ICABC Congress

5th ICABC 2024

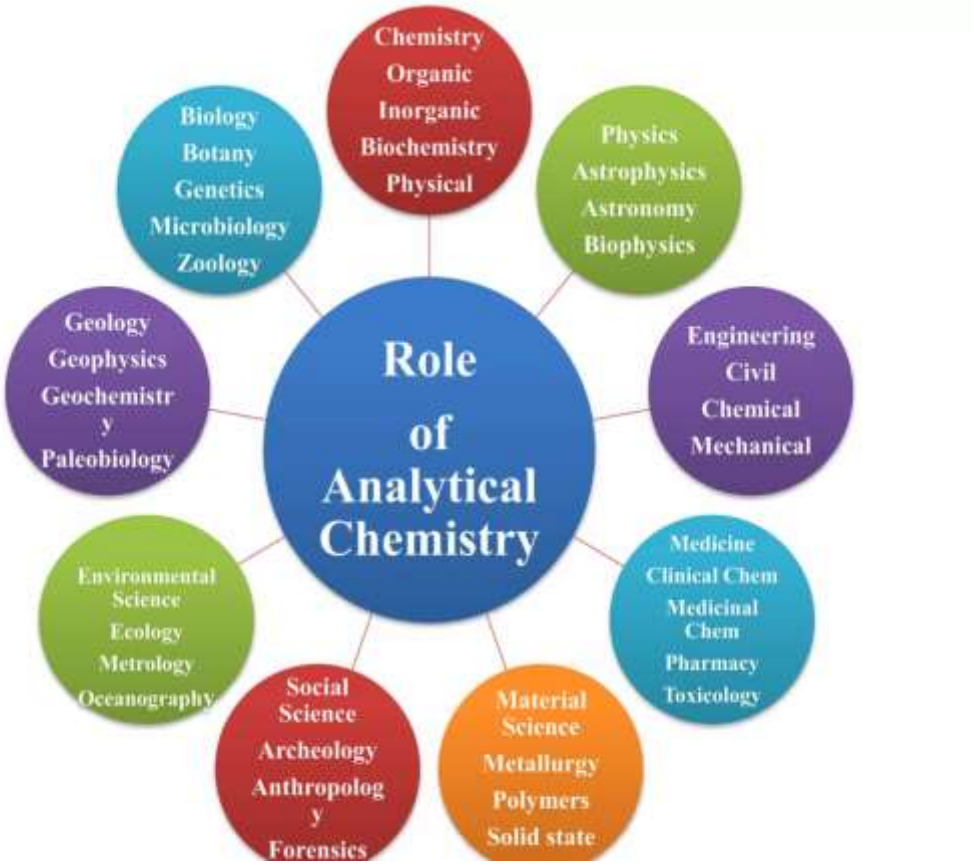
04-07 March 2024-Antalya-Turkey

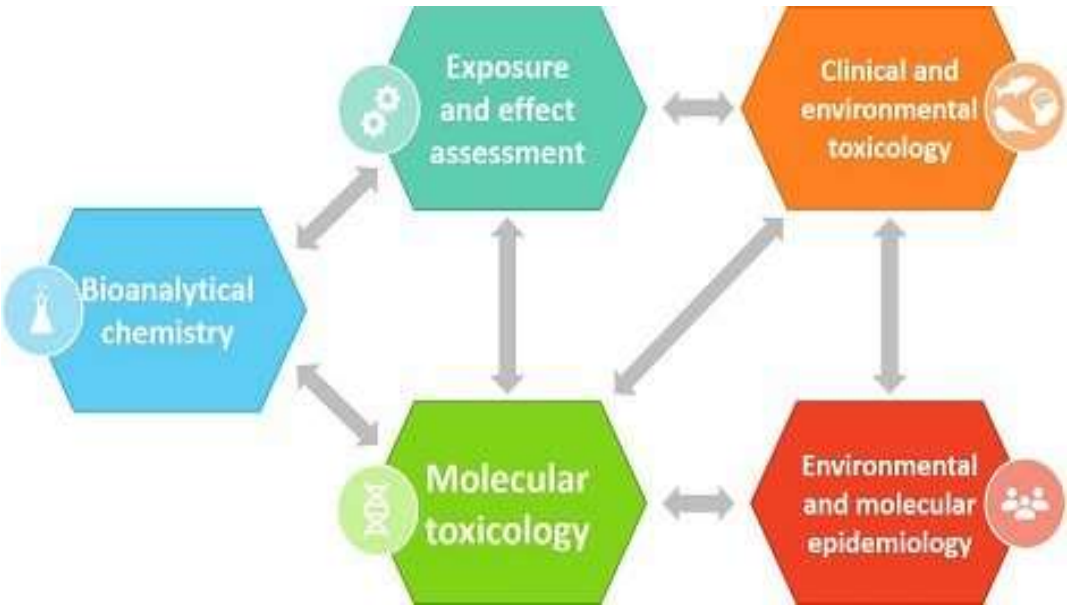
With the sponsors of the Altium Int. and Referans Kimya

