinner will be held on March 12 at 20:00 in a restaurant of Akka-Antedon Hotel. The menu will include a wide variety of traditional food, salads and drinks. Event will close with local traditional dances and a folklore party.

After welcome cocktail and Gala Dinner on nights of 11 and 12 March 2020, respectively, live modern and traditional music presentation will be done.

On 13 March 2020, an excursion to Kemer Ancient city is planned in near the congress hotel by bus.

### **OPENING SPEECH**

**Dear Honorable Professors, Colleagues and Participants,**

I am very happy to welcome all the participants coming to "2nd International Congress on Analytical and Bioanalytical Chemistry "

**Respectable academics and friends;**

Nowadays, role of analytical and bioanalytical chemistry is better understood and increasing day by day. This is valid across a broad spectrum, from global nutrition to health, from advanced technological research to detection of environmental pollution. So, cooperation among analysts to more and the update in analysis methods is gaining importance.

In particular, in the last years, the results of the researchers at different disciplines receiving the service from the institutions serving under the name of "central laboratory" which we have longed for years, has put at risk, because of the lack of analytical experience of the users and inability to validate results.

From this point of view, the first objective of this congress is to provide the opportunity for researchers interested in different disciplines to come together and to exchange ideas in analytical-perspective meetings.

On the other hand, the purpose of congresses and similar scientific meetings is to make presentations of young researchers, to listen to presentations and to update themselves in an environment of well-known scientists.

However, in recent times, there has been a worldwide increase in misguided conferences in the name of international congresses that the same meetings are held several times a year, even in different countries every week, the audience listening to the presenter is small enough to be counted with fingers, in summary, presentations have not even the opportunity to evaluate by expert scientists, even the video is sent to the meeting without the participant's. All these situations increase the need for a purposeful congress.

This congress was launched for such purposes.

Hereby, I would like to thank the invited speakers, the members of Science Committee and especially for your participation, and I would like to express our honor to host the congress.

**Statistical Information about the Congress**

The scientific conference program consists of 18 sessions that include 25 invited and 53 oral presentations as well as 61 posters. The participants are of 57 universities from 14 countries. I believe that the discussions and the exchange of ideas among the participants during the 4 days will make this conference a brilliant platform to initiate new research collaborations.

I wish you all to enjoy this conference and have a pleasant stay in Kemer/Antalya.

**Sponsors:**

The contributions of sponsors are important and I would like to thanks to

- ☐ SEM Lab.
- ☐ TERRA
- ☐ Referans Kimya

I wish you all to enjoy this conference and have a pleasant stay in Kemer/Antalya.

- I wish the conference will be useful.
- My best regards.

**Prof. Dr. Mehmet Yaman-  
Chair**

**CONGRESS PROGRAM****2nd International Congress on Analytical and Bioanalytical Chemistry  
(2nd ICABC 2020)****11-12 March, 2020, Antalya-Akka Antedon Hotel/Turkey**

	<b>11 March, 2020</b>
13.30 – 15.30	<b>Registration- Hall Sultan2 (Hall A)-Akka Antedon Hotel, Antalya-Turkey</b> The registration desk will be open everyday during conference hours
15.30 – 16.30	<p>➤ Welcome Ceremony</p> <p>➤ Respect-Silence of Independence and Opening Speeches: Prof. Dr. <b>Mehmet Yaman</b> (Chair) Prof. Dr. <b>Seref Gucer</b> (on behalf of continuation committee)</p> <p>➤ Honorable</p> <p><b>Inv. 1: Prof. Dr. Ulla Wollenberger-Potsdam U./DE-Bioelectrocatalysis with Enzyme Modified Electrodes and Potentials for Bioanalysis-IS6</b></p>
16.30-16.50	Tea/Coffee break
	<b>Hall A</b>
	<b>Session 1-Chairs: Slavomira Skrzypek –Sezgin Bakirdere</b>
16.50-17.20	<b>Inv. 2: Prof. Dr. Nosal-Wiercińska Agnieszka Maria Curie-Sklod. U/PL-The role of the active complexes of selected anti-cancer drugs with Bi(III) in the aspect of kinetics and electrode mechanism changes in the presence of non-ionic surfactants- IS25</b>
17.20-17.40	<b>Aysu Yarman- Potsdam U/DE - MIPs for Strep-Tagged Hydrogenase and Human Sulfite Oxidase OP14</b>
17.40-17.50	<b>V. Hazal OZYURT-Near East U/TRNC- Development of Protein Extraction Procedure For Proteomic Analysis of Tomato Seed-OP5</b>
17.50-18:00	Tea/Coffee break
	<b>Session 2- Chairs: Almira Ramanaviciene –Yusuf Dilgin</b>
18.00-18.30	<b>Inv. 3: Prof. Dr. Antony Calokerinos-Athens U/GR Chemiluminescence: A Sensitive Analytical Technique-IS3</b>
18.30-19.00	<b>Inv 4:Prof. Dr. Sezgin BAKIRDERE YTU/TR-Detection Power Improvement Strategies in Flame Atomic Absorption Spectrophotometry- IS12</b>
19.30-22.00	<b>Welcome Coctail-Dinner-Music</b>
	<b>12 March, 2020</b>
	<b>Session 3- Chairs: Durisehvar Unal -Ersin Kılıç</b>
08.45-09.15	<b>Inv 5: Prof. Dr. O. Yavuz Ataman METU/TR-Chat in an Instrumental Analysis lecture-IS9</b>
09.15-09.45	<p><b>Belgin İZGİ-Uludag U/TR-Metal Levels Monitoring in Coastal Region of Bursa-OP31</b></p> <p><b>Samuel Bunani Burundi U/-Utilization of Bipolar Membrane Electrodialysis (BMED) Systems for Selective Separation and Recovery of Valuable Elements from Water-OP46</b></p> <p><b>Dilsat Ozkan Ariksoysal- Ege U/TR-Electrochemical biosensor-based diagnostic kit designs and their applications in DNA analyses-OP13</b></p>
09.45-10.15	Tea/Coffee break
	<b>Session 4: Chairs: Antony Calokerinos - Levent Pelit</b>
10.15-10.45	<b>Inv. 6: Prof. Dr Bogusław Buszewski N. Copernicus U/PL-Searching Of VOCs As A Potential Cancer Biomarkers By Hyphenated Separation Techniques-IS1</b>
10.45-11.15	<b>Inv. 7: Prof. Dr. Gülay Bayramoğlu-Gazi U/TR-Design of QCM aptasensors for detection of pathogens using differently prepared magnetic separation systems- IS23</b>
11.15-12.00	<b>Neşe Ular-SEM LAB, An important role of the mass spectrometry on clinical diagnostics: Simultaneous Quantification of 16 Clinically Relevant Steroid Hormones Using LC-MS/MS</b>
12.00-13.30	Lunch

<b>Session 5:</b> Chairs: <b>Özlem Söğüt -Belgin İzgi</b> <b>Hall A</b>		<b>Session 6:</b> Chairs: <b>Burhan Ateş- Yusuf Dilgin</b> <b>Hall B</b>	
13.30-14.00	<b>Inv. 8: Prof. Dr. Mustafa Soylak</b> -Erciyes U, TR-Green sample preparation strategies for organic/inorganic trace species- <b>IS8</b>		<b>Inv 9: Prof. Dr. Arzum Erdem Gürsan</b> -Ege U/TR- Electrochemical Nucleic Acid Biosensors Developed by Advanced Nanomaterials- <b>IS20</b>
	<b>Biljana Balabanova</b> - Goe Delčev/MC Tracking silent geochemical markers for polymetallic pollution in area with Cu-Pb-Zn minerals exploitation- <b>OP23</b>		<b>İnci Uludağ</b> -Canakkale 18 Mart U/TR- A new immobilization route for immunosensors: CNBr activation for covalent attachment of anti-Leptin onto the electrode surfaces- <b>OP3</b>
	<b>Usama Alshana</b> TRNC-Dispersive Liquid-Liquid Microextraction Followed by Smartphone Digital Image Colorimetry for the Determination of Iodate in Table Salt- <b>OP35</b>		<b>Nur Tarimeri</b> -Canakkale 18 Mart U/TR- Optimization studies of a disposable immunosensor for sensitive detection of creatine kinase- <b>OP4</b>
14.00-14.40	<b>Betül Arı-TÜBİTAK/TR</b> - Measurements of Iron in a Candidate Seawater CRM by Triple Quadrupole ICP-MS/MS with Isotope Dilution Calibration Strategies- <b>OP24</b>		<b>Burcu Kartal</b> - Canakkale 18 Mart U/TR- Sensitive Differential Pulse Voltammetric Determination of an Antimarial Drug: Amodiaquine at a Disposable Poly-Calcein Modified Pencil Graphite Electrode- <b>OP20</b>
	<b>Buse Tugba Zaman</b> -YTU/TR- Trace Cobalt Determination in Chamomile Tea Samples by an Effective and Novel Analytical Method by Using Simultaneous Dispersive Liquid Phase Microextraction-Slotted Quartz Tube –FAAS- <b>OP25</b>		<b>Ahmet Ulu</b> - Inonu U/TR- Preparation and Optimization of Alginate Microbeads Loaded with Vincristine Sulfate and Its In vitro Release- <b>OP17</b>
			<b>Fırat Baran</b> - Artuklu U/TR-Synthesis and Enhancement of Composite for Removal of Dye: Effect of contact time and Initial Dye Concentration- <b>OP42</b>
14.40-15.00	<b>Latif GÖKSU, TERRA A.Ş.</b> -New Pressurized Liquid Extraction Method for the Extraction of multiple organic contaminants from various matrices		<b>Ayşe Şap</b> -Cumhuriyet U/TR- Determination of Curcumin in medicinal plants and its Isolation in Turmeric (Curcuma longa L)- <b>OP37</b>
15.00-15.15	Tea/Coffee break		
15.15-16.05	<b>Session 7: Poster Session 1(1-30)</b> Chairs: <b>Emine Akyüz Turumtay- Hanine Hafida- Fatma Bendimerad-Sadin Özdemir</b>		
<b>Session 8:</b> Chairs: - <b>Ulla Wollenberger Aysu Yarman</b> <b>Hall A</b>		<b>Session 9:</b> Chairs: <b>Mustafa Soylak-Perihan Ünak</b> <b>Hall B</b>	
16.05-16.35	<b>Inv. 10: Prof. Dr. Sibel Özkan</b> Ankara U/TR-Carbon Based Nanomaterials in Electroanalytical Sensors-What they are;What they do- <b>IS10</b>		<b>Elif Yazıcı</b> -YTU/TR- Trace cobalt determination in lake water samples by ultraviolet based photochemical vapor generation-T-shaped slotted quartz tube atom trap-FAAS- <b>OP27</b>
	<b>Mehmet Gumustas</b> - Ankara U/TR- Separation of the Enantiomers of Chiral Sulfoxide Derivatives by Supercritical Fluid Chromatography- <b>OP49</b>		<b>Zeynep Tekin</b> -YTU/TR- Zirconium Nanoparticles based Solid Phase Extraction Prior to Slotted Quartz Tube-FAAS for the Determination of Cadmium in Wastewater Samples and Evaluation of Green Profile- <b>OP29</b> –
16.35-17.05	<b>Göksu Özçelikay</b> -Ankara U/TR- A Novel Electrochemical Nanosensor Based on COOH-Functionalized Multi Walled Carbon Nanotubes Decorated with Quantum Dots for The Determination of Clopidogrel- <b>OP21</b>		<b>Didem Giray Dilgin, Serkan Karakaya</b> - Canakkale 18 Mart U/TR- Flow Injection Amperometric Detection of Formaldehyde at Bimetal Nanoparticles Modified Pencil Graphite Electrodes- <b>OP19</b>
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## INVITED SPEAKERS (IS)

### IS1- SEARCHING OF VOCs AS A POTENTIAL CANCER BIOMARKERS BY HYPHENATED SEPARATION TECHNIQUES

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Exhaled air gives unique possibility of diseases investigation and may offer evaluation of cancer and irritable bowel syndrome. Exhaled breath contains large number of VOC but most of compounds are at the level of parts per million by volume - parts per trillion by volume (ppmv-pptv). The most important preconcentration methods utilizes thermal desorption (TD).

Volatile organic compounds (VOCs) are generated by the human body as products of metabolic process. However, biochemical methods for creation of the most of compounds, which were detected in breath, have not been precisely explain from the scientific point of view. Chemical analysis of breath composition may help to identify potential markers. The most interesting technique seems to be GC-MS and/or GCxGC TOF-MS. An unconventional method applied was the dog smell to detect changes in carcinoma. Dogs after suitable training were able to indicated with high sensitivity and specificity samples of exhaled air.

In the current work, the SPME/TD-GC/MS and SPME/TD-GCxGC TOF-MS methods for determination of volatile organic compounds in breath and feaces of colon cancer patients and healthy persons has been applied. The analyses were carried out on breath samples and ambient air. Automatic identification of compounds was used for data processing. Colon cancer patients were detected. Our data were compared from the references. We identified more than 50 VOCs in breath and feaces of patients and healthy persons.

#### **Acknowledgments**

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**IS2- ASSESSMENT OF ENVIRONMENTAL IMPACT OF METAL PRODUCTION PROCESSES. CASE STUDY: THE REPUBLIC OF NORTH 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, potentially toxic elements (arsenic, cadmium, lead, mercury, etc.) are of a special ecological, biological and health significance. Therefore, detailed surveys of pollution with heavy metals of soil, waters, sediments, air and food on the entire territory of North Macedonia have been carried out. For that purposes various instrumental techniques such as atomic absorption spectrometry (AAS), inductively coupled plasma - atomic emission spectrometry (ICP-AES), inductively coupled plasma - mass spectrometry (ICP-MS) and neutron activation analysis (NAA) were applied. Air pollution was investigated by the application of moss biomonitoring and dust samples. 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. Special attention was paid to the pollution with heavy metals in the regions with mining and metallurgical activities. It was found that the highest pollution is present in the areas with abounded or active mines, metallurgical plants or thermoelectric power plants. High contents 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.

Keywords: Environmental pollution, heavy metals, air, water, soil, food, North Macedonia

### **IS3- Chemiluminescence: A Sensitive Analytical Technique**

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Chemiluminescence is the analytical technique which monitors the emission of light during a chemical reaction. The chemical reaction is exothermic and the energy is absorbed by an intermediate or a final product which then is de-excited by emission of light. If the reaction involves enzymes, then Bioluminescence occurs. The technique is characterized by simplicity, sensitivity, low limits of detection and relatively low cost of instrumentation. Since the emission intensity must be monitored during the reaction, applications of chemiluminescence were boosted after the introduction of flow-injection analysis and sequential flow analysis.

The mechanisms of chemiluminogenic reactions and typical instrumentation will be presented and discussed during the presentation. Emphasis will be given to applications in drug analysis, environmental analysis, clinical analysis, food and beverage analysis.

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## IS4- Formation of Conducting Polymer based Structures Suitable for Sensor Design

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In this presentation methods, which authors have used and/or developed for the formation of conducting polymer based structures suitable for sensor design will be overviewed. Significant attention will be focused on chemical [1] and electrochemical [2-4] formation methods suitable for the deposition of conducting polymer based layers on the surfaces of electrodes [2-4]. Biocompatibility aspects of some  $\pi$ - $\pi$  conjugated polymers [5] will be discussed and synthesis of  $\pi$ - $\pi$  conjugated polymers by 'bio-based' methods, which aims the application of enzymes and/or living microorganisms, will be discussed [6]. Methods, which are suitable for the modification of conducting polymer based layers with inorganic compounds and biomaterials, will be discussed. The application of conducting polymer based structures for various sensors and biosensors will be overviewed. Charge transfer mechanisms between some redox-able biomolecules and some  $\pi$ - $\pi$  conjugated polymers will be discussed [3]. Formation and analytical application of molecularly imprinted conducting polymers will be addressed [4].

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**Keywords:** conducting polymers, electrochemical sensors, glucose biosensor, molecularly imprinted polymers.

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## **IS5- Recent developments in enantioselective analysis of chiral drugs by capillary electrophoresis**

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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-selectand 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 separation of chiral drug enantiomers and on understanding of chiral recognition mechanisms of cyclodextrins by combined use of CE, nuclear magnetic resonance spectroscopy and molecular modeling calculations [1, 2].

### **Acknowledgment:**

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## IS6- Bioelectrocatalysis with enzyme modified electrodes and potentials for bioanalysis

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Bioelectrocatalysis involves the enhancement of an electrode reaction by a (bio)catalytic process and allows therefore to gain important kinetic information about the catalyst. The presence of the catalyst enables redox reactions of compounds at electrode to occur at low overpotentials. This phenomenon has already been described more than 30 years ago<sup>1</sup>. Together with the substrate selectivity of enzymes bioelectrocatalysis provides the basis for sensitive and selective electroanalytical devices. The signal transduction between the catalyst protein and redox electrodes proceeds by direct electronic communication and by mobile or polymer bound redox mediators. Recent progress in the field is due to advances in enzyme engineering, surface chemistry and nanotechnology.

In this talk, examples of electrochemical studies on molybdenum containing enzymes and biosensors based on them will be discussed. Molybdenum containing enzymes catalyze important metabolic conversions such as oxidation of aldehydes, xanthine, and sulfite, or the reduction of DMSO, CO<sub>2</sub> and nitrate<sup>2</sup>. Strategies to immobilize protein and enzymes based on self assembled monolayers, detergents and three dimensional sensor structures were successfully developed in order to achieve an efficient electronic communication between enzyme and electrode and a high catalyst loading particular for direct electron transfer and mediatorless bioelectrocatalysis. On the other hand immobilization is achieved in hydrogels, redox doped and conductive polymers for mediated electron transfer. Additional protective layers enable improved sensor performance. The lecture will discuss examples of such electrochemical biosensors, i.e. electrodes for aromatic aldehydes with an aldehyde oxidoreductase<sup>3-4</sup>, for sulfite with sulfite oxidase on nanoparticle modified electrodes<sup>5-7</sup>, and for N-methylamine oxides with an enzyme of the class of DMSO reductases<sup>8</sup>.

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## **IS7- Raman and SERS-Based Detection of Gene and Microorganisms**

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Detection of microorganisms and genetically modified organisms (GMO) has an increasing impact in terms of food safety and human health. Over the years, different approaches have been used to determine the major pathogenic bacteria, which can cause severe illness and possible death. Therefore, studies on developing reliable, sensitive and rapid methods for detection of pathogenic bacteria are on the increase. Several techniques have been developed for specific detection of foodborne pathogens such as automated or modified conventional assays, biosensors, immunological methods or nucleic acid based assays. On the other hand, GMO detection are gaining worldwide attention, especially in molecular biology and clinical diagnostics. Detection and identification of GMO have been achieved by using DNA and protein-based methods. In both cases, conventional methods have certain disadvantages in terms of analysis, sample preparations, selectivity, quantitative results, etc. At this point, Raman and SERS can an attractive alternative to determine pathogenic bacteria and GMO. Here, different applications of Raman and SERS will be presented to evaluate its potential to be used as rapid and sensitive method for detection of pathogenic bacteria and GMO. At first, the performance of Raman spectroscopy to identify *Pseudomonas aeruginosa* and *Pseudomonas fluorescens* was evaluated. Later on, the potential of the SERS method to detect *Escherichia coli*, 35S promoter gene and GMO was evaluated. As a result, Raman and SERS based methods showed they can be used as rapid, selective and sensitive method for detection of pathogenic microorganisms and GMO.

## **IS8- Green sample preparation strategies for organic/inorganic trace species**

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Nowadays green sample preparation techniques including separation and preconcentration is an important place in the analytical chemistry studies prior to accurate and precise determination of trace organic and inorganic species by modern instrumental techniques. Microextraction procedures including solid phase and solvent microextraction are very popular applications at this area.

Two important strategies at this area for trace species are:

1. The fabrication and characterization of new nanomaterials for solid phase microextraction studies
2. The usage of new generation green solvents for solvent microextraction studies

In this presentation some novel and green applications of solid phase and solvent microextraction will be discussed.

## **IS9- Chat in an Instrumental Analysis lecture**

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In any science lecture, sometimes you see that the attention and concentration of students become volatile. Then, the instructor feels that it is a time to insert an anecdote or an interesting event from history of science. This talk aims to transfer some of such short speech to be used. They are also related to the history and/or present status of instrumental analysis. These are summarized as follows:

- A problem in a case may be a solution for another case. Ion selective glass electrodes, composition, interference and analyte.
- Who invented the chopper? Was it first a tool for AAS or IR?
- Which unit should be used? M or mg/L? Everlasting discussion with Prof. Dr. Güler Somer, an electrochemist.
- Invention of IR region and a perfect example for an analytical measurement.

Keywords: Glass electrodes, chopper, units in trace analysis, parallel determinations and blank measurement.

*Acknowledgment: I would like to thank all my students and colleagues, since 1975 until today.*

## IS10-Carbon Based Nanomaterials in Electroanalytical Sensors-What they are;What they do

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**Abstract:** Nanoscience or nanotechnology are simply science and engineering carried out on the nanometer scale, that is,  $10^{-9}$  meters. There exist various materials of different types for fabricating nanosensors. Especially, carbon-based nanomaterials have become very important due to their unique combinations of chemical and physical properties, extensive research efforts are being made to utilize these materials for various industrial applications, such as high-strength materials and electronics. These advantageous properties of carbon-based nanomaterials are also actively investigated in several areas of biomedical and drug assay. Nowadays, a lot of different analytical methods are used in environmental, pharmaceutical, or clinical laboratories and also a number of the commercial point-of-care devices work using nanosensors. In this study, some important and recent developments brought about by the application of carbon based nanostructures to nanotechnology for both chemical and biological sensor development and their application are described in pharmaceutical and biomedical area.

**Key words:** Biomarkers; Electroanalytical chemistry; Determination; Nanosensors; Drugs.

**Introduction:** Carbon is called as a unique element, due to its magnificent applications in many areas. Today, application of different nanoparticles in construction of sensors as a modifier is quite frequent. The nanoparticles have different effects on response of the nanosensor besides improving their thermal, electrical, and mechanical properties.

In recent years, nanosensor technology has experienced a rapid development because of the extensive scientific efforts in understanding of nanoscale phenomena. Carbon-based nanomaterials, such as fullerenes, graphene, nanodiamonds, carbon nanotubes, nanorods, nanowires, nanodots, etc. have recently gained considerable attention among scientific communities due to their unique chemical and physical properties. These nanomaterials have found their place in a wide range of applications. They can easily use for the creation of the novel nanosensors for detecting heavy metal ions, gas molecules, food additives, antibodies, pharmaceuticals, environmentally important compounds, as well as reporters for bioimaging.

There exist various materials of different types for fabricating nanosensors. Especially, carbon-based nanomaterials have become very important due to their unique combinations of chemical and physical properties, extensive research efforts are being made to utilize these materials for various industrial applications, such as high-strength materials and electronics. These advantageous properties of carbon-based nanomaterials are also actively investigated in several areas of biomedical and drug assay.

Electrochemical nanosensors have recently found extensive applications in pharmaceutical and biomedical industries with some advantages such as lower detection limits, wider linear response range, sensitivity, good stability and reproducibility when compared with other sensors and techniques. As the demand for smaller, faster, cheaper, and ultrasensitive qualification and quantification of samples rapidly increases, these methods provide a viable path toward the next generation of electrochemical sensors<sup>1-5</sup>.

**Materials and Methods:** Electrochemical devices have recently received considerable attention in the development of nanosensors and nanobiosensors. They are devices that intimately couple a pharmaceutical and biological recognition element to an electrode transducer that relies on the conversion of the nanomaterial-drug or antibody-antigen or Watson-Crick base-pair recognition event into a useful electrical signal. Electrochemical devices offer elegant routes for interfacing – at the molecular level–the target recognition and signal transduction elements and are uniquely qualified for meeting the size, cost, low volume, and power requirements of decentralized working diagnostics<sup>1-7</sup>. In pharmaceutical and biomedical industries, electrochemical nanosensors have recently found extensive applications with some advantages such as lower detection limits, wider linear response range, sensitivity, good stability and reproducibility when compared with other sensors and techniques. As the demand for smaller, faster, cheaper, and ultrasensitive qualification and quantification of samples

rapidly increases, these methods provide a viable path toward the next generation of electrochemical sensors<sup>6,7</sup>.

**Results and Discussion:** Among all nanomaterials, carbon is the unique element for its endless advantages in sensor design. Carbon is unique in nature. Besides its capability to form complex networks that are fundamental to organic chemistry, elemental carbon shows unrivalled complexity among the elements, forming many allotropes, from diamond and graphite to fullerenes, through nanotubes and the most recently obtained graphene<sup>1-4</sup>.

Over the past two decades, we have contributed to the subsequent rise of the three different low-dimensional carbon allotropes. Carbon nanotubes are perhaps the most notable representatives of the present nanoworld. However, they are very likely bound to soon share the stage with graphene, the “rapidly rising star on the horizon of materials science and condensed matter physics”. Graphene may have a number of very peculiar electronic properties – from an anomalous quantum Hall effect to the absence of localization. Fullerene is the arrangement of 60 carbon atoms like a soccer ball structure. Moreover, carbon nanowires also suggested for the surface design in nanosensors as they can even act as an electrode independently. They have very small diameter even 1 nm. Another type of carbon derivative nanomaterial is carbon nanofiber. Carbon nanofibers can be microelectrodes due to their smaller surface area. There can be carbon nanofibers in the form of cylindrical or disk depending on the usage.

They have all high conductivity and large surface area properties that is very important in nanosensor design<sup>3-6</sup>.

**Conclusions:** In recent years, nanotechnology with its wide applications has become very popular in the biomedical and pharmaceutical area. Electrochemical devices have recently received considerable attention in the development of nanosensors and nanobiosensors. They are devices that intimately couple a pharmaceutical and biological recognition element to an electrode transducer that relies on the conversion of the nanomaterial-drug, nanomaterial-biological compound, antibody–antigen or Watson–Crick base-pair recognition event into a useful electrical signal. Electrochemical devices offer elegant routes for interfacing – at the molecular level–the target recognition and signal transduction elements and are uniquely qualified for meeting the size, cost, low volume, and power requirements of decentralized working diagnostics<sup>1-3</sup>.

The high sensitivity of electrochemical nanosensors or nanobiosensors, coupled with their inherent miniaturization, compatibility with modern microfabrication technologies, low-cost and power requirements, and independence of sample turbidity make such devices excellent candidates for centralized and decentralized the related testing.

As new procedures for the large-scale production of graphene are expected to be developed in the near future, most of such properties – including the electrochemical ones – will be soon experimentally demonstrated, thus permitting the development of the many important technological applications foreseen for this material.

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## IS11- Indium tin oxide based materials as disposable working electrodes for electrochemical biosensing applications

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**Abstract**-Electrochemical biosensing systems are a subclass of the biosensors, combine of an electrochemical transducer system and a biological recognition element with a high specificity. The biological recognition element is also called as “bioreceptor” that selectively interacts with its related target. Enzymes, antibodies, biological membrane receptors, nucleic acids, aptamers, or biological tissues are the most frequently used bioreceptors in electrochemical biosensing systems. In electrochemical biosensors, voltammetry, potentiometry, amperometry, and impedimetry are major methods, which used as electrochemical tools. There are many different type of working electrodes in electrochemical biosensors such as traditional solid electrodes (like glassy carbon, gold, or platinum). All of these are used many times by a cleaning procedure after measurement. However cleaning procedure usually destroys the surface of the electrodes and reduces the possibility of multiple uses in especially biosensing applications. In this regard disposable electrode materials are becoming very important for the biosensor fabrications. In this study firstly the commonly used disposable electrode materials are discussed and then special emphasis will be placed on indium-tin oxide based electrode materials used in biosensors.

**Keywords:** *Electrochemical biosensors; Disposable electrodes; Single use electrodes; Indium-tin oxide; ITO.*

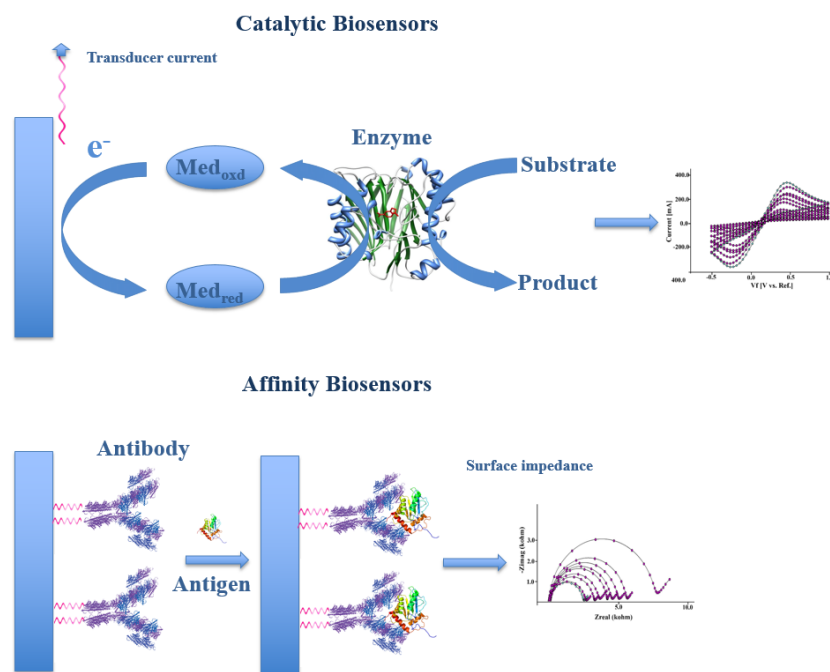
### Introduction

Electrochemical biosensors are generally divided into two categories i.e. biocatalytic and affinity based systems. In biocatalytic biosensors, a catalytic conversion takes place by the interaction occurred between the bioreceptor and the target. Consequently biocatalytic biosensors utilize bioreceptors that can produce electroactive species such as enzymes, whole cells, or tissues. In other respects affinity biosensors are based on the selective and specific binding between the bioreceptor and target. In this case there is no need for an electroactive species. Immunosensors based antibodies and DNA biosensors are the most frequent affinity biosensors. Figure 1 shows a schematic representation of this biosensor classification. In both two manners of the biosensing strategy, one of the most important components which can affect the performance of the biosensor is working electrode. For the fabrication of electrochemical biosensors a lot of different kinds of materials have been reported as a working electrode. While some of them are multiple use such as glassy carbon, gold, or platinum and the other important part of them is single use such as pencil graphite (PGE), screen printed (SPE), or carbon paste electrodes (CPE). The most three important drawbacks of traditional solid electrodes are high cost, memory effects, and requirement of tedious cleaning process. Nevertheless disposable working electrodes can successfully avoid these common problems of classical solid electrodes. For example PGEs could be preferred because of their extremely low cost and easy commercially available. CPEs are other alternative disposable electrode material which should be easily constructed by mixing graphite powder and a pasting organic liquid. Similarly the low cost of CPE is the most important reason for preference in electrochemical approaches. However, it must be kept in mind that, the success of this kind of electrode strictly depends on the practice of the researcher. In this regards, the repeatability and reproducibility of these electrodes are the most important drawback which limits their using in biosensing applications as a disposable working electrodes.

SPEs are the most frequently used electrodes as disposable working electrodes that could be commercially available. Because of the high demand, the companies introduces into the market SPEs with the different modified surfaces. Their miniaturized shape and possibility of integration with a portable electrochemical device make SPEs important candidate for applications of on-site determinations.

Besides these disposable electrodes indium tin oxide (ITO) based materials are promisingly used in biosensing applications such as in food, medical diagnosis, and environmental analysis<sup>1</sup>. Actually ITO is widely utilized in flat-panels because its good electrical conductivity and optical transparency. Beside when it comes to the question why ITO coverage should be used in biosensing applications, there is a few important answers; wide electrochemically working range, high and controllable electrical conductivity, good substrate adhesion, low capacitive current, and the nature of electrochemically and physically stability. In addition to this the possibility of lots of chemical modification on ITO surface

by varying chemical agents make ITO the most important candidate for the fabrication of biosensors with high performance.



**Figure 1.** Schematic representation of catalytic and affinity biosensors

## Materials and Methods

ITO based working electrode materials are discussed in terms of their modification procedures, analytical performances, repeatability and reproducibilities, other important aspects. First a brief data on the immobilization procedures for all biosensors are given. Chemical and physical procedures which have been carried out for the bioreceptor immobilization on the ITO surface were also evaluated. Following that the analytical performances are discussed and compared to other similar approaches based on the traditional solid electrodes and other single use electrodes. The performance of ITO based electrodes on the real sample analysis was examined. Finally the pros and cons of disposable ITO based electrode materials used in biosensing applications are concluded.

## Results and Discussion

ITO is composed of indium, tin and oxygen as a ternary composition in variable ratios. The most commonly encountered proportion is a formulation of 74 % In, 18 % O<sub>2</sub>, and 8% Sn by weight<sup>2</sup>. The molecular structure of ITO is shown in figure 2.

The most commonly used surfaces for the deposition of indium tin oxide are glass and polyethylene terephthalate (PET) sheets. And the techniques used for the deposition are commonly electron beam evaporation or a range of sputter deposition methods. ITO covered materials have been widely used in sensors and biosensors applications in recent years. Table 1 summarizes the bio(sensors) based on ITO covered materials. When these biosensing systems are evaluated it can be concluded that the most used transducer types in these systems are electrochemical and optical devices. Moreover it was also evaluated that for the surface modification silanization, electrochemical deposition, polymerization, and physical adsorption were the most preferred modification methods. For example; recently an ITO covered PET sheets were used for the development of an ultra high sensitive biosensor for the determination of Receptor for activated C kinase 1 (RACK1) which is also known as guanine nucleotide-binding protein<sup>3</sup>. RACK1 is metabolically so important that it is involved in a wide variety of signalling pathways. Besides it interacts with the different proteins and plays an important role in many cellular processes. In this study, 11-cyanoundecyltrimethoxysilane (CUTMS) was used as a silanization agent for the covalent immobilization of anti-RACK1 onto hydroxylated ITO surface. Chemically active aldehyde ends of CUTMS provided an effective tool for the practical immobilization of antibody.



**Figure 2.** Schematic representation of an ITO surface model (Reprinted from Ref.2)

As reported the biosensor provided a considerable wide calibration range for RACK1 between 0.036 fg/mL and 2.278 fg/mL with a LOD of 10.8 fg/mL. In some circumstances ITO surface was covered by different functional polymers for antibody immobilization. One of this type of surface modification was reported by our research group<sup>4</sup>. In this study two different biosensors were developed by using two different polymer matrixes; linear poly(glycidyl methacrylate) polymer (LPGMA) and carbon black-polyvinylidene fluoride (CLPGMA) composite for the covalent immobilization of anti-IL 1 $\alpha$ . This study was also a comparative experiment between these two immobilization platforms. Both materials were coated onto ITO substrates by spin coating technique. In the fabrication processes the surface of ITO by characterized by the help of the methods, SEM, AFM, and FTIR. Under optimum experimental conditions, the biosensors showed good linear relationships between impedance values and the IL 1 $\alpha$  concentrations ranging from 0.01 to 2 pg/mL and from 0.01 to 3 pg/mL for LP(GMA) and CLP(GMA) modified ITO electrodes, respectively. Both biosensors were also applied to the real human physiological samples such as serum and saliva. The results of these experiments revealed that the methods could be used in other biosensor technologies and the biosensors presented here could be important candidates for clinical diagnosis in complex biological samples. The immobilization scheme for the biosensors can be seen in Figure 3 below.

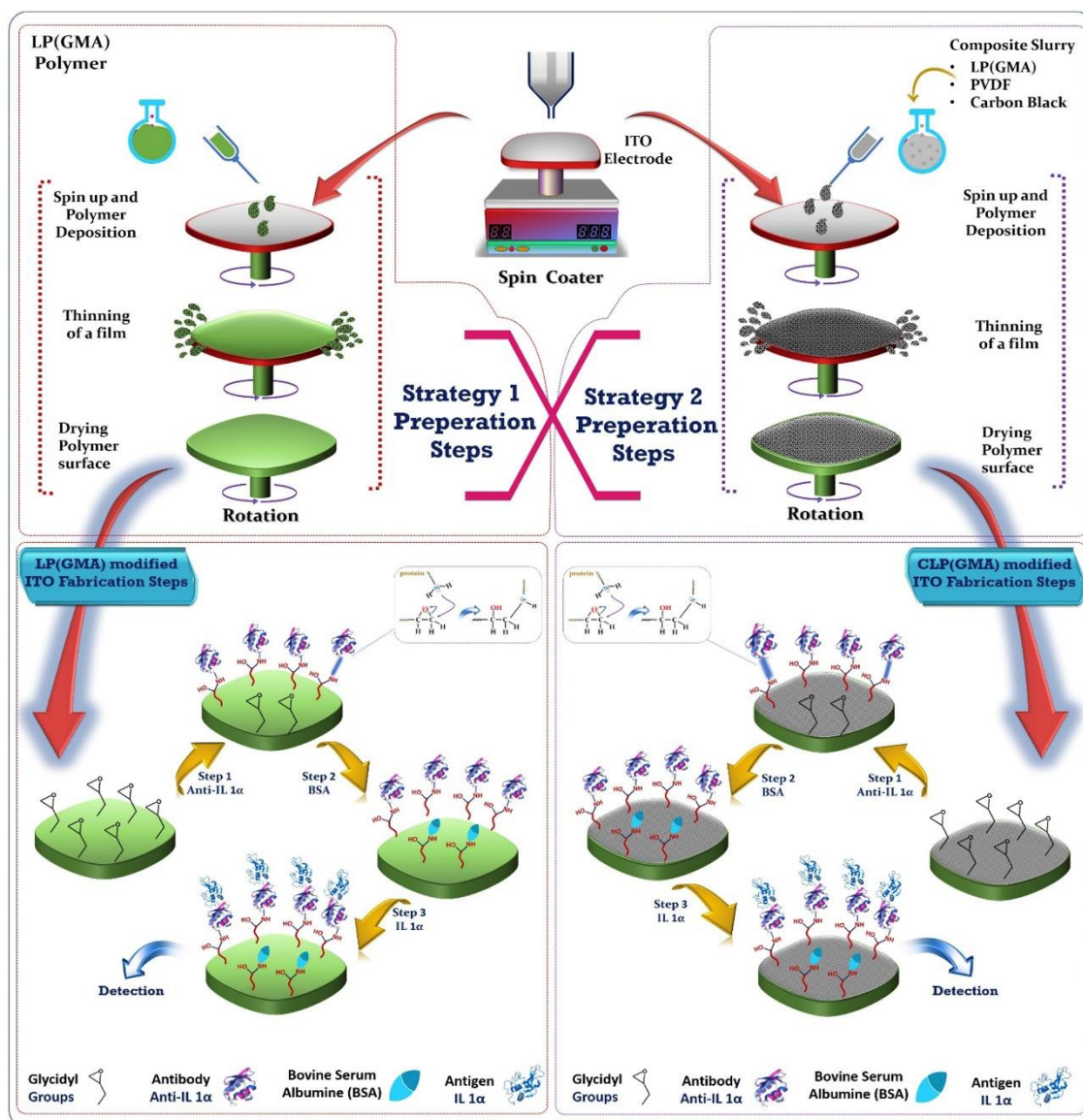
In another study, ITO sheets based biosensor was used in the determination of Alpha-synuclein which is an important protein in Parkinson-type dementia<sup>5</sup>. For this purpose, ITO-PET sheet was electrochemically covered by gold nanoparticles and polyglutamic acid. Anti-alpha synuclein was immobilized by the help of the functional carboxyl ends of polyglutamic acid on the surface. Optimization and characterization experiments were carried out and monitored by the electrochemical methods, EIS, CV, and SWV. The biosensor showed a highly linear calibration range for alpha-synuclein in the range of 4-2000 pg/mL with a LOD value of 0.135 pg/mL. Most importantly the presented biosensor was applied to the real human cerebrospinal fluid for the analysis of synuclein and found to be strikingly sensitive to the target protein. Moreover the results showed that ITO-PET based biosensor had a great reproducibility potency, long storage stability, and regeneration capacity although it was a disposable.

**Table 1.** ITO based electrochemical bio(sensors) previously reported (Reprinted from Ref.1)

Analyte	Immobilization steps	Linear range	Detection limit
Glucose	ITO/glucose oxidase	0-10 mM	0.05 mM
<i>E. coli</i>	ITO/(3-aminopropyl) triethoxysilane/ glutaraldehyde-laccase-horseradish peroxidase	$1.6 \times 10^3$ - $10^7$ cells/mL	$9.7 \times 10^2$ cells/mL
Morphine	ITO/Prussian blue/berlin green	0.09-1 mM	0.1 mM
Cholesterol	ITO/N-(2-aminoethyl)-3-aminopropyl-trimethoxysilane/1-ethyl-3-(3-dimethylaminopropyl)carbodiimide/N-hydroxysuccinimide/Cholesterol oxidase	50-500 mg/dL	25 mg/dL
Catechol	ITO/silica/polyvinyl alcohol/tyrosinase	10-200 $\mu$ M	10 $\mu$ M
Phenol		10-50 $\mu$ M	10 $\mu$ M
p-cresol		10-100 $\mu$ M	10 $\mu$ M
Ampicillin	ITO/aptamer	-	4 pM
CYFRA-21-1	ITO/hafnium oxide/APTES/anti-CYFRA-21-1/BSA	2-18 ng/mL	0.21 ng/mL
Bilirubin	ITO/APTES/stabilized Au nanoclusters/HAS	0.2-7 $\mu$ M	86.32 nM
Avian influenza virus H5N1	ITO-film transistors/3-glycidoxypopyl trimethoxysilane/anti-H5N1/BSA	$5 \times 10^{-9}$ - $5 \times 10^{-6}$ g/mL	$0.8 \times 10^{-6}$ g/mL
Cr (VI)	ITO/polyaniline/poly(vinylsulfonic acid)	$2.48 \times 10^{-6}$ - $50 \times 10^{-6}$ M	$2.48 \times 10^{-6}$ M
Dopamine	ITO/Prussian blue film/ polyethylene Imine	-	0.86 mM
E-cadherin	ITO/APTES/anti-E-cadherine/BSA	-	-
Monocytic leukemic cells	ITO/3-aminopropyl trimethoxysilane/jacalin	$10^2$ - $10^6$ cells/mL	4 cells/mL
Myeloblasticleukemi cells			3 cells/mL
Sulfite	ITO/APTES/polyvinyl alcohol/sulfite oxidase	2 -20 $\mu$ M	2.79 $\mu$ M
	ITO/APTES/Sox	0.5-2 $\mu$ M	0.5 $\mu$ M
Receptor for Activated C Kinase 1	ITO/11-(triethoxysilyl)undecanal (TESU)/anti-RACK1/BSA	14.25-712.5 fg/mL	30 fg/mL
Sex-determining region Y-box 2	ITO/carboxyethylsilanetriol/anti-SOX2/BSA	25 fg/mL-2 pg/mL	7 fg/mL
Melanoma-associated antigen1 (MAGE1)	ITO/3-Glycidoxypopyltrimethoxysilane/anti-MAGE 1/BSA	0.5 fg-15 fg/mL	0.0035 fg/mL
MAGE 1	ITO/3-Glycidoxypopyltriethoxysilane/anti-MAGE 1/BSA	0.01-1.28 pg/mL	0.0078 pg/mL

## Conclusion

In conclusion either ITO covered PET or ITO covered glass materials based biosensing applications have a great potential for the development of high sensitive, low-cost, and disposable biosensors. As outlined above, ITO is an excellent material that holding unique features such as high electrical conductivity, low capacitive current, most importantly electrochemical and physical stability. The surface of ITO enables the researchers to try many different chemical modifications after its hydroxylation. This opportunity increases the importance of ITO in the biosensor technology. Moreover, the cost of ITO based materials are extremely lower than the other solid electrodes or other disposable electrodes such as SPE. Although they have been developed as disposable it may be possible to reuse after a simple regeneration procedure. Repeatability and reproducibility of the ITO based biosensors have also been reported as perfect in the analytical perspective. Finally, ITO based systems have successfully been applied to the real human physiological samples.



**Figure 3.** Schematic representation of the immobilization processes of the biosensors (Reprinted from Ref.4)

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## IS12- Detection Power Improvement Strategies in Flame Atomic Absorption Spectrophotometry

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Quantitative determination of several elements at trace levels is one of the most significant objectives in analytical chemistry. Flame atomic absorption spectrophotometry (FAAS) has been prevalent and useful technique to carry out metal determination in different samples. Its popularity among scientists depends on its cheapness regarding its operating cost and investment<sup>1</sup>. However, low nebulization efficiency and short dwell time of the analyte atoms in the light path cause serious sensitivity problems<sup>2</sup>. Hence, there are various attempts to improve detection power of FAAS systems. Online and offline preconcentration techniques address the need to reach trace levels of the analyte(s) by FAAS system. Atom trapping methods are one of online preconcentration methods that achieve detection limits at  $\mu\text{g/L}$  or  $\text{ng/L}$  levels. Slotted quartz tube (SQT) has been coupled to FAAS system to increase residence time of the analyte atoms in the light path, therefore; detection power is increased 3-5 folds than conventional FAAS systems. Another technique is T-shaped slotted quartz tube (T-SQT) atom trapping which has been used to collect the analyte onto the inner surface of the tube. This follows an instant release of the analyte atoms from the quartz surface to the light path that gives a symmetrical and sharp signal<sup>3</sup>. Offline preconcentration techniques include microextraction methods, precipitation and adsorption. Microextraction methods based on solid phase and liquid phase techniques are most common methods to preconcentrate and purify the analyte(s). Dispersive liquid-liquid microextraction (DLLME)<sup>4</sup>, switchable solvent liquid phase microextraction (SS-LPME)<sup>5</sup> and deep eutectic solvent based liquid phase microextraction (DES-LPME)<sup>6</sup> have been well studied in literature to have lower detection limits in FAAS system.

**Keywords:** Flame atomic absorption spectrophotometry, Low detection limit, Atom trapping, Microextraction methods.

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## IS13- ISSUES AND TRENDS IN ANTIOXIDANT ACTIVITY MEASUREMENT

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**ABSTRACT-**Because O<sub>2</sub> uptakes electrons in a stepwise manner to produce reactive oxygen species (ROS) in the respirative cycle, the capability of using ROS for cell signaling and regulation may have been the initial breakthrough in the evolution of complex aerobic life forms, and the first cells should have developed antioxidant skills to protect themselves against oxidative damage. Ancient Egyptians used various antioxidant, antibacterial, and antifungal substances in mummification to keep the corpse intact, away from moisture, bacteria and air (*i.e.* oxygen radicals). Many historical examples of ‘antioxidant-rich’ food stored for relatively longer periods can be found in central Asia and Anatolia, such as ‘tarhana’ and ‘herby cheese’, to prevent oxidative deterioration when fresh vegetables are no longer available to the population. At the beginning of the 20<sup>th</sup> century, firstly pyrogallol for unvulcanized rubber, then various organosulfur and amine compounds in the vulcanization of rubber have been increasingly used to produce a durable cross-linked polymer to resist deterioration. The desire for antioxidant research have proliferated in the scientific community, but unfortunately, following a peak interest in 1990s-2000s, a decline was noted after 2010 mainly provoked by health scientists, seemingly arguing that antioxidants are longer important because their health beneficial effects have not been confirmed *via* clinical investigations up to the prevention of oxidative stress-linked diseases. Along with a great many chemists, the authors of this presentation do not share this opinion, and think that the development of novel antioxidant activity/capacity assays, though not perfectly standardized, still pose an important challenge to food analytical chemistry because each antioxidant or radical scavenging assay reveals a different aspect of the antioxidant action in real life.