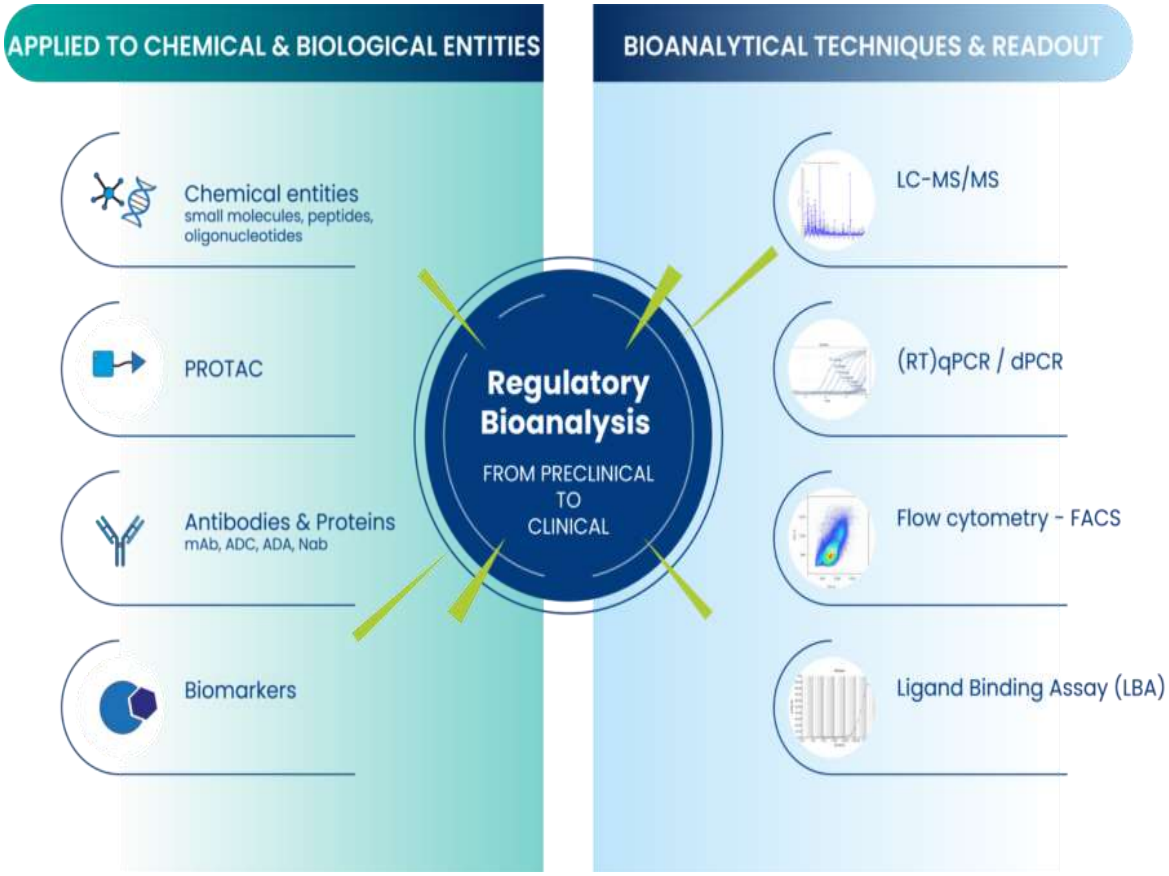
5th International Congress on Analytical and Bioanalytical Chemistry