In this presentation, the rationale and scope of the main issues of antioxidant measurement will be addressed, with basic definitions and terms in antioxidant research. An overview of technical problems and expectations will be given in terms of analytical sensitivity and selectivity of antioxidant measurement, interpretation of results, precision and comparability of methods, and capability of simulating the actual antioxidant action in preserving food and fighting against oxidative stress. Basic classification, reaction kinetics and thermodynamics of current antioxidant/radical scavenging assays will be discussed, along with the controversies and limitations of the widely used methods. Emerging techniques and future prospects in antioxidant testing will be mentioned, with a few hints for target-oriented assay selection.

## IS14- Potential and Challenges of Electrosynthesized Molecularly Imprinted Polymers

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**Abstract**-MIPs mimic antibodies by substituting the aminoacid scaffold of proteins by a synthetic polymer. Organic monomers are polymerized in the presence of the target molecule, which is removed after the formation of a polymeric matrix. This process results in the formation of cavities complementary to the size, shape and the position of functional groups of the template molecule. Typically, MIPs are made up from two to six functional monomers, but they have been also successfully synthesized from only one monomer

Here, we present

- (i)MIP-sensors for the enzymes laccase, tyrosinase, cytochrome P450cam and butyrylcholinesterase
- (ii)MIPs for the recognition of the N-terminal peptides of human adult hemoglobin (HbA) or glycated Hb (HbA1c).

*Keywords: Molecularly Imprinted Polymers (MIPs), electropolymerization, epitope imprinting, biomarker.*

### Introduction

Molecular imprinting is a method to create binding cavities in a polymer for the preferential recognition of the target. The application of the total biopolymer as the target for MIP-synthesis is the dominating approach since the introduction of protein-MIPs. Taking advantage of the simpler MIP synthesis and template removal for low-molecular weight compounds, exposed peptides (epitopes) have been used as the template in the synthesis of MIPs, which recognize both the epitope and the holo-protein.

Here, we present two approaches for electrosynthesizing protein-MIPs using scopoletin or o-phenylenediamine as the functional monomers.

- i) MIP-sensors for the enzymes laccase, tyrosinase, cytochrome P450cam and butyrylcholinesterase.
- ii) MIPs for the recognition of the N-terminal peptides of human adult hemoglobin (HbA) or glycated Hb (HbA1c).

### Results and Discussion

i) Holo-protein MIPs: MIP-sensors for cytochrome P450cam, butyrylcholinesterase, and the copper containing enzymes laccase and tyrosinase, were prepared by electropolymerizing the mixture of monomer and target protein. The measuring signal of the MIP sensor was generated either by measuring the formation of the product by the target enzyme or by evaluation of the permeability of the redox marker ferricyanide. For these highly active enzymes, synthesis of holo-protein-MIPs is the straightforward approach.

ii) Epitope imprinting: MIPs for the recognition of the N-terminal peptides of HbA or HbA1c MIPs were synthesized by applying either the mixtures of scopoletin and the respective terminal pentapeptides or a SAM of the Cys-extended N-terminal pentapeptide of the  $\beta$ -chain by electropolymerizing scopoletin on the gold electrode. These two peptides are the analytes in the IFCC (the International Federation of Clinical Chemistry) Reference Method for the determination of the long-term biomarker for diabetes HbA1c. All steps of MIP-synthesis and rebinding were analyzed by square wave voltammetry of the redox marker ferricyanide and by surface-enhanced infrared absorption spectroscopy of both the bound target, i.e., the peptides or the parent protein, and the polymer poly-scopoletin. Combination of the respective MIPs allows

the quantification of both the glycated and the non-glycated N-terminal peptides of HbA1c and HbA.

### Conclusion

Evaluation of diffusional permeability of a redox active low-molecular species is a simple and highly sensitive approach for the characterization of each step of MIP synthesis. However, rebinding causes only small decreases of the large reference value. Furthermore, different composition of the solutions for rebinding and the measurement of the redox marker can cause changes of the polymer layer and does not allow to discriminate for unspecific interactions. For highly active enzymes the indication of product formation is the straightforward approach.

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## **IS15- Sphingolipid Mediated Regulation of NF- $\kappa$ B and GRP 78 Gene Expression in Cancer Cells Treated with Thymoquinone**

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**Objectives:** Previous studies have shown that thymoquinone (TQ), an active compound of black seed, has anticancer properties. However, the antiproliferative mechanisms of TQ on cancer cells is unclear. Our study aimed to investigate the impact of TQ on sphingolipid metabolites, NF- $\kappa$ B and GRP 78 gene expression levels in MCF-7 and HepG2 cancer cell lines.

**Materials and methods:** Antiproliferative effect was exerted in cancer cells via TQ incubation at different doses and durations. Cell viability was measured by MTT assay. Levels of, sphingosine-1-phosphate (S1P), C16-C24 sphingomyelins (SM) and C16-C24 ceramides (CER) were determined in cell lysates by an optimized multiple reaction monitoring (MRM) method using ultra fast-liquid chromatography (UFLC) coupled with tandem mass spectrometry (MS/MS). NF- $\kappa$ B and GRP 78 gene expression levels were evaluated by quantitative PCR analysis, while protein levels were assessed by immunofluorescence and western blot analysis.

**Results:** Incubation with TQ significantly decreased cell viability in cancer cells when compared to control. A significant increase was observed in cellular levels of C16-C24 CERs in cancer cells treated with TQ for 24 hours compared to controls. Treatment with TQ significantly decreased S1P levels in cancer cells compared to untreated controls. Cleaved caspase-3 levels were also increased in cancer cells treated with TQ for 24 hours compared to controls. NF- $\kappa$ B mRNA and protein levels were significantly decreased in cancer cells treated with antiproliferative doses of TQ. GRP78 mRNA and protein levels were significantly increased in cancer cells treated with antiproliferative doses of TQ.

**Conclusions:** TQ-induced accumulation of ceramide and decrease in S1P may promote cancer cell death by inducing apoptosis.

**Keywords:** cancer, thymoquinone, neutral sphingomyelinase, ceramide

## **IS16- High Performance Biosensors in the Presence of Magnetic Nanoparticles**

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Magnetic nanoparticles (MNPs) with paramagnetic or superparamagnetic features are able to response to an exterior magnetic field. Taking advantage of their magnetic properties, samples adhered to magnetic nanoparticles can be efficiently separated from the liquid suspension. The elimination of unwanted and nonspecific interferences can be easily applied by simple controlling the magnetic particles surface chemistry [1]. Furthermore, omission of the extra steps (sample pretreatment) such as chromatography and centrifugation not only causes to shorten the handling time but also prevents the samples damage. Owing to these advantages, and some other benefits like as low background interference compared to optical and electrical approaches [2,3] and facile functionalizability, MNPs have been utilized in many biomedical and bioengineering applications such as signal markers, separation platforms, force transducers, and sensing devices, as imaging agents, heat generators, and drug carriers [2,4,5]. Here, after considering all the key parameters included starting materials, size, functionalization methods, and bio-conjugation strategies, we designed and prepared MNPs-based electrochemical biosensors (we used cobalt oxide nanoparticles and Iron(II, III) oxide nanoparticles) which utilized to diagnosis drug abuse such as cocaine, cannabinoids and even testosterone. These electrochemical biosensors are able to measure produced electrochemical signals (current, voltage, impedance) after combining target molecules to their receptors on the surface of a conducting electrode. The obtained results had shown a higher performance of the biosensor compared to traditional methods and also higher selectivity in the presence of different interferences. In conclusion, the designed biosensors propose great potential for portable and on-site substance abuse detection in addition to boasting a capability of reuse of the electrode materials, reducing the costs related to such applications.

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**S17- Future Perspectives on Enzyme Immobilization: L-asparaginase as an Example****Burhan ATEŞ**

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Enzymes have been remarkably accepted as biocatalysts in the field of bioprocess technology. The enzymes are widely used in food, paper, animal feed, textile, cosmetics, detergents and the pharmaceutical industry<sup>1</sup>. However, maintenance of structural stability of enzymes during any biochemical process is a critical factor and is highly challenging. Herein, immobilization of enzymes is used as alternative to enhance the functional efficiency, reusability, reproducibility and stability. Enzymes can be immobilized by different methods, such as adsorption, covalent attachment, immobilization of enzymes by cross-linking, matrix entrapment, encapsulation, magnetic nanoparticles hybrids formation and hybrid nanoflowers formation mechanism. One of the most important topics in this area enhances activity of immobilized enzymes in term of future perceptive. The second is to make enzyme activity controllable (turn on/off) with an externally inducible mechanism.

L-asparaginase (L-ASNase) is a unique enzyme for the enzymatic hydrolysis of L-Asn in medicine and food industry. L-ASNase is also the most important agent used as drug-enzyme in the treatment of cancer. Nevertheless, the instability, toxic effect and poor reusability of its significantly limit in the long-term applications<sup>1</sup>. Hence, the studies to increase the half-life and stability of this enzyme are important. Innovative carriers and immobilization methods must be developed to achieve this phenomenon<sup>2</sup>.

Our group has used different mechanisms to overcome disadvantages of enzyme immobilization as using model enzyme, L-ASNase. For example, L-ASNase were immobilized magnetic carrier consisting of magnetic Fe<sub>3</sub>O<sub>4</sub>-chitosan nanoparticles. We also hypothesized that magnetic excitation of conjugated L-ASNase can improved catalytic activity of L-ASNase. From our data, the conjugated L-ASNase displayed high catalytic activity in a weak magnetic field, thermal and pH stabilities. Moreover, the reusability of conjugated L-ASNase was significantly improved after immobilization and it retained 60.5% of its initial activity after undergoing 16 cycles. Furthermore, we revealed that the activity of conjugated L-ASNase onto magnetic Fe<sub>3</sub>O<sub>4</sub>-chitosan particles increased about 3-fold in the weak magnetic field at certain frequencies and flux density compared to free one.

As a result, the application of different mechanisms such as magnetic propelled and hybrid nanoflower in enzyme immobilization can be promising strategies for the increase of enzyme activity and stability, especially industrially important enzymes in terms of future perfectives.

Key Word: Immobilization, L-asparaginase, enzyme activity

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**IS18- Dietary fructose and human health: today's situation and future extrapolation**

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Sugars added to human diet contain fructose and glucose either as a disaccharide in sucrose or as a mixture of free monosaccharides in high-fructose corn syrup (HFCS). HFCS, one of the major sources of fructose, has largely replaced sucrose as a sweetener in prepared foods and soft drinks in recent decades. The increased consumption of high-fructose in diet may contribute to high prevalence of metabolic syndrome, which is a cluster of conditions including glucose intolerance, hyperlipidemia, fatty liver disease, central adiposity, hypertension and chronic low-grade inflammation. Fructose-induced metabolic disorders are more likely related to abdominal fat accumulation, but independent from the general obesity. Dysfunction in the insulin signaling pathway may cause compensatory hyperinsulinemia, thereby generating resistance to insulin. We showed that excess nutritional fructose leads to insulin resistance together with inflammatory status in blood vessel, adipose tissue, liver, kidney and testis, as well as disposition of the intestinal microbiota. Underlying mechanisms of these harmful effects of dietary fructose are related to changed expression level of insulin signaling effectors, lipogenic genes, barrier proteins, glucose transporters, inflammatory markers and disturbed hormonal balance. Dietary high-fructose causes metabolic irregularities in association with dysfunction of several organs thus signifying an important risk factor in the development of cardiovascular, hepatic, renal, gastrointestinal and endocrine disorders. Subsequently, more focus should be put on fructose, which is an important component of human diets.

## IS19- The Role of Analytical Chemistry in Clinical Trials

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Clinical trials are research investigations in which people volunteer to test new treatments, interventions or tests as a means to prevent, detect, treat or manage various diseases or medical conditions.

Bioanalysis is an integral part of the characterization of novel chemical entity from the time of its discovery and during various stages of drug development. Strategies in the drug discovery and development processes are undergoing radical changes in recent years. For example Pharmacokinetics in human and animal studies are increasing, Furthermore toxicokinetics has now become established as an essential part of toxicity testing. For those reason a sensitive and specific analytical methods are essential. Bioanalytical data generated in discovery and preclinical studies are a valuable guide to early clinical studies.

Full analytical characterization of drug should be made in compliance with the current The International Council for Harmonisation Guidelines (ICH).

In this study, the important bioanalytical- analytical parameters and pitfalls and its application to drug discovery and development approaches will be discussed which will help in the development of safe and more efficacious drugs.

**Keywords:** clinical studies, new drug entity, bioanalysis, analytical chemistry

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## **IS20- Electrochemical Nucleic Acid Biosensors Developed by Advanced Nanomaterials**

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Advanced nanomaterials, such as, graphenes, nanoparticles, nanofibers, nanowires, nanotubes etc. have recently received a significant interest in order to develop “nanoscale materials integrated (bio)sensors towards to the areas of biomedical technologies and drug discovery. Since electrochemical nucleic acid (NA) biosensors present the characteristic specificity for biorecognition process on nucleic acid hybridization with a high sensitivity and selectivity, they hold a great promise especially for sequence-selective nucleic acid detection in aspects of clinical investigations.

An overview to nanoscale materials integrated electrochemical biosensors has been presented in the present study for detection of the interactions of nucleic acids with drug or protein, and nucleic acid hybridization with their advantages.

**Keywords:** *Nanomaterials, Electrochemical Biosensors, Nucleic acid biosensors*

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## IS21- Radionuclide Labeled Theranostic Iron Oxide Nanoparticles (MNPs)

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Theranostics nanoparticles can combine diagnostic and therapeutic capabilities for both target-specific diagnosis and the treatment of a given disease. Iron oxide nanoparticles (MNPs) are significant alternatives when radiolabeled with radionuclides for hybrid imaging tools as theranostics. PET (Positron Emission Tomography) or SPECT (Single Photon Emission Tomography) radionuclides labeled MNPs as PET/SPECT-MRI hybrid imaging agents were prepared by reduction-precipitation method and coated with an amino silane coupling agent. Then they were coupled with several conjugates depend on the targeting aim and radiolabeled for the imaging or therapy purposes. Their characteristics and radiolabeling yields were determined. Their in vitro/in vivo bioaffinities were also determined.

Iron oxide nanoparticles have been successfully used as theranostic agents both in vitro and in vivo applications. Furthermore, their functionalization with drugs or functional biomolecules enhance their drug delivery efficiency and reduces the systemic toxicity of drugs. Recent progress on this topic will be outlined in the article.

**Keywords:** *Iron oxide nanoparticles; radiolabeling; hyperthermia; MRI contrast agent; theranostics*

### Introduction

Radionuclide labeled magnetic nanoparticles promise potential platform for imaging, diagnosis and therapy as theranostics. They can be directed to the magnetic area due to their magnetic properties. The magnetic property represents the particles by loading drug molecules, it is possible directed to localized disease sites in the body. Magnetite ( $\text{Fe}_3\text{O}_4$ ) is the most used carrier magnetic material for this purpose. The various application area has been studied due to biocompatible and low toxic properties of magnetic nanoparticles (MNP). They are bound to the membrane receptors when a magnetic field is applied. It has been reported that magnetic field also increases the apoptotic effect.<sup>1</sup>

Recent advances in technology over the past two decades allowed for the development of novel technique for medical diagnosis. In particular, the development of computer tomography, functional NMR and positron emission tomography (PET) greatly facilitate the diagnosis of early stage of cancer and therefore to begin the appropriate therapy much earlier as compared to classical diagnostics. Unfortunately, the effectiveness of current strategies for cancer therapy does not dove-tail the huge advances in tumor diagnostics. The most important problem is the acquired or innate drug and radiotherapy resistance of the tumor cells. Therefore, it is necessary to search for novel therapeutics, treatment and multimodal strategies for cancer therapies.

### Materials and Method

#### Synthesis and surface modification of magnetite nanoparticles:

Functional surface modification of magnetite nanoparticles is a significant issue for various reasons, such as stability, prevention of agglomeration or biocompatibility in nanoparticle research. Modification with polymeric, organic and enzyme may be appropriate, mostly based on ligand exchange reactions. In most studies, surface modification of  $\text{Fe}_3\text{O}_4$ -nanoparticles was done using amino silane coupling agent, N- [3- (trimethoxysilyl) propyl] -ethylenediamine (SG-Si900), followed by covalent ligand or prodrug. The amine group was fastened to using glutaraldehyde as a crosslinker.<sup>2</sup> Thiols, disulfides, amines, nitriles, carboxylic acids and phosphines are other groups that can be used for modification of iron oxide based magnetic nanoparticles.<sup>3</sup>

Normally surface modification involves coating materials to improve biocompatibility, blood circulation time and stabilize colloidal solution. 2. Polymers such as PEG (polyethylene glycol), HPG (Hyperbranched polyglycerol), dendrimers, hyaluronidase and dextran are the most commonly used coating materials. <sup>3</sup>

## Results and Discussion

Fe<sub>3</sub>O<sub>4</sub> magnetic nanoparticles exhibit MRI contrast feature. These nanoparticles will have the potential for PET-MRI imaging when radiolabeled with PET radionuclide or hybrid imaging potential with SPECT MRI when marked with SPECT radionuclide. Table 1 represents several radionuclides that have potential for labeling magnetic particles.

**Table 1:** PET and SPECT Radionuclides

PET Radionuclides		SPECT Radionuclides	
Radionuclides	Half Life	Radionuclides	Half Life
<sup>68</sup> Ga	67.7 min.	<sup>99m</sup> Tc	6.0
<sup>18</sup> F	109.7 min	<sup>201</sup> Tl	73
<sup>64</sup> Cu	12.7 hrs	<sup>67</sup> Ga	78
<sup>89</sup> Zr	3.3 days	<sup>111</sup> In	67.2
<sup>44</sup> Sc	3.97 hrs	<sup>123</sup> I	13.2
<sup>124</sup> I	4.18 days	<sup>131</sup> I	8.0 days

PET radionuclides decay by positron emission and emit 511 keV annihilation photons and used in PET imaging systems while SPECT radionuclides emit single gamma photons and used in SPECT systems. On the other hand if the ligands label with a gamma-emitting radionuclide with similar chemical properties, preferably an isotope of the same element as the therapeutic radionuclide or an isotope, which have similar chemical properties. Thus, gamma emitting radionuclide can be used for imaging and mechanisms of follow up, other one can be used for therapy. Tc-99m and Re-186, Y-88 and Y-90, I-123 and I-131, In-111 and In-114m are this type of couples. The radionuclides couples such as I-131/I-123, Cu-64/Cu-67, Sc-47/Sc-44, Lu-177/Ga-68 have been used for radionuclide therapy and imaging as theranostics. <sup>4</sup>

## Conclusion

Magnetic nanoparticles have promising potential in bioimaging and diagnostic applications due to their biocompatible properties as well as easily preparation and ease of bioconjugation. It may be possible to increase their specificity by using target-specific conjugates.

In the near future, we expect the development of various applications of radiolabeled magnetic nanoparticles for hybrid imaging systems such as PET-MRI or SPECT-MRI for targeted therapy, and various therapeutic radiolabeled magnetic nanoparticles.

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## **IS22- A New Method for Simultaneous Measurement of Short, Medium and Long Term Blood Sugar**

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Diabetes is a metabolic disease and regular glycemic control is necessary to prevent disease progression. This study focuses on the development of the flow injection analysis method that simultaneously performs the measurement of glucose, glycosylated-albumin and glycosylated-hemoglobin HbA1c.

A borate selective selective electrode has been developed and used as a detector. The characterization features of the electrode were studied. and the repetitive operation in a stagnant and mobile environment were tested. In the flow injection analysis environment, there was a detection chamber containing a composite borate selective sensor and a mini-column providing a partial separation. The method or device that measures three parameters together is absent in the literature, and is also economical and simple. Therewith, pretreatment requires only centrifugation and lysis of the blood sample taken.

The method developed, for the simultaneous measurements of glucose, glycosylated-albumin, and glycated-hemoglobin can be carried out in clinics, health centers, polyclinics and hospitals that do not have autoanalysers. This will be enabled to obtain the status of short, medium and long term blood glucose and diabetic condition fast and economically.

Keywords: Biosensor, diabetic diagnosis, hba1c, fructosamine, FIA.

## IS23- Design of QCM aptasensors for detection of pathogens using differently prepared magnetic separation systems

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Monitoring microbial contamination from food samples is complicated due to the present diverse molecules in such complex samples. The reliability of sensing method is usually affected of the presence of high quantities of biological macromolecules in the samples. Commonly applied procedures for pathogen infected sample concentration include gravitational techniques, membrane filtering and recognition element functionalized particles. Although many sensitive detection systems were developed for microorganisms, interference from the complex samples has been the major drawback that hindered applications. In recent years, magnetic separation technologies have been adapted for enzyme immobilization, biological macromolecules separations (such as protein and cells) and pre-concentration of target molecules from the complex mixture in a rapid way. In our recent studies, various magnetic pre-concentration methods have been adapted for pathogen detection system and then coupled to QCM and PCR sensing platforms. For example, magnetic p(HPMA/EGDMA) (poly (hydroxy propyl methacrylate) / ethylene glycole dimethacrylate) based system was used for pre-concentration of target bacteria and then subsequently combined with PCR, real-time PCR (rtPCR) or Quartz Crystal Microbalance (QCM) systems. Then, they were used for detection of pathogens from milk and milk products samples (1-3). Aptamers ligand are mostly used as specific ligand for detection of target pathogens. It is a bio-recognition molecules selected from complex libraries of nucleic acids by a combinatorial chemistry method known as SELEX<sup>(3,4)</sup>. DNA aptamers have several advantages comparable to antibodies such as better stability, smaller size, and easy chemical modifications. As functional nucleic acid molecules with specific recognition properties, aptamers have been commonly employed for the development of many aptamer-based biosensors and drug delivery systems. The applications of aptamer-based biosensors have been reported for a wide range of targets from small molecules to whole cells like bacteria. There are a few reports that introduce the strategy for the determination of pathogens in food products by combining magnetic pre-concentration system with QCM. Association of aptamers with nanoparticles provided enhanced performance for highly efficient purification of targets as well as biosensor applications.

**Keywords:** Aptasensors, QCM, Pathogen, Magnetic separation system

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## IS24- Challenges and Advancements in Safety Testing of Nanoparticles

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### Abstract

Nanoparticles (NPs) have exceptional physicochemical features that diverge from large-sized material. Relatively high surface to volume ratio, reactivity and small size properties favor a wide spectrum of applications in various industrial areas. Although amazing properties of NPs offer huge potential in biomedical and molecular biology applications, it should be comprehended that great powers come with great responsibility. Nearly all of the nanotechnological applications include products that direct contact with people in various aspects. Thus, it is essential to understand the toxicological features of certain nanoproducts and constitute safety reports according to the purpose of usage. Safety investigation strategies of NPs must be carried out in aspect to a certain application of these NPs. Different *in vitro* and *in vivo* safety analyses are performed to explore main routes of toxicity such as ingestion, inhalation and skin penetration characteristics of NPs. A confident safety investigation of NPs for human use should combine chemistry, approach routes, as well as the effectiveness and cytotoxicity testing. On the other hand, comprehensive analysis of safety assessments still needs standardization and strong correlation techniques. Another important issue for nanoparticle development is a cost for utility relationships because further investigations for the safety of various NPs results in higher expenses and laborious lab works. Size, surface structure and biochemical activity of NPs are the main factors that diversify toxicity mechanisms and safety investigations would be incomplete without molecular characterizations. The characterization of nanoparticles with microscopic, chromatographic, spectroscopic methods should be performed in detail, taking into account their size and physicochemical properties.

**Keywords:** Nanoparticles, Safety assessment, Nanotechnology, Toxicity analysis, Standardization

**IS25-The role of the active complexes of selected anti-cancer drugs with Bi(III) in the aspect of kinetics and electrode mechanism changes in the presence of non-ionic surfactants**

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Knowledge of electrochemical properties of anti-cancer drugs is essential with regards to more profound understanding of their metabolic path or *in vivo* redox processes. Therefore, it is necessary to investigate the mechanism of their actions and search for new systems of controlled drugs release mechanism, which can be used for monitoring of patient's health condition.

Based on the above, the studies on the kinetics and mechanisms of electrode's process in the presence of 6-mercaptopurine (6MP), the drug used in the anti-cancer chemotherapy, mainly in marrow and lymphoblastic leukaemia's<sup>1</sup>, seem to be by all means justified. The application of voltammetry techniques (SWV, CV, DC), as well as electrochemical impedance spectroscopy (EIS) will allow the determination of the kinetic and thermodynamic parameters which in turn, will define the 6MP catalytic effect and also their correlation in the presence of Triton X-100 and Tween 80.

The presence of mixed 6MP – non-ionic surfactants adsorption layers affects the mechanism and kinetics of irreversible Bi(III) ions electroreduction process in chlorates(VII). Tween 80 and Triton X-100 change the dynamics of the catalytic effects of 6-mercaptopurine on Bi(III) ions electroreduction with the tendency of inhibition. The mechanism of catalytic activity of 6MP is associated with formation of complexes under specific conditions which exist on the electrode surface. The Bi – 6MP type complex plays the main role in the Bi – 6MP – Tween 80 or Bi – 6MP – Triton X-100 systems, as 6MP dominates in the formation of adsorption equilibria of the studied mixtures<sup>2</sup>.

The results of investigations can propose a trend in medicine for determination and knowledge of somewhat different mechanisms of controlled drug release. Investigations on changes in dynamics of kinetics and electrode mechanism, in which chemical stages play an essential role, seem to be advisable.

**Keywords:** Bi(III) electroreduction, 6-mercaptopurine, surfactants, active complexes, kinetic parameters

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## **IS26- Electrochemical Glucose Biosensor Based on Dendritic Gold Nanostructures**

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Scientific and industrial impact of nanoscience and nanotechnology on analytical chemistry, medicine and pharmacy has been growing. Electrochemical biosensors have been applied in many areas, such as food industry, agriculture, military, veterinary, and clinical applications. Some challenging bioanalytical problems, such as sensitivity, specificity, reproducibility, duration and cost of analysis can be solved by nanostructure-based electrochemical biosensors<sup>1-3</sup>. The aim of this work was to synthesize dendritic gold nanostructures (DGNs) on the graphite rod (GR) electrode surface (DGNs/GR) by one-step procedure, in the absence of any template, surfactant and stabilizer, and to apply DGNs/GR for the development of amperometric glucose biosensor. All electrochemical measurements were performed with a computerized potentiostat PGSTAT 30/Autolab (EcoChemie, Netherlands) with GPES 4.9 using three-electrode system.

In this work the best conditions for DGNs synthesis were determined. The electrode surface after the modification with DGNs was characterized using electrochemical methods and SEM. Several methods of enzyme immobilization (adsorption, covalent immobilization on self-assembled monolayer, covalent immobilization with additional cross-linking) on GR electrode pre-modified with DGNs were tested and compared. DGNs/GR electrode modified with 11-mercaptoundecanoic acid self-assembled monolayer, covalently immobilized and cross-linked glucose oxidase was characterized by the highest analytical signal and the best limit of glucose detection.

**Keywords:** dendritic gold nanostructures, glucose biosensor, electrochemical methods.

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## ORAL PRESENTATIONS (OP)

### OP1- A novel immunosensor system based on polyvinyl alcohol modified disposable graphite paper electrodes for sensitive detection of a cardiac biomarker

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#### Abstract

Cardiovascular diseases (CVD) are one of the causes of death worldwide. In these types of diseases, which may occur due to many different factors, the systems developed for early diagnosis play a critical role. In this work, the biosensor system with disposable electrodes (GP) and several electrochemical techniques has been designed to determine Suppression of Tumorigenicity 2 (ST2) which is the important as a potential biomarker for early detection of cardiovascular diseases. Electrochemical impedance spectroscopy, cyclic voltammetry and single frequency technique were utilized for immobilization, optimization and characterization studies when biosensor was designed. It has been observed that the biosensor system developed for detection of cardiovascular diseases has very high sensitivity, excellent reproducibility and long shelf life.

Keywords: Suppression of Tumorigenicity 2 (ST2), graphite paper (GP), polyvinyl alcohol (PVA)

#### Introduction

Cardiovascular diseases (CVD) are included as the non-rhythmic heart, failure of blood vessels, acute myocardial infarction, coronary failure and many other cardiovascular disease<sup>1</sup>. World Health Organization (WHO) reported that 20 million people death (approximately, 31% of all global deaths) are related to cardiovascular disease in 2015<sup>2</sup>. ST2 genes are preferably expressed on activated Th2 cells instead of Th1 cells and play an important role in Th2 effector functions characterized by raised expression of IL-4, IL-5 and IL-13 cytokines<sup>3,4</sup>. The serum ST2 protein concentrations increase in fibrosis, various autoimmune diseases, and idiopathic pulmonary<sup>5</sup>.

In this study, the biosensor system with disposable electrodes and several electrochemical techniques has been designed to determine ST2 which is the important as a potential biomarker for early detection of cardiovascular diseases.

#### Materials and Method

Impedance and cyclic voltammetry measurements were taken with Gamry Potentiostat/Galvanostat (Reference 600, Gamry Instruments, Warminster, PA, USA) connected with a computer via an EChem Analyst software throughout the entire study. PURELAB flex 3 & 4 Ultrapure Water Purification System (ELGA LC 134 model) was used during the preparation stages of all immobilization and incubation of the immunosensor. All the chemicals, biorecognition materials (anti-ST2 antibody, ST2 antigen and BSA protein) were purchased from Sigma-Aldrich (St. Louis, M.O., USA). The whole proteins were formed by using phosphate buffer (50 mM, pH 7.0) and were kept at -20 °C. Also, 5 mM [Fe(CN)<sub>6</sub>]<sup>4-</sup>/5 mM [Fe(CN)<sub>6</sub>]<sup>3-</sup> (1:1) which is a redox probe solution containing 0.1 M KCl, was prepared. Using three - electrode system has a Ag/AgCl (saturated with KCl) as a reference electrode and a platinum wire as a counter electrode, except for the working electrode (GP) in this study. Reference electrode and counter electrode were purchased from BASi (West Lafayette, IN, USA). All measurements were performed at 25 °C in 15 mL of 5 mM [Fe(CN)<sub>6</sub>]<sup>4-</sup> /5 mM [Fe(CN)<sub>6</sub>]<sup>3-</sup> solution which is ferricyanide/ferrocyanide redox probe. The construction steps of the ST2 biosensor system are presented in Figure 1.

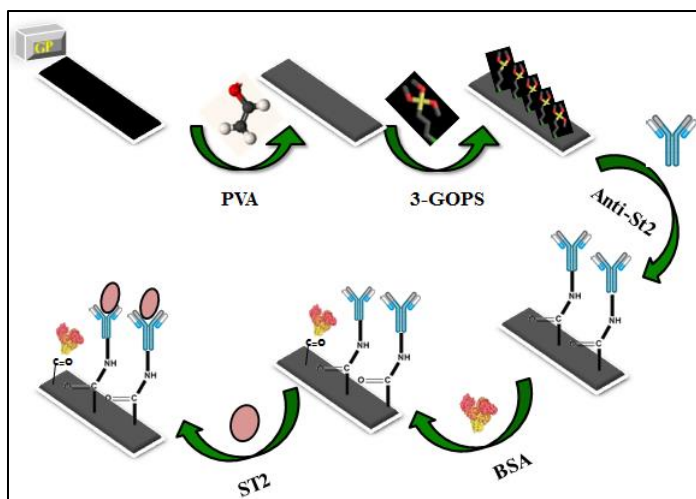


Figure 1. Schematic presentation of the ST2 immunosensor

### Results and Discussion

Electrochemical impedance spectroscopy and cyclic voltammetry are efficient techniques to research the immobilization of the electrochemical immunosensor. The EIS technique is utilized with a Randles equivalent circuit which involves the charge transfer resistance ( $R_{ct}$ ), the solution resistance ( $R_s$ ), the constant phase element (CPE) and Warburg impedance ( $W$ ). The diameter of the semicircle of the EIS data corresponds to the  $R_{ct}$  value in the Nyquist plot. The electrochemical impedance spectra and cyclic voltammograms are given in Figure 2.

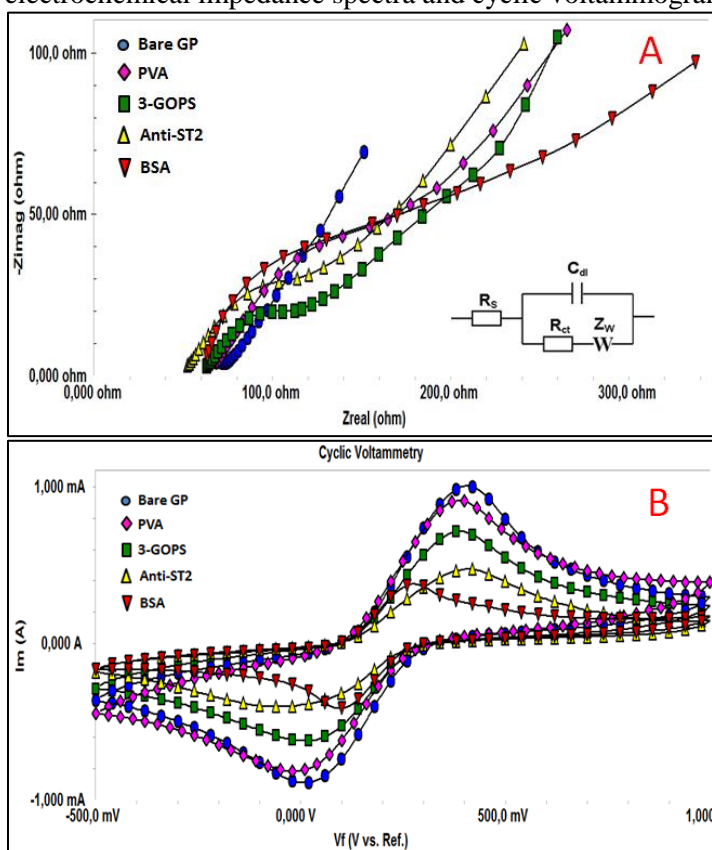


Figure 2. EIS spectra and CV voltammogram of the ST2 immunosensor

### Optimization studies

The optimization study was performed to investigate the effect of 3-GOPS concentration on the immunosensor response. The immunosensors were prepared using three different concentrations of 3-GOPS solution (1 %, 1.5 % and 2 %) for this optimization step. The obtained calibration graphs are seen in Figure 3.

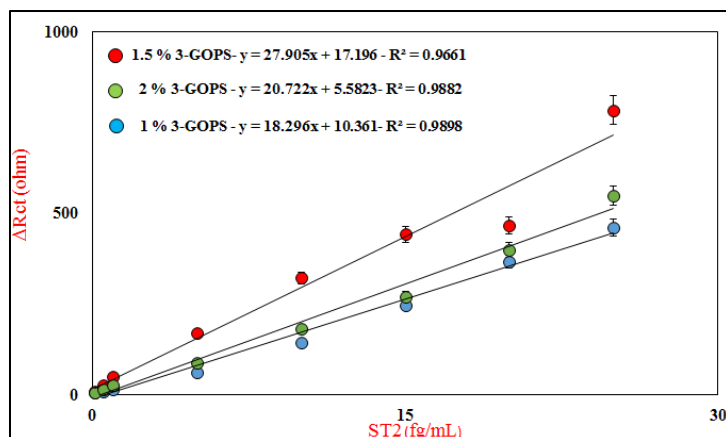


Figure 3. The effect of 3-GOPS concentration on the proposed immunosensor

## Conclusion

This study presents the fabrication of a reusable, very sensitive and excellent reproducible ST2 immunosensor with a single-use, low-cost, applicative GP electrodes. The developed immunosensor can perform the analysis of the ST2 biomarker in the level of sub-femtogram and at a wide detection range.

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**OP2- A new and disposable biosensing system for highly sensitive detection of Neuropeptide Y: A potential biomarker for childhood obesity**

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**Abstract**

The incidence of obesity, which is defined as the increased amount of fat in the body, varies according to sex, age and race. Neuropeptide Y (NPY) is thought to be a potential biomarker for early diagnosis of the obesity.

Neuropeptide Y is a hormone that suppresses appetite. Recent studies suggest that genetic differences in the synthesis of these peptides and their receptors play a role in the development of obesity. In this study, a disposable GP (graphite paper) based biosensor was designed for Neuropeptide Y detection. After the immobilization process was carried out, all the immobilization steps were optimized in order to develop a linear and stable biosensor. To investigate the immobilization steps and optimization of the biosensor, electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV) were used. Then, the analytical characterization of the proposed biosensor was determined.

Keywords: neuropeptide Y, graphite paper electrode, biosensor

**Introduction**

The prevalence of childhood obesity is increasing all over the world and The World Health Organization shows obesity as one of the most important public health problems. Most of the obese children continue to be obese adults in their lives. Obesity also causes different diseases such as type 2 diabetes mellitus, cardiovascular diseases, hypertension and some cancers. The identification and analysis of potential biomarkers associated with obesity is very important and necessary for the early diagnosis of the disease<sup>1</sup>. NPY strongly increases food intake and reduces energy consumption. In addition, NPY modulates memory processes, depressive state, anxiety and stress. It also acts as a chemical mediator that controls the cycle of circadian rhythms<sup>2</sup>. Electrochemical impedance spectroscopy (EIS) is a useful technique to examine the change in interface properties between electrode and electrolyte induced by antibody-antigen interaction, DNA hybridization or enzyme-substrate catalysis using ferri ferro cyanide as an indicator<sup>3</sup>. In this study, the aim is to develop a biosensor for neuropeptide Y detection using appropriate modification methods.

**Materials and Method**

All measurements for the designed biosensor were taken from Gamry Potentiostat / Galvanostat, Reference 600 (Gamry Instruments, Warminster, USA) connected to a computer with Cyclic Voltammetry, Chronoimpedimetry and Electrochemical Impedance Spectroscopy software (Gamry Instruments, Warminster, USA). Graphite paper working electrode, 3 M Ag/AgCl reference electrode and platin counter electrode were used as triple electrode system. All chemicals used in this study were bought from Sigma- Aldrich (St. Louis, MO, USA). GP electrode was purchased from Xiamen Tob New Energy Technology Co. Ltd. All electrochemical measurements were taken in ferri ferro cyanide. The schematic presentation of the immobilization steps is given Figure 1. And anti-neuropeptide Y concentration was optimized to develop a sensitive biosensor.

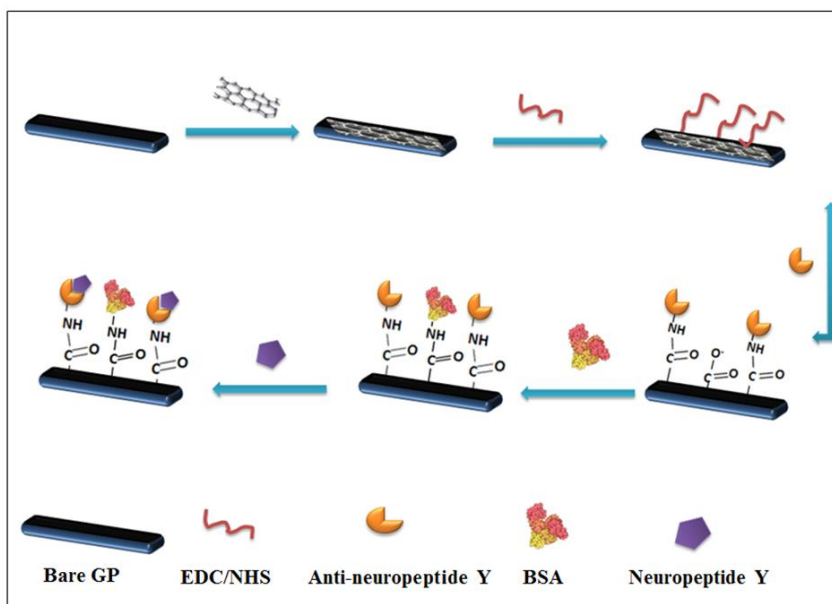


Figure 1. Schematic presentation of the neuro peptide Y biosensor

## Results and Discussion

Electrochemical impedance spectroscopy is an effective technique to investigate electrochemical systems and methods. This technique is mainly based on the measurement of resistance affected by capacitance and inductive changes when high frequencies are applied<sup>3</sup>. The electrochemical impedance spectra and cyclic voltammograms are given in Figure 2.

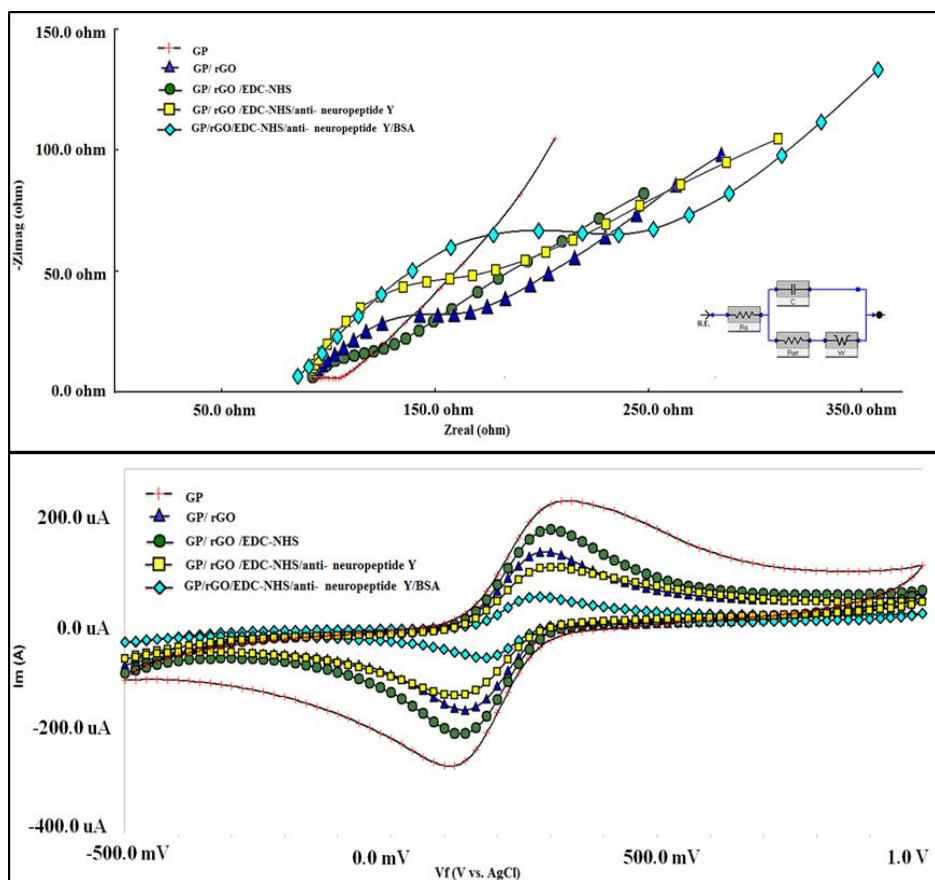


Figure 2. EIS and CV measurements of the immobilization process

### Optimization studies

The optimization of anti-neuropeptide Y concentration is very crucial and important step for fabrication a reproducible, repeatable, sensitive and linear biosensor. For this purpose, the GP electrodes were immobilized with different concentrations of anti-neuropeptide Y (5.024 ng / mL, 25.12 ng / mL, 50.24 ng / mL). The obtained calibration graphs are seen in Figure 3.

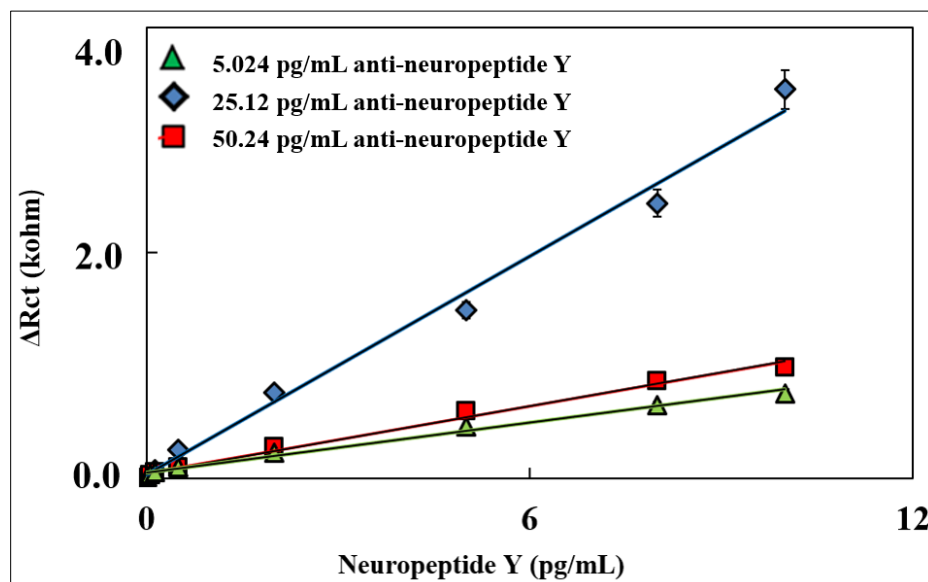


Figure 3. The effect of anti-neuropeptide Y concentration on biosensor responses

### Conclusion

This study presents a disposable GP-based biosensor was fabricated for neuropeptide Y detection. The proposed biosensor system has a lot advantages such as low-cost, excellent repeatability, reproducibility and usability to clinical field.

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### Acknowledgment

This work was funded by TÜBİTAK (The Scientific and Technological Research Council of Turkey, Project number: 113 Z 678) whose assistance is greatly acknowledged.

### OP3- A new immobilization route for immunosensors: CNBr activation for covalent attachment of anti-Leptin onto the electrode surfaces

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**Abstract:** Leptin is a peptide hormone synthesized in adipose tissue. It acts as a kind of anti-obesity factor to balances between energy intake and expenditure. High leptin concentrations are directly associated with obesity. Also, leptin has many functions such as regulation of bone mass, stimulation of angiogenesis, regulation of sympathetic nervous system activation, bone density determination, and brain development. In this study, we designed a novel immobilization method and aimed to design an ultrasensitive electrochemical immunosensor for Leptin detection. We developed single-use electrochemical biosensing system that enables a precise, specific, economical and practical analysis of Leptin biomarkers. The indium tin oxide (ITO-PET) sheets were used as a working electrode. ITO sheets were modified by using cyanogen bromide (CNBr) for immobilizing the anti-Leptin antibody via covalent interactions. Each stage of the desired biosensors were characterized by EIS and CV methods. Optimization and characterization studies were performed extensively.

**Keywords:** biosensor; disposable immunosensor; ITO-PET electrode; leptin; CNBr

#### Introduction

Metabolic syndrome acts a significant threat to human health and quality of life. Adipocytokines secreted by adipose tissue play a critical role in storage, food intake, energy expenditure, lipid, and glucose metabolism. Leptin is responsible for regulating food intake, body weight, and energy homeostasis through neuroendocrine functions. Recent research has shown that leptin affects insulin sensitivity and lipid metabolism. High leptin concentrations are involved in the development of metabolic diseases such as insulin resistance, type 2 diabetes and cardiovascular diseases and are directly related to obesity. Leptin has been evaluated as a predictive marker for metabolic syndrome<sup>1,2</sup>.

Electrochemical biosensors have important advantages in determining hormones due to their simplicity, sensitivity, portability, and ease of use. Unlike chromatographic techniques, the electrochemical techniques used do not require pre-treatment. Electrochemical biosensors are based on amperometric, potentiometric and impedimetric methods. Previously, Zhang et al. aimed to develop an electrochemical biosensor for the detection of leptin<sup>3</sup>. In this work, they modified glassy carbon electrode which they used as a working electrode with SWCNT-chitosan nanocomposites. As a result, the researchers suggested that this biosensor had a wide detection range in the range of 0-1000 ng/mL and the detection limit was 5 pg/mL. In this study, we developed an immunosensor based on a new immobilization process. By this new process ITO surface was simply modified and ready to bound antibodies for covalent immobilization. The resulted biosensor showed excellent analytical characteristics for the determination of Leptin.

#### Materials and Methods

##### Fabricating of ITO-based immunosensor

The first step, ITO-PET electrodes cleaned by ultrasonication with acetone, soap solution and ultrapure water respectively for 10 min. The electrodes were incubated for 90 minutes in a solution containing ammonium hydroxide, hydrogen peroxide and ultra-pure water (1:1:5) to form hydroxyl groups. Then the electrodes were washed with ultra-pure water and dried with argon gas. After this procedure, the ITO electrodes were immersed into %1 CNBr solution (pH 11) prepared in the pure acetonitrile and incubate for 45 minutes at room temperature. Then ITO electrodes were washed with acetonitrile and ultra-pure water, respectively to get rid of physically adsorbed CNBr molecules. After interaction of CNBr and OH groups, electrodes were incubated in 200 µL of 74 ng/mL Anti-Leptin solution for 60 minutes to immobilize antibodies to the

surface. After incubation with Leptin antibody, electrodes were washed with ultra-pure water to remove unbound antibody molecules. Finally, Anti-Leptin immobilized ITO electrodes were treated with BSA (0.5%) for 60 min to block the active ends. At the end of this, the electrodes were washed with ultrapure water and dried with argon gas. With this last step, the now-prepared biosensor was stored at +4°C until the Leptin measurements were performed. The modified ITO substrates were indicated as ITO, ITO/OH, ITO/OH/CNBr, ITO/CNBr/Anti-Leptin, ITO/CNBr/Anti-Leptin /BSA and ITO/CNBr/Anti-Leptin /BSA/Leptin.

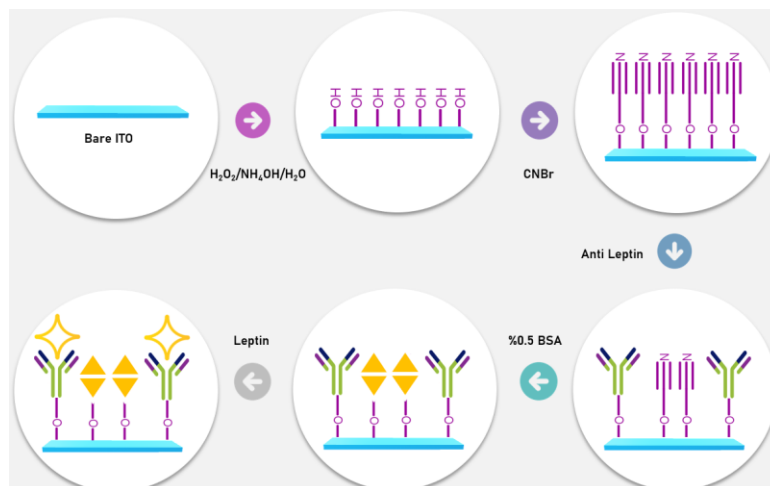


Figure 1. Immobilization scheme for Anti-Leptin by CNBr

## Results and Discussion

Electrochemical impedance spectroscopy is a powerful technique that allows for unlabelled and real-time measurement of probe-analyte interactions. EIS was successfully used to measure the amount of Leptin on the electrode surface of the described biosensor. Absolute impedance values were used to establish a linear relationship between Leptin amount and electrochemical signals. Increased concentration of Leptin caused an increase in semicircle diameters in Nyquist plots.

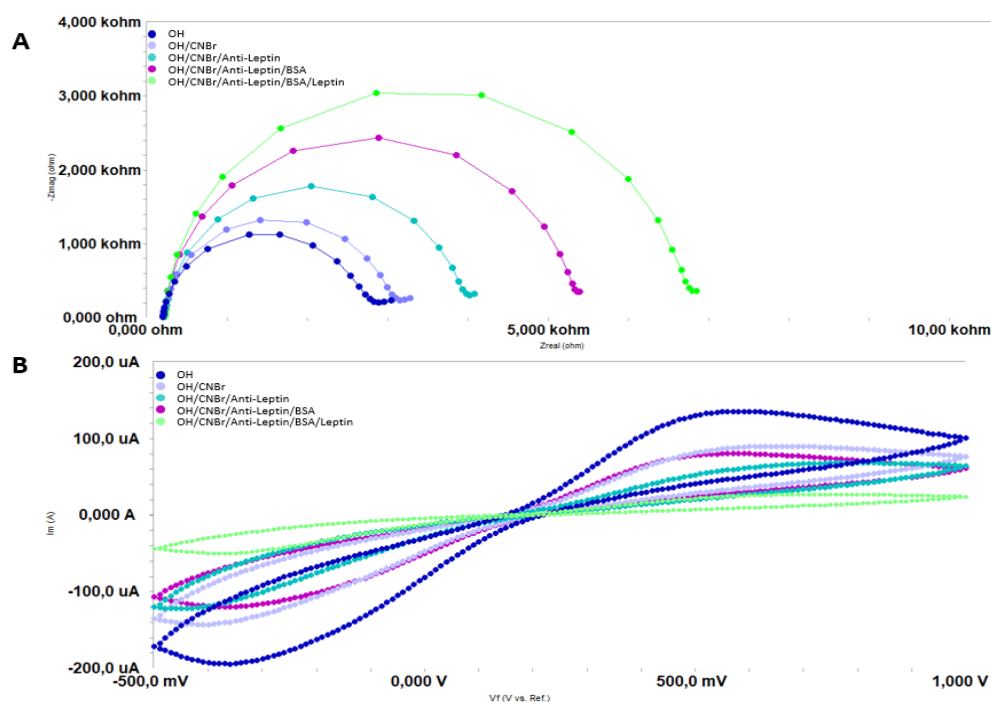
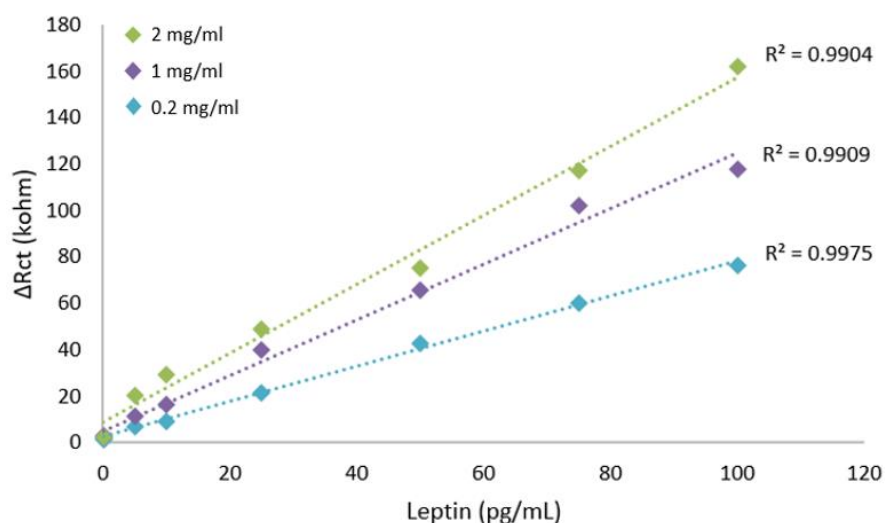


Figure 2. EIS (A) and CV (B) responses of immobilization steps

## Optimization studies

In the optimization study of biosensor, the effect CNBr concentration was investigated. Three different concentration values of 0.2, 1 and 2 mg / mL were tested. Each step of immobilization is characterized by impedance and voltammetric techniques. With obtained  $R_{ct}$  values differences standard curves were plotted.



**Figure 3.** Standard curves for Leptin obtained by different CNBr concentrations

### Conclusion

The aim of this study was to develop a biosensor for early diagnosis of Leptin-related diseases. The parameters for the formation of the desired biosensor have been optimized and characterization studies were performed comprehensively. This biosensor may be preferred because of its sensitivity, wide detection range and low-cost. The new immobilization process by CNBr for the fabrication of biosensor achieved here could be effectively used in other biosensing applications.

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**OP4- A promising neuro-biosensing probe for diagnosis of Parkinson's disease from cerebrospinal fluid and saliva samples: selective analysis of PARK-7**

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**Abstract**

Recent studies suggest that PARK-7 biomarker has a close relation with Parkinson's Disease (PD). The precise and selective determination of PARK-7 biomarker may help clinicians to diagnosis Parkinson's disease at an early stage and to guide treatment.

In this study, it is aimed that the ITO based disposable neuro-biosensor probe can selectively detect the target protein PARK-7 in the cerebrospinal fluid (CSF) samples. The design strategy of the neuro-biosensor is based on the modification of ITO and gold nanoparticle doped electrodes by multiwalled carbon nanotubes (MWCNT). The techniques used in the design of the immobilization steps, the optimization studies, and the evaluation of the analytical performance of the targeted neuro-biosensor are electrochemical impedance spectroscopy (EIS), cyclic voltammetry. As a result of the evaluation of its analytical characteristics, it has been seen that the developed system has high reproducibility capability, impressive regeneration capacity, good selectivity and long storage life. Changes in surface morphology were also evaluated by Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM).

**Keywords:** Parkinson disease; PARK-7; biosensor; electrochemical impedance spectroscopy

**Introduction**

PD is the most common movement disorder and represents the second most common neurodegenerative disease of the central nervous system<sup>1</sup>. Plaques, tangles and Lewy bodies in the autopsy findings of older individuals show that the preclinical phase of PD is very long, which has led to the characterization of the preclinical phase as a vital target<sup>2</sup>. The World Health Organization uses almost every measure, including chemical, physical or biological, that reflects the interaction between the biological system and a potential hazard for biomarkers<sup>3</sup>. PD is usually diagnosed based on clinical history and motor symptoms. When motor symptoms occur, the biology of the disease is thought to be in the middle or late stage<sup>4</sup>.

PARK-7 protein is mainly expressed in glial cells in the cortex and in neurons of the substantia nigra and striatum. Studies have shown that the loss of the PARK-7 protein function is one of the causes of Parkinson's disease<sup>5</sup>. Recent studies have shown that PARK-7 can be a potential marker not only in CSF but also in saliva, which increases in concentration of PD<sup>6</sup>.

**Materials and methods**

Reference (Ag/AgCl), counter (platinum wire) and working electrodes (disposable ITO-coated PET films, 2 × 0.5 cm) were supplied from BASi (West Lafayette, IN, USA) and Sigma Aldrich (St Louis, MO, USA). PARK-7 from human plasma and monoclonal anti-PARK-7 antibody from mouse were obtained from Sigma Aldrich (St Louis, MO, USA). PARK-7 and anti-PARK-7 stock solutions prepared with pH 7.0 phosphate buffer at certain concentrations were maintained at -20 °C until used. 5 mM [Fe(CN)<sub>6</sub>]<sup>4-</sup> and 5 mM [Fe(CN)<sub>6</sub>]<sup>3-</sup> containing 1 M KCl were prepared in 50 mM phosphate buffer using it as a redox probe. The CSF samples were obtained with research ethics committee approval numbered 2013/86/07/05 from Namık Kemal University, Faculty of Medicine. The morphological changes that occurred in each design step of the neurobiosensor were studied by Scanning Electron Microscopy (SEM, FEI-Quanta FEG 250). Measurements of changes in chemical bonds were performed by Attenuated Total Reflection Fourier Transform Infrared (ATR-FTIR) using a Bruker Company Vertex 70 FTIR infrared spectrometer (4000–400 cm<sup>-1</sup>). The design of the neuro-biosensor is intended to covalently bond multi-walled carbon nanotubes (MWCNT) on the surface of ITO modified ITO-based electrodes with gold nanoparticles. The design strategy of the neuro-biosensor is schematized in Figure 1.

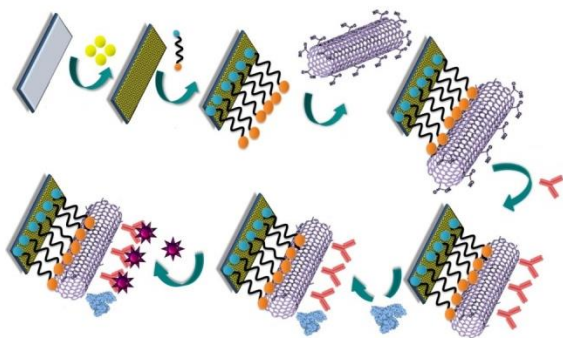


Figure 1. Schematic illustrations of immobilization steps of anti-PARK-7.

## Results and discussion

In this neuro-biosensor system developed for the determination of PARK-7, the SAMs layer (with 11-AUT) exhibits the perfect packaging properties in molecular order; Thanks to the conductive properties of the gold nanoparticle, the high surface-to-volume ratio of the redox mediator and the MWCNT have been combined to enable the PARK-7 to be detected very quickly, even at low concentrations. The impedance spectra taken from the EIS for the steps of the construction of the neuro-biosensor developed for the determination of PARK-7, the charge transfer changes for each step and the voltammograms of the CV are shown in Figure 2.

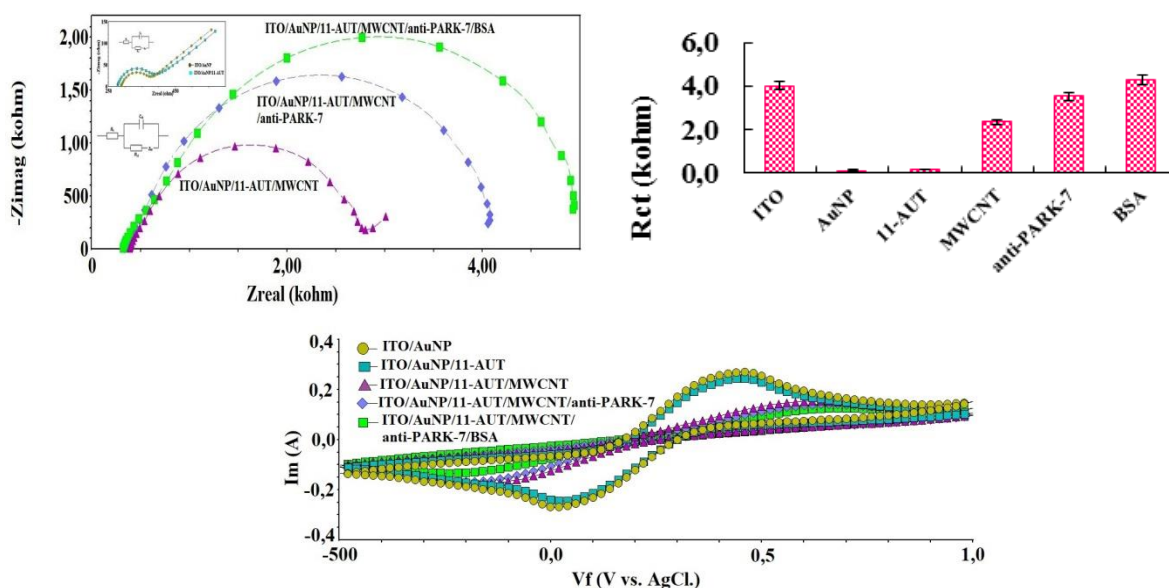


Figure 2. EIS spectrum, charge transfer changes and cyclic voltammograms of immobilization steps.

The high insulating properties of the ITO-PET electrodes do not allow the transfer of electrons, which leads to a high charge transfer resistance. Electrochemically, gold nanoparticle accumulation on the surface of the electrodes resulted in a reduction in the charge transfer resistance by providing conductivity to the electrodes. The charge transfer resistance of gold nanoparticle coated ITO electrodes after treatment with 11-aminoundecanethiol increased very little. The increase in the number of CH<sub>2</sub> units (n) in the hydrocarbon chain backbone significantly increases SAM formation.

The relationship between increased PARK-7 concentration charge transfer resistance was observed to reach a linear regression at femtogram level for PARK-7 concentration. These result emphasize that the hybrid structure developed is an excellent capture ability for PARK-7 antibody-antigen binding. ITO-based AuNP / 11-AUT / MWCNT doped system had very good reproducibility. Regeneration of the developed neuro-biosensor was performed by immersing the AuNP / 11-AUT / MWCNT modified ITO electrodes into 5 mM HCl for 3 minutes to decompose the PARK-7 antibody-antigen immuno-complex. The electrodes presented approximately the

same impedimetric response fix concentration of PARK-7 over 7 cycles.

The developed neuro-biosensor system was found to show high selectivity even in the presence of various proteins (SYN alpha, Tau-441, HSP-70). The neuro-biosensor developed for the determination of PARK-7 was stored dry for 5 weeks and the storage stability was evaluated by measuring the same concentration of PARK-7. The sensor system was completely stable for 5 weeks. To evaluate the feasibility and analytical reliability of the produced neuro-biosensor, real samples were analyzed using standard addition method in human cerebrospinal fluid and saliva. The addition of different concentrations of PARK-7 to the CSF and saliva samples and the relative standard deviation and the retrieval of the analytical values after the EIS detection were calculated.

## Conclusion

The changes of PARK-7 concentration in PD is remarkable point of the disease. Early detection of this protein may have a significant effect on the course of the disease and the quality of life of the patient. Thanks to the nanocomposite platform consisting of gold nanoparticles and MWCNT, this biomarker was identified in a selective, sensitive and wide range. In addition, the neuro-biosensor platform has high reproducible, repeatable and storage capability. The highlight of the study is that the MWCNT-AuNP-based 11-AUT-modified neuro-biosensor platform can analyze the PARK-7 protein in both CSF and saliva.

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## OP5- Effect of Bisphenol A, Lead and Endosulfan on Liver and Kidney Functions in Rats

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### Abstract

In this study, individual and combined toxic effects of Bisphenol A (BPA), Lead (Pb) and endosulfan on rat liver and kidney functions were investigated. In this study, 64 Wistar albino male rats were used. The rats were divided into 8 groups (n = 8); Control group, Lead group, Bisphenol A group, Endosulfan group, Lead + Bisphenol A group, Bisphenol A + Endosulfan group, Lead + Endosulfan group, Bisphenol A + Lead + Endosulfan group. The determined agents were administered to rats for 65 days. Tissues were stained with Hematoxylin-Eosin staining method. Serum samples of Aspartate aminotransferase (AST), Alanine aminotransferase (ALT), Gamma Glutamyl Transferase (GGT), Blood urea nitrogen (BUN), Creatinine and (CREA) levels were determined by spectrophotometric method. In the evaluations, it was determined that BPA, Pb and Endosulfan given in combination had an additive / synergistic effect and had a stronger effect on liver and kidney compared to individual exposures.

**Keywords;** Bisphenol A, lead, endosulfan, mixture toxicity, TUNEL

### Introduction

In vivo and in vitro studies have shown that some micro-pollutants in the environment may interfere with normal hormonal functions of organisms and disrupt endocrine systems. These chemicals, which can be found in soil, water and nutrients, create toxic effects on animals and humans through the food chain. These chemicals can be listed as Endocrine-Disrupting Compound EDC, medical drugs, hormonal active substances (Hormonally Active Agent, HAA) and antibiotics<sup>1</sup>.

BPA, polycarbonate plastics and epoxy resins, e.g. It is a high production volume chemical used for CDs and DVDs, dental fillings, cash receipts, plastic bottles, inner lining of cans and the reassembly of water pipes<sup>2</sup>.

Pb is a toxic heavy metal and one of the most commonly used metals in industries. Pb is used in printing, rubber, batteries, ceramics, paint, accumulator industry and as gasoline additive. In particular, workers of these industrial branches are exposed to lead at a higher rate<sup>3</sup>.

Endosulfan is a member of the organochlorinated pesticide group used worldwide in agriculture. It is widely used all over the world for vegetables, fruits and non-food crops such as cotton and tobacco<sup>4</sup>.

In current study, we aimed to investigate combined *in vivo* toxic effects BPA, Pb and Endosulfan in rats.

### Materials and Method

This experimental study was carried out in Experimental Research and Application Center of Namık Kemal University, Tekirdağ. Sixty four male Wistar albino rats were used. Rats were divided into eight groups; Control group, Lead group, Bisphenol A group, Endosulfan group, Lead + Bisphenol A group, Bisphenol A + Endosulfan group, Lead + Endosulfan group, Bisphenol A + Lead + Endosulfan group respectively. Endosulfan (10 ppm), Lead (100 ppm) and Bisphenol A (75 ppm) administered orally. End of the experimental period (65 days) rats were sacrificed under anesthesia. Histological structure of liver and kidney tissue were evaluated by Hematoxylin-Eosin staining method. Serum AST, ALT, GGT, BUN and CREA levels were determined by spectrophotometric method.

Data were evaluated statistically using SPSS (IBM, SPSS Statistics, Version 21). All data were expressed as mean standard deviation. The numerical parameters of the groups were evaluated using non-parametric test (Kruskal-Wallis). Significance of the values obtained in two-way comparison groups was measured by Mann-Whitney U-test. When the difference between the groups was less than 0.05, it was accepted as significant.

### Results and Discussion

Histopathological changes such as sinusoidal dilatation, hemorrhage and vacuolization in the liver tissue were evaluated. Tissue structure was normal in the liver sections of the control group. No histopathological changes were observed in the hepatocyte cords and portal areas. Histological structure of BPA and

Endosulfan treated groups was similar to control group, while vacuolization findings were observed in Pb group. Especially in Pb + Endosulfan + BPA group, bleeding and vacuolization were observed.

The types of hepatic injury can be measured very often using ALT and AST levels. ALT and AST are transaminases commonly used to evaluate hepatic trauma. AST is not a specific hepatic marker because it often increases when other cellular damages occur, for example during a heart lesion, over-loosening the skeletal muscle, or during some erythrocyte-based diseases (Shi et al. 2017).

In our study, serum AST and ALT levels were examined and a significant difference was found only in terms of AST levels in BPA + Pb + Endosulfan group compared to control group ( $p < 0.05$ ). There was no significant difference in ALT levels between the groups.

Bleeding, degeneration of tubular epithelial cells and glomerular changes were evaluated in renal tissue. No significant histopathological changes were observed in the groups with BPA, Pb and Endosulfan individual exposure, but bleeding, tubular epithelial shedding and vacuolization were observed especially in the groups combined with Pb. In the Pb + Endosulfan + BPA triple combination group, in addition to the changes, narrowing of Bowman interval was noted. A significant difference was found between the control and BPA + Pb + Endosulfan groups in terms of renal function tests BUN and CREA values ( $p < 0.05$ ).

### Conclusion

Statistically significant differences in the organ functions indicated in the groups in which the agents were given in combination showed that these agents were in synergistic / additive interaction.

The fact that these three agents are among the most frequently encountered environmental pollutants increases the value of the findings and suggests that further studies should be conducted on the subject.

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## **OP6- A Novel Electrochemical Nanosensor Based on COOH-Functionalized Multi Walled Carbon Nanotubes Decorated with Quantum Dots for The Determination of Clopidogrel**

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**Abstract:** In this study, a novel and sensitive voltammetric nanosensor has been developed for the first time, for the detection of Clopidogrel based on carboxyl-functionalized multiwalled carbon nanotubes (COOH-fMWCNT) with CdSe quantum dot modified glassy carbon electrode. Scanning electron microscopy, Atomic force microscopy (AFM) and cyclic voltammetry were employed for characterization of the modified electrode. The dependence of intensities of currents and potential on pH, concentration, scan rate, nature of the buffer was further investigated. The different pulse voltammetry results demonstrated excellent electrocatalytic activity of the modified electrode toward the oxidation of Clopidogrel as endorsed by the enhanced current responses compared to bare electrode. For experimental conditions optimization the influence of supporting electrolyte and pH was examined and pH 2.14 phosphate buffer was selected as the best electrolyte for getting intense current signals of the target analyte. Scan rate results showed that the oxidation of Clopidogrel at the nanosensor surface occurs under adsorption-controlled manner. Therefore, adsorptive stripping differential pulse voltammetric (AdSDPV) technique was employed for the determination of Clopidogrel. Optimum accumulation potential and time were found as 0 V and 60 s, respectively.

Under these optimum conditions, response of Rilpivirine demonstrated a linear behavior in the concentration range from  $2.0 \times 10^{-6}$  M and  $4.0 \times 10^{-5}$  M, with a limit of detection value of  $7.55 \times 10^{-8}$  M in pH 2.14 phosphate buffer solution. The designed method was successfully applied for the determination of clopidogrel in synthetic human serum. The value of limit of detection were found to be  $2.5 \times 10^{-6}$ – $1.5 \times 10^{-5}$  M with a limit of detection value of  $2.99 \times 10^{-7}$  in serum samples, of CLP in tablet dosage form Atervix® was also proposed with acceptable recovery values.

**Keywords:** Clopidogrel, Nanosensor, Quantum Dots, Carbon nanotubes

### **Introduction:**

Clopidogrel (CLP, (S)-Methyl 2-(2-chlorophenyl)-2-(6,7-dihydrothieno[3,2-c]pyridin-5(4H)yl)acetate, a thienopyridine derivative drug, is a prodrug that prevents adenosine diphosphate-induced platelet aggregation selectively and irreversibly; there is no direct antiplatelet activity in the in vitro environment. It transforms into active form by hepatic cytochrome P-450 3A4 and 3A5<sup>1</sup>.

Among all nanomaterials, carbon nanotubes (CNTs) are one of the most preferred materials for electrode modification. Carbon nanotubes have attracted attention thanks to their remarkable advantages such as promoting electron transfer reactions, high surface area, high electrical conductivity, mechanical robustness, and excellent chemical stability. On the other hand, in recent years quantum dots (QDs) have also been presented as an attractive electrode modification agent. QDs are semiconductor nanoparticles with size range of 1–100 nm nanocrystal particle.

Electrochemical methods are most promising as compared to other detection techniques such as mass spectrometry, spectrophotometry, fluorimetry, capillary electrophoresis, high performance liquid chromatography etc. Electrochemical techniques are currently the leading probes for sensing and monitoring of pharmaceuticals.

Our main objective here was to construct a simple, economical and highly sensitive nanosensor, fMWCNT and CdSeQDs modified GCE, to investigate the voltammetric determination of

Clopidogrel using adsorptive stripping differential pulse voltammetric (AdSDPV). The nanosensor was successfully applied to Clopidogrel assay from its pharmaceutical dosage forms.

### Materials and Method:

AUTOLAB-PGSTAT100N (Eco Chemie, Utrecht, The Netherlands) electrochemical instrument was used for performing cyclic voltammetry, differential pulse voltammetry (DPV) and AdSDPV, which is controlled by NOVA 2.1 software.

Clopidogrel bisulphate (CLP) and its dosage form, Atervix® tablets, were kindly provided by Biofarma İlaç (Istanbul, Turkey). Required amount of CLP was weighed and dissolved in methanol for preparing  $1.0 \times 10^{-3}$  M stock solution. -COOH group functionalized multiwalled carbon nanotubes (fMWCNT) and CdSeQDs were purchased from DropSens. fMWCNT suspension was prepared in DMF in the concentration of 0.5 mg/mL and sonicated for 2 h. fMWCNT/CdSeQDs suspension was prepared by mixing 200  $\mu$ L fMWCNT and 50  $\mu$ L CdSeQDs (dispersed in chloroform by commercially) under ultrasonic stirring during 30 min. The volume of fMWCNT/CdSeQDs suspension (4:1) was dropped as 5  $\mu$ L onto the cleaned GCE.

### Results and Discussion

Using fMWCNT/CdSeQDs/GCE, related with electrocatalytic effect of the nanomaterials, the peak of the CLP was moved to  $\sim 928$  mV, greatly enhanced with  $\sim 100$  mV shift. The peak current of  $1 \times 10^{-5}$  M CLP in pH 2.14 was approximately 23 times higher at the fMWCNT/CdSeQDs/GCE compared to the bare GCE under the same experimental conditions by AdSDPV.

For the analytical application using designed nanosensor, firstly operational parameters have been optimized. The dependence of intensities of currents and potential on pH, concentration, scan rate, nature of the buffer studies was further investigated. The best peak shape and highest current value were obtained when phosphate buffer solution at pH 2.14 was used. The logarithm of oxidation peak currents versus scan rates exhibited a linear relationship with a slope of 0.751 which can be expressed by mixture of diffusion and adsorption controlled process. Further voltammetric analysis were carried out in pH 2.14 phosphate buffer solution using 0 V as accumulation potential and 60 s as accumulation time. The LOD value of the designed fMWCNTs/CdSeQDs/GCE nanosensor is quite low, 0.075  $\mu$ M level, lower than the other voltammetric techniques in anodic way. The developed AdSDPV technique was applied to the direct determination of CLP in tablet dosage form Atervix® tablet and spiked serum by AdSDPV, using the related calibration equations. Furthermore, recovery studies, which were also applied for both determination of CLP in spiked serum and tablet dosage form by AdSDPV.

### Conclusion:

In this study, CLP was analyzed using fMWCNTs and CdSeQDs modified GCE. The obtained results show that the fMWCNTs/CdSeQDs/GCE exhibited good electrocatalytic performance toward CLP oxidation. The designed fMWCNTs/CdSeQDs/GCE sensor gave 0.075  $\mu$ M level LOD value which is lower than the other published voltammetric techniques in anodic way.

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## OP7- Ru@VC Nanohybrid Based Nanosensor for the Electrochemical Detection of Idarubicin

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### Abstract

A novel nanosensing platform was suggested based on ruthenium for the sensitive determination of anticancer drug idarubicin (IDA). GCE surface was modified with Ruthenium/Vulcan carbon-based nanoparticles (Ru@VC) and characterization of the nanosensor was performed using EIS and CV. Under optimized conditions at 0.0 mV accumulation potential, 30 s accumulation time, the calibration graph of  $I_p$  vs. concentration of IDA gave linear response between  $5 \times 10^{-8}$  M and  $1 \times 10^{-6}$  M. The LOD and LOQ values were found as  $9.25 \times 10^{-9}$  M and  $2.8 \times 10^{-8}$  M in buffer samples. In order to demonstrate the applicability and validity of developed nanosensor, it was used for the determination of IDA in Idamen<sup>®</sup> IV (10 mg/10 mL vial) and human serum sample. The results of recovery studies showed that the Ru@VC/GCE nanosensor was free from interferences of the excipients in the injection dosage forms and it can be successfully applied to biological samples.

Keywords: Idarubicin, Electrochemistry, Nanosensor, Ruthenium nanohybrid

### Introduction

IDA is a cytotoxic antibiotic which is used for treatment of different cancers such as leukemia, myeloma and hematological diseases in various chemotherapy protocols<sup>1</sup>. Due to its importance developing a new, fast, low cost and practical sensor for the determination of IDA in pharmaceutical dosage forms and biological fluids is important. Amongst a wide range of different techniques for the determination of compounds electroanalytical techniques are one of the most advantageous and widely used techniques due to their sensitivity, accuracy, reliability and low cost<sup>2</sup>. In the literature, most of the present studies are based on chromatographic analysis and there are only two electrochemical sensors<sup>3</sup>. Hence, the aim of the present work is to monitor inorganic nanomaterial platform based on ruthenium for the sensitive and rapid determination of IDA. For this aim, a novel nanomaterial (Ru@VC) is synthesized and a new and more sensitive electrochemical nanosensor using new nanomaterial for investigation of the electrochemical behavior and determination of IDA in pharmaceutical dosage forms and human serum sample is achieved.

### Materials and Method

The stock solution of  $1 \times 10^{-3}$  M IDA was prepared in methanol. In order to prepare working solutions of IDA with different concentrations, the stock solution was diluted with desired pH buffer solutions containing 20% methanol. For investigating influence of the pH effect on the electrochemical studies; pH 0.3, 0.5 M H<sub>2</sub>SO<sub>4</sub> and pH 0.5, 0.1 M H<sub>2</sub>SO<sub>4</sub> solutions, pH 1.5, pH 2.5, pH 3, pH 6.02, pH 6.5, pH 7.0 and pH 8.0 phosphate buffer solutions, pH 3.7, pH 4.7 and pH 5.7 acetate buffer solutions were used as supporting electrolyte. For the preparation of Ru@VC suspension; 1 mg of Ru@VC was dispersed in 1 mL of distilled water. The suspension was ultrasonicated for 2 hours using ultrasonic bath. the nanomaterial amount optimization study was performed by dropping different volumes of Ru@VC suspension (1  $\mu$ L, 3  $\mu$ L, 5  $\mu$ L, 7  $\mu$ L and 10  $\mu$ L) onto the electrode surface and drying in a vacuum oven. The working solution of Idamen<sup>®</sup> IV 10 mg/10 mL with 20% methanol was prepared by dilution of the stock solution to  $2.5 \times 10^{-7}$  M with pH 1.5 phosphate buffer solution. In order to prepare standard serum solution; 1 mL of IDA from  $1 \times 10^{-3}$  M stock solution of IDA, 3.6 mL serum and 5.4 mL acetonitrile were put together in a 10 mL centrifuge tube and this mixture was centrifuged for 30 minutes at 3500 rpm. The recovery studies were performed using standard addition method in order to prove reliability, applicability and accuracy of proposed nanosensor from real samples.

### Results and Discussion

These results of EIS studies indicate that the modified GCE has better electronic conductivity and enhances electron transfer kinetics compared to bare GCE. Besides, the surface of Ru@VC/GCE has faster electron transfer and decreased charge transfer resistance. The pH increase resulted with a shift of  $E_p$  to less positive values.  $E_p$  showed linear response versus pH. These results showed that  $E_p$  is pH dependent. Furthermore, the slope value of the equation above is close to the theoretical value of -59 mV and it suggests that equal amounts of electrons and protons are involved in the rate-determining steps. pH 1.5 phosphate buffer was chosen as the optimum pH value for the determination of IDA and was used in further experiments. As the results of scan rate studies; the linearity of  $I_p$  vs  $v$  indicates that the oxidation mechanism is adsorption controlled. Deposition potential and deposition time were further optimized. With 0 V accumulation potential and 30 s accumulation time, the highest oxidation peak of IDA was acquired and these values were chosen as optimum conditions for further experiments. With Ru@VC modification the peak current of IDA increased 9 times compared to the bare GCE. Using AdSDPV the peak current of IDA increased 16 times. As a result of optimization of nanomaterial amount studies 7  $\mu$ L was selected as the optimum nanomaterial amount and used in following studies. The calibration graph of  $I_p$  vs. concentration of IDA gave linear response between  $5 \times 10^{-8}$  M and  $1 \times 10^{-6}$  M. The LOD and LOQ values were found as  $9.25 \times 10^{-9}$  M and  $2.8 \times 10^{-8}$  M. IDA was also determined in human serum sample. Linear range was obtained between  $5 \times 10^{-8}$  M and  $2.5 \times 10^{-7}$  M with the LOD and LOQ values of  $7.24 \times 10^{-9}$  M and  $2.19 \times 10^{-8}$  M, respectively.

### Conclusion

The proposed nanosensor showed an enhancement for the oxidation peak current of IDA due to its improved electronic conductivity and electron transfer kinetics compared to bare GCE. The LOD and LOQ values were found as  $9.25 \times 10^{-9}$  M and  $2.8 \times 10^{-8}$  M with a linear range between  $5 \times 10^{-8}$  M and  $1 \times 10^{-6}$  M.

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## OP8- Pharmacometabolic Profiling of Solifenacin on Rat Plasma

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**Abstract:** In this study, it was aimed to create a metabolic profile of the drug from plasma samples of rats given orally solifenacin for 10 days. After the extracts obtained from control and drug applied rat plasma samples were dissolved in the mobile phase, they were analyzed in positive ion mode by LC/QTOF-MS. For the chromatographic separation of metabolites, a gradient elution program with a mobile phase consisting of a mixture of acetonitrile containing 0.1% formic acid and ultrapure water containing 0.1% formic acid was used. Data analysis, metabolic pathway analysis and chemometric analysis results of LC/QTOF-MS data of plasma samples were reported. The 43 metabolites that show significant differences in plasma levels of 4441 features determined by mass detector are identified by HMDB and METLIN. It was demonstrated with OPLS-DA that the metabolic profiles of the drug and control groups differ from each other with good selectivity and sensitivity.

### Introduction

Overactive bladder (OAB); it is a syndrome that causes lower urinary tract symptoms, disrupts patients' daily activities, work life, sleep patterns, sexual life and negatively affects the patient psychologically, physically and economically. Symptoms such as incontinence, frequent urination (two to eight times a day), urge to wake up from sleep at night (more than once a night) are observed depending on the urge to urinate suddenly and violently in OAB.<sup>1</sup>

The most preferred drugs in the treatment of OAB syndrome are antimuscarinic agents. Solifenacin, which is classified as antimuscarinic drugs, has specific antimuscarinic properties due to its very low efficacy for different ion channels and other receptors. Solifenacin; it inhibits frequent and uncontrolled contractions of detruster and peripheral muscle fibers by inhibiting neuromuscular regions that provide bladder contraction through acetylcholine.<sup>2</sup>

Metabolomics, which are included in the Omics technology class, examine the biological changes occurring in metabolism at the metabolite level, enable us to better interpret the metabolic reactions and examine the metabolic changes that the drugs cause in the organism with the help of the pharmacometabolomix approach.<sup>3</sup>

In this study, metabolic profiles of the plasma samples collected from the drug and control rat groups were created and interpreted.

### Materials And Methods

#### Materials

The chemicals used for the metabolomics study and their sources are as follows; deionized water, PURELAB Ultra system; The Netherlands), acetonitrile (Sigma-Aldrich, St. Louis, MO, USA), 98% formic acid (Riedel-de-Haën, Honeywell,). Olanzapine was used as internal standard. Materials, such as single-use sterile pipettes, 1.5 mL disposable tube (Eppendorf, UK), 1.5 and 2 mL conical bottom screw cap tube (BD Biosciences Discovery Labware, CT, USA), 96-well cell culture plates (Eppendorf, UK), 0.45 µm syringe filter tips (membrane), were used in the study.

#### Animal Experimental

Twenty four male albino wistar rats, weighing from 325 to 350 g, were obtained from the Medical Experimental Research Centre, Ataturk University. The animals were seperated different groups and fed under normal conditions (22°C). Animal experiments were approved by Atatürk University local animal care committee; carried out in accordance with national guidelines for the

use and care of laboratory animals. Rats were randomly divided into two groups (n=12 each): a healthy group and a drug group: solifenacin (5 mg.kg<sup>-1</sup>).

At the end of 10 days, all rats in the control and drug groups were anesthetized with 25 mg/kg thiopental sodium administered intraperitoneally and a suitable waiting period was observed before blood collection by surgical intervention. Then the blood of each animal was taken from their hearts.

### Method Subsections

The plasma of the blood samples was separated. After performing the extraction procedure from plasmas, the supernatants were completely dried in a vacuum concentrate. Samples dissolved in the mobile phase were analyzed by LC/QTOF-MS. Analysis results were interpreted using multivariate data analysis methods.

### Results And Discussion

All chromatograms of each group were pre-processed. Peak detection, matching and retention time alignment were carried out. After that obtained data were transferred into MATLAB software program for classification and annotation. OPLS-DA were preferred to classify groups. VIP scores were calculated for each parameter. Several metabolites that are important for many biochemical pathways were identified. Pathway analysis were succeeded via Metaboanalyst 4.0 software. Metabolite identifications were carried out by comparing the either MS/MS or Q-TOF results with the HMDB spectrums. In the study, 43 metabolites were identified which differ significantly from the drug group compared to the control group and whose features were identified with HMDB and METLIN.

### Conclusion

Metabolic differences between the control and drug groups have been successfully demonstrated by the metabolomic approach. The plasma levels of 43 metabolites involved in sphingolipid, aminoacyl-tRNA and vitamin D biosynthesis, cysteine and methionine, glycerophospholipid, arachidonic acid, phenylalanine and tryptophan metabolism and citrate (TCA) cycle metabolic pathways were determined to change.

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**OP9- Investigation of the inhibition effect of compounds isolated from *Paeonia daurica* root on viral (MMLV-RT) and bacterial DNA polymerases (PolC and DnaE)**

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**Abstract-** The methanolic extracts from roots of *Paeonia daurica* subsp. *macrophylla* (*P. daurica*) were investigated for inhibitory effect on replicative bacterial (PolC and DnaE2) and viral (MMLV-RT from Moloney Murine Leukemia Virus) DNA polymerases by primer extension assay and roots of the plant was focused to purify inhibitory compound(s). The chemical structures of compounds were completely elucidated using a combination of NMR, MS and FT-IR analyses. Five molecules with tree monoterpene glycosides, paeoniflorin (PD-2), paeoniflorigenone (PD-4), benzoyl paeoniflorin (PD-5), and benzoic acid (PD-3) with its derivate 2,4,6 trihydroxy-1-methyl benzoate (PD-1) were purified and identified. Both DNAdependent and RNA-dependent polymerase activity of MMLV-RT was strongly inhibited by these five molecules. On the other hand, bacterial polymerases PolC and DnaE2 were strongly inhibited by only paeoniflorigenone (PD-4). We proposed that the parts of *P. daurica* might be used to find new antimicrobial agents and generate supplementary material for foods. Furthermore, the isolated molecules with inhibitory effect may be used as new scaffold for the further modification in order to develop novel inhibitors against DNA polymerization.

**Keywords:** *Paeonia daurica*, DNA polymerase inhibition, Paeoniflorigenone

## 1. Introduction

There are a large number of DNA polymerases with the same or different functions involved in protecting, repairing, and replicating the integrity of genomic DNA in viruses, prokaryotes, eukaryotic cells, and organelles such as chloroplast and mitochondria. The multiplication of viruses and bacteria could be prevented by inhibiting the function of DNA polymerases. The development of antimicrobial drugs to inhibit polymerases will provide advantages in the fight against disease-affecting microorganisms.

*Paeonia* species have been used as traditional medicine especially in traditional Chinese medicine because of their wide range of curative effect for many diseases (He et al. 2010a). It has been claimed that the roots of these species have therapeutic properties due to their anti-inflammatory, antibacterial, antiviral, antioxidant, antispasmodic, tonic, astringent, sedative, and analgesic properties, and flowers have aromatherapeutic properties as well.

In this study, methanolic extracts of fruit, fruit barks, leaf, stem, and root from *P. daurica* were investigated for their inhibitory effect on viral reverse transcriptase as the control enzyme and bacterial replicative DNA polymerases (polC from *Bacillus subtilis* and DnaE2 from *Escherichia coli* as the control enzymes). Active molecules were isolated from the roots of *P. daurica* and their structures were elucidated by NMR, MS/MS, and FT-IR analyses.

## 2. Material and Methods

### 2.1. Extraction and isolation

The fresh plant samples were stored at -20 °C until extraction. Root parts of the plant was pulverized separately under liquid nitrogen using a mortar and pestle. For enzyme inhibition 10 g of pulverized root of *P. daurica* were extracted with 50 mL of methanol at 37 °C for 2.5 h in a thermo-shaker and 25 °C for 1 h in an ultrasonic bath, and then centrifuged at 4000 rpm for 20 min at 4 °C. Supernatants were transferred into a new Falcon tube and evaporated. Dried extracts of the plant parts were dissolved with methanol for direct analysis.

For isolation of the main compounds from the root, 300 g of the powdered root was extracted twice using 500 mL of methanol. Extracts were combined and evaporated at 30 °C by rotary evaporator. Seven grams of obtained methanol extract was subjected to Sefadex LH-20 column using methanol as elution solvent. Twelve fractions were collected.

Fractions (Fr.) 5–10 were evaporated (3.9 g). This fraction was subjected to VLC column (LiChroprep RP-18, 25–40  $\mu\text{m}$ , Merck 9303) using by elution solution of water: methanol mixtures (100:0→0:100). One-hundred sixteen fractions were collected. Fr. 26–30 were evaporated and 24.9 mg 2,4,6-trihydroxy- 1-methyl benzoate (PD-1) was obtained. Fr. 44–46 were evaporated and 55 mg paeoniflorin (PD-2) was obtained. Fr. 56–58 were evaporated and 45 mg benzoic acid (PD-3) was obtained. Fr. 90–92 were evaporated and 45.1 mg paeoniflorigenone (PD-4) was obtained. Fr. 93–95 were evaporated and 8.9 mg benzoylpaeoniflorin (PD-5) was obtained. Stock solutions were prepared from the purified molecules (7mM concentration) and used in experiments at 1 mM final concentration approximately.

## 2.2. Preparation of polymerases and DNA templates

Replicative DNA polymerases, PolC and DnaE2, were prepared as described in Turumtay et al. (2017). MMLVRT was purchased from New England (BioLabs, M0253S). 32-mer DNA and RNA template and 14-mer primer labeled by Cy5 were synthesized by Macrogen (Netherlands, Amsterdam) and Iontek (Turkey, Istanbul). The synthetic DNA substrates (Table 1) were prepared as in previous work described by Sandalli et al. (2009) and used in primer extension assays by single or multiple nucleotide addition.

## 2.3. Inhibition effect on DNA polymerases

Inhibition of DNA polymerase activity in the presence and absence of purified molecules was investigated by primer extension assay (single nucleotide or multiple nucleotide addition). The solvent of the extracts/compounds (methanol) was used as a control. The reactions were performed in 20  $\mu\text{L}$  final volume by mixing two reaction mixtures. The mixture 1 was prepared in 13.5  $\mu\text{L}$  final volume with appropriate polymerase (25 nM for bacterial replicative DNA polymerases and 2 units for MMLV-RT), 1 $\times$  polymerase activity buffers (harboring 10 and 3 mM of  $\text{MgCl}_2$  for replicative bacterial polymerases (Turumtay et al. 2017) and MMLV-RT, respectively), and 3  $\mu\text{L}$  of each extract or purified molecules. The mixture 2 was prepared in 6.5  $\mu\text{L}$  final volume with 100 ng synthetic 32/14-mer DNA/DNA substrate (for PolC, DnaE2 and DNA-dependent DNA polymerase activity of MMLV-RT) or 32/14-mer RNA/DNA substrate (RNA-dependent DNA polymerase activity of MMLV-RT), and 100  $\mu\text{M}$  of dNTP (both for multiple nucleotide and single nucleotide (dATP) incorporation). The mixture 1 was kept 5 min at room temperature and then the mixture 2 was added and the reactions were carried out for 10 min at 37  $^\circ\text{C}$ . The reactions were terminated by addition of 20  $\mu\text{L}$  of 2  $\times$  stop solution (20mM EDTA, 0.2% (w/v) SDS, 80% (v/v) formamide and 0.008% (w/v) each of bromophenol blue and xylene cyanol) and heated at 95  $^\circ\text{C}$  for 5 min. Six microliters of reaction mixture was loaded onto a 16% polyacrylamide-8M urea gel and the products were separated at 1500 V for 3 h. The resulting gels were visualized by Typhoon FLA9500 biomolecular imager (Amersham Biosciences).

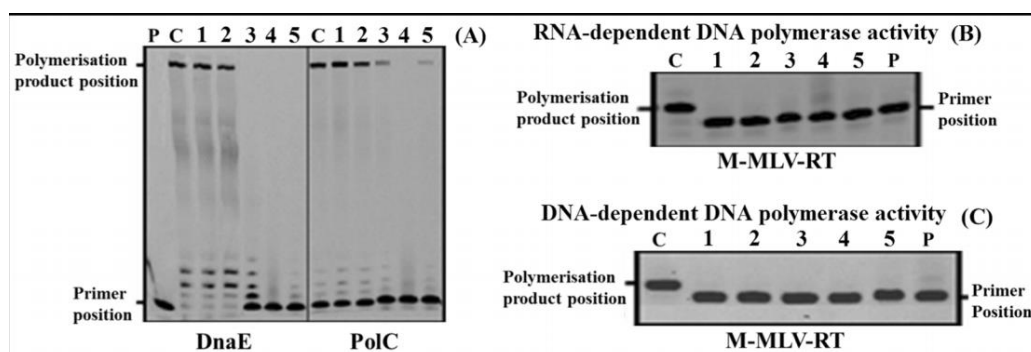
## 3. Results and discussions

### 3.1. Identification of the five isolated compounds from methanolic extract

Five molecules with three monoterpene glycosides, paeoniflorin (PD-2), paeoniflorigenone (PD-4), benzoyl paeoniflorin (PD-5), and benzoic acid (PD-3) with its derivate 2,4,6-trihydroxy-1-methyl benzoate (PD-1) were purified and identified. Isolated compounds were identified using NMR, FT-IR, and ESI-MS/MS spectra.