PROCEEDING BOOK

Editor: Mehmet Yaman




Exposure and effect assessment
Clinical and environmental toxicology
Bioanalytical chemistry
Molecular toxicology
Environmental and molecular epidemiology


APPLIED TO CHEMICAL & BIOLOGICAL ENTITIES
BIOANALYTICAL TECHNIQUES & READOUT
Chemical entities
small molecules, peptides, oligonucleotides
PROTAC
Antibodies & Proteins
mAb, ADC, ADA, Nab
Biomarkers
Regulatory Bioanalysis
FROM PRECLINICAL TO CLINICAL
LC-MS/MS
(RT)qPCR / dPCR
Flow cytometry - FACS
Ligand Binding Assay (LBA)

Preface

The organizing committee of the **5th ICABC 2024** would like to welcome all participants to the "**5th International Congress on Analytical and Bioanalytical Chemistry** ", will be held **in Antalya Porto Bello Hotel** between 04-07 March 2024. The **ICABC** meeting was started five years ago (in 2019) and covers all areas of Analytical and Bioanalytical Chemistry as well as applications of Chemical and Biochemical Analysis.

The scientific congress program consists of **10** sessions that include **11 invited and 44 oral** presentations as well as **28 posters** to be presented in the respective sessions. In addition, researchers of Academia (**37 universities and Institutes from 10 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 **5th 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 to joining, 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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GENERAL INFORMATION

Introduction

The **5th International Congress on Analytical and Bioanalytical Chemistry** will be held on 04-07 March 2024 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.

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.

5th ICABC 2024

Topics

To promote collaboration among analytical and bioanalytical (including biochemists, food engineering, molecular biology and genetics and similars) scientists from different countries, "**5th ICABC 2024**" 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

5th ICABC 2024 will be held in Antalya-Porto Bello Hotel, Turkey

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. Contributed papers describing original research work will be presented as oral and poster in order to promote efficient discussion on new scientific ideas and results. All presentations should be in English. Oral and poster presentation will be accepted if at least one of the authors is registered and present at the conference for personal communication.

OPENING SPEECH

Dear reputable Professors, Colleagues and Participants,

I am very happy to welcome all the participants coming to "**5th 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 for up to date research is gaining importance.

This Congress mainly aims to promulgate knowledge in Life Science Analysis and Industrial Analytical techniques. Both life and Industrial sciences need Analytical and Bioanalytical Techniques in course of research work and therefore, Analytical Meetings would be a perfect venue to share and develop knowledge on key Analysis tools.

Related with Bioanalytical Methodology,

The guideline impact of bio-investigation in the pharmaceutical region is to gain a quantitative proportion of the medication and its metabolites. The purpose behind existing is to play out the pharmaco-kinetics, toxic-energy, bioequivalence and introduction response like pharmacokinetics-pharmaco-dynamics examines.

So, analytical chemistry is indispensable of bio-scientific strategies and hyphenated instruments in assessing the bio-examination of the drugs including,

Hyphenated techniques, Chromatographic strategies, and Ligand bio-diagnostic procedures.

The first objective of this congress is to provide the opportunity for researchers interested in different disiplines to come together and to exchange ideas in analytical-perspective meetings.

This congress was launched for all those 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 10 sessions that include 11 invited and 44 oral presentations as well as 28 posters. The participants are of 37 universities and Institutes from 10 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.