### 3.2. Inhibition of isolated compounds on DNA polymerases

The method based on the extension of 5'-end fluorescent labeled-primer by DNA polymerases (Primer Extension Assay, PEA) and the imaging of the result by Typhoon FLA9500 offers a strong approach to investigate inhibitory effect of isolated molecules. The inhibitory effect of the isolated molecules on DNA polymerase activity of polC and DnaE was investigated by multiple nucleotide addition assays.



**Fig. 2.** (A) Primer extension assay with multiple nucleotides (four dNTPs) to show the inhibition of polymerization on DnaE and PolC. (B) and (C) Primer extension assay with single nucleotide (just dATP) to show the inhibition on both RNA-dependent DNA and DNA-dependent DNA polymerase activity of MMLV-RT, respectively. P:primer, C:control, 1:2,4,6-trihydroxy-1-methyl benzoate, 2:paeoniflorin, 3:benzoic acid, 4:paeoniflorigenone, 5:benzoylpaeoniflorin.

Methanol did not inhibit the polymerization activity of both polymerases and enzymes elongated the 14-mer primer to the 32-mer position (Fig. 2.A). Paeoniflorigenone (PD-4) strongly inhibited the polymerase activity of both enzymes, which the enzymes could not add a single nucleotide to the primer terminus (Fig. 2.A). Benzoylpaeoniflorin (PD-5) had also showed inhibitory effect on both polymerases and but it was relatively more effective on DnaE than PolC (Fig. 2.A). Benzoic acid (PD-3) had a relatively low inhibitory effect on PolC. DnaE could not complete the polymerization and it could not elongate the 14-mer primer into the 32-mer product position in the presence of PD-3. It was concluded that the polC was less inhibited than DnaE by PD-3 and polC was completed the polymerization and elongated the 14-mer primer into the 32-mer product position in the presence of this molecule (Fig. 2.A). Paeoniflorin (PD-2) and 2,4,6-Trihydroxy-1-methyl benzoate (PD-1) were found to be ineffective against both polC and DnaE (Fig. 2.A). A strong inhibitory effect was observed on MMLV-RT by all purified molecules (Fig 2.B-C). That means the roots of *P. daurica* may have many chemicals as inhibitors of the polymerization activity of viral reverse transcriptase.

#### 4. Conclusion

Paeoniflorigenon (PD-4) was found the main active compound since it strongly inhibited all polymerases which means paeoniflorigenon (PD-4) affects the common part of the polymerases. Either benzoyl group or monoterpene skeleton of these compounds or both should have played effective role in the mechanism of DNA polymerase inhibition. These molecules inhibiting DNA polymerases from may have potent to control the increasing resistance rate in bacterial or viral diseases.

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## **OP10- Optimization studies of a disposable immunosensor for sensitive detection of creatine kinase**

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Creatine kinase(CK) is an enzyme that is found in high amounts in the heart and skeletal muscle, while in the brain tissue [1]. High values of CK is usually seen in cardiovascular system diseases. Its level is used as a biomarker to determine heart muscle and skeletal muscle damage [2]. In this study, an ITO (indium tin oxide) based biosensor is configured to detect Creatine kinase. The ITO-PET electrodes were modified with  $\text{NH}_4\text{OH}/\text{H}_2\text{O}_2/\text{H}_2\text{O}$  to form the hydroxylated electrode surface. Then, the ITO-PET electrode surfaces were modified with 3-Aminopropyltriethoxysilane(3-APTES). Then, cyanogen bromide was used to activate the amino ends of 3-APTES. The 3-APTES concentration, cyanogen bromide concentration and incubation time, anti-CK concentration and incubation time, CK incubation time were optimized. Electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV) were used for all characterization experiments of the electrode surfaces and also used for plotting the CK calibration graphs. An extremely wide linear range for CK was determined as 1 fg/mL - 1000 fg/mL.

**Keywords:** ITO-PET, Creatine kinase, 3-APTES, Biosensor, Electrochemical Impedance Spectroscopy

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## **OP11- Development of Protein Extraction Procedure For Proteomic Analysis of Tomato Seed**

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Nowadays, there is an increasing interest in major compounds such as carbohydrates, proteins, and lipids obtained from fruit and vegetable wastes. Novel fruit and vegetable by-product proteins can be an alternative for animal-derived proteins, due to higher production efficiency, reduced life cycle environmental impact and possibility to meet consumers' dietary or cultural preferences<sup>1</sup>. Although having a versatile functionality (emulsifying, foaming, gelling, texturizing agents), the application of proteins is limited. Tomato, the member of family *Solanaceae*, is one of the most popular garden crops of much interest in the world. Tomato processing generates large quantities of solid wastes<sup>2</sup>. These wastes are contained lots of seeds, fibrous, skin, which contains high-quality nutrients but could be extensively used as fertilizer or feed. In this study, the protein extraction of tomato seed was studied to find optimal conditions for protein extraction and isolation. A central composite design including water/solids, ultrasonication duration, and extraction duration was used. Protein yield was used as a response. Based on results obtained under the conditions tested, the new tomato seed protein extraction procedure was formed. Additionally, the suggested protein extraction procedure may be used as a starting point for proteomic studies of other related plant species.

**Keywords:** Tomato seed, protein, response surface methodology

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## OP12- Carbon ceramic electrodes for electroanalytical applications – possibilities and limitations

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Ceramic carbon electrodes (CCEs) were first introduced by Lev and his co-workers<sup>1</sup> in 1994, and they have remained of interest to many scientists ever since. However, CCEs are not as widely used in electroanalysis as glassy carbon (GCE) or carbon paste electrodes (CPE). Similarly to CPEs, the preparation of CCEs is very easy and cheap. This kind of ceramic electrodes is produced using the sol-gel technology which is based on hydrolysis and condensation processes. Next, into the inert silicone matrix obtained in the sol-gel process, a conductive filler (typically the graphite powder) is incorporated. Such preparation procedure gives the CCEs unique features in comparison with other carbon-based electrodes. The combination of a large surface area of the sol-gel ceramic matrix and possibility of electrodes' modification in the entire volume using other materials enables the construction of a surface-renewable electrode. Such renewability of the surface is typical for carbon paste electrodes (CPEs), but CCEs are more rigid, porous, and have higher stability than CPEs.

This talk will discuss some possibilities and limitations of carbon ceramic electrodes in terms of their application in electroanalytical procedures. Some results of our recent studies concerning preparation of CCEs via sol-gel method and their modification in entire volume will be presented. In our studies we have used different types of carbon materials – not only graphite powder, but also carbon nanotubes, carbon black, as well as reduced graphene oxide nanoparticles. We have also examined electrodes modified with ferrierite and bismuth oxide. In every case, surface analysis was performed using atomic force microscopy or scanning electron microscopy. The preliminary electrochemical characterization of the produced electrodes was carried out in the  $\text{Fe}^{2+}/\text{Fe}^{3+}$  redox system using cyclic voltammetry and electrochemical impedance spectroscopy. In the next step, after optimization procedure, the electrodes were applied in electroanalytical determination of the chosen biologically active compounds via voltammetric techniques. The influence of the applied modifier on electroanalytical capability of the ceramic electrodes was determined on the basis of validation parameters such as sensitivity, linear dynamic range (LDR), limit of detection (LOD) and limit of quantification (LOQ). Moreover, the developed analytical procedures were used to determine those bioactive compounds in the real samples.

Summarizing, for each modification we have stated improved sensitivity, lower LOD and LOQ values, good stability over time, high repeatability and reproducibility of measurements, and most importantly, each electrode has an easily renewable surface. Nevertheless, we have to state that ceramic carbon electrodes are relatively demanding in use. During the research we encountered several problems, that will be pointed out during the talk.

Keywords: carbon ceramic electrode, sol-gel process, electroanalysis, voltammetry, biologically active compound,

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**OP13- An application of a glassy carbon electrode modified with multi-walled carbon nanotubes for electrochemical determination of pesticides**

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Electrochemical techniques (mainly voltamperometric techniques) are increasingly used for the determination of pesticides due to the fast, simplicity and low cost of analysis as well as the principles of green analytical chemistry. A Glassy carbon is one of the most commonly used electrode material in electroanalytical techniques. To improve the analytical parameters of the glassy carbon, it is often subjected to various surface modifications. A carbon nanotubes are often used for modifications.

The aim of the presented work was to develop electrochemical procedures for the quantitative determination of selected pesticides (*Propham* and *Oxycarboxin*) at a glassy carbon electrode modified with multi-walled carbon nanotubes (GCE/MWCNTs) based on square wave adsorptive stripping voltammetry (SWAdSV). The effect of an amplitude ( $E_{sw}$ ), a frequency ( $f$ ), a step potential ( $\Delta E_s$ ), an accumulation potential ( $E_{acc}$ ) and time ( $t_{acc}$ ), and equilibration time ( $t_{eq}$ ) was investigated. Additionally the surface characterization of GCE/MWCNTs was evaluated with an atomic force microscopy (AFM) and a scanning electron microscopy (SEM).

The best analytical signal was obtained in the medium of sulfuric acid for both pesticides. The proposed electroanalytical procedure allows the determination of *Propham* in the concentration range from  $2.0 \times 10^{-6}$  –  $4.8 \times 10^{-5}$  mol L<sup>-1</sup> for SWAdSV (LOD =  $3.6 \times 10^{-7}$  mol L<sup>-1</sup>, LOQ =  $1.1 \times 10^{-6}$  mol L<sup>-1</sup>) and *Oxycarboxin* in the concentration range from  $6.0 \times 10^{-6}$  –  $9.0 \times 10^{-5}$  mol L<sup>-1</sup> (LOD =  $1.1 \times 10^{-6}$  mol L<sup>-1</sup>, LOQ =  $3.8 \times 10^{-6}$  mol L<sup>-1</sup>). The obtained results from cyclic voltammetric method (CV) showed that the electrode mechanism is an irreversible process controlled by the diffusion-adsorption mixed process adsorption at GCE/MWCNTs for both *Propham* and *Oxycarboxin*.

The use of a glassy carbon electrode modified with multi-walled carbon nanotubes enhanced the analytical signals and provided the possibility to develop a novel and alternative electroanalytical procedures for *Propham* and *Oxycarboxin* determination. The advantages of these procedures, such as its simplicity, cheapness, and rapidity, were confirmed by successful application for the determination of *Propham* and *Oxycarboxin* in spiked river water samples via the standard addition method without samples pretreatment.

**Keywords**

square wave adsorptive stripping voltammetry, glassy carbon, multi-walled carbon nanotubes propham, oxycarboxin,

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## OP14- Current Analytical Solutions for Biomarker Detection in the Pre-Diagnosis of Lung Cancer

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Lung cancer is one of the cancer types that cause the most deaths worldwide, and the diagnosis can only be made in the later stages. This not only reduces the chances of treating the disease, but also increases the cost. Expensive imaging techniques applied for early diagnosis in patients at risk of lung cancer are also unable to provide reliable results, and these findings need to be confirmed by disruptive biopsy and histopathological examinations. The search for non-invasive techniques that can be used at the bedside, which may be an alternative to these techniques, is still continues. The search for cancer biomarkers has become an integral component of cancer research because of the potential of biomarkers to enable early detection of diseases and to ensure prognostic, diagnostic and predictive information. There is an increasing number of studies on the early diagnosis of lung cancer, with significant changes in the levels of certain chemical groups in clinical samples and some of these substances can be used as biomarkers<sup>1</sup>.

In the past twelve years, as biological matrix, protein groups in blood, sweat, saliva and urine, tumor antigens, peptides, metabolic products, DNA and RNA and expression of certain genes are monitored as biomarkers. In these studies, when compared with healthy cases, changes in the profile of some compounds were observed in the samples of patients with cancer, it was observed that the concentration of some species increased significantly and some of them decreased. However, the most important problem for the clinical use of these assays is the use of preconcentration and pre-separation methods that can lead to desired sensitivity, support the diagnosis in the biological environment and provide the reproducibility. The most widely used detection technique for the determination of biomarkers is mass spectrometry combined with gas and/or liquid chromatography. The sensitivity of these techniques were improved using advanced (MS/MS, QTOF, MALDI TOF, TIMS-TOF) detectors<sup>2</sup>. In this study, the limitations of biomarker methodology, the future perspectives and the trends for biomarker analysis in clinical samples for lung cancer diagnosis are reviewed.

Present study describes a thin film microextraction methodology for biomarker analysis in urine samples. In this study, adsorption of biomarker metabolite species of different chemical structure was carried out on different polarity thin film coated bars integrated with 96 well plate (96WP) system. After desorption in a small volume of suitable solvent in 96 WP, LC-MS/MS system was used to analyze the best response media for the target analytes for that matrix medium was determined. Response surface analysis method for 12 coatings which yielded the best yield was obtained. The type of coating, desorption solution type and pH effect were also optimized. Optimized method with featured coatings was applied for determination of metabolites in urine specimens of cancerous and asthmatic subjects.

**Keywords** Chromatography, Sample Preparation Methods, Biomarker, Lung Cancer

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**OP15- A novel chemometric strategy for the estimation of sesame  
(*Sesamum indicum*) oil adulteration with edible oils**

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Adulteration of food products by the substitution of premium quality ingredients with lower grade and cheaper substitutes is a type of economic fraud that can pose a major health threat to consumers. A useful procedure for the qualitative and quantitative determination of vegetable oils (colza, soy, and sunflower) as adulterants in samples of sesame (*Sesamum indicum*) oil has been developed. Partial least squares (PLS) was employed for the analysis of Fourier transform infrared spectroscopy (FTIR) spectral data of the blend oil samples. Thirty three samples of each adulterant with sesame oil were prepared with different concentration (from 0,5% to 30%). A PLS model, using orthogonal signal correction and mean centering data pretreatments, and VIP scores variable preselecting, was able to predict the concentration of sunflower and soy and colza oil adulterants in the 1–30 % weight ratio range with relative prediction errors lower than 2 % (w/w), for external validation samples. The method developed was very suitable for the determination of modeled adulterants but it may also reveal an adulteration even if it does not derive from the adulterant employed in this study.

**Keyword:** Adulteration, Sesame oil, adulteration, infrared spectroscopy, edible oil

## OP16- Impedimetric Analysis of Sarcosine by Using Aptamer Modified Gold-Coated ITO Electrode

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Aptamers are ssDNA or RNA-based recognition elements. The advantages of aptamers such as low cost, easy production, thermal stability, and manipulation capacity according to the buffer conditions have dramatically increased their usage as recognition element in biosensor studies in recent years<sup>1</sup>.

In this study, an aptamer-based impedimetric biosensor was developed using gold-modified ITO electrode against sarcosine, a potential marker of prostate cancer<sup>2</sup>. In the developed biosensor, the aptamer sequences using different linkers from different regions were immobilized independently of the gold-coated ITO electrode. Differences in impedance responses against varying concentrations of sarcosine were analysed.

The aptamer-based impedimetric biosensor was used to measure sarcosine in nM sensitivity. Saturation curve for sarcosine was obtained and K<sub>d</sub> value was calculated and compared with previous methods.

In conclusion, aptamer modified gold-coated ITO electrode-based biosensor platform was used for rapid and specific determination of sarcosine. It is anticipated that the biosensor system will provide a powerful alternative for sarcosine analysis with its low cost and high sensitivity.

**Keywords:** Aptamer, EIS, ITO electrode, Aptasensors, Sarcosine

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## **OP17- Electrochemical biosensor-based diagnostic kit designs and their applications in DNA analyses**

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In line with the developing technologies, biosensors have been widely used in the analysis of fields such as medicine, environment, food, etc. in the last 20 years. In this context, electrochemical DNA biosensors are basically; based on the rapid analysis of hybridization between the synthetic, single-stranded capture DNA probe immobilized on the transducer surface and the target DNA sequence related to a genetic disease, a gene part of a microorganism, etc. In other words, DNA biosensors are designed as an alternative to genotyping to identify specific sequences of DNA. Micrometer-sized devices (microchips, etc.) capable of multiple DNA measurements for gene sequencing are examples of single DNA biosensor designs developed in laboratories, as shown in scientific studies. However, prior to conversion from the biosensor to the instrument, the development of the analytical method to be used in the instrument is the most important issue. Because the method should be accurate, reproducible, fast and reliable with only a single measurement. Although they have low cost and low detection limit, the pre-preparation step required before analysis with electrochemical DNA biosensors is an important problem which leads to prolongation of the assay time. Additionally, in order to integrate advanced DNA (nano) biosensors into a bedside analyzer (POC) device, it is necessary to reduce sample and reagent volumes and design stable biosensor surfaces ready for analysis at any time.

For these purposes, new generation kit-type electrochemical DNA biosensors (diagnostic kit) were developed in our laboratory to choose the right biosensor material and reduce total design costs for the application. Here, we review exemplary approaches, including the design of our two different diagnostic kits based on a disposable carbon graphite electrode (CGE) and the other one based on magnetic beads (MBs) and screen printed carbon electrodes (SPEs) which can reach picomole level of detection limits. These ready-to-use electrochemical kits can detect target DNA sequences even after about 6 months of kit preparation.

**Keywords:** Electrochemical DNA biosensor, diagnosis kit, carbon graphite electrode, screen printed electrode, magnetic beads.

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## OP18- MIPs for Strep-Tagged Hydrogenase and Human Sulfite Oxidase

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Over the past decades increasing attention has been paid to substitute biochemical reagents in bioanalysis and separation techniques by fully synthetic organic polymers like molecularly imprinted polymers (MIPs) or nucleotide-based aptamers. Molecular imprinting is a method, pioneered by Wulff and Mosbach, to synthesize artificial binders using functional monomers with or without cross-linkers in the presence of target analyte (template). Subsequent removal of the template leads to the formation of binding cavities with a molecular memory mirroring shape and/or functionality of the template [1,2]. MIPs for low molecular-weight substances have been successfully prepared whereas it is still challenging for biomacromolecules like proteins [3]. Here, we present two different approaches for protein imprinting.

(i) In the first approach, so-called epitope imprinting, Strep-Tag II was used as a template. It is an synthetic eight amino acid peptide that can be fused to recombinant proteins. For the MIP synthesis the Cys-extended Strep-Tag II-peptide was chemisorbed on the gold electrode prior to electropolymerization of the functional monomer scopoletin. Binding and electrochemical removal of the peptide were analyzed by means of electrochemistry using ferricyanide as a redox marker. In addition, Surface-Enhanced Infrared Absorption (SEIRA) spectroscopy and atomic force microscopy were applied for characterizing the different preparation steps. The electrochemical response was linearly dependent on the concentration of the tag up to 6 nM and reached saturation at 10 nM. Moreover, the Strep-tagged membrane-bound hydrogenase (MBH) of *Ralstonia eutropha* was bound to the peptide-imprinted layer and exhibited enzymatic activity in presence of hydrogen.  $K_D$  values for Strep-Tag II and Strep-tagged MBH to the Strep-Tag II-MIP were calculated to be 3.05 and 33.08 nM respectively, by fitting the binding isotherm to the Langmuir model.

(ii) The second approach is based on holoprotein imprinting. It is the most straightforward approach for the MIP preparation especially in the case of highly active enzymes. MIPs for the molybdenum containing human sulfite oxidase (hSO) were prepared by electropolymerization of *o*-phenylenediamine in the presence of hSO. The template was removed from the polymer by the application of cyclic voltammetry. Preliminary results demonstrate that all steps of MIP preparation were successful. Furthermore, hSO rebinding to the MIP resulted in catalytic activity towards its substrate sulfite in the presence of a mediator, N,N,N',N'-tetramethyl-*p*-phenylenediamine (TMPD).

**Keywords:** Molecularly Imprinted Polymers, Epitope Approach, Holoprotein Imprinting, Membrane Bound Hydrogenase, Human Sulfite Oxidase

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**OP19- Preparation and characterization of carboxyl functionalized core-shell Fe<sub>3</sub>O<sub>4</sub>/SiO<sub>2</sub> for L-asparaginase immobilization**

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**ABSTRACT:** Magnetic nanoparticles are well known as a facile and effective support for enzyme immobilization since they have high surface area, large surface-to-volume ratio, easy separation, a fast and high enzyme loading<sup>1</sup>. In this study, modified particles are more effective for L-asparaginase (L-ASNase) immobilization compare to free enzyme. Therefore, carboxyl functionalized (Fe<sub>3</sub>O<sub>4</sub>/SiO<sub>2</sub>/COOH) particles were prepared. The functional groups, crystalline structure, magnetic properties, morphology, chemical composition of the prepared modified nanoparticles were examined via Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), vibrating-sample magnetometer (VSM), scanning electron microscopy (SEM), and energy dispersive X-ray spectroscopy (EDAX). Under the optimum conditions, the immobilized enzymes were more stable within certain range of temperature and pH value in comparison to free enzyme. On the other hand, the immobilized enzymes showed improved stability after incubation for 3 h at 50 °C. The free enzyme maintained only 30% of its initial activity for 4 weeks at 4 °C, while Fe<sub>3</sub>O<sub>4</sub>/SiO<sub>2</sub>/COOH/ASNase retained more than 56.5% of initial activities under the same conditions, respectively. Moreover, Fe<sub>3</sub>O<sub>4</sub>/SiO<sub>2</sub>/COOH/ASNase (%57.4) displayed excellent operational stability after 17 repeated cycles. These findings suggested that the Fe<sub>3</sub>O<sub>4</sub>/SiO<sub>2</sub>/COOH may be utilized as efficient and sustainable supports to developed immobilized ASNase in several biotechnological applications.

**Keywords:** magnetic nanoparticles, silica coating, L-asparaginase, enzyme immobilization, enhanced stability.

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## OP20- The Comparative Investigation of Cytotoxic Properties for Synthetic Commercial Tissue Adhesives

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Tissue adhesives, which have recently become popular in medicinal, cosmetics and emergency medicine fields, are widely used today. In general, tissue adhesives are classified under two main titles as fibrin, gelatin, albumin, polysaccharide (chitosan, dextran, chondroitin sulfate), mussel protein based biological and cyanoacrylate, poly(ethylene glycol), polyester, polyurethane based synthetic tissue adhesives. Commercial tissue adhesives have advantages such as ease of application with local medicines, no need for removal, quick and easy applicability, less pain, and providing desired results for cosmetics. Mechanical tissue joining systems not only slow down the healing process of the tissue, whose cellular and anatomic integrity is impaired due to their physical and chemical properties, but also cause vital disadvantages such as leading tissue fluid leaks.<sup>1,2</sup> Tissue adhesives have therefore been of great interest in recent years as a supportive and/or alternative to suture systems for joining tissues in surgical procedures.

In this study, the cytotoxic properties of five synthetic commercial tissue adhesive materials based on different chemical bases which are widely used today were investigated *in vitro*. The contents of commercial tissue adhesives researched in our study are as follows: **L** having 2-octyl and n-butyl cyanoacrylate content, **D** having a mixed product form with calcium hydroxide content, **V** having N-butyl-2-cyanoacrylate content, **C** having polymethyl methacrylate content, and **S** having polyurethane acrylate content. Firstly, the monomers specifically found in different tissue adhesives were polymerized under the appropriate conditions in the user manuals. Then, TGA, DTA, DSC and FTIR characterizations of these adhesives were performed. The cytotoxic effects of synthetic tissue adhesives, which provide great benefits to patients during healing process, were examined by MTT test using L929 (healthy mouse fibroblast), HepG2 (liver cancer) and CaCo2 (colon cancer) cell lines and IC<sub>50</sub> values of each tissue adhesive were determined.

When the FTIR spectrum of **D** is examined, C-H stretching vibrations are asymmetrically seen at 2870, 2930 and 2980 cm<sup>-1</sup> due to CH<sub>2</sub> units in the organic monomeric structure. From the analysis of FTIR spectrum related to **V**, it is obviously seen that C=C monomeric units in the main chain structure are converted to C-C bonds during the polymer formation. The disappearance of these peaks into C-C units was understood as the absence of peaks around ~1650 cm<sup>-1</sup>. **C** commercial tissue adhesive is characterized by two different methyl units in its structure. C-H stretching vibrations of these methyl units are seen as asymmetric stretching vibrations at 2800 cm<sup>-1</sup> and markedly at 638 cm<sup>-1</sup>. When the FTIR spectrum of **S** is analyzed, it is seen that the peaks prove the classical polyurethane acrylate structure and the polymerization is completed. Moreover, IC<sub>50</sub> values for **D** against CaCo2 and L929 cells are 450.76 µg/mL and 545.09 µg/mL, respectively.

Consequently, the determination of cell death rates in terms of cytotoxicity during the biodegradation processes will be a vital guide for practitioners as well as patients to investigate the comparative results of five different synthetic commercial tissue adhesives.

**Keywords:** Synthetic commercial tissue adhesive, cytotoxicity, MTT, IC<sub>50</sub>.

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## OP21- Preparation and Optimization of Alginate Microbeads Loaded with Vincristine Sulfate and Its In vitro Release

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Vincristine sulfate (VCR) is a dimeric indole alkaloid isolated from the periwinkle plant *Catharanthus roseus*<sup>1</sup>. VCR, an antimitotic, was used as an antimitotic chemotherapeutic agent to treat different cancers, including malignant lymphoma, acute leukemia and breast cancer<sup>2</sup>. However, rapid release of the drugs is still a challenge for anti-tumor treatment. Alginate microbeads are very suitable carriers to delivery VCR (a positively charged and water-soluble drug formulation) and for prevent drug leakage.

The aim of the present study was to prepare and characterize alginate microbeads as carrier for delivery of VCR. The structures, thermal stabilities and surface morphologies of alginate microbeads and the VCR-loaded alginate microbeads were characterized by using various methods including FTIR, TGA-DTA-DSC, SEM, EDX. Additionally, drug loading efficiency and *in vitro* release kinetic of VCR was examined in detailed. The VCR loading efficiency of was found more than 50%. *In vitro* release behavior was performed in PBS buffer (pH 7.4).

As a result, the *in vitro* release results indicated that alginate microbeads with slow release behavior could be a promising drug carrier for therapeutic drugs such as VCR.

**Keywords:** Alginate; Encapsulation; Vincristine sulfate; *In vitro* drug release

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## **OP22- Voltametric and potentiometric applications of antidiabetic drug Metformin- Selective sensor**

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Type 2 diabetes is a long term metabolic disease in which the body gains resistance to the effects of insulin, which is a hormone that regulates the absorption of sugar<sup>1</sup>. Metformin is an oral antihyperglycemic agent used to treat non-insulin-dependent (type 2 diabetes) diabetes. Metformin hydrochloride (MET-HCl) is a white crystal powder, which is used as a hypoglycemic drug, it is hygroscopic and freely soluble in water<sup>2</sup>. Metformin-tetraphenylborate (MET-TPB) ion-pair was used as an ionophore in the structure of the sensor membrane. It was determined that the sensor membrane at the ratio of 69% o-nitrophenyl octyl ether, 27% polyvinyl chloride and 4% MET-TPB performed the best potentiometric performance. The voltammetric performances of the sensor were also investigated. The prepared microsensor was successfully utilized for the determination of Metformin in a pharmaceutical drug sample by potentiometry and voltammetry.

**Keywords:** Potentiometry, voltammetry, antidiabetic.

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## OP23- Flow Injection Amperometric Detection of Formaldehyde at Bimetal Nanoparticles Modified Pencil Graphite Electrodes

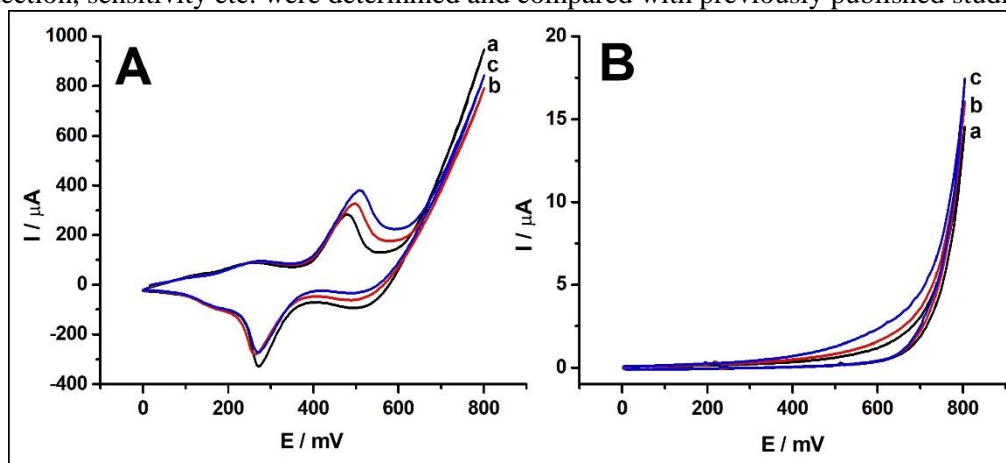
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Formaldehyde (FA) is a carcinogenic compound against human health and it can include in the formation of ground level of ozone, which can damage crops and causes environmental contamination<sup>1</sup>. Moreover, its electro-oxidation has been used in the chemical, wood processing, paper, and textile industries etc<sup>1-2</sup>. Therefore, electrochemical oxidation and determination of FA have received great interest by using metal/bimetal nanoparticle modified electrodes.<sup>1-2</sup> In this study, various monometallic (Cu, Ni, Co, Pt and Pd) and bimetallic nanoparticles (Ni-Co, Cu-Ni, Pt-Pd and Co-Cu NPs) modified graphite pencil electrodes (GPE) were prepared for electrocatalytic oxidation of FA. Cyclic voltammograms (CVs) recorded in the presence of FA show that bimetallic NPs modified PGEs (especially Ni-Co/PGE) exhibit higher electrocatalytic activity towards oxidation of FA than monometallic NPs modified PGEs. In the CVs of FA, oxidation peak, which was proportionally increased by FA concentration, was seen at about 530 mV at Ni-Co/PGE (Fig. 1A) while any oxidation peak was not observed at non-modified PGE (Fig. 1B). These results indicate that the Ni-Co/PGE exhibits a good electrocatalytic activity toward oxidation of FA. In the final step, Flow Injection Electroanalysis of FA at Ni-Co/PGE was performed under optimized conditions. Analytical figures of merit such as linearity range, limit of detection, sensitivity etc. were determined and compared with previously published studies.



**Fig. 1.** Cyclic voltammograms of Ni-Co/PGE (A) and bare PGE (B) in 0.10 M NaOH + 0.10 M KCl (a) and in the presence of 1 (b) and 2 mM (c) FA (scan rate: 25 mV/s)

**Keywords:** Formaldehyde, Bimetal Nanoparticles, Pencil Graphite Electrode, Flow Injection Analysis

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**OP24- Sensitive Differential Pulse Voltammetric Determination of an Antimalarial Drug: Amodiaquine at a Disposable Poly-Calcein Modified Pencil Graphite Electrode**

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Malaria is a serious disease and it infects and transmits from human to human by biting from infected Anopheles female Anopheles mosquitoes<sup>1</sup>. The disease has become a global health crisis and between 400 and 900 million cases of malaria are told to be causing 1 to 3 million deaths every year<sup>1,2</sup>. In this work, the determination of an antimalarial drug, amodiaquine (ADQ) used in treatment of malaria disease was described at a disposable poly(calcein) modified pencil graphite electrode (poly-CCN-PGE) by cyclic voltammetry (CV) and differential pulse voltammetry (DPV).

For the fabrication the ADQ sensor, calcein were electro-polymerized on PGE by recording of 6 successive cyclic voltammograms of 0.50 mM CCN in 0.10 M HClO<sub>4</sub> solution and dried at the same temperature before use. The electrochemical characterizations of obtained electrode and bare PGE were performed by recording electrochemical impedance spectra. SEM and EDX measurements were performed for the investigation of surface morphologies of the surface of the PGE and poly-CCN-PGE. Then, electrochemical responses at both bare and modified electrode were examined towards increased concentrations of ADQ in pH 3.0 Britton Robinson buffer solution (BRBS). Obtained voltammograms indicated that electrochemical response of the ADQ at poly-CCN-PGE was found to be much higher than found at PGE. In order to determine the significant analytical parameters (Linear response range (LRR), detection limit (DL) and sensitivity), the response of the increased concentrations of ADQ at poly-CCN-PGE were performed by recording the differential pulse voltammograms under optimized conditions ( $E_{\text{step}}$ : 10 mV, pulse amplitude: 250 mV and pulse time: 10 ms). LRR, DL and sensitivity were determined as 0.50-25  $\mu\text{M}$ , 0.094  $\mu\text{M}$  and 20.566  $\mu\text{A}/\mu\text{M}$ , respectively. Selectivity of the proposed sensor was tested in presence of various ionic and molecular interferences. The recovery studies were performed in various matrixes each includes ADQ and the obtained high recovery values suggested that proposed sensor can be determine ADQ with high accuracy in different sample matrixes.

**Keywords:** Amodiaquine, Calcein, Pencil Graphite Electrode, Differential Pulse Voltammetry, Electrochemical Sensor.

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## OP25- PVC Membrane Potentiometric Ion Selective Electrodes Based Ion-Pair in Pharmaceutical Analysis

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In this study, selective solid-state-contact PVC membrane potentiometric electrode was developed for imipramine-hydrochloride (IMIP-HCl), maprotiline hydrochloride (MAP-HCl) and Isoprenaline hydrochloride (IZO-HCl), which are drug active substances .Electroanalytical methods are preferred because of the short analysis time, low determination limits and more economic compared to the existing analytical methods. The superiority of these methods over other analytical methods is; small amounts of materials are sufficient for analysis, they are cheap, they are easy to apply, analytes do not need much pre-purification, they do not require much solvent and they are sensitive methods (Zuman ve Brezina, 1962; Kissinger ve Heineman, 1996). In this method , ion pairs with tetrafenylborate (TFB), reynekat (REY), phosphomolybdate (FM), tungstosalicylic acid (TSA) and phosphotungstic acid (FTA) of imipramine, maprotiline and isoprenaline drug active substances were synthesized and used as an electroactive substance in the membrane composition of the electrode. Imipramine-selective electrode, maprotiline-selective electrode and isoprenaline-selective electrode with different membrane compositions were produced. Potentiometric performance characteristics of the electrodes were investigated with static medium measurement. The membrane composition showing the best potentiometric performance for imipramine hydrochloride, maprotiline hydrochloride and isoprenaline hydrochloride are 3% IMIP –TPB, 32% PVC, 64% DBP, 1% KTPCIPB (Potassium Tetrakis (4-chlorophenyl) borate); 3% MAP-TPB ion pair, 65.0% DBP, 32.0% PVC; ISO-TPB ion pair, 64.0% NPOE, 32.0% PVC and 1.0% KTPCIPB. For imipramine, the linear working range is  $1 \times 10^{-5}$ - $5 \times 10^{-2}$  M; the slope of the electrode was found to be 45.1 mV ( $R^2 = 0.98$ ), the limit of detection was  $1.0 \times 10^{-5}$  mol / L, and the response time was less than 5 s. For Maprotiline, the linear working range of the electrode is  $1.0 \times 10^{-5}$ - $1.0 \times 10^{-2}$  M, the slope 55.4 mV at a 10-fold concentration change; determination limit was determined as  $5.0 \times 10^{-6}$  M, pH working range 3–5, and response time 5s. For isoprenaline, the slope of the linear working range is  $5.0 \times 10^{-6}$  M -  $1.0 \times 10^{-1}$  M and the slope 45.3 mV at 10 times concentration change; determination limit of  $5.0 \times 10^{-6}$  M, pH working range 2.6-3.6 and 5.7-7.9; it was determined that the response time was <5s. In the samples of a pharmaceutical drug with the developed electrodes, the determination of imipramine hydrochloride, maprotiline hydrochloride and isoprenaline hydrochloride was successfully performed.

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**OP26- Tracking silent geochemical markers for polymetallic pollution in area with Cu-Pb-Zn minerals exploitation**

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The distribution of certain elements, which in higher content represents hazard to the environment, causes certain unwanted consequences on human health. Therefore, the environmental monitoring not only for the lithogenic but also for the anthropogenic distribution leads to determination of the main hot spots in environment. The anthropogenic activities for exploitation of natural resources and their processing represent a global problem of pollution of the environment. Bregalnica River Basin in the eastern part of the Republic of Macedonia was selected as a study area with the presence of three potential emission sources: lead and zinc mines („Zletovo” and „Sasa” mines) and copper mine („Bučim” mine). Lithogenic and anthropogenic distribution of 69 elements was evaluated in alluvial, automorphic and paddy soil from Bregalnica River Basin, Republic of North Macedonia. Determination of the total elements contents was performed using mass spectrometry with inductively coupled plasma (ICP-MS) and atomic emission spectrometry with inductively coupled plasma (ICP-AES). The obtained values for the contents of certain potentially toxic elements show significantly higher values in all types of samples collected from the vicinity of the mines, characterized with anthropogenic impacts. Soil analysis indicates that the lithogenic distribution is represented by six geochemical associations. The anthropogenic distribution represents the factor F2 (Cu, Pb, Sb, Cd, Sn, Zn, Te), where the regions of mines cover 90 percentile of the contents of these elements. Intensive polymetallic depositions were recorded only in the surroundings of the localities where the hydrothermal extractions are implemented.

*Keywords: toxic metals, monitoring, soil, environmental pollution, Bregalnica River Basin*

**OP27- Measurements of Iron in a Candidate Seawater CRM by Triple Quadrupole ICP-MS/MS with Isotope Dilution Calibration Strategies**

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Quantification of trace level iron in seawater matrix has been a trend topic for the last three decades as it has an important role in the oceanic carbon cycle<sup>1</sup> by binding the active sites of molecules responsible for oxygen transport and mitochondrial electron transfer<sup>2</sup>. It is very crucial to develop highly accurate analytical method(s) for the certification of UME CRM 1206 Trace Elements in Seawater as a candidate certified reference material. Isotope dilution mass spectrometry, IDMS, as a potential primary technique is the proper option for the characterization of a certified reference material (ISO 17034<sup>3</sup>). The developed method is based on isotope dilution mass spectrometry combined with triethylamine ( $\text{N}(\text{CH}_2\text{CH}_3)_3$ , TEA) assisted  $\text{Mg}(\text{OH})_2$  co-precipitation. Determination of Fe was carried out by ICP-MS with tandem mass spectrometry (ICP-MS/MS) with  $\text{NH}_3/\text{He}$  cell gas. Limit of detection values were found to be 11 ng/kg for single IDMS. Accuracy of the developed method was checked using CASS-6 and NMIA MX014 certified reference materials. In addition, repeatability and intermediate precision were evaluated using the results of UME CRM 1206. The relative standard uncertainty of within day precision was found as 0.16%, 0.19% and 0.20% for single, double and triple IDMS, respectively. Comparison of calibration approaches and the application of TEA assisted  $\text{Mg}(\text{OH})_2$  co-precipitation with IDMS for iron is reported for the first time in this study.

**Keywords:** Isotope dilution mass spectrometry; Seawater; Iron; Triethylamine;  $\text{Mg}(\text{OH})_2$  co-precipitation

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**OP28- Trace Cobalt Determination in Chamomile Tea Samples by an Effective and Novel Analytical Method by Using Simultaneous Dispersive Liquid Phase Microextraction-Slotted Quartz Tube –Flame Atomic Absorption Spectrophotometry**

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Cobalt is fundamentally necessary for the function of humans and other organisms as it is a component of B<sub>12</sub> vitamin<sup>1</sup>. Although cyanocobalamin is essential, other cobalt compounds may be detrimental considering their adverse effects on the human body and environment. According to the report of The French Food Safety Agency (AFSSA), the daily mean Co intake for adults and children should not exceed 0.48 and 0.31 µg/kg body weight, respectively<sup>2</sup>. In this study, development of a novel and sensitive analytical method was aimed for the determination of cobalt at trace levels. This was achieved using a combined system of flame atomic absorption spectrophotometry (FAAS) and preconcentration/separation of cobalt by simultaneous dispersive liquid phase microextraction (S-DLLME). In the S-DLLME process, complexation and extraction was performed simultaneously in order to overcome high error rates which are consequences of multiple operating steps. A Schiff Base synthesized from the reaction of 5-bromosalicylaldehyde and 1-naphthylamine was used as complexation agent. Optimum experimental conditions were determined in order to enhance the absorbance signal. Analytical performance of the developed method was investigated under the optimum conditions, and about 48-folds enhancement in detection power was obtained with respect to the detection power of the typical FAAS system. Spiked recovery studies were also performed in chamomile tea samples with the purpose of determining the accuracy and applicability in real samples of the method. Satisfactory recovery results (≈100%) with standard deviation values below 10% were calculated for two different concentrations.

**Keywords:** Cobalt; Chamomile; Slotted Quartz Tube; Simultaneous Dispersive Liquid Phase Microextraction; Flame Atomic Absorption Spectrophotometry.

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**OP29- Determination of Cobalt by Pressurized Liquid Phase Microextraction using an Automated Simple Two Syringe System Combined with Slotted Quartz Tube Flame Atomic Absorption Spectrometry**

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Cobalt is a vital component of various biological processes but it can cause harmful effects in humans when found above its threshold level. This element can accumulate in tissues and organs over a time period and eventually cause adverse health issues even at trace levels<sup>1</sup>. Hence, determination of cobalt at low levels is important to protect human health. In this study, a new, simple and green liquid phase microextraction method called sieve conducted two syringe based pressurized liquid phase microextraction (SCTS-PLPME) was coupled with slotted quartz tube-flame atomic absorption spectrometry (SQT-FAAS) to respectively preconcentrate and determine cobalt at trace levels. Two syringes were held on a mechanical apparatus and connected to each other. There was a plastic material inside the tube with 6 tiny perforated holes with the aim of enhancing surface area in a spray form. Pressure was formed between the two syringes by the simultaneous movement of syringe plungers (to and fro) and this generated an efficient dispersion, eliminating the need for a dispersive solvent. In the method development method, (2-[(Z)-[2-aminophenyl]imino]methyl]-4-bromophenol) was synthesized in the laboratory and used as a selective complexing agent and chloroform was selected as the extraction solvent. Effective factors that influence the microextraction efficiency were optimized to enhance the detection power of the FAAS system.

**Keywords:** Cobalt; SQT; FAAS; Microextraction.

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**OP30- Trace cobalt determination in lake water samples by ultraviolet based photochemical vapor generation-T-shaped slotted quartz tube atom trap-flame atomic absorption spectrophotometry**

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Cobalt determination is one of the most important issues because it has toxic effects on human health [1]. It has been widely used in several industrial applications such as magnets, ceramics and colors [2]. Flame atomic absorption spectrophotometry (FAAS) system was integrated with photochemical vapor generation-T shaped-slotted quartz tube-atom trap (T-SQT-AT-PVG) technique to perform cobalt determination in lake water samples. T-SQT-AT-PVG method simply consisted of T-SQT, UV reactor, peristaltic pump and gas-liquid separator. Radicals of low molecular weight organic acid (LMWOA) may occur in UV irradiation and these radicals may attack to metal analyte to create volatile derivatives. These volatile derivatives of analyte were adsorbed into inner surface of T-SQT and then suddenly released by sending hydrogen gas in the system. Optimum conditions of PVG system were taken from previous performed study in our research group [3]. Flame type, trapping period, type of coated material in the T-SQT, flow rate of carrier gas, T-SQT-AT height, type of T-SQT-AT and flow rate of hydrogen gas were investigated to evaluate optimum conditions of the PVG-T-SQT-AT system. Under the optimum conditions, limit of detection and limit of quantification values were calculated as 0.05 and 0.16 mg/L, respectively. Percent recovery results were found in the range of 97.5-102.5% for lake water samples in spiking experiments.

*Keywords: Cobalt, photochemical vapor generation, T-shaped SQT, lake water*

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**OP31- Trace Determination of Cadmium using Ultrasound Assisted Deep Eutectic Solvent Microextraction-Slotted Quartz Tube-Flame Atomic Absorption Spectrometry**

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Cadmium is known as an element with toxic effects and it has the potential to be collected in the kidney of the human body [1]. Although it is found at low concentrations in different organs of human being, exposure to Cd can have serious health effects including problems in the liver, bone and blood, vomiting, cramps and nausea [2]. Studies have shown that low levels of long-term exposure to Cd can cause to risk of cancer [3]. In this study, deep eutectic solvent based on liquid phase microextraction (DES-LPME) has utilized for preconcentration of Cd in a simple and effective manner at trace level by a flame atomic absorption spectrometry (FAAS) system equipped with a slotted quartz tube (SQT). Ultrasound assisted (UA) strategy was used to improve the detection power. All optimization parameters belong to UA-DES-LPME-SQT-FAAS system were optimized to enhance the sensitivity of the FAAS. Under the optimized conditions, limit of detection (LOD), limit of quantitation (LOQ) and enrichment in detection power (EDP) were found as 0.35 µg L<sup>-1</sup>, 1.2 µg L<sup>-1</sup> and 80, respectively. For the purpose to test the accuracy of the developed analytical method, the method was successfully applied to apple and celery samples, and the recovery results calculated were close to 100%. This established that the proposed method can be applied to complex matrixes for the accurate determination of cadmium.

**Keywords:** Cadmium; FAAS; SQT; Deep eutectic solvent.

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**OP32- Zirconium Nanoparticles based Solid Phase Extraction Prior to Slotted Quartz Tube-Flame Atomic Absorption Spectrophotometry for the Determination of Cadmium in Wastewater Samples and Evaluation of Green Profile**

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Cadmium is an extremely toxic metal even at low amounts and known to cause damages to various organs including the lungs, liver and kidneys<sup>1</sup>. Due to its occupational and ecological effects on health, the contamination of cadmium in soil, water and plants is a great concern<sup>2</sup>. Hence, it is very crucial to develop sensitive and accurate analytical methods for the determination of this element at trace levels. In this work, a novel analytical method was proposed by combining zirconium nanoparticles (Zr-NPs) based solid phase extraction (SPE) with slotted quartz tube-flame atomic absorption spectrophotometry (SQT-FAAS) for the determination of cadmium at trace levels. Zr-NPs were synthesized with a novel/simple method and characterized with scanning electron microscope and X-ray diffraction instruments. Optimization studies included main factors affecting the efficiency of extraction including pH, amount of nanoparticles, mixing type/period and eluent type for the developed strategy. The limit of detection was found as  $0.44 \mu\text{g L}^{-1}$ , while the limit of quantification was obtained as  $1.46 \mu\text{g L}^{-1}$  under optimal parameters. The optimized extraction procedure was applied to wastewater samples in order to test both applicability and accuracy in complex matrixes. For this aim, wastewater samples were spiked at three different concentrations and satisfactory recoveries were achieved in the range of 96 – 101%.

**Keywords:** Cadmium; Zirconium nanoparticles; Flame atomic absorption spectrophotometer; Solid phase extraction; Wastewater.

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**OP33- A Novel Analytical Strategy for the Determination of Cadmium in Milk Powder, Linden and Mint Samples: Molybdenum Coated T-Shaped Slotted Quartz Tube-Flame Atomic Absorption Spectrophotometry (Mo-T-SQT-AT-FAAS)**

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Cadmium is one of the heavy metals with high toxicity and proven to have carcinogenic potential. It was proved that chronic exposure to this element can cause damage to the kidneys and bones<sup>1</sup>. Hence, it is highly crucial to determine this toxic metal at ultra-trace levels in several food and environmental samples. Atom trapping strategy describes atoms that are trapped on the inner surface of a T-shaped quartz tube for a specified period and released from the surface by creating a reducing medium to obtain a transient signal and corresponding high sensitivity<sup>2</sup>. In this study, Mo-T-SQT-AT-FAAS method was introduced for the sensitive determination of cadmium. Molybdenum was used to coat the the inner surface of the T-SQT to enhance the trapping efficiency by increasing metal-metal attraction. Hydrogen gas was used to release the trapped analyte atoms instead of organic solvents. Developed method was environmentally friendly and inexpensive compared to other extraction methods. LOD and LOQ values were calculated as 0.024 and 0.081 µg/L, respectively. The method had a linear working range between 0.10-1.0 µg/L. 2854 times enhancement was calculated in detection power compared to the conventional FAAS. Recovery experiments were done to check the applicability of the developed method to linden, milk powder and mint samples. and satisfactory results (94 – 101%) were obtained. Tomato Leaves 1573a SRM was analyzed in order to check the accuracy of the Mo-T-SQT-AT-FAAS method, and the result found in this study was very close to the certified value.