Sponsors:

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

- **Altium International Lab. Cihazları**
- and
- **Referans Kimya**

I wish you all to enjoy this conference.

- I wish the congress will be useful.
- With my best regards.

Prof. Dr. Mehmet Yaman-
Chair

CONFERENCE PROGRAM

5th International Congress on Analytical and Bioanalytical Chemistry (5th ICABC 2024)

04-07 March, 2024, Antalya/Turkey

	04 March, 2024
16.00-16:30	➢ Welcome Ceremony ➢ Prof. Dr. Mehmet Yaman (Chair) Prof. Dr. Seref GUCER (on behalf of continuation committee) *Honorable*
16:30-17.00	***Inv. 1: Prof. Dr. Antony CALOKERINOS*** – ***Athens U/GR*** ***Artificial Intelligence and Analytical Chemistry***
17:00-17:40	**OP1: Izabela Nowak-** Testing of cosmetic products designed for various purposes - a brief overview of available analytical methods
	OP2: Katarzyna Bierla-HPLC-ICP MS and ESI MS – convenient analytical tools for the assessment of selenosugars, selenocysteine, and other selenometabolites expression in livers of animals fed with organic and inorganic selenium
	OP3: Emad A. Khudaish- A novel sensor based on grafting a polymeric film with electrochemically reduced graphene oxide: Fabrication, characterization, and potential analysis on ephedrine
	OP36: Ozge CAGLAR- A New Approach to Immobilization Porcine Pancreatic Lipase Using Copper-Based MOFs: Mechanochemical Encapsulation
	05 March, 2024
	Session 1- Chair: **Prof. Dr. F. Durişehvar ÜNAL- Dr. Cecylia Wardak**
09:00-09:40	***Inv. 2: Prof. Dr. Patrik SPANEL -The Czech Academy of Sciences/CZ*** ***Selected Ion Flow Tube Mass Spectrometry, SIFT-MS, for analyses of volatile compounds: food flavour, storage and spoilage***
09:40-10:10	**OP4: Neşet Neşetoğlu-** Analytical Method validation challenges in analyzing dissolution profile by Liquid Chromatography-Tandem mass spectrometry
	OP5- Kartal Çetintürk- Multiple Headspace Extraction along with cryostatic cooling inlet - GCMSD for determination of volatile organic compounds in marine water and wastewater samples
	OP6- Hilal Akbıyık- Simultaneous Determination of Pyridalyl and Metalaxyl by Gas Chromatography-Mass Spectrometry after Combination of QuEChERS and Orbital Shaker-Assisted Switchable Solvent Microextraction
10:10-10:30	Tea/Coffee break
10:30-11:10	***Session 2-*** Chairs: ***Prof. Dr. Almira RAMANAVICIENE-Assoc. Prof. Dr. Sevinç Kurbanoğlu*** ***Inv. 3: Prof. Dr.* Arunas RAMANAVICIUS** - **Vilnius U./LT** ***Affinity sensors for the diagnosis of COVID-19***
11:10-12:10	**OP7: Pınar Kara-** Aptasensor Development Based on Transition Metal Oxide for Diagnosis of Prostate Cancer
	OP8- Dilek Öztürk- Carbon Quantum Dots Based Fluorescent Nanosensor Platforms for Detection of Gastric Cancer-Associated Genes
	OP9-Mahitha Pulithitta Mohanan- An electrochemical sensor based on molecularly imprinted polymer supported by ruthenium pincer complex and multi-walled carbon nanotube for the detection of anticancer drug Nintedanib
	OP10- Elifcan Emiroglu Bolukbas- Electrochemical DNA Biosensor for the Investigation of Antioxidative Activity of Turmeric
	OP11- Murat Ata Ülkü- Rapid and Sensitive Determination of I-Glutamate in Food Samples with Using Capillary Electrophoresis Coupled with Contactless Conductivity Detector
	OP12- Berna Nis- Efficient Synthesis of 5-Hydroxymethylfurfural from Glucose using Ionic Liquid MIL 101(Cr) Composite Catalysts: Towards Sustainable Biomass-Derived Chemicals
12:10-14:00	**Lunch**