**Keywords:** Atom trapping; FAAS; Linden; Milk powder; Mint; Molybdenum.

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### OP34- Metal Levels Monitoring in Coastal Region of Bursa

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Bursa is an industrial, agricultural and economically developed city of Marmara region. It is important to monitor environmental sea pollution. Metal monitoring in coastal areas of the Bursa was planned over 42 seawater samples taken from 7 regions of selected coastline in the 6-month period covering July- December 2017<sup>1-7</sup>.

In trace metal analysis with Flame Atomic Absorption Spectrometer (FAAS), various enrichment techniques such as co-precipitation, adsorption, solid phase extraction etc. So that, seawater samples were taken for simultaneous determination of Cd, Cr, Cu, Ni, Pb and Zn with enrichment in the activated carbon filled column.

The validation studies was performed with synthetic sea water (ASTM D1141-98). Also method was tested with commercially available reference material QC-3163.

All samples results were found under the limit of detection (<LOD) in period of 6 month. This work also need to apply to the sediments because of the precipitations<sup>8</sup>. On the other hand, this case, the study provided the basis for future investigations.

**Keywords** seawater, FAAS, activated carbon, 8-HQ,

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#### Acknowledgment

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### **OP35- Feasibility Studies on the Migration and Bioaccessibility of Environmental Pollutants in Plants, and Simulation under Intestinal and Gastric Conditions**

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Man-made activities result in several kinds of waste materials emanating from households, industries, mining sites, metal processing plants and spilled chemicals. A great percentage of these materials are synthetic and tend to stay for very long periods in the environment due to their non-biodegradable nature. They therefore pose great risks to humans and other organisms in the environment <sup>1</sup>. Depending on the solubility of contaminants and nature of soil, contaminants may leach down into ground water. On the other hand, plants (depending on the species) are capable of taking up these contaminants through the root system and this depends on physicochemical properties, temperature and interactions with microorganisms in the soil <sup>2</sup>. These contaminants can easily enter the food chain and biomagnify as they move up the trophic levels. This study was therefore aimed at investigating the feasibility of selected endocrine disruptive compounds moving into plants grown in a soilless medium. The analytes included seven (7) organochlorine pesticides, one (1) organophosphate pesticide, two (2) hormones, two (2) alkylphenols and bisphenol A. The main criterion for selecting green onion and lettuce as case study plants was their high affinity for water. An optimized dispersive liquid-liquid microextraction (DLLME) method was used to preconcentrate the analytes from the soilless media for the determination by gas chromatography mass spectrometry (GC-MS). On alternate days, the aqueous media were analyzed and the results obtained suggested that analytes migrated into the plants. To confirm this, the green onion samples were freeze dried, powdered, extracted with acetonitrile and sent to the GC-MS system after preconcentrating with DLLME. All analytes were detected in the samples and the amounts calculated were in the range of 0.59 – 6.4 µg/g. The onion samples were also simulated under gastric and intestinal conditions and all analytes showed about 89% degradation within a two hour period.

**Keywords:** Endocrine Disruptive Compounds; Pesticides; Hormones; Alkylphenols; GC-MS; DLLME.

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**OP36- Comparison of microwave-assisted and conventional hydro-distillation extraction of essential oil from *Artemisia abrotanum* L.**

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Herbs and spices are commonly used as food additives, fragrances, flavours in pharmaceutical products for many years. Nowadays, their impact on the human health is gain more importance and especially essential oils contained in them have been the subject of many researches<sup>1</sup>. An essential oil is a volatile substance derived from fragrant plant materials. Essential oil extracted flower, buds, leaves, stems, seeds, fruits, roots, barks of the plants generally by steam distillation or cold press<sup>2,3</sup>. In this study we aimed to find out the best extraction method for the essentials oil of *Artemisia abrotanum* L.. Instead of using classical methods for the extraction of essential oil, microwave-assisted and conventional hydro-distillation extraction methods are used in this study. Essential oils of the plant extracted by microwave-assisted hydro-distillation (MAHD) for 30 minutes and conventional hydro-distillation (CHD) for 3 h. The obtained essential oils were dried with NaOH and stored at +4°C until gas chromatography-mass spectrometry (GC-MS) analysis. 50 compounds were determined by GC-MS analysis in the samples prepared by both extraction methods. Major compounds are 1-8-cineol, borneol, bornylene, p-cymene and camphor respectively. Although the composition of the compounds were similar, significant differences were observed in terms of energy and time spent.

**Keywords:** Microwave extraction; Hydro-distillation; *Artemisia abrotanum*; Essential oil; Green chemistry

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**OP37- Dispersive Liquid-Liquid Microextraction Followed by Smartphone Digital Image Colorimetry for the Determination of Iodate in Table Salt**Jude Caleb<sup>1</sup>, Usama Alshana<sup>1\*</sup>, Nusret Ertaş<sup>2</sup><sup>1</sup> Department of Analytical Chemistry, Faculty of Pharmacy, Near East University, 99138 Nicosia, TRNC, Mersin 10, Turkey<sup>2</sup> Department of Analytical Chemistry, Faculty of Pharmacy, Gazi University, 06330, Ankara, Turkey\*E-mail: [usama.alshana@neu.edu.tr](mailto:usama.alshana@neu.edu.tr)

Digital image colorimetry (DIC) is a miniaturized analytical technique that has recently been proposed as a cost-effective and simple complementary spectrophotometric technique for qualitative and quantitative chemical analysis <sup>1</sup>. It makes use of processing digital images captured by digital cameras, hand-scanners, webcams or smartphone cameras. Small differences in colors and their intensity can be exploited to find the concentration of analytes absorbing in the visible region. The applications designed for this purpose detect the primary colors, red, green and blue (RGB) channels based on a whole number value between 0 and 255 for each channel. The higher the intensity of the color, the lower the RGB value <sup>2</sup>. The absorbance can then be calculated as the logarithm of  $I_0$  to  $I$  ratio, where ' $I_0$ ' represents the intensity of the blank at a specific channel and ' $I$ ' represents the intensity of the analyte solution at the same channel. Despite its simplicity, DIC would, however, be expected to have low sensitivity and selectivity due to the use of a continuum light source and the absence of a monochromator. Thus, combining DIC with a suitable preconcentration technique such as dispersive liquid-liquid microextraction (DLLME) prior to analysis would overcome these two limitations. In this study, smartphone-based digital image colorimetry (SDIC), combined with DLLME, is proposed for the determination of iodate ions. A colorimetric box was designed to capture images of the yellowish sample solutions after derivatization of iodate into iodine via the well-known redox reaction of iodate with iodide ions in an acidic medium. Factors affecting the efficiency of SDIC included type of phone, area of region of interest, position of camera, and distance between camera and sample solution. Optimum DLLME conditions were achieved with 1-undecanol (500  $\mu$ L) as the extraction solvent, ethanol (1.5 mL) as the disperser solvent within 20 s extraction time. Under optimum DLLME-SDIC conditions, limit of detection (LOD) was found as 0.1  $\mu$ g mL<sup>-1</sup> (or 0.2  $\mu$ g g<sup>-1</sup>) with enrichment factors ranging between 17.4 and 25.0. Calibration graphs showed good linearity with coefficients of determination ( $R^2$ ) higher than 0.9954 and relative standard deviations (%RSD) less than 5.6%. The proposed method was efficiently applied to determine iodate in table salt samples with percentage relative recoveries (%RR) ranging between 89.3 and 109.3%.

**Keywords:** Digital image colorimetry; Dispersive liquid-liquid microextraction; Iodate; Smartphone; Table salt

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### OP38- Current Situation and Challenges in Green Analytical Chemistry

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Bioanalytical and pharmaceutical drug analyses are a massive area of chromatographic methodology. Common solvents used in HPLC such as acetonitrile and methanol are threatening human health and natural life cycles. The amount of solvent waste has increased as a consequence of increased number of chromatographic experiments carried out in drug analysis laboratories without a planned waste management policy. While performing experiments for the sake of human health or science, the inflicted harm may outweigh the benefit gained.

Green Analytical Chemistry (GAC) emerged from green chemistry in 2000 (1). This new approach aims to make the laboratory practices of analytical chemists environmental-friendly (2, 3). In addition to the developments in method and device technologies, the efforts to reduce the negative effects of chemicals on the environment and the applications of sustainable development of analytical chemistry laboratories are becoming increasingly important. In this context GAC is considered as a stimulus in the development of analytical chemistry. The greatest success in this discipline is the increase in environmentally friendly studies in parallel with the increase in the quality of analytical results. Anastas and Warner scripted 12 principles of green chemistry, which are used as guidelines for defining a ‘greener’ analytical method.

In addition to the advances in methods and device technologies, the applications of sustainable development to analytical chemistry laboratories and efforts to reduce the negative effects of chemicals on environment and analysts are gaining importance. The most common green analytical chemistry applications are miniaturization of the analytical devices, reducing column diameter, eco-friendly sample preparation steps, decreasing the amount of solvent consumption, generating less waste to the environment. In addition, the reduction of toxic solvents used in the sample preparation steps or their replacement with less harmful ones is also appropriate for the green chemistry approach. Also performing analysis with the help of chemometric methods in optimization and validation steps will reduce the amount of solvents used. It is not possible to green all the analysis process but each attempt is precious.

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### OP39- Determination of Curcumin in medicinal plants and its Isolation in Turmeric (*Curcuma longa* L)

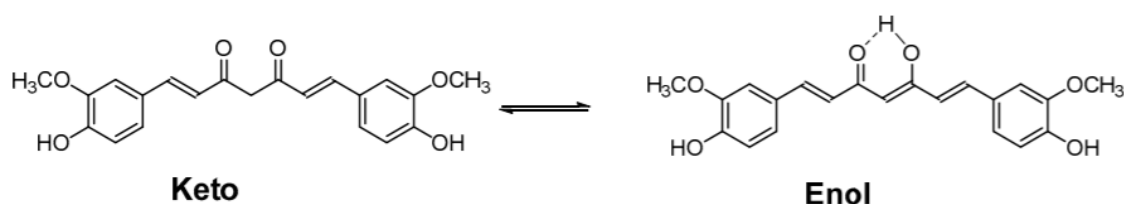
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Curcumin and its derivatives; demethoxycurcumin, and bisdemethoxycurcumin are bioactive polyphenolic compounds which have been collectively referred to as curcuminoids. Curcuminoids have attracted a lot of attention in recent decades due to their therapeutic potential as anti-inflammatory, anti-diabetic, anti-cancer, and anti-aging. Curcumin is practically insoluble at room temperature in aqueous solutions at neutral and acidic pH. However, it is soluble in organic solvents like methanol, ethanol, acetone, and dimethyl sulfoxide agent <sup>1-4</sup>.



Both at neutral and acidic pH, keto form is predominant, however, enol tautomer is exclusively present in alkaline conditions. Curcumin has pKa of 8.54 and possesses three labile protons at neutral pH of which one is enolic, and two are phenolic protons. Curcumin shows maximum absorption at 430 nm in methanol and 415–420 nm in acetone.

In this study, firstly, curcumin and its derivatives were determined in eight medicinal plants. For this purpose, HPLC-DAD was used. To determine the best solvent, methanol, HCl-methanol, ethanol, acetonitrile, butanol, ethyl acetate were examined. At the measurement step, the parameters such as injection volume, flow rate, column temperature and mobile phase were optimized.

Depending to its concentration, turmeric was examined for isolation studies. In the isolation studies, TLC and preparative chromatography were used. Some optimum conditions were found as 425 nm-wavelength, 20 µL-injection volume, 0,4 mL/min-flow rate, 25°C-column temperature and THF:% 1 citric acid (4:6)-mobile phase.

**Keywords:** turmeric, curcumin, medicinal plants, isolation, determination.

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**OP40- Simultaneous Determination of Dioxacarb, Trifluralin, Tefluthrin And Flumetralin in Tap Water and Wastewater Samples by Gas Chromatography Mass Spectrometry after Vortex Assisted Switchable Solvent Based Liquid Phase Microextraction**

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In order to ensure food security around the world, pesticides have been mainly used on agricultural fields to deter pests that cause harm to crops, thereby, increasing the overall output (quality and quantity) of farm produce<sup>1</sup>. In addition to crop protection, pesticides are used to exterminate disease carrying pests to ensure good health. However, humans and other non-target organisms stand the risk of pesticide exposure and adverse effects. It is therefore essential to develop sensitive analytical methods that are capable of determining pesticides at trace levels in the environment<sup>2</sup>. This study employed switchable solvent based liquid phase microextraction to preconcentrate dioxacarb, trifluralin, tefluthrin and flumetralin from wastewater and tap water matrices into measurable amounts for gas chromatography mass spectrometry detection. In a stepwise manner, significant extraction parameters including volume of switchable solvent, concentration and volume of sodium hydroxide, mixing effect and salting-out effect were optimized to boost the signals of all four analytes. To validate the developed method, aqueous calibration standards were used to develop calibration plots for each analyte with R<sup>2</sup> values above 0.9997. The respective detection limit and quantification limit ranges calculated for the analytes were 0.38 – 2.0 µg/L and 1.3 – 6.5 µg/L. Spike experiments were then performed to further validate the optimized method based on applicability to real sample matrices and accuracy of quantification. Two wastewater samples from a municipal treatment plant and a hospital sewer were spiked alongside tap water taken from the laboratory. The samples were spiked with the mixed standards at three peculiar concentrations and analyzed according to optimum conditions. Using aqueous calibration standards as reference, percent recoveries were calculated between 79 and 107% for all four analytes in the selected matrices at three different spike concentrations. This proved the method's accuracy in quantifying the pesticides in the matrices tested.

**Keywords:** GC-MS; Switchable solvent microextraction; Pesticides; Wastewater.

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**OP41- An Accurate and Precise Determination Method for Propineb in Black Tea and Infant Formula Samples by GC-MS after Applying a New Derivatization Technique and Dispersive Liquid-Liquid Microextraction Method**

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Propineb is one member of propylenebisdithiocarbamates and its poor miscibility in common solvents results in tedious steps in its determination<sup>1, 2</sup>. In this study, an easy and new analytical method was developed to detect propineb by gas chromatography mass spectrometry (GC-MS). For this purpose, a new derivatization technique was combined with dispersive liquid-liquid microextraction method for accurate and sensitive determination of analyte in black tea and infant formula samples by GC-MS. Potassium persulfate as oxidative desulfurization reagent and potassium carbonate were used to obtain isothiocyanate derivatives of the analyte. Comprehensive optimization was performed for all parameters including type/period of mixing for the derivatization reaction, concentration/volume of potassium persulfate and potassium carbonate salts, type/volume of extraction solvent, type/volume of dispersive solvent and type/period of mixing for the microextraction method. Limit of detection and quantification of the established method were found to be 0.16 mg/kg and 0.52 mg/kg, respectively. There was no detectable propineb in black tea and infant formula samples. In order to ascertain the applicability and accuracy of the developed method, spiking experiments were done for black tea and infant formula samples, and results obtained ranged between 99 – 102% and 98 – 103%, respectively.

**Keywords:** Propineb; Gas chromatography mass spectrometry; Black tea; Infant formula.

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**OP42- A Simple, Accurate and Sensitive Analytical Strategy for Propoxur Determination in Raisin Samples using Gas Chromatography-Mass Spectrometry with Matrix Matching Method after Vortex Assisted Dispersive Liquid-Liquid Microextraction and Assessment of Green Profile**

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Propoxur is classified as one of the most significant carbamates that is broadly used in agricultural applications for its effective insecticidal properties<sup>1</sup>. Despite its advantages against insects, it has carcinogenic effects on untargeted living organisms like honey bees and fish<sup>2</sup>. It is therefore crucial to determine trace levels of propoxur in environmental and food samples. In this study, sensitive determination of propoxur in raisin samples was achieved by gas chromatography mass spectrometry (GC-MS) after preconcentration using an optimized vortex assisted dispersive liquid-liquid microextraction (VA-DLLME) method. Optimization of all important experimental and instrumental parameters was performed with the aim to improve the detection power of the GC-MS system for propoxur. Under the optimum conditions, the limits of detection (LOD) and quantification (LOQ) were calculated as 0.24 and 0.81 µg/L, respectively. The detection power of the GC-MS was enhanced by 105 folds. The linear range of the method was between 1.0 – 75 µg/L with relatively low percent relative standard deviations (%RSD < 10.0%) which represents high experimental precision. Real sample application was performed on raisin samples with high percent recovery results that ranged between 100 – 104%, indicating the accuracy and the applicability of the developed method.

**Keywords:** Dispersive liquid-liquid microextraction; insecticide; propoxur; gas chromatography-mass spectrometry; raisins.

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### OP43- Synthesis and Enhancement of Composite for Removal of Dye: Effect of contact time and Initial Dye Concentration

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**Abstract:** In order to research the adsorption characteristics of cationic dye, the fungus based composite (FBC) was synthesized. For this, batch adsorption experiments were applied by using three different initial dye concentrations 50, 100, 150 mg/L kinetically and the data are performed via pseudo first and pseudo second kinetic models<sup>1</sup>. Fourier transform infrared spectrophotometer method was used for investigation whether cationic dye adsorbed onto FBC composite or not by using the spectrum of dye-adsorbed composite. The highest adsorption capacity of composite was predicted as 40.11 mg/g (100 mg/L, 120 rpm, 298 K).

Keywords: adsorption kinetic, composite, contact time effect, FTIR

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**OP44- The Effects of the Extraction Methods on Biologically Active Components and Antimicrobial Effects of *Ziziphus jujube* Mill.**

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The jujube (*Ziziphus jujube* Mill.) fruit grows mostly in Europe, southern and eastern Asia, and Australia. Edible dried jujube fruits have been used as a remedy additive in traditional medicine. Flavonoids, vitamin C, phenolics, triterpenic acids, and polysaccharides are the main active components of the fruit and the anti-inflammatory, anticancer, antiobesity, antioxidant, hepatoprotective, and gastrointestinal protective effects have been reported in the literature (1). In this study the effect of the extraction procedure on the flavonoid concentration and antimicrobial effect of the ethanolic extracts of the jujube has been investigated. Ethanolic extracts of the dried fruit prepared by soxhlet, maceration, microwave and ultrasound assisted extraction methods and a validated reversed phase HPLC-UV method used for quantitation of the flavonoid concentrations of the extracts. Separation of the flavonoids were performed by acetonitrile & phosphate buffer (0,025 M, pH 2,6) mobile phase system by gradient elution on a C18 (250 x 4,6 mm, 5 µm) column at 30 °C column temperature with 0.6 mL.min<sup>-1</sup> flow rate and 25 µL injection volume.

The bioactivities of the extracts were determined by using thin layer chromatography (TLC) bioautography method against pathogenic microorganisms. Ethanolic extracts were applied on TLC plates which were then subjected to bioautography against pathogen microorganisms for determination of the antimicrobial effect differences of the extracts.

*Acknowledgement: This work was supported by Tübitak 218S812 financially.*

**Keywords:** Jujube, extraction, HPLC, TLC-bioautography

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## OP45- The Relation between LC Retention Data of Some NSAIDs on a C18 Column and Octanol-Water Partition Coefficients

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Non-steroidal anti-inflammatory drugs (NSAIDs) are well known pharmaceutical drug candidates which are frequently used as analgesics, anti-inflammatory and antipyretic agents. In addition to these therapeutic actions, NSAIDs have also demonstrated beneficial effects in treating cancer, Alzheimer's, and arthritis diseases.

During the last ten years a great amount of drug discovery studies use physicochemical properties to facilitate the interpretation of biological effects and to guide the design of compounds such that the probability of achieving a favorable biological profile is increased. The physicochemical properties of a compound, particularly its lipophilicity, will influence to varying extents its potency and pharmacokinetic and toxicological profile. Although, in many research works, lipophilicity is determined using direct methods, especially shake flask method, this technique has been successfully replaced by chromatographic methods such as reversed-phase high-performance liquid chromatography (RP-HPLC) and reversed-phase high-performance thin-layer chromatography (RP-HPTLC)<sup>1</sup>.

In the present study, the partition coefficients of some NSAIDs (ketorolac, tolmetin, naproxen and dexketoprofen) have been determined by RPLC as described by Donovan and Pescatore<sup>2</sup>. Lipophilicity values published up to now significantly differ. In order to overcome this ambiguity, some additional measurements and the critical evaluation of lipophilicity properties of compounds are necessary. The chromatographic retention were studied in the isocratic mode, using a Zorbax Eclipse XDB C18 (4.6 × 150 mm, 5 µm) column. The oven temperature was set at 37°C (biorelevant temperature) and the flow rate was 1mL/min. Four reference standards were chosen from literature to determine logP<sub>o/w</sub> values of the studied compounds. The lipophilicity values found for the compounds are in good agreement with the literature thus confirming the reliability of the HPLC method used for logP<sub>o/w</sub> determination.

**Keywords:** *Lipophilicity, NSAIDs, Biorelevant temperature, RPLC.*

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**OP46- Development of Tungsten Oxide Modified Pencil Graphite Electrode  
As A Novel Platform For Ochratoxin-A Detection**

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OTA (Ochratoxin-A), one of the most abundant mycotoxins, is found in tissues and organs of animals and is known to produce a number of toxic activity in several animal species. Therefore, its determination in cereals, roasted coffee, wines, dried fruits, baby food and animal feeds has great importance and various chromatographic techniques have been used for this purpose. Electrochemical sensors based on electrochemical impedance spectroscopy (EIS) have recently attracted considerable interest for their detection where the interactions of biomolecules on a conductive electrode can be assayed by monitoring interfacial electron transfer characteristics [1].

In order to increase the sensitivity of electrochemical measurement systems, modification of the electrode surfaces with conductive polymers, nanomaterials and composite materials is applied. Transition metal oxides are also utilized in biosensor applications due to their unique electrocatalytic properties, large surface area and strong adsorption of biomolecules [2].

In the present study, pencil graphite electrode (PGE) surfaces were modified with tungsten oxides (WO<sub>x</sub>) via cycling the potential between -0.6 to 0.9 V at a rate of 50 mV/s for 5 cycles, or via using pulsed deposition (PD) technique at two potential levels being -0.5V and 0.2 V for 3.0 sec. for 50 cycles. Amino labeled aptamer was immobilized onto the WO<sub>x</sub> modified surfaces that was previously treated with EDC/NHS solution. That modified surfaces was used as an aptasensor for specific and sensory determination of OTA via EIS technique.

The evaluations for OTA detection were made by calculating  $\Delta R_{ct}$  values and it was observed that pulsed deposited tungsten oxides has greater interaction with OTA molecules.

**Keywords:** aptasensor, OTA, tungsten oxide

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**Acknowledgment:** Authors would like to thank Ege University Scientific Research Council for financial support (Project number: 18-FEN-036)

## OP47- Utilization of Bipolar Membrane Electrodialysis (BMED) Systems for Selective Separation and Recovery of Valuable Elements from Water

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### Abstract

Water sources are natural resources of both potable water and valuable elements useful for various industrial applications. Natural sources of water are not directly usable due to their content in various elements and/or pollutants. On one hand, these elements and or pollutants are harmful to human being health, animals and plants. On the other hand, some of them are valuable due to their applications in the industrial world and ultimately become necessary for human life needs. There is therefore a need of technologies for water resources purification and valuable elements recovery. Lithium and boron content as valuable elements in geothermal waters attracted our attention. Selective separation and recovery of lithium and boron by using bipolar membrane electrodialysis (BMED) was investigated. Two BMED systems provided by PCCell GmbH and Mega electromembrane companies were used. Both systems were set in various operational conditions for the separation and recovery of boron and lithium. As BMED is based on transport of ionic species and recover them as bases and acids, lithium was separated as lithium ion and recovered as lithium hydroxide while boron was separated as tetraborate ion and recovered as boric acid. For higher separation and recovery performance, it was discovered that an attention should be made during the choice of BMED based on system design and ion exchange membrane materials present in the stack. This helps to achieve satisfactory separation and recovery at reasonable specific energy consumption. Electrodialysis with bipolar membrane was found to be a successful process to separate and recover selectively boron and lithium from aqueous solutions.

**Keywords:** Bipolar electrodialysis membrane (BMED), boron and lithium, separation and recovery, water resources,

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**OP48- Determination Of Plasma Vemurafenib Levels And Development Of LC-MS / MS Method In Rats Fed With High Fat Diet**

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**Objective:** Melanoma is less than 5% of the skin cancers in the world, but it is a dangerous cancer with a high mortality rate. Mutation analysis of melanoma in the metastatic stage is an extremely important molecular marker in determining treatment. It is the BRAF V-600E mutation that causes 90% of the mutations to cause a single nucleotide change. BRAF is a serine-threonine kinase involved in the RAS-RAF-MAPK signal cascade. Vemurafenib (VEM) is a specific B-Raf inhibitor, an effective drug that extends average lifetimes and even cures by applying together with immunotherapy in the world for the treatment of metastatic melanomas. In the study, plasma levels were determined in rats fed with high-fat diet (YYD) of VEM used in the treatment of melanoma and it was aimed to develop Liquid Chromatography Tandem Mass / Mass Spectrometer System (LC MS / MS) method.

**Material-Method:** In the study, 300-350g of Wistar rats were obtained from Afyon Kocatepe University Experimental Animal Unit and ad libitum and water were given for 7 days at room temperature (25°C) with 50-55% humidity. During the application, it was divided into 10 groups and VEM was administered in various doses in addition to YYD by feeding with a fatty diet for 3 weeks. At the end of the application period, plasma samples were collected from the rat groups considering various time intervals. The Games-Howell test was used to determine which groups are different from each other because the variances are not homogeneous (Levene Test p value <5%). VEM analysis using LC-MS / MS was carried out by Agilent 6460 triple quad mass spectrometry.

**Conclusion:** When p values were analyzed, it was seen that there was no significant difference only between Groups 7 and 8 ( $p = 0.846$ ), and all groups were different from each other ( $p < 5\%$ ). It was observed that the 6th group had the smallest vemurafenib values, followed by 7th and 8th groups, followed by 9th and 10th groups. The calibration curve was linear between 0.01 and 0.8  $\mu\text{g mL}^{-1}$  vemurafenib with a 0.999 regression coefficient. The detection limit (LOD) and quantification (LOQ) of the method are calculated from the signal to noise ratio as 3: 1 and 10: 1, respectively. These are 1.10-4  $\mu\text{g mL}^{-1}$  for LOD and 10-4  $\mu\text{g mL}^{-1}$  for LOQ. Intraday and intraday analyzes were used to investigate the accuracy and precision of the developed method. This method has been found to be reproducible and highly accurate and provides a combination of faster analysis time and advanced detection limits.

**Discussion:** Although various methods have been reported for the determination of VEM, to our knowledge, there is no data for VEM on the high-fat diet on the rat plasma sample. Although studies on the changes in blood levels of VEM of the high-fat diet are limited, we think our study will shed light on studies evaluating the effects on human health. The evaluation of the results of our study still continues.

**Keywords:** Vemurafenib, LC MS / MS, High-fat diet, method.

**OP49- Separation of the Enantiomers of Chiral Sulfoxide Derivatives by Supercritical Fluid Chromatography**

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Caroline West<sup>4</sup>, Bezhan Chankvetadze<sup>2</sup>**

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Supercritical fluid chromatography (SFC) is a form of liquid chromatography that uses a supercritical carbon dioxide as the mobile phase component. It is known to be most effective for enantioselective separations as the resolution per unit time is often advantageous compared to HPLC separations, making it suitable for high-throughput enantioselective analysis. Furthermore, from the ecological point of view, due to the use of pressurized carbon dioxide as the main mobile phase component, it is also an attractive method for the industry, both at the analytical and preparative scales due to the reasons following: CO<sub>2</sub> has a much lower cost than the organic solvents used in the normal-phase HPLC mobile phases (hexane or heptane). Aside solvent costs, solvent disposal costs have also impact on the overall economy.

In the frame of this study fully porous (FP) and superficially porous (SP) silica particles containing polysaccharide-type chiral selectors were compared in SFC from the viewpoint of retention and separation behaviors. While chlorinated polysaccharide CSP especially the cellulose tris(4-chloro-3-methylphenyl) carbamate has proven to be promising for the separation of enantiomers of chiral sulfoxides in the past, we were interested in examining the SFC retention and separation in more detail by using some chiral sulfoxides.

A practical conclusion made from these experiments is that carbon dioxide offers the possibility to enhance retention for those analytes that would be too weakly retained in the polar organic mode. Many of the chiral sulfoxides' enantiomers could be separated within less than 1 minute.

**Acknowledgment**

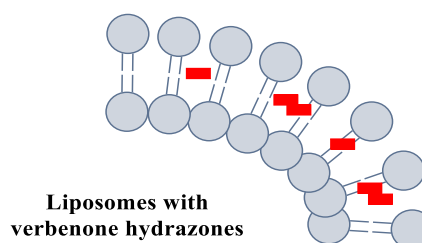
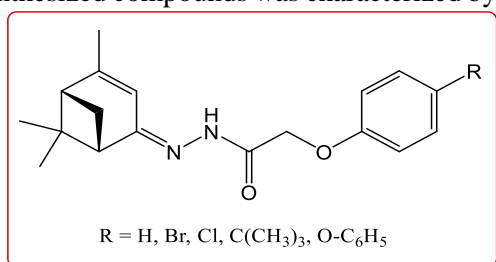
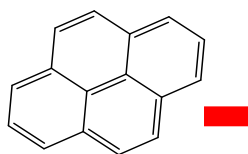
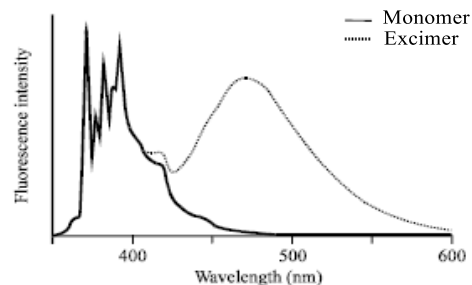
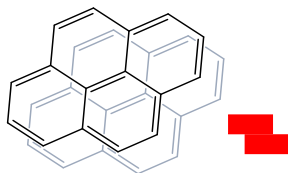
*This project was partially supported by the Project Based Mevlana Exchange Program with the project number MEV-2018-1295.*

**Keywords:** SFC, Separation of Enantiomers, Superficially Porous Silica.

**OP50- Influence of verbenone hydrazones on model membrane properties****Mariia Nesterkina<sup>1\*</sup>, Sergii Smola<sup>2</sup>, Iryna Kravchenko<sup>1</sup>**<sup>1</sup>Department of Organic and Pharmaceutical Technology, Odessa National Polytechnic University, Odessa 65044, Ukraine<sup>2</sup>A.V. Bogatsky Physico-Chemical Institute, National Academy of Sciences of Ukraine, Odessa 65080, Ukraine\*E-mail: [mashaneutron@gmail.com](mailto:mashaneutron@gmail.com)

The main obstacle for transdermal drug delivery is the outermost layer of skin – stratum corneum (SC) which is considered as a primary barrier to the penetration of compounds through the skin. Aiming at permeability increasing of SC various methods are applied, especially the use of skin penetration enhancers. Among them, terpenes and their derivatives attract great attention due to their high efficiency and low skin irritation. Here, we report the synthesis of novel hydrazones of bicyclic terpenoid verbenone as potential skin permeability enhancers.

To achieve this goal, a series of hydrazones has been obtained via (–)-verbenone condensation with hydrazides of *para*-substituted phenoxyacetic acids. The structure of synthesized compounds was characterized by <sup>13</sup>C-NMR, <sup>1</sup>H-NMR, FT-IR and mass spectrometry.

**Monomer****Excimer**

The mechanism of action for verbenone hydrazones on phospholipids of artificial membranes and lipids isolated from the rat stratum corneum was studied by fluorescence and FT-IR spectroscopy. We report here, that inclusion of synthesized compounds in phospholipid liposomes leads to growth of excimer to monomer ratio ( $I_E/I_M$ ) indicating a decrease of membrane microviscosity. Thus, the influence of verbenone derivatives on molecular organization of the lipid matrix substantiates the feasibility of their use after transdermal delivery *in vivo*.

Bearing in mind the aforementioned, (–)-verbenone hydrazones was elucidated after transdermal delivery as potential analgesic agents via chemical-induced pain models. In this study, pain in experimental animals was caused by selective agonists of TRP channel – capsaicin and allyl isothiocyanate (AITC) via their subplantar injection. All the tested compounds were found to suppress painful sensation produced by noxious stimuli indicating TRP channels (specifically, TRPV1 and TRPA1) as molecular targets of verbenone derivatives.

Thus, the current study reveals a strategy for drug development with analgesic effect for transdermal delivery. This idea is implemented by targeted synthesis of verbenone low molecular weight derivatives – hydrazones followed by their influence investigation on molecular organization of the lipid matrix and further pharmacological testing.

**Keywords:** verbenone, hydrazones, phospholipids, pyrene, analgesic activity

## OP51- Preconcentration of Trace Metals with Novel Synthesized Polymeric Resin

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Trace metals such as cadmium (Cd), cobalt (Co), copper (Cu), nickel (Ni), and lead (Pb) have toxic effects even at low concentration levels, and so it should be determination of theirs in various samples. Flame atomic absorption spectroscopy (FAAS) is one of the most used techniques for determination of those elements in samples. Although the technique has advantages including accurate, precise, inexpensive and easy to use, it does not have sufficient sensitivity for the determination of low concentration metals<sup>1</sup>. This disadvantage can be eliminating with preconcentration methods. Solid phase extraction, which has advantages such as high preconcentration factor, recovery, and selectivity, is one of the most preferred preconcentration methods. The adsorbents such as active carbon, amberlite XAD polymeric resins, clay, zeolite, silica gel, chitosan are used in this method<sup>2</sup>.

The purpose of this study aim is to preconcentrate of Cd, Co, Cu, Ni and Pb with novel synthesized polymeric resin having contain functional groups without use ligand. In this context, analytical conditions such as pH, contact time, and elution volume were investigated and the optimum conditions were determined to be in ranges of pH 3.0-3.5; 20 min for contact time, 3 mL for elution volume. The measurements were carried out by FAAS.

**Keywords:** Preconcentration, trace metal, Solid phase extraction, FAAS

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**OP52- Trace Element Concentrations of Leachates Associated with Different Polymetallic Mine (Cu, Cr and Pb-Zn) Areas**

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High trace element concentrations from leachates at mine areas are known to cause the important environmental problem [1]. Since mining activities are carried out in geological formations, the quality of leachates formed are related to the geochemical structure of the rocks. Geochemical studies include determining the acid-base levels and metal concentration contents from rock and ore [2]. Due to mining activities, the most common trace elements polluting groundwater and surface water resources are reported as As, Cd, Cr, Cu, Ni, Pb, Zn and Hg [3]. In this study, it was aimed to determine the trace element content of the leachates resulting from mining activities in three different polymetallic (copper, chromium and lead-zinc) mine areas. The leachate samples were analyzed by ICP-MS to determine the concentrations of As, Cu, Cd, Cr, Pb, Zn, Ni, Fe, Mn, Co and V. Leachates release from copper mine area are highly acidic and contain very high concentrations of Cu, Cd Mn, Ni, Zn and Co. However, leachates release from chromium mine area are slightly alkaline and contain low concentrations of trace element except Zn and Cr. On the other hand, leachates from the lead-zinc mine area have high levels of Zn and As, although they have neutral pH levels. Therefore, appropriate care needs to be treatment of leachates at mine areas in order to control the environmental pollution.

**Keywords:** Mining activities, Leachates, ICP-MS, Trace element, Polymetallic mine area

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## Poster Presentations

### **PP1- A novel impedimetric immunosensor for sensitive detection of IL 6 prostate cancer biomarker based on succinimide substituted polypyrrole modified disposable ITO electrode**

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**Abstract-**In this work, we constructed an ultrasensitive immunosensor based on succinimide substituted polypyrrole polymer (Poly(Pyr-NHS) modified indium tin oxide sheet. Firstly, the ITO sheet was coated with Poly(Pyr-NHS) polymer and this modified ITO sheet was used as a working electrode. Interleukin 6 (IL 6) receptor was employed as a biorecognition molecule to detect IL 6 antigen. Electrochemical and morphological characterizations of biosensor construction stages were performed by electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV), and atomic force microscopy (AFM) and scanning electron microscopy (SEM), respectively. In order to obtain a sensitive immunosensor, analytical parameters were optimized.

Keywords: immunosensor, prostate cancer, electrochemical impedance spectroscopy.

#### **Introduction**

Prostate cancer (PCa) is a dangerous disease which forms in the outermost part of the prostate owing to mutation of DNA. PCa leads to uncontrolled proliferation, differentiation and it causes metastasis in different organs. Moreover, PCa is a major cause of death in men aged between 50 and 80 years [1].

Biomarkers are bioindicators that identify the biological conditions in human body. PCa biomarkers are typically measured in blood or serum and urine samples. Interleukin (IL)-6 is a pro-inflammatory cytokine and it is expressed in prostate tumors. It has role in regulation of proliferation, apoptosis, angiogenesis, and differentiation. Therefore, IL 6 is an important biomarker for prostate cancer [2]. For the determination of tumor biomarkers, enzyme linked immunosorbent assay (ELISA), radioimmunoassay, chemiluminescence immunoassay, fluoroimmunoassay and electrophoretic immunoassays have employed. Although ELISA is an effective way for the determination of IL 6 antigens, it demands a long time to carried out the ELISA procedure and high-cost device [3].

Electrochemical impedance spectroscopy is a label-free and effective way for the determination of several analytes. The specific biorecognition between biomolecules and target analyte causes changes in capacitance or charge-transfer resistance. This change can be measured easily [4].

#### **Materials and Method**

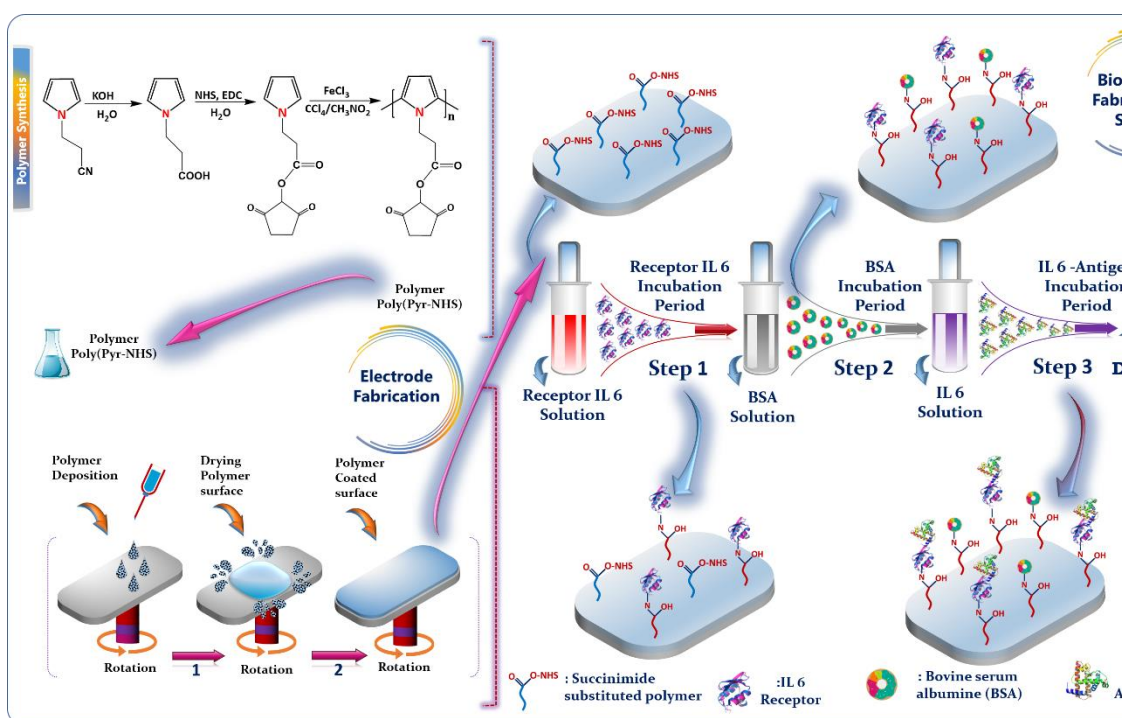
Indium tin oxide (ITO) electrodes (0.5 cm×2 cm, 60 Ω/cm<sup>2</sup>), IL 6 receptor, IL 6 antigen were purchased from Sigma-Aldrich. IL 6 receptor, recombinant human IL 6 antigen and bovine serum albumin (BSA) solutions were prepared by employing phosphate buffer saline (50 mM, pH 7.4). Ferricyanide/ Ferrocyanide probe solution prepared in phosphate buffer (1 M KCl, 5 mM [Fe(CN)<sub>6</sub>]/[Fe(CN)<sub>6</sub>]<sup>-3/-4</sup>).

The electrochemical analyses were performed in a traditional three-electrode cell composed of a single-use ITO sheet, a platinum wire and Ag/AgCl. Electrochemical analyses were measured in the ferricyanide/ferrocyanide probe solution and a Gamry Potentiostat/Galvanostat (Reference 1000, Gamry Instruments, USA) was employed for EIS and CV analyses. The potentials of CVs were set from -0.5 V and 1 V at a scan rate of 100 mV/s. EIS frequencies range were set from 0.5 to 50000 Hz.

## Results and Discussion

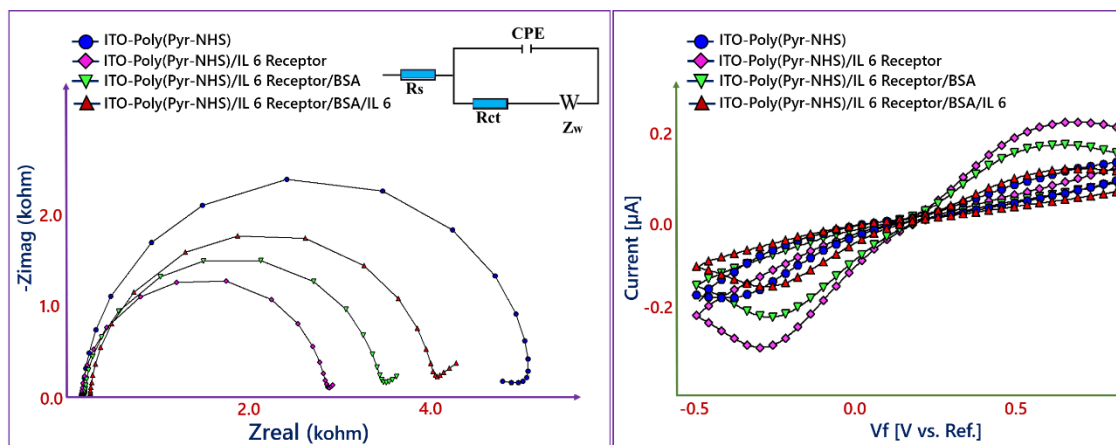
In this study, we fabricated an ultrasensitive, low-cost and simple impedimetric biosensor for the determination of IL 6. IL 6 receptors were immobilized onto a single-use ITO sheet covalently after coating with Poly(Pyr-NHS) polymer. The specific bio-interaction between IL 6 receptor and IL 6 antigen was confirmed EIS and cyclic voltammetry measurements. The step-by-step modification process was also investigated by scanning electron microscopy and atomic force microscopy analyses. The proposed immunosensor was successfully used in serum samples for IL 6 antigens determination.

The proposed biosensor fabrication process is schematically displayed in Scheme 1. Firstly, succinimide substituted polypyrrole polymer was coated by employing a spin-coater. Then, polymer coated ITO sheets were dipped in receptor IL 6 solution for 60 min, thus IL 6 receptor was attached onto ITO sheet via covalent binding. The free succinimide groups of polymer were blocked BSA solution for 60 min to prevent non-specific immobilization. After that, the fabricated immunosensor was ready to analysis IL 6 antigen.



**Scheme 1.** Schematic presentation of immunosensor

EIS is a successful technique to follow modifications onto the electrode materials and they usually utilized for electrode surface characterization and variations formed onto immunosensor surfaces. Figure 1 shows EIS spectra and CVs of fabrication process. As seen in figure 1, after each modification step, the EIS and CV responses were varied. The EIS and CV results proved the successful fabrication.



**Figure 1.** EIS and CV responses obtained during immobilization steps.

## Conclusion

An impedimetric immunosensor was constructed for IL 6 antigen detection by employing a single-use ITO sheet. By using spin-coating method, Poly(Pyr-NHS) polymer coated on the ITO sheet surface and used for covalent coupling of IL 6 receptors. This immunosensor had an excellent sensitivity for IL 6 antigens.

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## PP2- Adiponectin determination by a high sensitive and cost-effective disposable biosensor

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**ABSTRACT-** In some developed and developing countries, obesity is an important health problem and paves the way for many health problems such as Type 2 diabetes, cardiovascular diseases, weakened immunity and joint disorders. Adiponectin is a multifunctional peptide hormone released into the circulation from fat cells. It is one of the most genetically encoded proteins found in adipose tissue. It is a molecularly collagen-like plasma protein. Adiponectin levels were found to be low in obesity, diabetes mellitus and coronary artery disease in clinical studies. Early detection of adiponectin level decrease with a biosensor may be a new target for the prevention and treatment of cardiovascular diseases, impaired immunity, joint disorders and especially obesity-related pathologies. In this study, a disposable ITO thin film based electrode was used as working electrode for biosensing the interaction between adiponectin antigen and anti- adiponectin antibody. In this study, (3-Glycidyloxypropyl) trimetoxysilane (3-GOPS) was also utilized for electrode modifying so as to obtain self-assembled monolayers. Electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV) analyses were employed for monitoring of electrochemical properties of the immunosensor.

**Keywords:** Disposable biosensor; ITO; Adiponectin; 3-GOPS; Obesity

### 1. INTRODUCTION

Obesity, which is among the diseases of the modern age, is a serious problem threatening human life. It has been found to be one of the causes of human life, lifestyle and diseases such as cardiovascular system, respiratory system, hormonal system, and digestive system<sup>1</sup>. Adiponectin plasma levels are particularly low in people with visceral obesity.

ITO is one of the most popular transparent conductive oxides. It is a transparent material with excellent electrical properties that makes it very popular for various electronic applications. Moreover, it has been tested to have unique advantages such as wide working range, very useful surface, adhesion strength and material durability. An ITO-PET electrode is very cost-effective compared to other conventional working electrodes (Pt, Au, Ag etc.), and is therefore a prioritized electrode. The present study utilizes a novel immunosensor based on an ITO-PET disposable electrode using a 3-GOPS agent. This material is both an excellent silane agent and is used without a cross-linked material for the immobilization process.

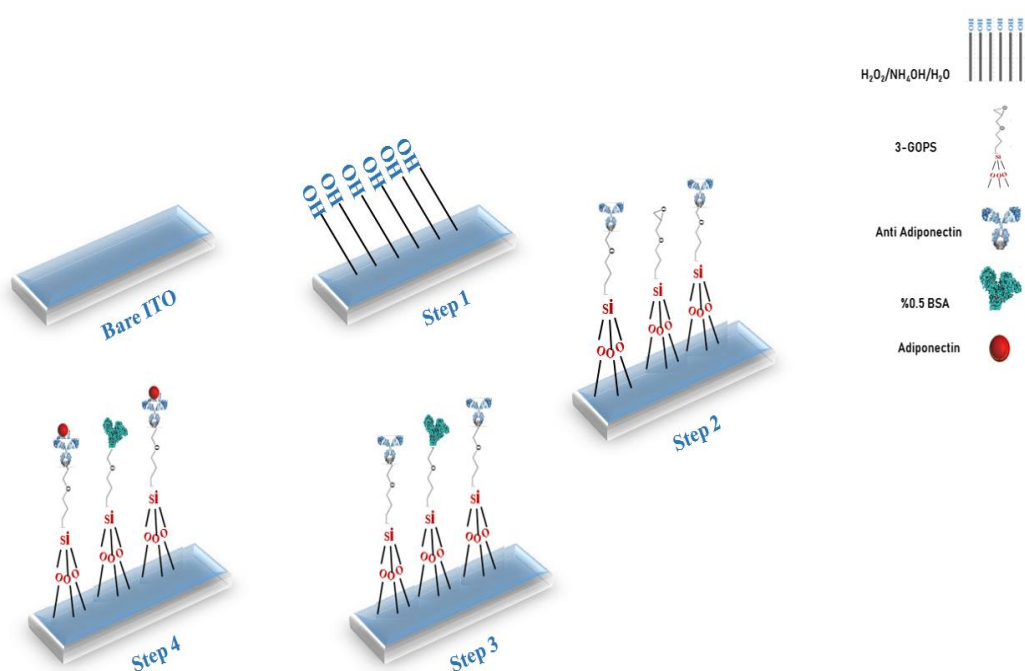
### 2. MATERIALS AND METHODS

#### 2.1. Chemicals and apparatus

All reagents, which are utilized in the study, were of analytical grade and were obtained from Sigma-Aldrich (St. Louis, M.O., USA). ITO-coated Polyethylene Terephthalate (PET) films, which have a surface resistivity of 60  $\Omega$ /square and the transmittance 550 nm (>79%), and biorecognition elements were obtained from Sigma-Aldrich. Anti-adiponectin antibody, adiponectin and bovine serum albumin (BSA, 0.5%) were prepared by generated by using a Gamry potentiostat/galvanostat (Reference 600, Gamry Instruments, Warminster, PA, USA) interfaced with a PC via an EChem Analyst and the electrochemical measurements were performed in 50mM PBS solution (pH 7.4) containing 0.1M KCl 5mM  $[\text{Fe}(\text{CN})_6]^{3-/4-}$  as a redox probe.

#### 2.2. Fabrication of ITO-based immunosensor

The ITO electrodes were prepared by ultrasonic washing in an acetone, soap solution and ultra-pure water for 10 min each. Then they were dried at room temperature under argon gas for future use. Afterwards electrodes were dipped into a mixed solution containing ultra-pure water/hydrogen peroxide/ammonium hydroxide (5:1:1, v/v) for 90 min to obtain hydroxylated active ITO surfaces in dark conditions. Then, they were rinsed carefully with ultra-pure water and dried under argon gas. The activated ITO thin films were immersed 0.5% GOPS solution prepared in toluene overnight. The ITO/GOPS thin films were then rinsed with toluene and ultra-pure water to remove non-bonded 3-GOPS silane molecules from PET film surface and dried under argon gas. After that GOPS modified ITO thin films immersed into solution containing anti-Adiponectin antibodies. For immobilization of anti-Adiponectin antibodies, the amount of antibody used was optimized. The ITO/ GOPS /anti-Adiponectin electrode washed thoroughly with ultra-pure water. At the last step of fabrication, BSA (0.5%) was used to block free silane ends of modified ITO. The fabrication process of impedimetric immunosensor was illustrated in Scheme 1. At the end of these steps our immunosensor was ready for Adiponectin detection. The fabrication process of impedimetric immunosensor was illustrated in Fig 1.



**Figure 1.** Schematic fabrication process of impedimetric immunosensor

### 2.3. Electrochemical measurements

All electrochemical experiments were performed with a potentiostat/galvanostat and the electrochemical features of the modified ITO electrodes were monitored by using CV and EIS. The operation parameters for CV measurements are as follows; scan rate: 100 mVs<sup>-1</sup> and step size 10 mV, for EIS measurement; initial frequency: 50000 Hz, final frequency: 0.05 Hz, points:5, for SFI measurements; frequency: 20 Hz, repeat time 0.01 min, total time 0.5 h.

## 3. RESULTS AND DISCUSSION

In this study, a novel, low cost, easily prepared disposable biosensing system for the determination of adiponectin was successfully developed. In this work, silane modified ITO electrode was utilized as a biosensing layer for the development of impedimetric immunosensor. The fabrication process of impedimetric immunosensor was illustrated in Fig 1. The stepwise biosensor fabrication procedure was monitored by EIS and CV, and the resulting Nyquist plots and cyclic voltammograms are illustrated in Fig. 2 and Fig. 3. Step 1 (in the first

step), which is an insulating material, has a small semicircular diameter due to its conductivity when immersed in a solution prepared with  $\text{H}_2\text{O}_2$ ,  $\text{NH}_4\text{OH}$ , and  $\text{H}_2\text{O}$ . The circle grew as the conductivity on the surface decreased due to the 3-GOPs silane agent that binds to the surface at Step 2. In Step 3, it was observed that the conductivity of the surface decreased with The Binding of anti-adiponectin. Similarly, the increased nonconducting property in Step 4 blocked the electron transfer between the electrode and the electrolyte solution. Thus, the diameter of the Nyquist graph was increased at each digit. These results showed that ITO was successfully immobilized on the electrode. It can detect adiponectin antigen in a linear range of 25 pg/mL–2500pg/mL.

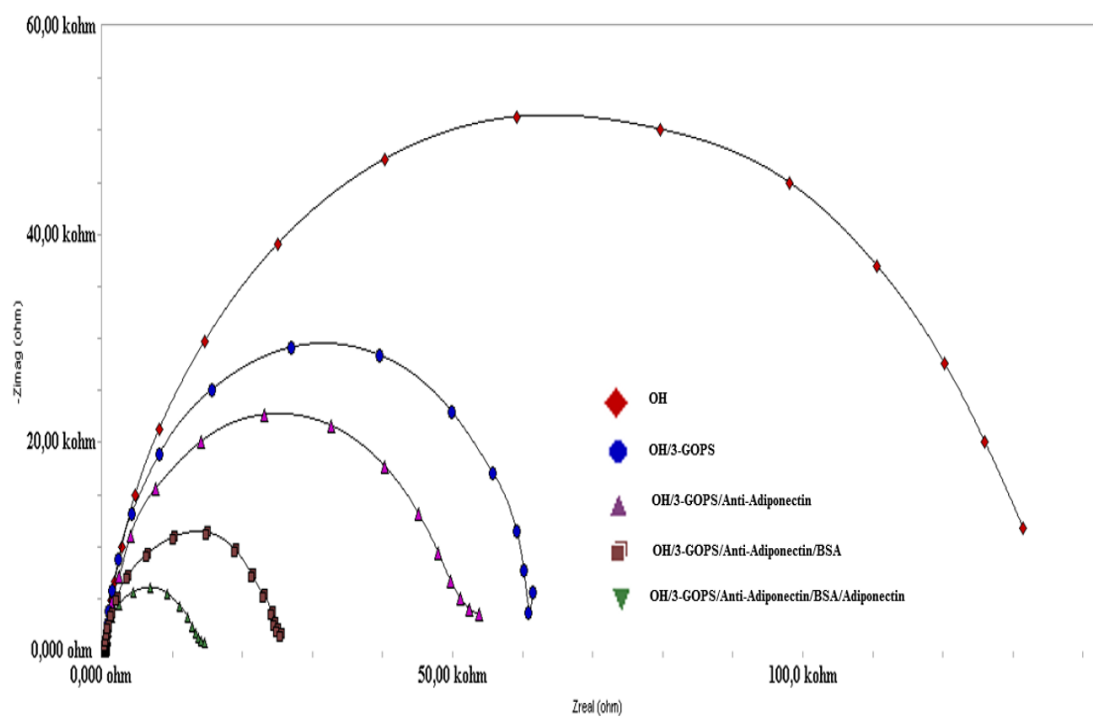


Figure 2. Nyquist plots for immobilization steps

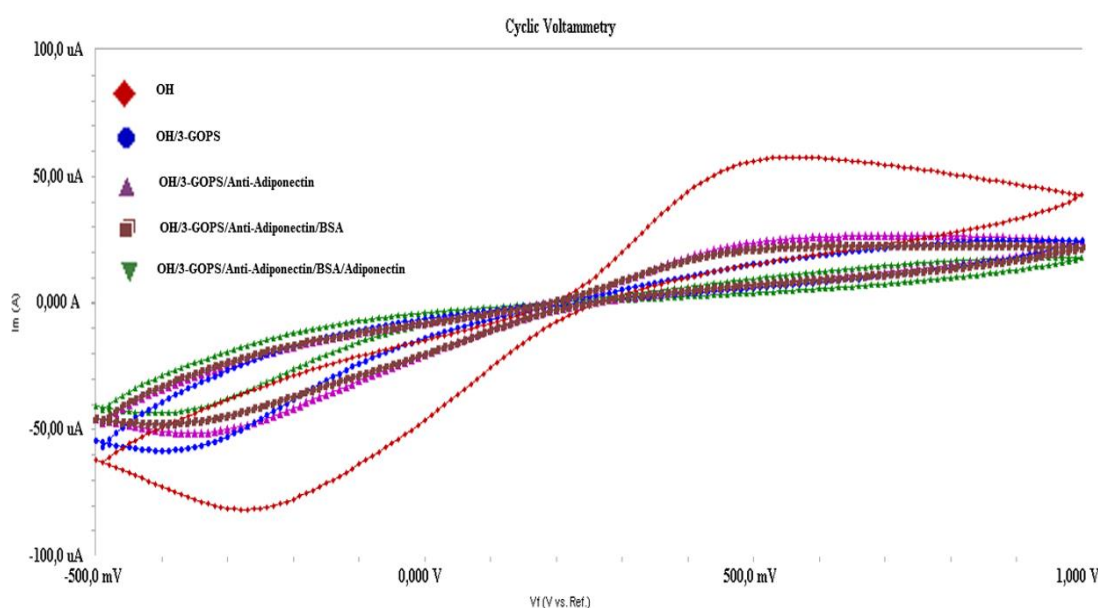


Figure 3. Cyclic voltammograms for immobilization steps of Anti-Adiponectin

#### 4. CONCLUSION

Anti-adiponectin antibodies were effectively immobilized on the ITO electrode surface owing to the unique properties of GOPS, which made the label-free detection of adiponectin feasible. The proposed immunosensor has good repeatability, reproducibility and storage stability as well as acceptable precision.

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### PP3- Determination of Some Toxic Volatile Organic Compounds in Air Samples by Lab-made Carbon Sorbent

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**Abstract-** Monitoring of volatile organic compounds (VOCs) in air samples have attracted considerable interest due to their hazardous effect to human and also to environment in recent years. In this work, a cheap method was developed for the analysis of ethyl benzene, p-xylene, m-xylene, o-xylene, styrene, p-dichlorobenzene, nonanal and benzaldehyde in air samples. For this purpose, the filter coffee wastes were used as a starting material for the preparation adsorbents. Four different adsorbents were prepared from this material by using different processes. Then adsorption tubes were prepared from these adsorbents for the sampling of VOCs in air samples. For this purpose, suitable amount of adsorbent was tightly filled inside of the glass tube. Then, targeted VOCs in air samples adsorbed inside of this adsorption tubes. After adsorption process, adsorbed VOCs eluted by carbondisulphide (CS<sub>2</sub>) and analysis of VOCs in CS<sub>2</sub> phase were performed by gas chromatographic technique. The results were also compared with the commercial activated carbon and the best results were obtained with 200-1 coded adsorbent. Optimization of the proposed method with 200-1 coded adsorbent was performed under optimize conditions and calibration curves of the each targeted VOCs were performed. The limit of detection (LOD), limit of quantification (LOQ), linear range, intraday repeatability and interday reproducibility of the method for each compounds were calculated and acceptable results were obtained. The proposed method was successfully applied for the analysis of targeted compounds in different air samples.

**Keywords:** Volatile organic compounds, air samples, lab-made carbon sorbent, pyrolysis

#### 1. Introduction

Carbon-based volatile compounds are called Volatile Organic Compounds (VOC) and some of them have great harmful effects both on human health and on the environment (Lee vd., 2001; Norback vd., 1995; Bayır, 2013). VOCs at room temperature are completely in the vapor phase and they have a vapor pressure of  $> 10$  Pa at  $25^{\circ}\text{C}$  and a boiling point  $<260^{\circ}\text{C}$  at standard atmospheric pressure (Koppmann 2007). Due to their low boiling point, they are found in the indoor air as a gas. To examine the health effects of VOCs and to measure regulatory limit values and the origin of these are essential. VOC concentration in air varies according to time and place. Therefore, effective monitoring of air samples is very important.

VOCs spread from the various sources to the atmosphere. While the VOC resources inside the building are generally used materials, the resources outside the building are generally production, loading, transportation and unloading operations. The main sources of outdoor air are motor vehicles, exhaust emissions, chemical producing industry and power plants. In general, VOC resources are grouped into two groups, anthropogenic resources (resulting from human activities) and natural resources (Gaga vd., 2008).

Analysis of air samples is generally more difficult and non-standardized methods compared to those of food, water and soil. Therefore, monitoring air quality in people's living spaces is a very important issue. After a sampling of VOC in air samples generally, there need expensive and complicated instruments such as thermal desorption to unite combined with gas chromatography-mass spectrometry (GC-MS) for analysis. In the active and passive samplings, VOCs are analyzed by adsorbing on an adsorbent first, then desorbed with a suitable solvent or thermally and then analyzed by GC. In the sampling step, VOCs are actively or passively adsorbed on a suitable adsorbent such as activated carbon (Bayır, 2013). In the active sampling, VOCs are passed through the adsorbent by a gas pump. In passive sampling, VOCs are transferred on the adsorbent by diffusion (Yeşilyurt ve Akcan, 2001).

In this study, it was aimed to prepare a lab-made carbon sorbent from carbon wastes as adsorbent and the analysis of some VOCs in air samples by using these sorbents.

#### 2. Experimental

**2.1. Apparatus and Reagents:** In this project, analytical reagent grade n-pentane (Sigma Aldrich), dichloromethane (Sigma Aldrich), ethyl benzene (Sigma Aldrich) p-xylene (Sigma Aldrich), m-xylene (Sigma Aldrich), o-xylene (Sigma Aldrich), styrene (Sigma Aldrich), p-dichlorobenzene (Sigma Aldrich), nonanal (Sigma Aldrich), benzaldehyde (Sigma Aldrich), benzene (Sigma Aldrich), tetrachloroethylene (Sigma Aldrich), toluene (Sigma Aldrich), decanal (Sigma Aldrich) and acetone (Sigma Aldrich) were selected as targeted VOCs. Analytical reagent grade hydrochloric acid (Sigma Aldrich), carbon disulfide (Sigma Aldrich) and methanol (Sigma Aldrich) were also used.

VOC analysis was carried out with the Agilent 7820A gas chromatography equipped with a flame ionization detector (GC-FID). Air samples were passed through adsorption tubes with a Gillian air pump. Gas flow rates are calibrated with the Gillian Go-call instrument. Adsorbents were prepared in a lab-made 316 type steel pyrolysis reactor.

**2.2. Preparation of Lab-Made Sorbent:** Coffee waste was pyrolyzed at a nitrogen atmosphere at 300°C first. Then the activation of the adsorbents was done according to the chemical activation method with  $\text{ZnCl}_2$  (Serkan Timur et al., 2010).

The hydrothermal carbonization of the raw hydro coal, filter coffee waste at 200°C is carried out. The hydrothermal carbonization study was carried out in a 450 mL high temperature and pressure-resistant steel reactor. After the mixture of 30 grams of dry coffee waste and 150 mL of water were placed and mixed at 100 rpm. The pressure of the system was read with a manometer connected to the reactor with leak-proof connections. In order to remove the air inside the reactor, nitrogen gas was connected to the inlet valve, 10 bar nitrogen was pressed and discharged twice and 10 bar  $\text{N}_2$  was left in the reactor as the initial pressure. Reactor 5 °C / min. After being brought to the desired temperature with the heating rate, it was left for 30 minutes. At the end of the experiment, the reactor temperature was reduced to room conditions with the maximum cooling capacity. The reactor content was filtered under vacuum and hydrocarbon (MP-HC-200-30) was separated and dried at 105 °C. In order to remove volatile organic matter, the hydro-coal fixed bed was first subjected to 500°C for 1 hour in the pyrolysis reactor.

Subsequently, the chemical activation process was initiated with KOH to increase the porosity and surface area of the hydrocarbon obtained. 20 g of sorbent and of 20 g of KOH were impregnated (impregnated) with the ratio of KOH mass 1: 1. After the mixture was dried at 105°C for 1 day, it was activated under nitrogen gas at 800°C. The mixture taken from the reactor was boiled with 10% HCl solution for 1 hour, then washed with distilled water until the chloride ion was removed. The resulting activated hydrocarbon was dried overnight at 105°C (HC-1-800-2).

**2.3. Preparation of Adsorption Tube:** Adsorption tubes are prepared from a borosilicate glass pipe with a diameter of 1 cm and a height of 15 cm. One end of the pipe is thinned and 0.0500 g glass wool is filled tightly. Then, 0.2000 g of adsorbent is placed on the glass wool and 0.0500 g of glass wool is placed on it again and the adsorbent is pressed well. Then 5 minutes of pure nitrogen was passed through and the impurities that could be found in it were removed. Finally, the two ends of the ready-made adsorption tubes were tightly sealed from the atmosphere.

**2.4. Air Sampling:** The active sampling method was used in the experiment (Wang, et al., 2016). Air samples and standards were passed through the gas inlet port of the adsorption tube by means of a pump at a flow rate of 250 mL/min (Figure 1). Then, after sampling a total of 0.5 L air sample, the entrance and exit of the glass tube were closed with a stopper and isolated from the outer atmosphere. Finally, desorption of the VOCs on adsorbent was performed by passing 1 mL of  $\text{CS}_2$  through the adsorption tube.  $\text{CS}_2$  desorption solution containing target VOCs was taken into 1.5 mL gas chromatography vials and chromatographic determination of VOCs was done by liquid injection.

**2.5. Gas Chromatographic Analysis Conditions:** GC condition were summarized at Table 1.

### 3. Results and Discussion

First of all, the adsorption of target VOCs were tested. The adsorbent is only adsorbed ethylbenzene, p-xylene, m-xylene, o-xylene, styrene, p-dichlorobenzene, nonanal and benzaldehyde. Then the sampling parameters such as sampling flow rate, sample volume etc. were optimized.

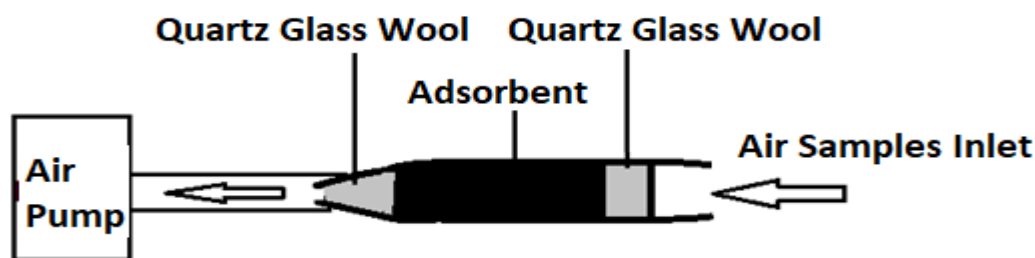


Figure 1. Air sampling of targeted VOCs

**3.1. The effect of Sampling low rate:** The effect of sampling flow rates was studied between 100-300 mL/min flow rates. The highest peak area was obtained at 250 mL/min flow rate and optimum flow rate was selected as 250 mL/min for further analysis.

Table 1. GC analysis conditions

Parameter	Value
Injection volume	1 $\mu$ L
Injection Modu	Splitless
Carrier gas flow rate	1.5 mL/min
Injection Temperature	200°C
Oven Program	40 °C wait 6 min. 2°C/min to 65°C 20°C/min to 100°C wait 30 min
Total Analysis Time	50,25 min
Column Type	Restek 10999, 60 mx250 $\mu$ m x 0.4 $\mu$ m

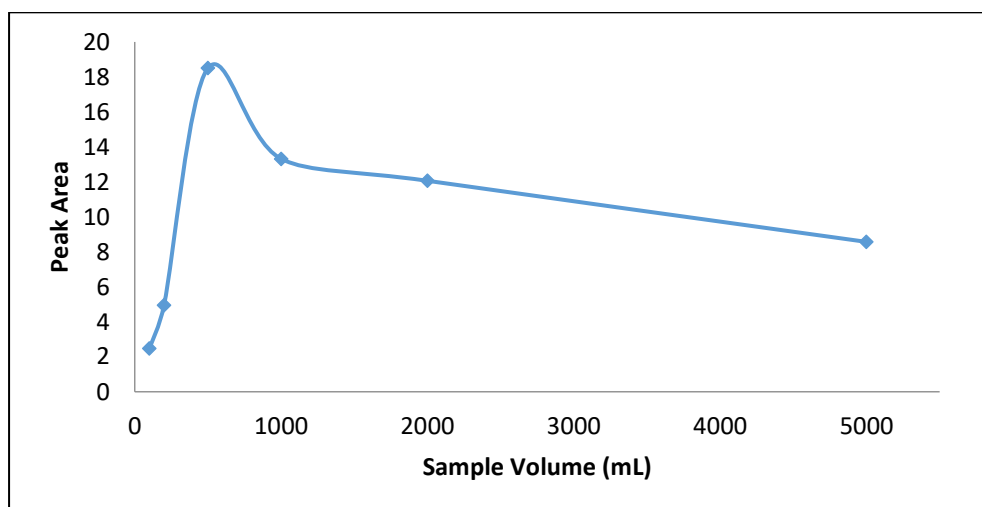


Figure 2. The effect of sample volume

**3.2. The effect of Sample Volume:** The effect of sample volume was studied between 100-5000 mL. The all compounds showed similar adsorption properties as Figure 2. After 500 mL a small decrease was observed because of the desorption of the analytes at higher adsorption time. Therefore 500 mL was selected as optimum flow rate.

**3.3. Analytical Figure of Merits: Calibration** curves of the targeted compounds were constructed and under optimized conditions analytical figure of merits for the proposed method were listed at Table 2.

**3.4. Application of the Real Samples:** The developed method was successfully applied to 4 laboratories and 1 outdoor atmosphere samples in Ege University, Faculty of Science, Department of Chemistry. The measured substance amounts are presented in Table 3. Only styrene and nonanal compounds were detected in all samples and their levels were below the limits specified for short-term exposure (STEL).

**Table 2.** Analytical figure of merits for the targeted compounds.

Analyte	Calibration Curve	LOD (mg/m <sup>3</sup> )	LOQ (mg/m <sup>3</sup> )	Linear Range (mg/m <sup>3</sup> )	Interday reproductibility (%)	Intraday reproductibility (%)
ethylbenzene	$y = 0,0642x + 2,6499$	0,25	0,85	0,85-3100	3,8	5,8
P-xylene	$y = 0,2513x + 14,8$	0,49	1,59	1,59-2483	7,1	12,5
M-xylene	$y = 0,2438x + 11,117$	0,58	1,89	1,89-3104	3,0	6,2
O-xylene	$y = 0,3783x - 3,2649$	0,36	1,21	1,21-3166	8,9	12,2
styrene	$y = 2,2759x + 0,3675$	0,19	0,62	0,62-20	5,9	10,4
p-dichlorobenzene	$y = 0,1317x + 3,6674$	1,05	3,49	3,49-1670	30,5	26,1
Nonanal	$y = 0,1254x - 0,7193$	1,10	3,68	3,68-604	10	13,6
benzaldehyde	$y = 0,9469x - 16,115$	0,14	0,46	0,46-749	3,4	14,4

**Table 3.** The level of targeted VOCs in different indoor atmosphere.

Analyte	Lab 1 (mg/m <sup>3</sup> )	Lab 2 (mg/m <sup>3</sup> )	Lab 3 (mg/m <sup>3</sup> )	Lab 4 (mg/m <sup>3</sup> )	Outdoor (mg/m <sup>3</sup> )	STEL (mg/m <sup>3</sup> )
Etilbenzen	<LOD	<LOD	<LOD	<LOD	<LOD	884
P-ksilen	<LOD	<LOD	<LOD	<LOD	<LOD	442
M-ksilen	<LOD	<LOD	<LOD	<LOD	<LOD	442
O-ksilen	<LOD	<LOD	<LOD	<LOD	<LOD	442
Stiren	6,53	7,81	7,91	6,71	4,32	425
P-diklorobenzen	<LOD	<LOD	<LOD	<LOD	<LOD	306
Nonanal	108,28	119,24	124,36	136,5	89,32	1131
Benzaldehyit	<LOD	<LOD	<LOD	<LOD	<LOD	40

**4. Discussion:** In this study, a simple, cheap, sensitive method was developed for the analysis of some VOCs in air samples. The main advantage of the proposed method is the preparation of lab-made adsorbent from coffee wastes. A cheap and effective alternative adsorbent was prepared against their commercial product.

**Acknowledgments:** This study was supported by Ege University Scientific Research Project (17-FBE-012)

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## PP4- Investigation of Potentiometric PVC Membrane Electrodes Containing Different Ionophores

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The main selective components of ion-selective membranes are lipophilic complexing agents called ionophores. A great variety of analyte selectivity can be achieved with the appropriate ionophore selection. For this purpose, finding new ionophores and determining polymeric membrane properties and potentiometric behaviors have become the main research topics for the development of new membrane sensitive potentiometric electrodes.

In this study, the potentiometric behaviors of solid-state contact PVC membrane potentiometric electrodes, which are sensitive to different species in environmental and biological samples, were investigated.

**Keywords:** *Ion-selective electrode, ionophore, ions*

### INTRODUCTION

A large number of examples can be cited where ion chromatography(IC) finds application worldwide; such as determination of ions in environmental and biological compartments. The technique is now applicable to the determination of a wide range of solutes in diverse sample matrices and food analysis, although environmental studies continue further to be the largest application area of IC<sup>1</sup>.

However, low detection limits and high measurement sensitivity to ions combined with a small sample volume are needed in a number of applications of ion chromatography. Much attention has been paid to optimizing these factors and various analytical methods have been developed<sup>2-3</sup>. Potentiometric ion selective electrodes (ISEs) are currently the most widely used chemical sensors in medical, environmental, and industrial analysis, as they offer many advantages such as fast response, simple analytical procedure, and relatively low cost.

### MATERIALS AND METHOD

The construction of all solid-state contact PVC membrane electrodes without an inner reference solution was carried out, as described by Isildak and colleague<sup>4,5</sup>. The sensing membrane consisted of 31.0 weight % polyvinylchloride (PVC), 2.0 wt.% as active ligands (ionophore), 66 wt.% as plasticizers after evaporation of THF at room temperature open to air for 4 h. To reduce the membrane resistivity and cationic selectivity, in the case of neutral carrier electro-active materials, the membrane contained 1.0 wt. percentage potassium tetrakis (p-chlorophenyl) borate (KTpClPB). The epoxy resin mixture used to bind the graphite in preparing the internal conducting support of the electrode was made from epoxy and hardener in tetrahydrofuran solvent in the proportions of 1 : 0.5. in the proportions of 1 : 1. Electrical connection was made to copper rod inserted into a hole in the epoxy-graphite conductive support. When not in use for a long time the all solid-state contact tubular PVC-matrix electrodes were stored dry after washing with deionized water.

### RESULT AND DISCUSSION

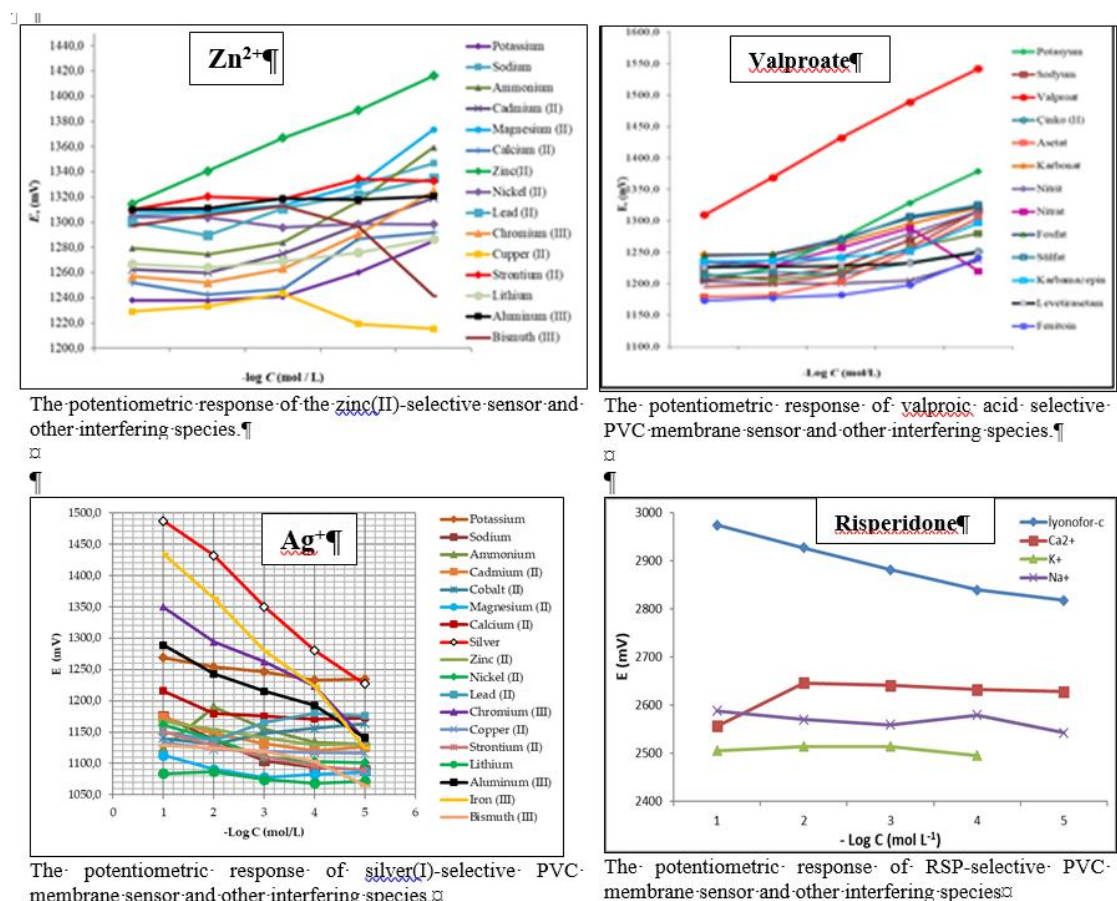
Potentiometric sensors has been developed through immobilizing a membrane matrix on all-solid-state contact. It was for sample monitoring without any reagent consumption and at a very short analysis time. The all-solid-state contact mixture electrode of graphite-epoxy resin was prepared in suitable ratio where the sensing membrane adhered to the surface of all-solid-state contact electrode without an internal reference solution. As a result, lifetime of the electrode increased without any loss in its potentiometric response property and thus electrode became more stable.

All-solid-state membrane electrodes are cost effective because they are very easily prepared in miniaturized construction.

Potentiometric measurement cells used in this study are displayed schematically as follows:

Conducting wire |Solid-state contact |sensitive membrane |Test solution || External ref. electrode

The potentiometric behavior of all solid state PVC membrane electrode is shown in Figure 1. Table 1 shows linear coefficients and linearity of determination ( $r^2$ ) for any of the ions studied in the concentration ranges.



**Figure 1.** Potentiometric responses of the developed sensors

**Table 1.** The linear coefficients and linearity of all solid state PVC membrane electrode

	LINEARITY	$r^2$
<b>Zinc (II)</b>	$E = 25.15 (-\log[\text{Zn}^{2+}]) + 1290.1$	0.9991
<b>Valproate</b>	$E = 53.50 (-\log[\text{VPA}]) + 2923.3$	0.9987
<b>Silver (I)</b>	$E = 51.57 (-\log[\text{Ag}^+]) + 1381.2$	0.9986
<b>Risperidone</b>	$E = -40.11(-\log[\text{RSP}]) + 3008.5$	0.9848

## CONCLUSION

A novel potentiometric sensors based on all solid-state contact is developed for fast and simple analysis of different samples. The all solid-state PVC membrane electrodes could easily be prepared and economic. Its response times is considerable. Additionally, PVC membrane sensors can be miniaturized and used in mobile. An alternative method to the literature has been introduced for the analysis of these ions in biological and environmental samples.

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## PP5- SPECTROPHOTOMETRIC DETERMINATION OF CHROMIUM IN AQUEOUS SOLUTION ON COAL ASH

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**Abstract-**Water is one of the most important substances on earth for life. Heavy metal such as chromium is a big problem for water pollution. Therefore, a study of the removal of Cr from wastewater is of considerable significance from an environmental point of view. There are approximately 82 million of tons asphaltites reserves in Şırnak, East Anatolia of Turkey. The present study was investigated to employ ashes of Şırnak BCW (burned coal waste) as an ion exchange in removal of important toxic metal, Chromium, in high yields by adsorption. The ion exchange characteristics of chromium onto BCW from aqueous solution were investigated with respect to the changes in pH of solution, adsorbent dosage, initial metal ion concentration, contact time and temperature of solution. We show that BCW that was used; as low cost, readily available ion exchange; can be used for removal of chromium from waste waters. The amount of Chromium left after adsorption process was determined by UV-VIS Spectrophotometer.

**Key words:** Chromium, Coal ash, Ion-exchange, UV-VIS spectrophotometer, Turkey

### Introduction

Water pollution is a major problem for health. Chromium (Cr) is a major pollutant for environment, usually as a result of some industrial pollution including tanning factories, steel works, industrial electroplating, wood preservation, etc. and artificial fertilizers. Ingestion of chromium may pose great risks to human health. Because, Chromium trace element is toxic to live organisms if present in excessive amounts.

Direct determination of the chromium by instrumental techniques including UV-Visible Spectrophotometer, atomic absorption spectrometry (AAS), inductively coupled plasma-atomic emission spectrometry (ICP-AES), inductively coupled plasma-mass spectrometry (ICP-MS), etc. is suitable.<sup>1,2</sup>

In the present study, a sensitive and simple method for the adsorption and determination of Cr in water samples using a coal ash. Parameters influencing the analytical performance, pH of solution, adsorbent dosage, initial metal ion concentration, etc. were investigated.

### Materials and Methods

**Adsorbate: A solution of metal:** The Cr metal solution was prepared from K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> (Merck, USA) salt.

**Adsorbent:** The adsorbent, coal ash was collected from Şırnak, Turkey. It was sieved to –140 mesh size and dried at 383 K and finally kept in bottles.

**Reagents:** All chemicals used in this work, were of analytical reagent grade and were used without further purification. Double deionized water (Milli-Q Millipore 18.2 MΩ cm<sup>-1</sup> resistivity) was used for all dilutions.

**Procedure:** Coal sample was obtained from Şırnak, SE Anatolia region of Turkey (37°36'0"N, 43°10'12"E). After oven drying at 105 °C, sample was crushed by a jaw breaker (Retsch BB 1/A) and ground in a rotor beater mill (Retsch SRZ). The particle sizes of the samples were less than 250 µm. Finely ground powdered coal samples were dried in an oven at 105°C until constant weight was reached, and stored over silica gel in a desiccator.<sup>3</sup>

**Apparatus:** The equilibrium concentrations of Cr concentration were determined by UV-VIS 1800 double beam spectrophotometer (Shimadzu Ltd.) Cr gives the maximum absorbance value at 540 nm in the wavelength range of 200–600 nm.

### Results and Discussion

The pH of a solution is an important parameter in adsorption process because of the pH dependencies of the complexation reactions or electrostatic interactions in physisorption processes at the silanol groups on the surface of adsorbate. We show that the effect of pH on the adsorption of Cr ions onto BCW in Fig.1.<sup>3</sup>

The effect of initial concentration on removal of Cr ions by BCW is shown in Fig.2. The effect of adsorbent dosage on the removal of Cr ions was studied and the results are presented in Fig. 3. The effect of contact time on Cr ions uptake capacity by BCW was shown in Fig. 4.

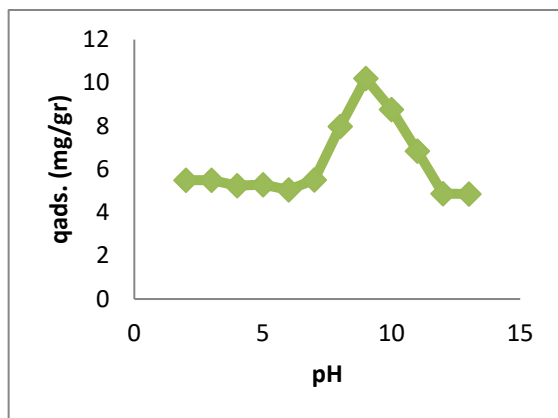


Fig. 1. Effect of pH

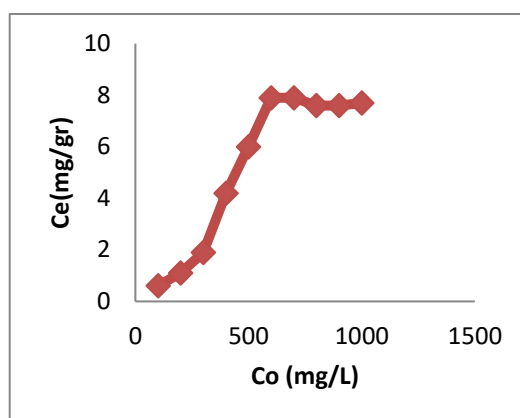


Fig. 2. Effect of initial concentration

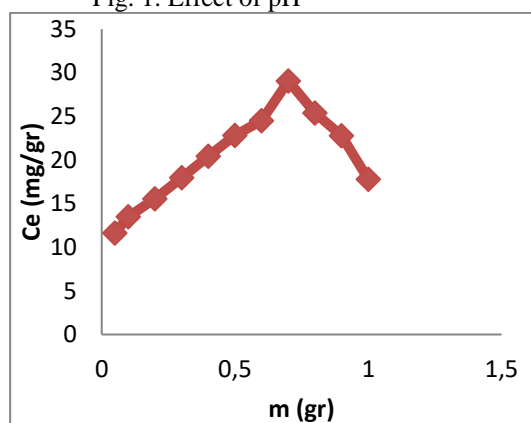


Fig. 3. Effect of adsorbent dose

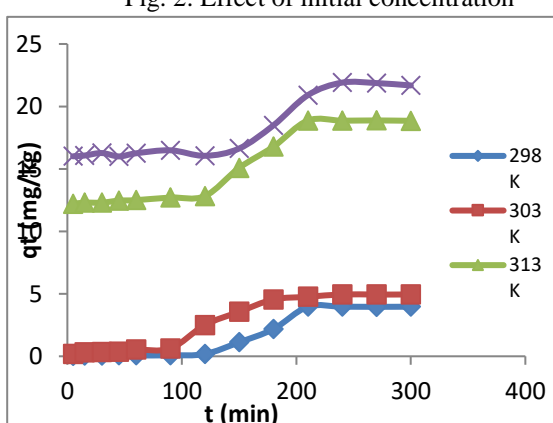


Fig. 4. Effect of contact time and temperature

The removal process increases along with the Cr concentration till  $600 \text{ mg L}^{-1}$  in the effect of initial concentration on removal of Cr ions by BCW. After then, it remains almost stable. At high adsorbent dosage,  $m > 0.7 \text{ g}$ . Later 90 min., a large amount of Cr ions with increasing contact time were removed in the first 240 min. Equilibrium was reached in 4 h for Cr.

### Conclusion

The determination of chromium in water samples was successfully performed in the present study by UV-VIS spectrophotometer. In batch ion exchange experiments were used ashes of Şırnak BCW (burned coal waste) as an ion exchange in removal of important toxic chromium metal. BCW is a low cost and readily available adsorbent and experiments have shown that coal ash adsorbed chromium ions. The ion exchange characteristics have been determined at contact time, temperature, pH, etc.

The results show that the proposed method could be applied for the determination of total chromium. This adsorption procedure is simple, facile, accurate and economic.

### Acknowledgements

The authors are grateful for the financial support of the Unit of the Scientific Research Projects of Dicle University (Project No: FEN.18.004).

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## **PP6- An immunosensor for analysis of suppression of tumorigenicity 2 protein and optimization experiments**

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Suppression of tumorigenicity 2 (ST2) protein has been associated with cardiovascular diseases which are very common in today. ST2 is part of the interleukin-1 family. It has two form, which one is soluble form (sST 2) and the other one is a membrane bound receptor form (ST2L).<sup>1</sup> In this study a disposable biosensor was fabricated for the determination of ST2. For this purposes indium tin oxide covered flexible material was used as a working electrode. After a series of cleaning, indium tin oxide polyethylene terephthalate (ITO-PET) sheets, electrode surface has become eligible for groups to bond. The surface of ITO was modified with 3-aminopropyltriethoxysilane for introducing amino ends onto the surface. Anti-ST2 proteins were immobilized on the electrode surface by using glutaraldehyde as a crosslinking agent. In each step of the immobilization process, cyclic voltammetry(CV) and electrochemical impedance spectroscopy(EIS) were used to characterize the modified electrode surface. To determine the optimal conditions of the modified sensor, a series of optimizations was made. 3-APTES, glutaraldehyde, anti-ST2, ST2 concentrations and glutaraldehyde, anti-ST2, ST2 incubation times were optimized.

**Keywords:** Suppression of tumorigenicity 2, ST2, Cardiovascular diseases, Immunosensor, ITO-PET

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**PP7- Highly sensitive, reproducible, and cost effective determination of Suppression of tumorigenicity 2 protein by a disposable immunosensor**

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ST2 (suppression of tumorigenicity 2) protein is a biomarker for cardiovascular disorders. ST2 protein could be expressed by cardiac cells in reaction to myocardial stress, point to researchers to a role in the cardiovascular system. <sup>1</sup>

In this study, it is aimed to design an ultrasensitive electrochemical immunosensor for ST2 detection. All electrode modification and immobilization processes were characterized by using cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS). To detect analytical characterization of 3-APTES modified immunosensor; repeatability, reproducibility, regeneration, linear range studies were performed. Also, the interactions between anti-ST2 antibody and ST2 antigen were observed by single frequency impedance technique (SFI). After all, we designed a disposable high sensitivity immunosensor. The linear range of the immunosensor was detected as 1 fg/mL - 1500 fg/mL. The immunosensor showed extremely wide linear calibration range and perfect analytical characteristics.

**Keywords:** Suppression of tumorigenicity 2, Cardiovascular diseases, Biosensor, ITO-PET, Electrochemical impedance spectroscopy (EIS)

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## PP8- Development of a Novel Glucose Biosensor Based on Nanodiamond Particles and Poly-L-Aspartic Acid Modified Glassy Carbon Electrode

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Development of practical and reliable methods for glucose determination is of great importance in diabetic health care, clinical diagnosis and treatment. Amperometric biosensors have been extensively investigated for the detection of glucose due to their simplicity, good selectivity and high sensitivity<sup>1,2</sup>.

In this study, glassy carbon electrode (GCE) was modified with nanodiamond particles (ND) and aspartic acid (Asp) was electropolymerized on this modified electrode. The electrode surface was further modified with glucose oxidase (GOX) enzyme for the construction of a glucose biosensor. The effect of modification materials on the electrochemical behavior of the modified electrodes was explored by cyclic voltammetry in  $K_3[Fe(CN)_6]/K_4[Fe(CN)_6]$  (1:1) solution including 0.10 M KCl in the potential range of  $(-0.80) - (+1.00)$  V at  $100 \text{ mV s}^{-1}$ . The results showed that electron transfer on the electrode surface increased in following order: unmodified GCE, ND/GCE and p-Asp/ND/GCE. Moreover, the peak to peak separation value obtained with the p-Asp/ND/GCE modified electrode was found to be lower than the peak to peak separation values obtained with ND/GCE and unmodified GCE which indicates that electron transfer at p-Asp/ND/GCE is easier than the other electrodes. Thus, the p-Asp/ND/GCE was selected as the optimum electrode configuration for the construction of the glucose biosensor. After the optimization of experimental conditions, analytical performance of the biosensor was investigated in terms of linear dynamic range, detection limit, sensitivity and stability. The GOX/p-Asp/ND/GCE biosensor showed good sensitivity, short response time and wide dynamic range for glucose determination. Therefore, our future study will be focused on the use of the biosensor for glucose determination in real samples.

Keywords: Glucose, Biosensor, Nanodiamond Particles, L-aspartic acid

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## PP9- A Rapid Genosensor that Enables E. coli and S.aureus to be Identified Using the DPV Method

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Bacteria are responsible for many infectious diseases that spread throughout the world through the consumption of contaminated water and food. For example, *Escherichia coli* (E. coli) is a Gram-negative and non-spore rod-shaped bacterium and it give rise to gastrointestinal problems as diarrhea, renal and hepatic troubles.<sup>1,2</sup> Another example, *Staphylococcus aureus* (S. aureus), is a Gram-positive and sphere-shaped bacteria which is frequently lead to lung-related infections such as, pneumonia, pericarditis, etc.<sup>3</sup>

Due to the lack of reliable and easy-to-use devices that can perform DNA detection in a short time on the market, new technologies for electrochemical genosensor designs<sup>4</sup> are constantly being developed as an alternative of other DNA analysis methods as traditional culturing methods, PCR and ELISA.

In this study, a genosensor concept which is applicable to medical/environmental fields was demonstrated for the detection of both E. coli and S. aureus based on synthetic DNA sequences related to these bacteria. First, the disposable pencil graphite electrode (PGE) was modified with the related capture probe which is specifically recognizes E. coli or S. aureus DNA., and then hybridization was performed with their target sequences. Electrochemical behavior of DNA-modified and unmodified PGEs was investigated by using differential pulse voltammetry (DPV). Sensitive and selective analysis of E. coli and S. aureus was carried out and obtained results were evaluated by using changes in the intrinsic guanine response at about 1.0 V. Optimum detection parameters of the genosensor such as target concentration, hybridization time, etc. were studied. As a result of this study, the developed genosensor provides an extremely simple, fast and sensitive DNA detection after the optimization conditions were met, the diagnosis time was found as 5 minutes with the detection limit of nM level. However, our experiments on adding the nanomaterial to our detection scheme and converting the genosensor to the diagnostic kit are also continuing. Due to its easy determination and long-term stability, the developed genosensor can perform real sample analysis in field applications outside the laboratory in the medical or environmental field.

**Keywords:** *Escherichia coli*; *Staphylococcus aureus*; Differential pulse voltammetry; Genosensor

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**PP10- Determination of Indium using Sieve Conducted Two Syringe based Pressurized Liquid Phase Microextraction-Slotted Quartz Tube-Flame Atomic Absorption Spectrometry System**

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Indium is a heavy metal which has different applications in several fields such as biomedicine, alloys and semi-conductors. Its compounds have the potential to cause toxic effects mainly to the teeth and joints when their amounts exceed a threshold value in the human body<sup>1,2</sup>. In this study, a new liquid phase microextraction method namely sieve conducted two syringe based pressurized liquid phase microextraction (SCTS-PLPME) was developed to determine indium at trace levels by slotted quartz tube-flame atomic absorption spectrometry (SQT-FAAS). SCTS-PLPME method eliminates the use of dispersive solvents by generating the dispersion through the pressure created between the two syringes by the simultaneous movements of the syringe plungers (to and fro). All significant parameters that affect complex formation, instrumental measurement and extraction efficiency were optimized to enhance the detection power. Under the optimum experimental conditions, SCTS-PLPME-SQT-FAAS method provided 72-fold improvement in detection power of FAAS. The limits of detection and quantification (LOD and LOQ) of the method were obtained as be 19 and 72, respectively. Percent relative standard deviation (%RSD) for the lowest concentration (3.0%) represented high precision. Applicability and accuracy of the proposed method was examined by real sample application on lake water samples and the percent recoveries obtained were between 101 and 107%.

**Keywords:** Syringe; Flame atomic absorption spectrometry; Indium; Liquid phase microextraction.

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**PP11- Determination of Trace Nickel by Switchable Solvent Based Liquid Phase Microextraction- Flame Atomic Absorption Spectrometer System after Complexation with a proper Schiff Base**

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Nickel as a transition metal is resistant to heat and corrosion, assembling alloys of high strength and uniformity<sup>1</sup>. Due to these properties, it has wide usage in several industrial areas. Beside its considerable consumption, nickel has toxic effects on the skin, kidney and lungs. On the other hand, nickel is an essential metal for the human body because of being an enzyme cofactor<sup>2</sup>. Therefore, determination of trace nickel has become crucial in order to prevent its adverse effects on humans and other organisms. For this reason, a sensitive and selective method was developed to determine nickel by switchable solvent based liquid phase microextraction-slotted quartz tube-flame atomic absorption spectrophotometry (SS-LPME-SQT-FAAS). All experimental conditions were optimized for the purpose of maximizing extraction and instrumental outputs. The Schiff base used for complexation of the analyte was synthesized from the reaction of 5-bromosalicylaldehyde and 1-naphthylamine. Under the optimum conditions, analytical figures of merit for system were determined. The relative standard deviation of the lowest concentration in the dynamic working range fell below 10% and this showed high precision. The enhancement in detection power obtained for both SS-LPME-FAAS and SS-LPME-SQT-FAAS systems was satisfactory. The applicability and accuracy of the developed system was investigated by spiked recovery studies.

**Keywords:** Nickel; Schiff Base; Slotted Quartz Tube; Switchable Solvent Based Liquid Phase Microextraction; Flame Atomic Absorption Spectrophotometry

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**PP12- Determination of Palladium by Pressurized Liquid Phase Microextraction Using a Simple Two Syringe System-Flame Atomic Absorption Spectrophotometry**

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Palladium is a useful element consumed in several areas such as modern synthetic chemistry, material science and medicinal chemistry. However, excess usage of palladium in the chemical industry and automotive catalytic converters has caused increased concentration of palladium in the environment <sup>1</sup>. In this study, determination of trace amount of palladium by the traditional flame atomic absorption spectrometer (FAAS) system was performed by using a liquid phase microextraction method with double syringe based automation system for preconcentration/extraction. The syringe system was used as an efficient tool for the dispersion of extraction solvent in the aqueous solution using the pressure formed between the two syringes by their simultaneous movement. A filter was placed between the syringes to disperse the aqueous solution in a form of nebulization. By using this syringe based method, there was no need for dispersive solvents which are required in relatively large volumes than the extractant for efficient dispersion. Thus, this study minimized the use of organic and toxic chemicals and presented a cost-effective and environment friendly method. In order to obtain high sensitivity, all variable parameters affecting the extraction output were optimized. The optimized extraction and system conditions were used to determine analytical performance. The developed method was successfully applied for the determination of palladium in real sample matrix to validate applicability and accuracy of the method.

**Keywords:** Palladium, FAAS, Syringe, Microextraction.

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**PP13- Trace Determination of Organochlorine Pesticides and Hormones by the Combination of QuEChERS and Ultrasonic-Assisted Switchable Solvent Liquid Phase Microextraction with GC-MS**

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A large percentage of pesticides are used for agricultural purposes with the main aim enhancing the quality of food crops, production quantity and prolonging shelf life <sup>1</sup>. Estrogens (natural and synthetic) are hormones that are released into the environment and they are capable of harming aquatic organisms when released into the water bodies <sup>2</sup>. In this study, QuEChERS and switchable solvent liquid phase microextraction (SS-LPME) were used to extract and preconcentrate selected organochlorine pesticides (heptachlor, aldrin, dieldrin) and hormones (estrone, 17- $\beta$  estradiol) before the determination by gas chromatography mass spectrometry (GC-MS). A Box-Behnken Design was used to develop an experimental model for prominent SS-LPME variables to evaluate their effects on extraction output as well as their interaction effects. Optimum extraction conditions obtained from the design model predictor were as follows: 1.0 mL switchable solvent, 1.0 mL sodium hydroxide (0.50 M) and 30 s sonication period. In comparison to the detection limits calculated for direct GC-MS, the optimized method enhanced the system's detection power by 33 – 115 folds. Tap water, lake water, well water, tea samples and medical wastewater were used as test samples to determine the method's accuracy and suitability to real samples. Matrix matched calibration standards were used in place of the conventional calibration method to enhance the percent recovery of analytes in tea samples. Tomato was used as a solid test subject and the SS-LPME method was applied after QuEChERS was used to extract and clean the sample matrix. Matrix matching was also applied to the spiked tomato sample to obtain satisfactory analyte recoveries.

**Keywords:** Endocrine disruptors; Pesticide; Switchable solvent; QuEChERS; Hormones.

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**PP14- Determination of Selected Endocrine Disrupting Compounds in Baby Food and Different Water Samples by GC-FID after Reduced Graphene Oxide Magnetic Nanoparticles Based Vortex-Assisted Micro Solid Phase Extraction**

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Pesticides and alkylphenols are two major groups of pesticides that are a major concern for the health of humans and other organisms. Since pesticides are widely used in agricultural fields and alkylphenols used in most household and industrial materials, it is very common to detect them in various environmental and food samples<sup>1</sup>. Carcinogenic and endocrine disruptive effects<sup>2</sup> have been reported for these two groups of chemicals, making their determination in food and environmental samples very crucial in safeguarding human health as well as other organisms. This study employed the composite material of reduced graphene oxide and magnetic nanoparticles (Fe<sub>3</sub>O<sub>4</sub>/rGO-MNP) in a micro-solid phase extraction (μ-SPE) process to preconcentrate three pesticides (diazinon, heptachlor and aldrin) and two alkylphenols (4-*n*-nonylphenol and 4-*n*-octylphenol) from aqueous and food matrices. Gas chromatography flame ionization detection (GC-FID) was used to identify and quantify the analytes. The analytical signals of the five analytes were boosted by fully optimizing the extraction method in a step-by-step approach. Different parameters were used to validate the optimized method and the detection limits calculated for the analytes were all lower than 2.5 μg/L. The detection power of the GC-FID system was enhanced by about 60 – 100 folds with respect to the high enrichment provided by the μ-SPE method. Seven water samples were directly analyzed by optimized method but none of the analytes were detected. A baby food sample was pretreated with the standard QuEChERS method before the μ-SPE method was applied. Water from an irrigation canal and the baby food were used to validate the methods accuracy and applicability by performing spike recovery experiments. Matrix matching was used to offset interference from the complex baby food matrix to obtain recovery results close to 100%.

**Keywords:** Micro solid phase extraction; QuEChERS; GC-FID; Pesticides; Alkyl Phenol.

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**PP15- A Sensitive and Accurate Analytical Strategy: Zirconium Nanoparticles based Dispersive Solid Phase Extraction Prior to Slotted Quartz Tube Flame Atomic Absorption Spectrophotometry for the Determination of Selenium in Green Tea Samples**

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Selenium is a significant trace element due to many reasons such as being a component of selenoproteins and having antioxidant properties<sup>1</sup>. However, different concentrations of selenium could lead to acute or chronic toxicity leading to several diseases including heart disease and cancer in the human body and other living organisms<sup>2</sup>. Thus, it is very important to determine selenium with sensitive and accurate analytical methods. A simple, green, rapid and novel analytical method was developed for the precise and accurate determination of selenium at trace levels by using zirconium nanoparticles based dispersive solid phase extraction prior to slotted quartz tube-flame atomic absorption spectrophotometry detection. For this purpose, zirconium nanoparticles were synthesized with a novel approach and utilized as adsorbent to provide extensive surface area. In order to enhance extraction efficiency for the analyte, all experimental and system parameters including pH, nanoparticle amount, eluent type and amount were optimized in single variant experiments. Under the optimal conditions, analytical performances of each system, FAAS, SQT-FAAS, DSPE-FAAS and SQT-DSPE-FAAS were determined. Limits of detection and quantification values were calculated as 5.3 and 18 µg/L, respectively. The developed method exhibited good linearity in the concentration range of 25 – 100 µg/L. Detection power of conventional FAAS was improved by 415 folds with the developed method. Accuracy and applicability was checked in spiked green tea samples and percent recovery results ranged between 92 and 102%, with low relative standard deviations. The obtained results proved not only the suitability of the developed method to complex matrixes but also validated accuracy and precision.

**Keywords:** Zirconium nanoparticles; selenium; dispersive solid phase extraction; flame atomic absorption spectrophotometry; green tea.

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**PP16- A Sensitive and Effective Analytical Method for Determination of Iron in Hair Samples by Slotted Quartz Tube-Flame Atomic Absorption Spectrometry after Preconcentration with Switchable Solvent Based Liquid Phase Microextraction Method**

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Iron as one of the most important elements for biological processes of plants and animals is commonly found in environmental and biological materials<sup>1</sup>. Presence of iron is important in red blood cells that transport oxygen to the body cells<sup>2</sup>. In this work, switchable solvent based liquid phase microextraction (SS-LPME) was developed for both extraction and preconcentration of iron in hair samples at trace levels. Switchable solvent was used as the extraction solvent that was synthesized from N,N-Dimethylbenzylamide (DMBA) and ultrapure deionized water with the addition of dry ice for protonation. Iron was complexed with a Schiff Base ligand synthesized by the reaction of 3,5-dibromosalicylaldehyde and 2-aminophenol and extracted into the DMBA phase. Type and volume of the buffer solution, mixing period, concentration and volume of the ligand, amount of switchable solvent, concentration and volume of NaOH solution that affect the extraction procedure were carefully optimized. Slotted quartz tube (SQT) was combined with flame atomic absorption spectrometry (FAAS) to improve the detection power of the system. Analytical performance of all four systems (FAAS, SQT-FAAS, SS-LPME-FAAS, SS-LPME-SQT-FAAS) were determined. Applicability of the proposed method was checked with recovery experiments on hair samples and satisfactory percent recovery results were obtained.

**Keywords:** Iron; Switchable solvent; Slotted Quartz Tube; Flame Atomic Absorption Spectrometry.

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**PP17- Cloud Point Extraction Method by using Novel Schiff Base Ligand for Cobalt Determination in Fennel Tea Samples Prior to Slotted Quartz Tube Flame Atomic Absorption Spectrophotometry System**

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Cobalt as a biologically active element is an integral part of the vitamin B12 complex, which is needed in trace amounts for both humans and animals. The daily dose of vitamin B12 is 2.4 µg for an adult. Therefore, it is very crucial to perform cobalt determination at low levels<sup>1</sup>. As an effective preconcentration and separation technique, cloud point extraction (CPE) has many advantages such as its simplicity, rapidity, low toxicity and sensitivity over other methods<sup>2</sup>. This study describes the development of a CPE method for the extraction/preconcentration of [cobalt](#), and its determination by slotted quartz tube flame atomic absorption spectrometry ([SQT-FAAS](#)). A hydrophobic ion-associated cobalt complex compound was obtained from a novel ligand synthesized from the reaction of 1-naphthylamine and 5-bromosalicylaldehyde. Main parameters affecting the efficiency of cobalt extraction such as concentration and volume of ligand, concentration and volume of surfactant, cloud point temperature, ionic strength, eluent type and volume were investigated to obtain optimum experimental conditions. Under the optimum experimental conditions, the limits of detection and quantification, linear working range, percent relative standard deviation and coefficient of correlation were used to validate the CPE-SQT-FAAS method. Relative to the traditional FAAS system, the optimized method produced significant enhancement in detection power for analyte. The accuracy and applicability studies were performed for the developed method by spiking fennel tea samples at different concentrations, and the percent recovery results obtained were very satisfactory.

**Keywords:** Cobalt; SQT; FAAS; CPE; Fennel Tea.

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**PP18- Switchable Solvent Based Liquid Phase Microextraction: An Alternative Method for the Determination of Manganese in Water and Soil Samples**

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Determination of manganese in environmental samples such as soil and water is very important as it enables scientist to examine biochemical cycles that determine manganese distribution in the environment and accumulation in biological organisms<sup>1</sup>. Manganese is an essential element for various enzymatic and metabolic processes but can lead to neurological and metabolic disorders at toxic levels or when deficient in the body<sup>2</sup>. In this study, preconcentration of manganese was performed using a switchable solvent liquid phase microextraction (SS-LPME) which is an efficient and green alternative to the classical extraction methods. Novelty of this method is related to the extraction solvent which is converted from a non-polar liquid into a polar form by adding carbon dioxide, and reverted back to its non-polar state after the extraction process by carbon dioxide removal. The SS-LPME method was optimized to select the optimum pH and buffer volume, ligand concentration and volume, switchable solvent volume, NaOH concentration and volume, in addition to the optimum mixing period after each chemical addition. Detection of manganese was performed by the flame atomic absorption spectrometry (FAAS) after applying the optimized microextraction method. The developed method yielded limit of detection (LOD) and limit of quantification (LOQ) values of 0.71 and 2.36 µg/L, respectively, with an enhancement detection power of 101 folds with respect to the conventional FAAS system. The accuracy of the method was determined by performing spiked recovery studies on soil and aqueous samples. Satisfactory percent recovery results were obtained for all samples at different spike concentrations.

**Keywords:** Manganese; Carbon dioxide; LOD; LOQ; FAAS; SSLPME.

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## PP19- Development of Alternative Methods for Determination of Acrylamide and Caffeine in Coffee Samples

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Acrylamide (Figure 1.) has gained attention in recent years, is a composition resulting from the heat treatment process of foods. By the International Agency for Research on Cancer (IARC), it is listed in Group 2A as a possible carcinogenic substance for humans. Genotoxic and carcinogenic effects of acrylamide were determined in animal experiments.

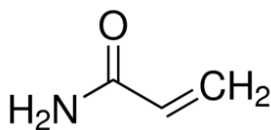


Figure 1. Acrylamide (AA)

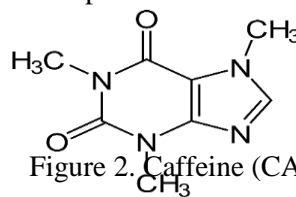


Figure 2. Caffeine (CA)

Caffeine (Figure 2.) is a nitrogenous organic compound of the alkaloid group, which has important physiological effects. It is a substance found naturally in nature. Caffeine, an important stimulant, is reported to cause insomnia, headache, lack of concentration, increased heart rate and blood pressure, and stomach problems. Therefore, caffeine is one of the most researched and discussed the nutritional components of recent years.

There are several analytical methods in literature for acrylamide (AA)<sup>1</sup> and caffeine (CA)<sup>2</sup> determination. This methods are GC-MS, LC-MS, and CE for both of AA and CA; voltammetry, ion chromatography and FTIR for CA; GC-EDC, LC-MS/MS, and GC-IT/MS for AA. In addition, organic molecules could be identified via the complexation with metals<sup>3</sup>. It is well known that the Job Method is determining the formation rate of the complex between metals and organic molecules in a certain mixtures.

In this study, the metal complexes of acrylamide and caffeine formed with copper and cobalt were determined according to M:L ratio by UV-VIS spectrophotometer with Job's method. Based on the data obtained, the amount of acrylamide and caffeine was determined in commercially available coffee samples.

Keywords: acrylamide, caffeine, Job's method, Chromatography

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**PP20- A novel microextraction procedure for azorubine for its spectrophotometric determination**

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In order to separate and enrich the trace species from environmental samples, microextraction techniques have been used recently. Microextraction techniques are preferred due to their advantages such as minimizing the amount of solvent, providing effective separation in a short time, enrichment with separation, and high enrichment factor. The use of deep eutectic solvents as solvents in combination with microextraction techniques is common. Deep eutectic solvents are formed by the combination of a hydrogen bond donor and a hydrogen bond acceptor<sup>1-2</sup>.

The aim of this study is to separate and preconcentration Azorubine dyestuff. In this study, parameters such as pH, deep eutectic solvent volume, tetrahydrofuran volume, sample volume are optimized and the method is planned to be applied to some water samples and environmental samples. Validation of the method will be performed by adding and recovering these samples.

Keywords: deep eutectic solvents, separation and preconcentration, azorubine

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**PP21- UV-vis spectrophotometric determination of allura red in real samples after microextraction strategy using deep eutectic solvents**

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In recent years, the use of liquid phase microextraction techniques for the analysis of trace environmental samples has gained popularity due to the use of a small amount of solvent, the ability to perform the operation in a short time and ease of operation. The use of deep eutectic solvents (DES) in combination with liquid phase microextraction is also common. DESs are new generation solvents formed by combining a hydrogen bond acceptor and a hydrogen bond donor<sup>1-2</sup>.

In this study, a method for liquid phase microextraction of allura red dye using deep eutectic solvents was developed. Among the variables of this method, parameters such as pH and deep eutectic solvent type and volume, tetrahydrofuran volume, sample volume, ultrasonic effect time and centrifugation time were optimized. The dyestuff phase was extracted to the DES phase at pH 4.0, and tetrabutylammonium bromide and decanoic acid were used as DES solvent. Matrix effects were also investigated. The validation of the method was verified by addition recovery test of the allura red dye on various environmental samples.

**Keywords:** *deep eutectic solvents, microextraction, allura red*

*Acknowledgement. The authors are grateful for the financial support of the Unit of the Scientific Research Projects of Erciyes University (FYL-2019-9503) (Kayseri, Turkey).*

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## PP22- Fluorimetric Analysis of Controlled Drug Release from Polyethylene Glycol-Isobutyl Vinyl Ether-Co-Maleic Anhydride Polymer Modified with Acetyl Salicylic Acid

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Controlled release technique has the advantage that the target can be achieved with less frequent administration, the side effects can be minimized and the active reagent is more effective. In case of drugs, they must first exit the systems which are called dosage forms which carry the active substance and then they must be safely and effectively mixed with the blood, dispersed into the tissues and eventually metabolized. After mixing into the blood, the dose should be maintained at an interval above the effective amount and below the toxic amount. In new drug applications, the drug is intended to be effective in the body according to a pre-planned process and to be treated at longer intervals with low doses of the drug without side effects. These objectives can be achieved by controlled release systems, which ensure prolonged circulation of the drug, accelerate its absorption and target the effect of the drug. Basic controlled release formulations comprise an active ingredient such as a drug, dye, perfume, and a carrier, usually a polymeric substance. The carrier permits release of the active reagent at the intended rate and periodic intervals. The building block of many controlled release systems are polymers. The drug release rate and times are adjusted with water-soluble, biocompatible and biodegradable polymers<sup>1,2</sup>. In this way, it is aimed to direct the drug active substance to the target cells without contacting other healthy cells and to maintain the effects on the target cell-tissue system.

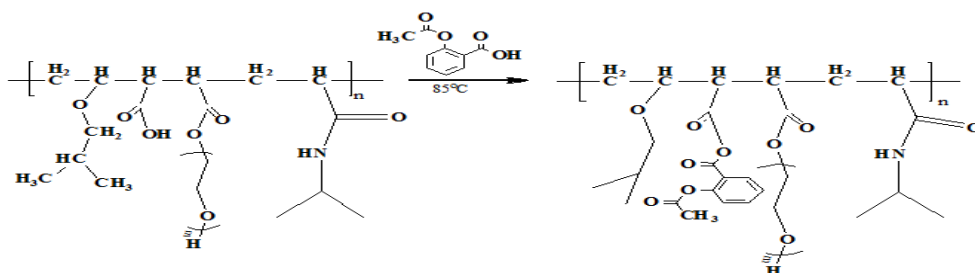


Fig. 1. Acetyl salicylic acid (ASA) modification reaction

In this study, in order to determine the amount of ASA hydrolyzed from the polyethylene glycol-isobutyl vinyl ether-co-maleic anhydride (PEG-IBVE-MA) modified with acetyl salicylic acid (ASA) (Fig. 1) copolymer to the solution medium, firstly the spectrofluorimetric properties of ASA, PEG-IBVE-MA and PEG-IBVE-MA-ASA were investigated. Afterwards, the calibration graphs were prepared under the most suitable spectral conditions determined with solutions of different concentrations prepared from ASA stock solution at appropriate concentration in physiological environment (physiological pH and temperature). Using the previously determined most appropriate fluorimetric wavelengths, ASA modified PEG-IBVE-MA at different physiological pH (pH = 2.0, 4.5, 7.4 and 9.0) prepared with phosphate buffers of 200 ppm solutions, with a constant mixing rate at 37°C and at certain time intervals maximum fluorescence intensity values were measured in order to determine the ASA release to the solution from the polymer. The amount of ASA introduced into the solution at each pH was determined using calibration charts.

**Keywords:** PEG-IBVE-maleicanhydride, acetyl salicylic acid, spectrofluorimetry, controlled drug release.

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## PP23- Determination of heavy metal levels in commercial herbal teas by ICP-MS

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Herbal teas are preparing by infusion or decoction of different parts of the plants such as leaf, flower or radix. Many people use herbal tea bags in their daily life due to the healthful and delighting effects. Heavy metals are toxic for health and they present in herbs because of the environmental pollution. Studies mainly focused on the heavy metal amounts of teas but people consume them by preparing their infusions (1).

The aim of the study is determination of As, Co, Cr, Cu, Li, Ni, Sr, V, Zn, Ag, Cd, Bi, Pb contents of tea bag infusions by inductively coupled plasma-mass spectrometry (ICP-MS), prior to microwave digestion. The precision and accuracy of the method was evaluated by applying standard (<sup>6</sup>Li, <sup>45</sup>Sc, <sup>72</sup>Ge, <sup>115</sup>In, <sup>209</sup>Bi) addition method to the samples. Ten different herbal tea bag samples collected from local markets. Infusion of samples prepared at 90 °C by using ultrapure water (18.2 MΩ).

Based upon the Turkish Food Codex Tea Communique (2015/30) and U.S. Food and Drug Administration, all samples infusions were found to be safe for human consumption.

*Key Words: Heavy Metal, ICP-MS, Herbal tea, Microwave digestion,*

*Acknowledgement: This work was supported by TÜBAP (2019/70) financially.*

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**PP24- About short-term variations of element concentrations in native grasses and in the rhizosphere soil**

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Elemental composition of plants is not constant. Among others, temporal variations of concentrations of different elements are of special interest. Till now, however, little is known about short-term, for example, over a period of hours, variations of element concentrations in plants and in the rhizosphere soil.

In our research, two widely distributed native plant species (couch grass and plantain) together with the rhizosphere soil were collected during a day every 4 hours from a small site located in the south of St. Petersburg city. The pH values of soil samples were determined and an elemental analyzer CHN628 (LECO) was applied for measuring total amounts of C, N, and H in the soils. A standard Agilent 8900 ICP-QQQ Inductively Coupled Plasma Mass Spectrometer with micromist nebuliser was applied to determine the concentrations of Ca, V, Cr, Mn, Fe, Ni, Cu, Zn, Ga, As, Rb, Sr, and Pb in the plants and soils.

In the course of the day, the variations of the total amounts of C, N, and H in the rhizosphere soil were rather marked and different for soils taken from roots of plantain and couch grass. The concentrations of some other elements in the rhizosphere soil of the plants varied in a similar way. The short-term variations of element concentrations in roots and leaves of the plants were also rather large. In many cases, a decrease of element concentration in roots correlated with an increase of its concentration in leaves.

Although couch grass and plantain were collected simultaneously and from the same site, mean concentrations of many elements in the two plant species were statistically significantly ( $P < 0.05$ ) different. This may be result of the fact that the plants belong to different clades. The differences between concentrations of most part of elements in roots and leaves of the plants were also statistically significant. The concentrations of many trace elements were higher in roots than in leaves, while the concentrations of essential plant nutrients were often higher in leaves compared to roots. The distribution of elements between different plant parts were not the same in couch grass and plantain.

The results of the experiment showed that concentrations of many elements in the rhizosphere soil and in different parts of plants could change during a short time. The variations often depended on the plant species, and varied between different species of plants growing in the same place.

**Keywords:** Trace elements; Macronutrients; Natural grasses; Rhizosphere soil; Temporal variability

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**PP25- Thermophilic *Anoxybacillus flavithermus* for Th(IV) resistance and bioaccumulation and its usability as a bioindicator**

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Studies on the construction of radionuclides and toxic metal ions resistance, bioaccumulation, and antioxidant enzymes have been tested applying mesophilic microorganisms. But, there is not enough research on the influences of radionuclides and toxic metal ions on resistance, removal, biological accumulation and antioxidant enzymes using thermophilic microorganisms<sup>1</sup>. The thermophilic *Anoxybacillus flavithermus* were used as biosorbent within the scope of this investigation for on thorium resistance and bioaccumulation. The minimum inhibition concentration of was determined as 2800 mg/L and 305 mg/L in solid and liquid culture medium, respectively. The effect of different concentrations of thorium on *A. flavithermus* growth and bioaccumulation were tested. SEM and FT-IR analyzes were investigated for characterization of cell surface of thermophilic bacteria before and after Th(IV) bioaccumulation. The highest thorium cell membrane capacity was determined as 312.9 mg Th(IV)/g wet cell membranes. The influence of various thorium concentrations on  $\alpha$ -amylase production was also studied. In addition to these, the effects of thorium on superoxide dismutase and catalase enzyme activities were studied in order to test the use of these microorganisms as thorium bioindicators in real water samples.

**Keywords:** *Anoxybacillus flavithermus*, Th(IV), resistance, bioaccumulation, bioindicator

**Acknowledgment**

This study was supported by the Research Fund of Mersin University in Turkey (Project No. 2018-3-AP4-3092).

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**PP26- In vitro activity of bacteriophage and molasses on *Escherichia coli* O157:H7**

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**ABSTRACT**

Antibiotic resistance in pathogenic bacteria is one of the most important health problems in the world. Therefore, it is necessary to find alternative treatment methods in order to reduce the use of antibiotics. Phage therapy is one of these alternative methods. Also, certain products containing antimicrobial components such as molasses are used to eliminate resistant bacteria. In the present study, phages-molasses interactions were investigated in order to examine the effects arising from concomitant use. For this purpose, *E. coli* O157:H7 phages were isolated, and antimicrobial activity of molasse was investigated. In order to understand the effect of molasses samples on phage plaque size and latent period, one-step growth curve experiments were used. According to results, d/4 and d/8 concentration of molasse samples increased burst size of phage plaque. Although d/8 concentration of molasse samples prolonged latent period of phage, it was seen that d/4 concentration of molasses decreased latent period of phage. Finally, time kill curves were conducted to evaluate possible synergism. Although no synergistic effect was found between phage and molasses, the antimicrobial activities of the components and the effect of molasses on phage activity have been demonstrated.

**Keywords-***Bacteriophage, phage therapy, Escherichia coli O157:H7, synergism, molasse*

**PP27- A new magnetized thermophilic bacteria for determination and preconcentration of uranium from environmental samples by magnetic solid-phase extraction (MSPE)**

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Magnetic solid-phase extraction (MSPE) is also one of the solid phase extraction (SPE) process. It generally utilizes in separation and preconcentration applications. By reason of the input of magnetic materials, MSPE has attracted extensive interest<sup>1,2</sup>. A magnetic solid-phase extraction (MSPE) was developed for simultaneous preconcentration of U(VI) before its determinations by inductively coupled plasma optical emission spectrometry (ICP-OES). The main idea of this biotechnological application is based on the use of bacteria, thermophilic *Bacillus cereus* SO-14, as solid-phase biosorbent. It was immobilized to  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles and used for MSPE. Characterization of the biosorbent was performed using the scanning electron microscope (SEM), the energy dispersive X-ray (EDX) and Fourier transform infrared (FT-IR) spectroscopy. Also, The best value of experimental parameters was investigated, and the accuracy of the developed method was checked by applying the certified reference materials. LOD of the U(VI) was calculated as 0.008 ng mL<sup>-1</sup>. RSDs was found to be 1.6%.  $r^2$  were found to be higher than 0.9991. Preconcentration factors were achieved as 100. It should be highlighted that LODs were critically improved and sensitivity of ICP-OES was enhanced.

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Acknowledgment

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## PP28- Harmful Chemical Elements in Children's Toys from Latvian Market.

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No matter how harmless children's toys may look, looking at European Union and Latvian statistics, it can be concluded, that children's toys are not necessarily the safest product group as they have often been found to contain substances, that are very harmful to health. Worldwide studies of heavy metals in toy materials confirms – this particular issue continues to be alarming<sup>1</sup>. The contamination with harmful chemical elements in children's toys occurs because harmful chemical elements are used in the toys manufacturing process as stabilizers and catalysts, as well as colour pigments, resulting in the desired softness, luster and elasticity of toy<sup>2,3</sup>.

Quantitative analysis of some harmful chemical elements stated at 15 February 2011 Latvia's Cabinet of Ministers Regulation No. 132 "Toy safety regulations" and European Parliament and Council directive of 18 June 2009 2009/48/EC on the safety of toys – antimony, arsenic, barium, mercury, cadmium, cobalt, nickel, selenium and lead has been carried out in children's toy samples, which were randomly bought in Latvia. Samples of children's toys purchased in Latvia were analysed according to European standard EN-71:3 „Migration of certain elements”.

Mercury and selenium levels were below the detection limit for ICP - MS in all analysed children's toys. Arsenic was found only in soft children's toys. Cadmium was found in some parts of almost all analysed children's toys; mostly cadmium was found in soft children's toys. Cobalt was only found in some parts of analysed children's toys. Most cobalt was found in soft children's toys as well as in the plasticine - like mass. Nickel, antimony and lead was found in all analysed parts of children's toys. Most of the mentioned above was found in soft children's toys as well in one of the parts of the analysed doll and in the plasticine - like mass. Barium was found to have significantly higher levels in all analysed parts of children's toys in relation to other detectable harmful chemical elements. The barium is distributed evenly throughout children's toys available in Latvia.

Only one of the analysed children's toys purchased in Latvia exceeded the permissible migration limit of the harmful chemical element - lead for Category II toys stated in Cabinet of Ministers Regulations Nr. 132 and Directive 2009/48/EC of the European Parliament and of the Council set 0.5 mg/kg of the toy to be analysed.

Keywords: Harmful chemical elements, pollution, children's toys.

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## PP29- Novel Ion Imprinting Polymer for Selective Removal of Cr(VI) ion from Aqueous Solution

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In the water treatment technologies, the toxic metal ions are removed using the adsorption, precipitation, ion exchange and membrane processes methods. Ion-imprinted polymers are recently identified nano-porous polymeric materials which on leaching the imprint ion can rebind, sense or transport selectively the target analyte in presence of closely related inorganic ions <sup>(1,2)</sup>.

Ion imprinting system contains polymerization of the functional monomers in the presence of metal ion as a template. After polymerization, the template is removed from the polymer structure under mild conditions so that the specific binding sites of the prepared polymeric imprinting system remain.

In this study, a novel Cr(VI) ion imprinted polymer (Cr(VI)-MIIP) was successfully synthesized and used as a selective adsorbent for the removal of Cr(VI) ions from aqueous solution. To this, vinyl imidazole (VIM) was used as functional monomer. The Cr(VI) ion imprinted poly(vinyl imidazole-co-hydroxypropyl methacrylate), poly(VIM-HPMA), particles were prepared by bulk polymerization. The Cr(VI) ion-imprinted polymer particles were grained from the bulk polymer, and the template ions were removed using thiourea in mineral acid. Similarly, the non-imprinted polymer (NIP) was also prepared under the same conditions without the chromate anion template. Various parameters, such as solution pH, initial concentration, aqueous phase volume, sorbent dosage, contact time and leaching solution volumes, were investigated. Scanning electron microscopy (SEM), Fourier Transform Infrared (FTIR) spectroscopy, BET surface area and pore size analysis were used for the characterization of IIP and NIP materials.

The effect of experimental conditions onto the adsorption rate and capacity for Cr(VI) ions were studied using the ion imprinting system. A maximum binding capacity was obtained 121.6 mg/g polymer for Cr(VI) ions which was observed at pH 3 and at 25 °C. The equilibrium time was 30 min, and the amount of adsorbent which gave the maximum adsorption capacity was 2.0 g L<sup>-1</sup>. Under competitive condition, the adsorption capacity of ion imprinted polymers for Cr(VI) is 14.7 and 9.2 folds greater than that of the Cr(III) and Ni(II) ions, respectively.

Isotherm studies showed that the adsorption equilibrium data were fitted well with the Langmuir adsorption isotherm model and second order kinetics models were well described the ion imprinted system. The selectivity studies indicated that the synthesized sorbent had a high single selectivity sorption for the Cr(VI) ions in the presence of competing ions. Thermodynamic studies revealed that the adsorption process was exothermic and spontaneous. The Cr(VI)IIP was used for several times and no significant loss in adsorption capacity was observed.

Keywords: Ion imprinting polymer, Removal, Cr(VI), Selective Recognition

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**PP30- Production of Algal Oil from Hot Spring Isolate *Kamptonema formosum* and Biodiesel Synthesis Using Immobilized Lipase on Magnetic Diatomic Particles**

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Algae are aquatic photosynthetic plants including a large group of various organisms that are extensively spread in the sea, freshwater, swamp, etc. Algae can be used as an industrial and chemical raw material, as a food source and as an energy source, and thus, different algal biomasses can be used as an alternative source for biodiesel production instead of fossil fuels.

**Keywords:** *Kamptonema formosum*; Algal oil; Immobilized lipase; Transesterification; Biodiesel

**References**

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### PP31- Bioremediation of Reactive Red 195 Azo Dye Using Free and Immobilized *Oscillatoria sp*

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Due to their versatility, adaptability and low cost, bioremediation using biomasses have been demonstrated for soil and water remediation. In this regard, many published studies have indicated that microorganisms could be used for the degradation of various classes of organic molecules. The azo dye known as Reactive Red 195 was studied for their decolorization by an algae (*Oscillatoria sp*)<sup>(1,2)</sup>.

In industrial or technical operations, the use of non-living microbial cells in the powdered form have some problems, difficulty in separation of microbial cells after biosorption, mass loss during separation and low mechanical strength and small particle size, which make difficult to use in the batch and continuous systems. These problems can be solved by immobilization of microbial cells using natural or synthetic polymers<sup>(1,2)</sup>. *Oscillatoria sp* biomass was immobilized in Ca-alginate gel beads.

The free and immobilized algal biomass forms were used for the bioremediation of Reactive Red 195 (RR 195) using plain of Ca-alginate gel beads as a control system. The effect of pH was investigated and the maximum decolorization of dye on the free and immobilized algae in alginate forms was observed between pH 2.0 and 3.0. The free and immobilized algae forms had high removal capacity, i.e., 74% and 99% for RR 195, respectively. The experimental data were successfully fitted by the Langmuir and Freundlich equations.

Bioremediation kinetics has also been studied, and the second order model described better than the Lagergren model. The immobilized algae could be regenerated using HCl with up to 91% recovery and reused in six reuse numbers without any considerable loss in their bioremediation capacity.

Keywords: Algae, Ca-alginate, Azo dye, Biodegradation

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## PP32- GSH-Functionalized Gold Nanoparticles Based Electrochemical Biosensor For The Cancer Cell Detection

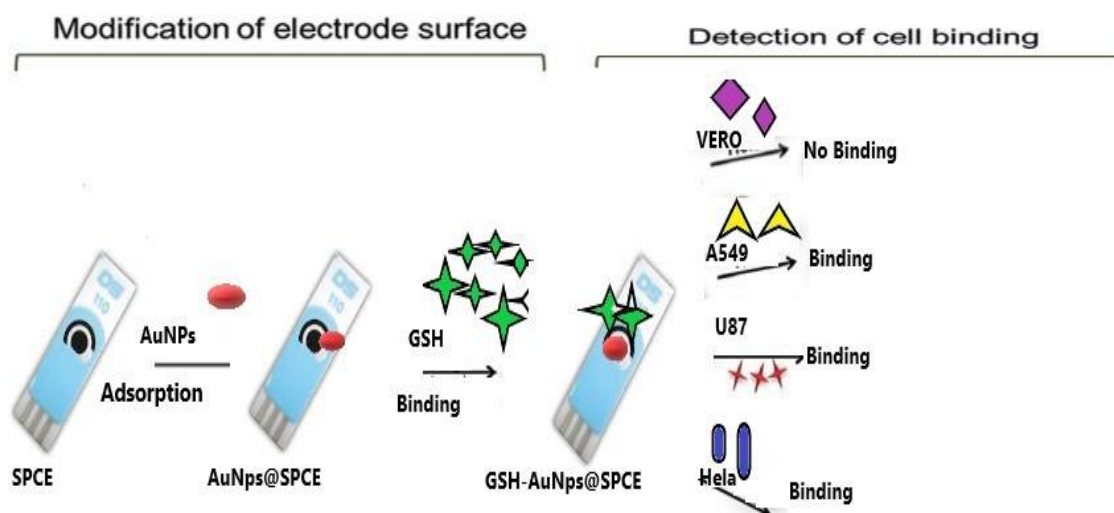
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**Abstract:** Glutathione (GSH,  $\gamma$ -Glu-Cys-Gly) played a crucial role in protecting intracellular components against oxidative damage and plays a major role in detoxification of various electrophilic compounds. This work presents the preparation of glutathione (GSH) functionalized gold nanoparticles (GSH-AuNPs) for the detection of cancer cell based on surface enhanced Screen Printed Carbon Electrode (SPCE) by electrochemical detection. Gold nanoparticles (AuNPs) were attached to the screen-printed carbon electrode (SPCE) surface by adsorption and then the thiol group of glutathione conjugates with gold nanoparticles through Au-S bond to form (GSH-AuNPs@SPCE). The sensitivity of the modified electrode is electrochemically measured in various cancer cell lines (Hela, A549, U87) and intact cell line (VERO). The selective adhesion of GSH holds on cancer cell lines and intact cell lines the surface is followed by the electrochemical methods. Differential pulse voltammetry (DPV) and cyclic voltammetry (CV) have been used to characterize stepwise modified of the electroactive surface. All the results demonstrated that develop electrochemically disposable, cheap and simple biosensors and are applied in the cancer diagnosis.

**Graphic Abstract :**

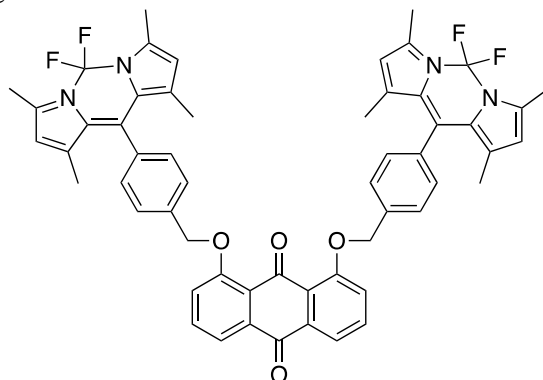


**PP33- The synthesis of dual-Bodipy bearing Dantron and its spectroscopic properties****Cagla Dursun, Elif Bastug, Ahmed Nuri Kursunlu, Ersin Guler, Tugce gover, Zafer Yazicigil**

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The reason for this increase in interest in Bodipy is that they have a sharp fluorescence profile and a high degree of photocompatibility, depending on the properties of the bonded substituents. Many Bodipy and dipyrin review articles have emphasized their synthesis, reactions and applications as fluorescence chemosensors. Therefore, researchers synthesized new Bodipy fluorophores with various reaction methods in order to obtain a shift in absorption and emission wavelengths. Bodipy derivatives, which can be used to detect metal cations by using changes in wavelength and fluorescence intensity just like porphyrin and phthalocyanines, have become increasingly important for cytotoxicity and bio-imaging applications. Because, Bodipy compounds have the potential to undertake the task of photodynamic effect. Increasing interest in photo-organic based substances also makes the tendencies towards synthesis of new Bodipy derivatives meaningful. However, Bodipy's are the only materials in the class of compounds that can perform energy transfer through the gap and energy transfer through the bond and exhibit self-laser behavior. The superior fluorescence of Bodipy as a photo-active material is also a great potential for bio-imaging studies.



In this study, the synthesis of dual-Bodipy bearing Dantron was carried out and its spectroscopic properties were investigated.

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**PP34- Investigation of Electrochemical Behaviors and Modification of 8-{4-(azidomethyl)phenyl}-4,4-difluoro-1,3,5,7-tetra methyl-4-bora-3a,4a-diaza-s-indacene (BODIPY-I) Compound on Glassy Carbon Electrode Surface**

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BODIPY is an organoboron compound known as fluorescence dyes. Fluorescent dyes are widely used in chemical sensors, laser dyes and therapeutic applications <sup>1,2</sup>.

In this study, modification of newly synthesized BODIPY-I compound to glassy carbon (GC) electrode surface will be realized and electrochemical behavior on modified surface will be examined. Optimum conditions for surface preparation and analysis will be determined and processes will be performed under established optimum conditions. Morphology of bare and modified electrode surfaces will be evaluated by scanning electron microscopy (SEM), transmission electron microscopy (TEM), electrochemical impedance spectroscopy (EIS). The metal adsorption properties of the modified surface will be investigated by using metal solution to determine the application area of these modified electrode.

It was determined that GC surface was modified with BODIPY-I compound from the results obtained by probe tests and surface characterization techniques. This also showed that the bare GC surface allowed the transfer of electron, while the modified GC surface did not allow the transfer of electron. Voltammograms obtained in the metal application study to prove the presence of BODIPY-I compound by bonding the metal are capable of proving the presence of BODIPY-I compound on the surface.

Keywords: BODIPY, electrode modification, voltammetry

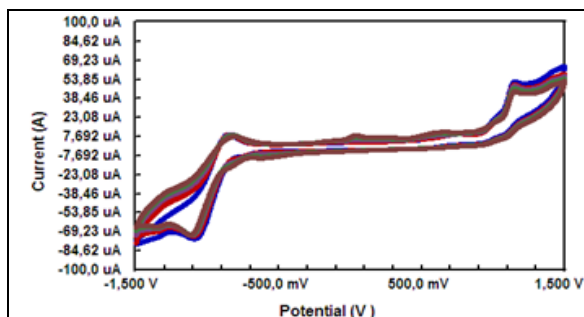


Fig. 1. The cyclic voltammograms of 8-{4-(azidomethyl)phenyl}-4,4-difluoro-1,3,5,7-tetra methyl-4-bora-3a,4a-diaza-s-indacene in CH<sub>3</sub>CN containing 0.1 M of TBATFB solution at the GC electrode surface.

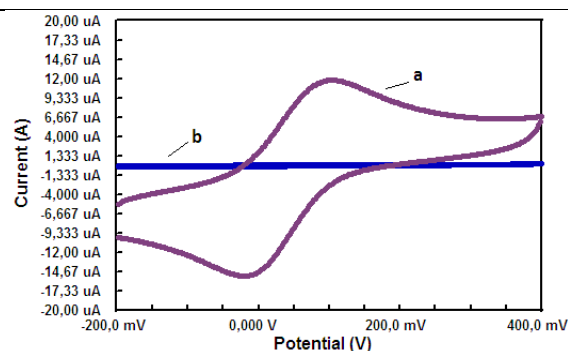


Fig. 2. The comparison of cyclic voltammograms of 1.0 mM of Fe(CN)<sub>6</sub><sup>3-</sup> in BR buffer (pH=2.0) of (a) bare GC and (b) BODIPY-I/GC electrodes..

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"Bu çalışma S.Ü. Fen Bilimleri Enstitüsü Kimya Anabilim Dalında yüksek lisans tez çalışması olarak sürdürülmektedir. "

**PP35- Separation, preconcentration of tartrazine by amine based micro-extraction method and analysis by UV-Vis spectrophotometry**

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Azo dyes are used generally in food and textile industry. They may cause mutagenic, genotoxic and carcinogenic effects [1]. Therefore, analytical quantitative analyses of these dyes are important. Recently there is a trend towards microextraction techniques, which is developed a minimum amount of reagent and solvent consumption in accordance with the principles of green chemistry in sample preparation which is a necessary step for the analytical process for the azo dyes from the environmental samples [2].

In this study, a microextraction procedure amine based for tartrazine dye has been established. The tartrazine concentrations were performed by UV-Vis spectrophotometry. Analytical parameters such as pH, solvent amount, sample volume, matrix effect were optimized and the developed method was applied to real samples. The validation of the method was verified by addition recovery test of tartrazine dye on various real samples.

**Keywords:** Octylamine, microextraction, tartrazine dye

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**PP36- Simultaneous Determination of Duloxetine and Sildenafil by using LC-MS/MS****Yalim TOSCALI, Hasan ERTAŞ\***

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Dapoxetine (DPX) and Sildenafil (SIL) are active ingredients used together in the treatment of erectile dysfunction problems therefore, their simultaneous determination in pharmaceutical dosages as well as plasma has become important<sup>1,2</sup>.

The aim of the present study is to develop an LC-ESI/MS/MS method for these active substances and an Ultra Fast Liquid Chromatographic system (Shimadzu) (UFLC and ABI SCIEX API 4000 MS) was used for this purpose. Analytical column was a Zorbax XDB-C18 (2.1 x 50 mm, 5 µm) which was kept at 40°C. A gradient elution program was conducted for chromatographic separation with mobile phase A (aq. solution of 0.05% formic acid), and mobile phase B (acetonitrile) at a flow rate of 0.50 mL/min and the overall run time was 5.2 min.

Quantitative analysis of samples was performed in more sensitive multiple reaction monitoring (MRM) mode. The peak areas of the precursor ions obtained for Dapoxetine (305.8 m/z) and Sildenafil (474.8 m/z) were compared with the peak area of the internal standard, Tadalafil (TAD) (389.7 m/z). The fragmentation ions, 100.1, 157 and 268 m/z were also observed for SIL, DPX and TAD, respectively. An aliquot of synthetic plasma samples (500 µL) spiked with internal standard was subjected to liquid-liquid extraction (LLE) by using 3 mL of ethylacetate/diethylether (v/v; 1/1) mixture and centrifuged at 4500 rpm. The upper phase was blown to dryness under nitrogen gas at 40°C and dissolved sample was centrifuged at 15300 rpm + 4°C for 10 minutes. The upper phase was passed through 13mm 0.45 µm nylon filter and transferred to HPLC vials for injection.

Operational conditions were optimized by taking into account parameters such as separation power, selectivity and retention times. In the LLE technique, EMA validation parameters were examined for the method validation. The linear calibration curve for DPX and SIL were drawn in the range of 0.04 - 1.00 and 2.00 - 40.0 ng/mL, respectively. The lower limit of quantitation (LLOQ) of the method was calculated as 0.04 ng/mL for DPX and 2.00 ng/mL for SIL. Recovery values are in the range of 87.5 - 112.5% for DPX and 85 - 113.5% for SIL at low, medium and high concentrations. In conclusion, the method developed provide a sensitive and selective analysis for simultaneous determination of DPX and SIL in synthetic plasma samples.

**Keywords:** (Dapoxetine, Sildenafil, Tadalafil, liquid-liquid extraction, LC-MS/MS)

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**PP37- Application of Green Methodology to Pharmaceutical Analysis Using Eco-Friendly Ethanol-Water Mobile Phases**

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**Abstract**

Green analysis techniques based on solvent consumption and substitution are two major applications in greening chromatographic analysis. Decreasing the toxicity of solvents used in mobile phases by substituting them with less or non-hazardous ones or revealing the consumed amounts by using lower diameter columns or miniaturizing instruments are some examples that can be implemented to an analysis. Environmentally friendly water and ethanol based mobile phases reduce the use of toxic solvents such as methanol and acetonitrile, and consequently the necessary cleaning of waste is reduced. In the presented study, ethanol having less toxic and hazardous effects has been selected as organic modifier. Famotidin, paracetamol and thiocolchicoside were selected molecules for demonstration of the applicability of green HPLC in pharmaceutical analysis. Experiments were carried out using an LC system connected to a Diode Array Detection (DAD) at 254 nm. C8 (150 × 4.6 mm, 5 µm) analytical column was tested as stationary phases. In the mobile phase optimization, ethanol and sodium dihydrogen phosphate buffer contents were adjusted to the desired concentrations for the elution of selected analytes both in isocratic and gradient elution modes. Initial flow rate of the mobile phase was set to 1.0 mL/min and the injection volume was 20 µL. Developed mobile phase was consisted of sodium dihydrogen phosphate (pH 4.6; 50 mM), and ethanol in gradient elution mode. Famotidin, paracetamol and thiocolchicoside were well separated both from baseline and each other with the capacity factors of 2.14, 2.53 and 4.26, respectively. Caffeine was selected as internal standard. The developed EtOH based mobile phase method in RP-HPLC was validated in terms of ICH requirements and found to be selective, linear, sensitive, accurate, precise, repeatable, rugged and robust. Developed method was also successful in pharmaceutical analysis of famotidin, paracetamol and thiocolchicoside from Turkish drug market. The findings of the presented study suggested that environmentally friendly ethanol and water based mobile phases could successfully apply in the pharmaceutical analysis.

**Keywords:** *Green separation, pharmaceutical analysis, famotidin, paracetamol and thiocolchicoside*

### PP38- Anodic Oxidation of Axitinib by Voltammetric Techniques

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#### Abstract

Axitinib is a tyrosine kinase inhibitor. It has been shown inhibiting angiogenesis, vascular permeability and inducing tumor cell apoptosis *in vitro* by blocking VEGFR-2 phosphorylation. A voltammetric technique has been developed for the quantification of Axitinib using glassy carbon electrode. The oxidation mechanism of axitinib was investigated by cyclic, differential pulse and square wave voltammetric techniques in the supporting electrolytes which acetate, phosphate and Britton Robinson buffers in the pH range between 1.00 and 12.00. The scan rate studies of axitinib was performed by cyclic voltammetric technique in pH 2.00 Britton Robinson buffer.

According to the scan rate studies, the oxidation reaction of the Axitinib was found to be irreversible and mix diffusion-adsorption controlled process. The oxidation mechanism of Axitinib was investigated as details and compared with some model compounds and drugs. The determination of Axitinib was performed by differential pulse and square wave voltammetry in pH 2.00 Britton Robinson buffer. The linear calibration curve was obtained in the range of  $2.00 \times 10^{-6}$  M –  $1.00 \times 10^{-4}$  M using differential pulse voltammetry.

The limit of detection and limit of quantification were calculated  $6.18 \times 10^{-7}$  M and  $2.06 \times 10^{-6}$  M, respectively. The developed method was applied to the pharmaceutical dosage form of Axitinib.

To use in clinical studies and in environmental monitoring, the interest in developing electrochemical sensing devices is growing rapidly. Nanostructured materials provide an important and feasible platform for electroanalysis. To increase the sensitivity, the signal of axitinib was measured by differential pulse and square wave voltammetry on various nanomaterials modified glassy carbon electrode. The best response nanomaterials selected for the further studies.

**Keywords:** Axitinib, voltammetry, validation, pharmaceutical dosage forms, nanosensor

# **PP39- Determination of Chemical Components of Essential Oil Obtained by Water Vapor Distillation from *Origanum onites* Plant with GC/MSD**

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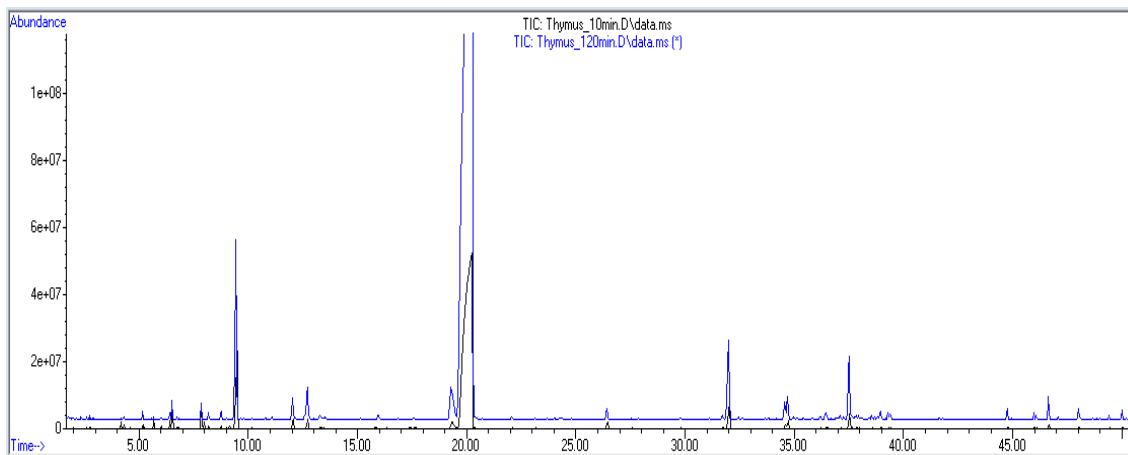
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*Origanum* species, widely used and marketed as spices, hold a large place among plants collected from nature. The genus *Origanum*, which belongs to the Labiatae family, consists of many species and subspecies with great Inter-and intra-species variation (Mheen, 2006). More than half of thyme exported in our country is supplied from the production of cropland (thyme culture). The types of thyme made in culture are *Origanum onites* and *Origanum vulgare* subsp. is grown in Isparta, Denizli and around Izmir. Among the species traded in Turkey, *Origanum onites* L. is the most widely collected and exported species. (Başer, 2002).

The buttons of the *Origanum onites* L. plant forming the leaf and flower collection are consumed as spices. Essential oil of *Origanum onites* L. containing significant amounts of carvacrol and thymol (Özcan ve ark., 2001) has antibacterial, antispasmodic, antiseptic, antimicrobial, cytotoxic, antioxidant and antifungal activity. The essential oil of the plant is also used in pharmaceuticals and perfumery besides food.

In this study, essential oil was obtained from *Origanum onites* plant by distillation of water vapor. Essential oils were collected at intervals of 10 minutes from the start during distillation, and their chemical composition was analyzed with GC/MSD device (Agilent 7890GC-5975 MSD). According to the results, the percentages of major component carvacrol were 75.315% (10 min.), 82.641% (60 min.), 86.232% (90 min.) has been identified. Chromatograms obtained from the analysis are given below.



Keywords: Essential oil, Carvacrol, *Origanum onites*, GC/MSD

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#### PP40- Determination of Arsenic and Mercury in Commonly Consumed Medicinal Plants and Their Infusions by ICP-MS and Statistical Elucidation of the Data

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**Aim:** Medicinal plants grown in many regions of our country have an important role in human health<sup>1</sup>. The purpose of our study is to determine and compare the levels of arsenic and mercury in nineteen medicinal plants and their infusions; and also statistically to evaluate both acid dissolved and infused forms of plants. The studied plants sold in Diyarbakır herbalists which are commonly used as treatment in many diseases by local people.

**Experimental:** The amounts of arsenic and mercury in the studied medicinal plants and their infusions were analyzed by ICP-MS technique. The accuracy and precision of the method were evaluated using CRM 1515 apple leaves. Moreover our results were statistically evaluated by using Paired T Test, Wilcoxon T Test, Spearman Correlation Analysis in SPSS.

**Results:** It was determined that arsenic and mercury values of the studied plants and infusions are below the limit values (2.0-5.0 mg/kg for arsenic and 0.2-0.5 mg/kg for mercury) for raw herbal materials predicted by different countries authorities (WHO)<sup>2</sup>. When the statistical results were analyzed, it was seen that the data was not normally distributed for arsenic and mercury.

**Conclusion:** It was concluded that arsenic and mercury levels in studied plants are suitable for consumption as medicinal plants and herbal tea; since they are below the limit values determined by WHO<sup>2</sup>.

**Keywords:** Medicinal plants, Heavy Metals, ICP-MS, Paired T Test, Wilcoxon T Test.

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## PP41- Smartphone Digital Image Colorimetry for the Determination of Aluminum in Antiperspirant Products

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Antiperspirants are designed to reduce the body's production of sweat. They use aluminum salts that dissolve in the moisture on the skin's surface forming a gel that sits on top of the sweat glands. With the sweat glands covered, sweat and bacteria cannot react with one another and cause bad odors<sup>1</sup>. Transdermal absorption can lead to aluminum overload, which may result in anemia, bone disease, or even dementia. The metal is potentially neurotoxic, so its accumulation can cause diseases such as Parkinson's, Alzheimer's, encephalopathy, and osteomalacia<sup>2</sup>. Therefore, there is a need for developing simple, rapid and cost-effective analytical methods for the determination of aluminum in antiperspirant products.

Digital image colorimetry (DIC) has recently emerged as an alternative calorimetric technique. DIC involves two processes, namely, image acquisition and color analysis. An image acquisition tool is used for collecting the image of the sample, followed by processing by a suitable software<sup>3</sup>. Mobile phones can be used for both steps, offering great advantages over conventional colorimetric methods, which include simplicity, portability and minimum requirement of energy. Smartphone-based digital image colorimetry (SDIC) benefits from minute differences in color tones and their intensities in the red, green and blue (RGB) model, which can easily be correlated to analyte concentration. In this model, a whole-number value from 0 to 255 is assigned to each of these three channels such that [0, 0, 0] corresponds to absolute black and [255, 255, 255] to true white. Thus, the higher the concentration of the analyte in the solution, the higher the intensity of the color and the lower the RGB value. A linear response can be obtained by using the logarithm of  $I_0$  to  $I$  ratio, where ' $I_0$ ' represents the intensity of the blank at a specific channel and ' $I$ ' represents the intensity of the analyte solution at the same channel or by using  $255-I-I_0$ . In this study, SDIC is proposed for the determination of aluminum in antiperspirant products. Aluminum was converted into a stable yellow complex with quercetin, the solution was placed into a quartz UV/Vis cuvette inside a homemade colorimetric box and its color was monitored from the blue channel. The parameters affecting the efficiency of SDIC were optimized, which included the type and position of the digital camera, area of the region of interest, and distance between the camera and sample solution. Optimum complexation conditions were achieved with 3.0 mL of the sample solution at pH 5.5, with 250  $\mu$ L, 1000  $\mu$ g mL<sup>-1</sup> quercetin, and within 1 min complexation time. Under these optimum conditions, the limit of detection (LOD) was found as 26.3 ng mL<sup>-1</sup> (or 0.20  $\mu$ g g<sup>-1</sup>). The calibration graph showed good linearity with a coefficient of determination ( $R^2$ ) of 0.9981 and relative standard deviation (%RSD) less than 3.5%. The proposed method was successfully applied to determine aluminum in eight different antiperspirant products, seven of which were found to contain an average concentration of aluminum at  $3.4 \pm 0.9\%$  w/w.

**Keywords:** Aluminum; Antiperspirant products; Digital image colorimetry; Quercetin; RGB; Smartphone

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## PP42- Possible Biomonitor Tools for Toxic Elements: Annual Plant Parts

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Metal-hyperaccumulator plants have the ability to take up extraordinary quantities of certain elements without causing toxic effects. Although the standard for a hyperaccumulator plant has still not been defined scientifically, the current criterion defining a hyperaccumulator is; “a plant that can accumulate metal to a concentration that is 100 times greater than “normal” plants growing in the same environment”. In other words, hyperaccumulator plants growing on polluted sites can accumulate certain elements up to 10–500 times higher levels compared to the same plants growing on non-polluted soils <sup>1-2</sup>.

In order to determine pollution extent as well as source of pollution, hyperaccumulator plants can be used as biomonitor tool.

Due to the common use of industrial and high technology products, hazards from toxic elements continue threat human and animal health. According to WHO, allowable values (fresh weight basis) for some toxic element in agronomic plants are as follow (mg/kg): 0.2 for As, 0.05 for Cd, 2.0 for Cr, 5.0 for Co, 1.0 for Ni and 0.5 for Pb <sup>3</sup>.

In this study, edible parts and leaves of some vegetables irrigated with water having higher arsenic, taken from Elazig vicinity were analysed by ICPMS. It was found that corn leaves have highest arsenic concentration (up to 9.0 mg/kg basis of FW) compared with the other studies matrices.

**Keywords:** *hyperaccumulator plants, Biomonitor, ICP-MS, toxic elements.*

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**PP43- A New Analytical Method for Trace Nickel Determination: Spray Assisted Droplet Formation based Dispersive Liquid Phase Microextraction Combined with Slotted Quartz Tube- Flame Atomic Absorption Spectrophotometry**

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Nickel is known to have principal duties in living organisms<sup>1</sup>. High amount of accumulation in body may cause toxic effects for organisms<sup>2</sup>. Therefore, accurate and precise determination of nickel at trace levels is crucial. In this study, a novel analytical approach was developed in order to determine nickel at trace levels. For this purpose, spray assisted droplet formation based dispersive liquid phase microextraction method (SADF-LPME) was developed in order to separate/preconcentrate nickel. Determination of nickel was performed by combination of SADF-LPME method with slotted quartz tube-flame atomic absorption spectrophotometry (SQT-FAAS). In SADF-LPME method, extraction solvent was dispersed into aqueous solution by formation of droplet assisted by spraying in order to avoid relative error by reducing multiple operation steps and minimizing the organic solvent consumption. Complexation of nickel was provided by using a Schiff Base yielded from the reaction between 3,5-dibromosylaldehyde and 2-aminophenol. All experimental parameters were optimized step-by-step in order to determine optimum condition to get maximum absorbance values. Under the optimum conditions, analytical performance values were examined. The enhancement in detection power was determined about 30-folds over the detection limit of conventional FAAS system. Chinese green tea sample was used in spiked recovery studies to check applicability/accuracy of the method developed.

**Keywords:** Droplet Formation; Nickel; Slotted Quartz Tube; Dispersive Liquid Phase Microextraction; Flame Atomic Absorption Spectrophotometry.

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# PP44- A New Validated HPLC-UV Method for the Quantitative Determination of Benidipine in Pharmaceutical Dosage Form, Human Urine and Serum

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Benidipine, used as an antihypertensive agent and long-acting calcium antagonist, is synthesized for commercial use as a drug substance in highly pure form [1]. The purpose of this study was to develop a high-performance liquid chromatography with an ultraviolet visible detector (HPLC-UV) method for the determination of benidipine pharmaceutical dosage form, human urine and synthetic serum samples. The proposed method was validated according to International Conference on Harmonization guidelines after optimization of various chromatographic conditions and other experimental parameters.

The desirable results were obtained with ACE 5 C18 (150 mm x 4.6 mm; 5.0  $\mu$ m) column at 45 °C. Mobile phase consisted of Acetonitrile: Buffer (40 Mm Ammonium acetate); (75/25; v/v) with flow rate of 1.0 mL min<sup>-1</sup>. Wavelength was selected as 238 nm. The method was fully validated and validation parameters were: linearity range 0.150–25  $\mu$ g.mL<sup>-1</sup>; correlation coefficient, 0.999 for all samples. Limit of detection values were found 0.00046, 0.0012 and 0.0083  $\mu$ g.mL<sup>-1</sup> for acetonitrile, serum and human urine, respectively. Limit of quantification values were 0.0015, 0.0038 and 0.027 for acetonitrile, serum and human urine. Mean recovery values were between 97.7% and 106.59%.

The proposed method proved to be rapid and accurate and can be successfully used in pharmacokinetic studies and routine clinical practice.

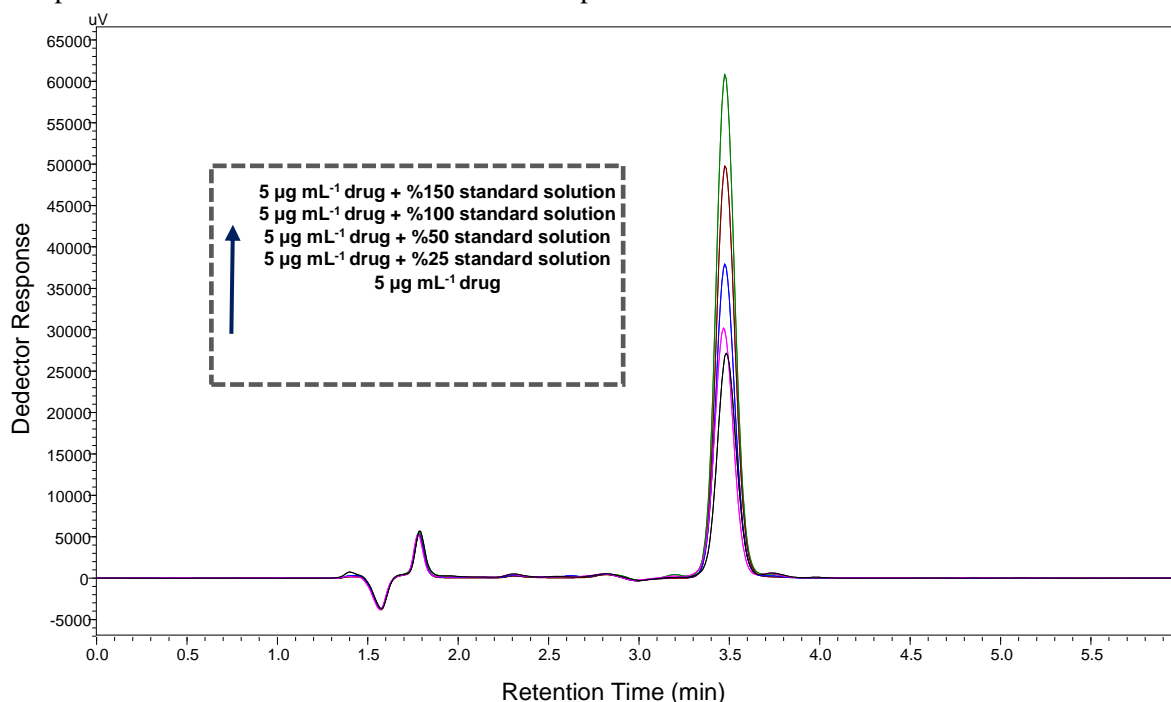


Figure 1: Tablet sample under optimized conditions and the addition of 25%, 50%, 100%, and 150% active ingredient added into the tablet sample.

**Keywords:** Analysis, Benidipine, HPLC

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# PP45- Comparison of Water Vapor Distillation and The Pressurized Water Extraction Techniques on Carvacrol Ratio and Yields of Essential Oil Obtained from *Origanum minutiflorum* Plant

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*Origanum minutiflorum* is an endemic plant to mountain habitats in the province of Isparta in Turkey and has been announced as one of the 10 most endangered species to be protected (Özhatay et al., 1997). This thyme species is one of the thyme with the highest carvacrol ratio (Başer et al., 1993). This study was conducted to increase yield of essential oil and the quality of extraction components of *Origanum minutiflorum* by using the pressurized water extraction system.

In this study, two different technique as distillation of water vapor and the pressurized water extraction system were used to compare the essential oil yields obtained from the *Origanum minutiflorum* plant. 5 samples were collected from Isparta Sütçüler region on 17 August 2019 at the beginning of the flowering phase. The water vapor distillation technique was traditionally performed by placing 20 gram samples. Pressurized water extraction was carried out using the Accelerated Solvent Extractor system (ASE 350 Dionex) as Figure 1 with a pressure of 1500 psi (10 MPa). After 20 g of the ground sample was placed in a 100 mL stainless steel extraction cell, extraction was carried out at 80 °C temperature, with the ratio of sample to water 1/20.

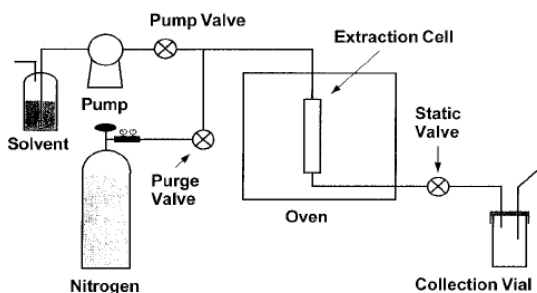


Figure 1. Schematic view of the pressurized water extraction system (Richter et al., 1996).

As a result, the average of total essential oils has been identified 3,92% by distillation of water vapor, 4,17% by the pressurized water extraction system. Chemical composition of essential oils was analyzed with GC/MSD device (Thermo Trace 1300GC-ISQ 7000 MSD). According to the results, the average percentages of major component carvacrol were 82.32% by distillation of water vapor, 85.26% by the pressurized water extraction system has been identified.

**Keywords:** Essential oil, Carvacrol, *Origanum minutiflorum*, GC/MSD

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**PP46- HPLC-DAD-MS analysis of *Althaea officinalis* L. extracts with their Urease Inhibition and Radical Scavenging Activities**

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**Abstract**

*Althaea officinalis* L., also named as Marshmallow is traditionally widely used for treatment of cough, irritation of mucosal membranes, common cold-sore and inflammation. The purpose of this study is to identify bioactive content of *Althaea officinalis* by spectroscopic and chromatographic methods. Flowers, thalamus and seeds of the plant were extracted with methanol and aqueous methanol at 37°C for 2.5 h in a shaker and ultrasound bath. Extracts were subjected to the acidic hydrolysis to identify the main compounds extracted from their derivatives. Phenolic based compounds were identified and quantified using HPLC-DAD-MS. Total phenolic content and antioxidant activities (radical scavenging activity) and enzyme inhibitions of the extracts were also performed spectroscopically. Urease inhibition of the extracts were performed according to the phenol-hypochlorite method at 625 nm. Total phenolic compounds of the flower extract were found highest in three methanol extracts which was correlated with the radical scavenging activity and inhibition effect against urease as well. According to the HPLC-DAD-MS analysis, the extracts had a wide variety of phenolic compounds, including benzoic acid and flavonoids. They had gallic acid, caffeic acid, *p*-coumaric acid and their derivatives, and luteolin, quercetin, myricetin, apigenin, kaempferol, isorhamnetin, eriodictyol and their derivatives.

**Key Words:** *Althaea officinalis*, urease, antioxidant, flavonoids

**PP47- Determination of Oxcarbazepine in Urine and Blood Samples by Gas Chromatography–Mass Spectrometry after Switchable Solvent Liquid Phase Microextraction Method**

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Oxcarbazepine is a new antiepileptic drug and known as 10-keto analogue of carbamazepine. It is an effective treatment for tonic-clonic seizures in adults and children. However, some adverse effects like dizziness, nausea and vomiting can be observed in patients receiving the relevant drug (1). Some detection techniques for oxcarbazepine can be categorized as high-performance liquid chromatography (2) and gas chromatography (GC) (3) techniques. In addition, preconcentration methods like liquid-liquid microextraction and solid phase microextraction can be used to extract and purify the interest of analytes from biological samples (4). Switchable solvent liquid phase microextraction (SS-LPME) has drawn interests for its high preconcentration yields with less toxicity. Switchable solvents has been known for their changeable miscibilities (5). In this research, trace determination of oxcarbazepine was preconcentrated by switchable solvent liquid phase microextraction (SS-LPME) method. Some parameters such as volume of switchable solvent, concentration and volume of sodium hydroxide and vortex period were optimized to achieve high signal to noise ratios. After performing optimization studies, linear range, limit of detection and limit of quantitation for oxcarbazepine determination were found as 27-354, 6.2 and 20.7 ng/g, respectively. Enhancement in detection power was calculated as 19.1 folds when detection values for GC-MS and SS-LPME-GC-MS method were compared each other. Further study was conducted to check accuracy and applicability of the developed microextraction method. For this purpose, recovery experiments were done for urine and blood samples after their blank analyses. Percent recovery results for urine and blood samples were ranged between 97.7 and 99.6% that proved the method applicability and accuracy for urine and blood samples.

**Keywords:** Oxcarbazepine, Switchable Solvent Liquid Phase Microextraction, GC-MS, Biological Samples

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## **PP48- Effects Of Graphene On Tomato, Cucumber And Leek Seed Germination And Seedling Growth**

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Sustainable use of nanotechnology for agricultural applications requires the understanding of the possible toxicological effects of nanomaterials on plants as well as the life cycle of the plant. The environmental impact of graphene has recently attracted great attention. The present study was aimed to investigate the effects of graphene on tomato, cucumber and leek seed germination and seedling growth. We synthesized water-soluble graphene from natural graphite by using a modified Hummers method and characterized by using scanning electron microscopy. The experimental treatments included three concentrations of graphene (500, 1000, 2000 mg/mL and control (without any graphene) At certain time intervals (2 days), the seed samples were observed for germination and seedling performance. After 20 days of exposure, roots and shoots were separated and washed with water to remove the growth medium and dried to remove the surface water. Their length and fresh weights were recorded. In conclusion our results revealed that graphene played complicated roles in affecting the initial stage of seed germination and subsequent seedling growth. In order to understand underlying mechanisms, further studies at cellular and molecular levels are needed.

**PP49- Determination of Elemental Impurities from Different Pharmaceutical Forms by Inductively Coupled Plasma –Mass Spectrometry and Risk Assessment**

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Elemental impurities in drug products come from several sources. Elemental impurities in drug products exposure a risk to patient health due to toxicological effects, and their levels should be controlled within acceptable limits.

International Conference on Harmonization (ICH) Q3D represents a new approach in the analysis of elemental impurities in pharmaceuticals. In developing method for describing elemental impurities in drug products, the principles of quality risk management, described in ICH Q9, were evaluated according to ICH Q3 D.

Elemental Impurities in three different pharmaceutical products matrixes (pomade, syrup, tablet) were analysed by Inductively Coupled Plasma –Mass Spectrometry (ICP-MS). Twenty four impurities have been classified as Class 1, 2 and 3 according to ICH Q3(D) and all impurities were analysed concomitantly by developing method<sup>1</sup>. Same sample preparation was proceed in all matrixes.

Au, Ag, Se, Sn, Mo, Ru, As, Rh, Pd, Cd, Sb, Ba, Os, Ir, Li, Pt, Tl, V, Hg, Pb, Co, Ni, Cr, Cu were analysed from pharmaceutical preparations. All elements were evaluated for risk assesment according to ICH guideline<sup>1</sup>.

**Key words:** elemental impurities, ICP-MS, elemental impurities in pharmaceuticals.

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**PP50- The Effect of Enantiomer Elution Order on Determination of Minor Enantiomeric Impurity: The Case of Ketoprofen**

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The problem of evaluation of enantiomeric purity of “enantiomerically pure” chiral drug formulations is quite hot topic nowadays. In the recent study it was observed that enantiomer elution order of ketoprofen enantiomers is opposite on two amylose-phenylcarbamate-based chiral columns with the same chemistry of the chiral selector but in one case with coated and in other case with “covalently immobilized” chiral selector. In the present study the effect of this uncommon effect on method validation parameters for determination of minor enantiomeric impurity in dexketoprofen was studied.

The analytical reference standards of racemic ketoprofen and its S-(+)-enantiomer were commercially available from Sigma-Aldrich (St. Louis, MI, USA). Commercially available dosage forms for racemic ketoprofen and dexketoprofen were acquired from the pharmacy shops in Ankara, Turkey and Tbilisi, Georgia. HPLC-grade n-hexane, isopropanol and ethanol, as well as chemical grade formic acid were supplied by Karl Roth (Karlsruhe, Germany). Both coated and covalently immobilized columns were of 250x4.6 mm dimensions packed with silica particles of 5 µm nominal particle size. Agilent 1200 HPLC instrument (Agilent Technologies, Palo Alto, CA, USA) equipped with a G1367C HiP ALS-SL autosampler, G1316B TCC-SL temperature controller, G1311A quaternary pump and G1314D VWD variable wavelength detector was used. HPLC separations were performed at 35°C at 2.00 mL/min mobile phase flow rate if not mentioned otherwise. All mobile phases contained 0.1% formic acid (v/v). UV detection was performed at 254 nm.

As a result of current study, the developed and validated methods with the alternative elution order of enantiomers were applied for evaluation of enantiomeric impurity in marketed dexketoprofen formulations. In most of these formulations the content of enantiomeric impurity exceeded 0.1%.

**Keywords:** Enantiomeric impurity, enantiomer elution order, method validation, dexketoprofen

**Acknowledgements**

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## **PP51- Determination of Some Essential and Toxic Elements in Herbal Teas Consumed in Turkey**

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Tea as being one of the most popular non-alcoholic beverage next to water, may become a dietary source of major and minor elements after brewing<sup>1-3</sup>. For this reason, determination of elemental contents in tea brews is seen among one of the important topics<sup>4-5</sup>.

The components of herbal teas include essential or non-essential elements<sup>5</sup>. Thus, this study assess the amounts of total ten essential or toxic elements from ready-to-use herbal teas obtained from Bursa market. Ten different samples, selected as individual or mixed herbal teas, were studied. Inductively coupled plasma-mass spectrometry was used for elemental determinations. Samples were prepared according to the label information of teas considering the recommended brewing time and quantified after diluting at the appropriate levels. For verification studies, standard reference material of tea leaves were used. Interference possibilities on the results were also evaluated.

*Keywords: Herbal tea, element, ICP-MS*

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**PP52- Comparison methods to determine antioxidant activities: what they mean?**

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Antioxidants help prevent oxidative damage to biomolecules caused by free radicals and are thought to aid in the prevention of many health problems, such as heart disease, cancer, and inflammatory diseases. Antioxidants stabilize or deactivate free radicals, often before they attack targets in biological cells. Although almost all organisms possess antioxidant defense and repair systems to protect against oxidative damage, they cannot prevent the damage entirely. The most commonly used assays of antioxidant activities are Oxygen radical absorbance capacity (ORAC), Ferric reducing antioxidant power (FRAP), Trolox equivalent antioxidant capacity assay (TEAC), cupric ion reducing antioxidant capacity (CUPRAC). Antioxidant capacity assays may be broadly classified as electron transfer (ET)– and hydrogen atom transfer (HAT)–based assays. Although the authors suggest that “the association results from production of reactive oxygen species by preclinical tumors”, the main results show that those evaluations are not completely true. For example, “FRAP was not associated with Colorectal Cancer (CRC) risk”, whereas “reactive oxygen molecule (ROM) levels were associated with increased risk of CRC only in subjects with relatively short follow-up”. Again, So, the capability of an antioxidant to slow down the oxidation of an oxidizable component in a soft drink may be very different from its capability to protect biomembranes against oxidative damage in vivo. Briefly, comparison between different antioxidants is commonly ambiguous. Based on these findings, It is necessary to clear that what mean the results obtained by different studies and methods. Firstly, this is depending on the solvent properties used in the antioxidant methods such as their solubility power for specific species and unhealthy.

In this study, the antioxidant potency/activities obtained by different authors using separate methods mentioned above were compared by giving also the antioxidant molecule concentrations. Again, their roles in the pathogenesis of a given disease contribution to the health benefits such as anticancer, antiinflammation.

*Keywords: ORAC, FRAP, CUPRAC, TEAC, antioxidant activity*

**PP53- Biologically active substances of the *Cyclamen persicum* aerial part****Iryna Kravchenko, Aleksandra Aleksandrova**

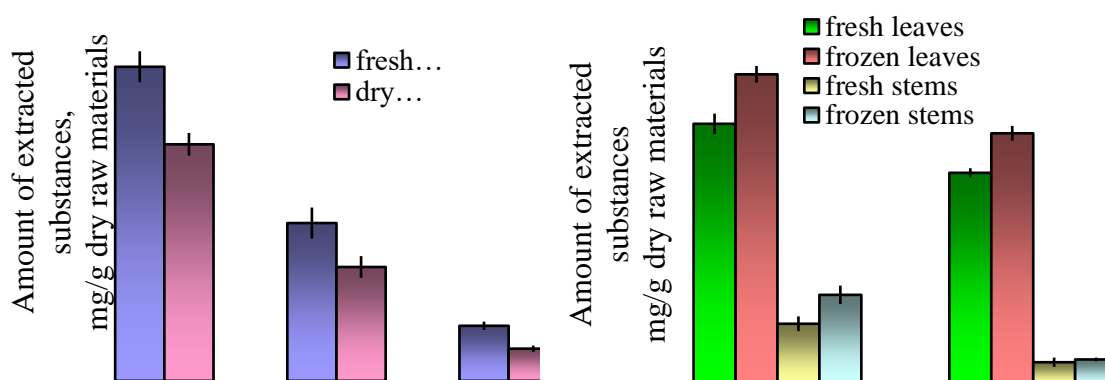
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Currently, the chemical composition of cyclamen tubers is being actively studied. Cyclamen-based preparations intended for the treatment of respiratory organs are particularly effective. The plant juice contains alkaloids, saponins, essential oils, astringents. They have anti-inflammatory and antiseptic effects. In the treatment of colds, sinusitis and antritis drugs containing cyclamen juice are prescribed.

However, in the literature there are no studies on the chemical composition of the aerial part of cyclamen. Therefore, the goal of our work was the determination of biologically active substances in extracts of flowers, leaves and stems of *Cyclamen persicum*. Pink petals were used fresh and dried, leaves and stems were used fresh and frozen. The extracts were prepared using 70% alcohol, the ratio of raw materials to alcohol was 1:10. To extract anthocyanins, the alcohol was acidified with a solution of hydrochloric acid.

In extracts of fresh and dried petals, polyphenolic compounds, flavonoids and anthocyanins were quantified by spectrophotometric methods. The maximum amount of biologically active substances is released when using fresh raw materials under maceration for 7 days: polyphenolic compounds - 60.7 mg, flavonoids - 30.5 mg and anthocyanins - 10.6 mg/g of dried raw materials.

Amount of biologically active substances in the aerial part of the *Cyclamen persicum*

The extraction mechanism of the studied compounds depends on the processing method of the raw materials used for extraction. So, dried raw materials are characterized by leaching of dyes from damaged plant cells and significant factors are the processes of swelling and wetting. Obviously, 7 days is not enough for extraction from dry raw materials.

The use of preliminary freezing and thawing of raw materials leads to an increase in extractable substances due to damage of cell structures and an easier process of diffusion of compounds from the cell contents to the solvent. The content of polyphenolic compounds and flavonoids in the leaves (33.7 and 27.3 mg/g, respectively) is much higher compared to their content in the stems (9.8 and 6.7 mg/g, respectively).

Keywords: *Cyclamen persicum*, polyphenolic compounds, flavonoids, anthocyanins

**PP54- Bioactive compounds from *Rhaponticum acaule* (L.) DC. organs: Study of their antioxidant activity, and their RP-HPLC-PDA analysis.**

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*Rhaponticum acaule* (L.) DC. is a medicinal plant commonly used in North Africa for the treatment of some illness such as gastrointestinal infections, treatment of fever and other affections.

In this work, we report, for the first time, the antioxidant activity of on phenolic compounds in different part of this medicinal plant, and their, RP-HPLC-PDA analysis. The results obtained from the study of the antioxidant activity showed that methanolic extract of roots presented the highest activity, in DPPH<sup>•</sup> scavenging ability test with an IC<sub>50</sub> of 0.31±0.04 mg/mL and in FRAP test with an EC<sub>50</sub> of 1.06 ± 0.02 mg/mL. The highest total antioxidant capacity was observed in butanolic extract of roots part. The RP-HPLC-PDA analysis revealed that there are five phenolic acids including sinapic, caffeic, chlorogenic, ferulic and syringic acids, one flavanone; naringenin), and one flavonol; rutin, and vanillin compound.

**Keywords:** *Rhaponticum acaule*, Asteraceae, Medicinal plants, Phenolic compounds, Antioxidant activity, RP-HPLC-PDA

## PP55- Development of Laboratory Made Needle Trap Devices By Using Conductive Polymers and Its Application to Some Aldehydes

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In recent years determination of some metabolite in biological medium is one of the attractive topic for the diagnosis of disease in scientific community. For this purpose, breath, urine, and blood are used major sample for early diagnosis of diseases. Diseases can be diagnosed by analyzing volatile organic compounds (VOCs), in different biological samples. Literature is growing on the analysis of VOCs biomarker in the exhaled breath for the diagnosis of disease. The concentration of VOCs in biological samples are ppb and ppt range. Therefore, development of standard analysis method having high sensitivity and precision is necessary. Development of gas-sensing technologies is also necessary for monitoring of different VOCs biomarker related to diseases diagnosis. Solid-phase microextraction (SPME) fibers are commonly used for detecting VOCs. Needle trap (NT) sensors are used for alternative to restricted surface area of solid phase microextraction (SPME) technique. Similar to SPME, NT can also be prepared by using electrochemical or sol gel technique (1,4). In this study, NT devices, which contain different type of polymers coated on the inner surface of the needles, were prepared (2,3). For this purpose, it was selected four kind of conductive polymers; polythiophene, polypyrrole, polyaniline and polyfuran. Afterwards, working condition of these NT sensors were optimized and their responses for the targeted VOCs were compared. Calibration graphs were drawn with each NT sensor and their analytical figure of merits each compound were calculated.

**Keywords:** Solid-phase microextraction, conductive polymers, volatile organic compound

**ACKNOWLEDGEMENT-***The authors thank to the Ege University for financial support (Project FDK-2020-21615).*

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## PP56- Cd<sup>+2</sup> Determination by Anodic Stripping Voltammetry Using Hg-Bi Bimetallic Film Electrodes

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Cadmium is a highly toxic metal which used in some industrial fields such as batteries, polymers, paints and electro-plating. Once absorbed from/by the human body, it accumulates in kidneys, liver, lungs and pancreas and lead to significant health problems. These health problems include teratogenic effects, renal dysfunction, hypertension, hepatic injuries and lung injury<sup>1</sup>. This study describes the selective and sensitive electroanalytical method for the determination of cadmium at trace levels with dispersive liquid-liquid microextraction prior to anodic stripping voltammetry using metallic film electrode.

Firstly, studies were performed to make the suitable metallic film for pencil graphite electrode (PGE) which used as a working electrode. pH: 4.5 0.1 M acetic acid and 0.1 M NaOH solution containing 10 ppm of mercury and 1 ppm of bismuth was used as the support electrolyte then metallic film electrodes were prepared using the chronoamperometry method. Then, the obtained results were examined and metallic film electrodes were chosen which gave the best results for cadmium determination. The significant analytical parameters that may affect the efficiency of anodic stripping voltammetry were optimized step by step while the other variables was kept constant (Optimized analytical parameters  $E_{\text{step}}$ : 5 mV, pulse amplitude: 125 mV, pulse time: 5 ms, accumulation time: 120 s, accumulation potential: -1.0 V). Under the optimum conditions, limit of detection, limit of quantification and percent relative standard deviation were obtained as 0.0006  $\mu\text{g L}^{-1}$ , 0.0018  $\mu\text{g L}^{-1}$  and 0,882%, respectively. In order to determine the measurements more precisely under optimized conditions, pre-concentrations were carried out by applying dispersive liquid-liquid micro extraction method to analytes. In addition, the responses of the developed system toward ionic interference species effects were examined. Results obtained showed that the developed method could be applied for the determination of cadmium with high precision and accuracy.

**Keywords:** Cadmium; anodic stripping voltammetry; dispersive liquid-liquid microextraction

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**PP57- Determination of Some Volatile Organic Compounds in the Exhaled Breath of Asthma and Healthy Subjects by Needle Trap Based Sampling Method Coupled with Gas Chromatography Mass Spectrometry**

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There has been an increasing interest in noninvasive tools for asthma diagnosis since early intervention is recommended for preventing aggravation and progression of asthma. In today's clinical practice, asthma is diagnosed and monitored by symptoms and physiological measurements, including lung function tests and the assessment of responses to inhaled pharmacological agents. These tests have been internationally standardized and are generally considered to be reliable. However, they are rather complex, time-consuming, and not widely applicable<sup>1</sup>. Therefore, there is a need for new diagnostic methods in asthma that are simple, fast, accurate, and cost-effective.

Exhaled VOCs are among the most promising biomarkers, and several compounds have been identified as significantly associated with asthma.<sup>2</sup> In this study, NTD based sensitive analysis method was developed and applied for the analysis of VOCs in the exhale breath samples. For this purpose, firstly, experimental parameters such as desorption time and temperature were optimized for gas chromatography mass spectrometry (GC-MS) method. Later, NTD method was applied to breath samples of healthy subjects and asthma patients. Among the 20 variables (compounds), only acetic acid, ethylbenzene and isoprene concentration have relatively high level, and other compounds were low level concentration. The levels of VOC were chemometrically analyzed for the classification of asthma and healthy subject.

**Keywords:** *Exhale breath analysis, asthma, VOC, NTD, biomarker, diagnosis*

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## **PP58- Development of a Method for the Analysis of Human Volatilome Metabolites in Urine Samples**

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Many studies have described that VOCs profiling is an important tool for the monitoring of diseases<sup>1</sup>. Especially in the early diagnosis of cancer, some changes might occur in the level of certain volatile organic compounds (VOC) metabolites when blood contacts with urine. Therefore, studies regarding that these certain metabolites could be significant biomarkers has been increasing. Some of these applications involve the correlation of VOCs with medical conditions of various groups of people, as well as, the correlation with biochemical processes and metabolism.

The most important problem for the clinical use of these volatile markers in urine, is the development of supportive preconcentration kits, which can lead to desired sensitivity, and provide bias selectivity and reproducibility. This in particular requires the development of selective adsorbent materials suitable for the chemical structure of the target compounds. To solve this kind of extraction problems, thin-film microextraction (TFME) as new format of solid-phase microextraction has been created with thin film extraction blades to increase sampling efficiency<sup>2</sup>.

In this study, thin film extraction and thermal desorption followed by gas chromatography with mass spectrometry was applied for determination of volatile organic compounds (Octanal, Nonanal, Decanal, Benzaldehyde, Toluene, Styrene, Phenol, Cyclohexanone, Acetophenone and Pentanone) in human urine samples. The extraction performance of these TFME blades produced in the laboratory was examined by sampling with headspace and immersion methods. The best extraction efficiency for 10 volatilomes was obtained with polydimethyl siloxane coating. Time of extraction, time of desorption in TD system, stirring speed, adsorption temperature and salt effect were optimized. The calibration curves, limits of detection and quantification for all compounds were calculated. This procedure is characterized by very low limits of detection and quantitation and good repeatability for all volatile compounds. This new analytical procedure was identified to be easy, reliable, sensitive, and requires only small amounts of sample.

**Keywords** Gas Chromatography mass spectrometry, Thermal Desorption, Human Volatilome, Urine

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## **PP59- Assessment of the Bioaccessibility and Elemental Levels of Olive Oil by Inductively Coupled Plasma Mass Spectrometry**

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Virgin olive oil is with its phenolics, represents the principal fat component of the Mediterranean diet<sup>1</sup>. Beyond these organics, elemental analysis of olive oils is challenging because of the high organic contents of samples but the low analyte concentrations. In addition, detecting more elements are also important in the context of geographical origin studies by improving the performance of the classification models<sup>2</sup>.

The aim of this work is to assess the elemental levels of olive oil samples obtained from markets. Three different samples were selected and inductively coupled plasma-mass spectrometry was used for elemental determinations. Samples were decomposed using microwave digestion system. Additionally, in-vitro bioaccessibility studies using pepsin and pancreatin enzymes under specified pH values were also studied to calculate the percent bioaccessibilities of selected elements to assess the correlation by dietary consumption for health assurances.<sup>3,4</sup> The potential of the elemental levels for further geographic origin studies were also interpreted.

**Keywords:** Olive oil, element, bioaccessibility, ICP-MS

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**PP60- Contribution to the study of *Rhaponticum acaule* L. from the region of Tlemcen (ALGERIA).**

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Many studies are increasingly interested in the therapeutic effects of medicinal plants, which are mainly due to the activities of secondary metabolites<sup>1</sup>.

In this context, we were interested in the phytochemical study in particular of phenolic compounds (total polyphenols, flavonoids and tannins) as well as alkaloids of a plant from the region of Tlemcen: the roots of *Carlina acaule* L. known by the name vernacular of "Tafgha".

The yield of the crude extract by the two solvents of the roots of *Carlina acaulis* L.DC, (methanol / acetone) reveals percentages of 7.3% and 8.3%, respectively, but the yield of the fraction of flavonoids, tannins and alkaloids of this plant gave levels estimated at 1.7%, 7.6% and 16.5% respectively<sup>2</sup>.

The spectrophotometric assay of the total polyphenols shows that the extract registers 2.67 mg eq AG/g MS, followed by the content of condensed and hydrolysable tannin estimated at 0.49 mg / g and 0.29 mg/g respectively, while the flavonoid assay was performed using the aluminum trichloride method ( $AlCl_3$ ), the results show that it is the lowest content at an average of 0.21 mg /g.

Indeed this plant is rich in compounds endowed with antioxidant activity such as: Polyphenols, flavonoids, tannins and alkaloids, the various constituents exert its antioxidant actions by inhibiting the production of the superoxide anion, the hydroxyl and inhibit the lipid peroxidation at the level of microsomes<sup>3,4</sup>.

**Keywords:** *Carlina acaulis* L., secondary metabolites, dosage of polyphenols.

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## **PP61- Elemental Monitoring Of Rainwater Samples By Inductively Coupled Plasma Mass Spectrometry**

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Trace elements become a part of biogeochemical cycles in the atmosphere after being introduced into the environment by natural and anthropogenic sources.<sup>1</sup> After atmospheric deposition and recycling in the environment globally, these elements are deposited by rain, snow, dry fallout, and over 80% of wet deposited trace metals are dissolved in rainwater, reaching the vegetation canopy at the end.<sup>2</sup> Therefore, studies of the occurrence and determination of heavy metals in ground, surface or harvested rain waters is an important issue for the human health and environment.<sup>3</sup> In international monitoring programs also, measurement of heavy metals in wet deposition is becoming an increasingly central role in the production of basic data.<sup>4</sup>

Rainwater samples were collected from the terrace of EGE-MATAL for 24 hours at different times in the same season from 2017 to 2018 in the Izmir city center/Turkey. Up to 20 elemental levels in acidified 56 samples were determined without digestion by an Elan 9000 ICP-MS composed of a Rytan cross-flow nebulizer coupled to a Scott-type double-pass spray chamber, standard glass torch and nickel sampler/skimmer cones. Recovery studies and three certified water samples were used for validation purposes. Interference possibilities were assessed by isotopic analysis.

*Keywords: Rain water, element, ICP-MS*

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## **PP62- An Example of Drug-excipient Compatibility by Differential Scanning Calorimetry**

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Drug-excipient compatibility is an important field of study to be carried out prior to the formulation of a pharmaceutical dosage form in order to predict physical and chemical interactions or incompatibilities that may affect the safety and effectiveness of the drug. Several techniques have been applied in compatibility studies with excipients, such as differential scanning calorimetry (DSC), thermogravimetry (TG), Fourier-transform infrared (FTIR), X-ray diffraction (XRD), solid-state nuclear magnetic resonance (ssNMR) and isothermal stress testing (IST) coupled with liquid chromatography (LC). Some of these techniques are more commonly used due to fast analyses and low material consumption, such as DSC.

The DSC analysis were performed in a DSC-60A calorimeter (Shimadzu Co., Japan) using the following conditions: a heating rate of 10 °C /min. The excipients (Mannitol and EDTA), Esomeprazole sodium and the mixtures were submitted to further heat scanning from 50 to 300 °C in a sealed aluminum pan. Data analysis was carried out using TA 60 Analysis software.

DSC curve of pure esomeprazole sodium was compared to the curves obtained from physical mixtures of esomeprazole sodium-EDTA and esomeprazole sodium-mannitol. A significant shift in the melting of the Mannitol was observed (from 169.22 to 162.84 °C) and the melting point peak of the esomeprazole sodium (258.52 °C) was disappeared in the physical mixture indicate incompatibility. There were not observed any unusual peaks in esomeprazole sodium-EDTA mixture DSC curves. The analysis carried out by DSC allowed us to demonstrate the reproducible thermal events of pure drug substance and in physical binary mixture of EDTA. This study demonstrated for that esomeprazole sodium was compatible with EDTA promoted physical interaction and chemical incompatibilities by a thermal method.

**Keywords:** DSC, Drug-excipient compatibility, pre-formulation

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