	Session 3 - Chairs: **Prof. Dr. Sezgin Bakırdere- Prof. Dr. Özlem Söğüt**
14:00-14:40	**Inv. 4: *Prof. Dr. Ryszard LOBINSKI*– Pau U-FR** ***Analytical approaches for the monitoring of the environmental pollution by microplastics***
14:40-15:10	**OP13- Usama Alshana**- Smartphone digital image colorimetry combined with liquid–liquid- and solid-phase microextraction: A new analytical tool for chemical analysis
	OP14- Selim Gürsoy- Investigation of the Removal of Cadmium from Aquatic Medium Using Tin Oxide: Adsorption Kinetics and Isotherm Studies
	OP15- Gamze Dalgıç Bozyiğit- Adsorptive Removal of Cadmium by Al2O3 Nanoparticles from Domestic Wastewater: Langmuir Isotherm Model Studies
15:10-15.30	*Tea/Coffee break*
15:30-16:30	***Session 4 –*** Chair: ***Prof. Dr. F. Nil ERTAŞ*** ***POSTER PRESENTATIONS***
	Session 5: Chairs: **Prof. Dr. İzabela Nowak- Prof. Dr. Pınar Kara**
16:30-17:00	**Inv. 5: Prof. Dr. Hüseyin ÇELİKKAN-** ***Gazi U/TR*** ***Impedimetric measurements in analytical chemistry***
17.00-18.00	**OP16- Konrad Rudnicki-** Electrochemical Study of Veterinary Drug – Danofloxacin – at Glassy Carbon Electrode and Electrified Liquid-Liquid Interface
	OP17- Mutlu Sönmez Çelebi- Poly(thionine) Supported PtPd Bimetallic Catalyst for Electrochemical Oxidation of Ethanol
	OP18- Karolina Sobczak- Electrochemical Detection of one of the β-Blocker - Nebivolol at the Polarized Liquid-Liquid Interface
	OP19- Karolina Kowalewska- Synthesis of polyamide materials at the polarized liquid – liquid interface
	OP20- Sevinc Kurbanoglu- Electrochemical Enzyme-Based Biosensors and Their Inhibition Applications
	OP21- Pınar Bozbeyoğlu- Chemometric Characterization of Kome with Pikola Hazelnut and Triangle-Shaped Mulberry Leather (Pestil): Exploring the Role of Proximate Analysis and Multi-Element Analysis
	06 March, 2024
09.00-09:40	**Session 6-** Chairs: **Prof. Dr. Aysu Yarman- Dr. Konrad Rudnicki**
	Inv. 6: Prof. Dr. Frieder W. SCHELLER- ***Potsdam U/DE*** ***Achievements and remaining challenges of electrochemical MIP -sensors***
09:40-10:10	**OP22-Raghad Alhardan**- Development of a screen-printed electrochemical biosensor based on enzymatic inhibition for Anabaenopeptin B detection
	OP23- Andrzej Leniart- Evaluation of the suitability of glassy carbon paste electrode modified with bismuth(III) oxide for electrochemical analysis of diphenoxuron
	OP24- Barbara Burnat- Incorporation of bismuth(III) oxide nanoparticles into carbon ceramic electrode for improved electroanalytical performance in 4-chloro-3-methylphenol determination
10:10-10:20	*Tea/Coffee break*
10:20-10:50	**Session 7-** Chairs: **Prof. Dr. Agnieszka Nosal -Wiercińska- Prof. Dr. Robert Pietrzak** ***Inv. 7:*** **Prof. Dr. *Mutay ASLAN*- Akdeniz U/TR** ***The Role of Ceramide in Diabetic Dyslipidemia from a Biochemical Perspective***
10:50-12:00	**OP25- Cigdem Yengin-** Comprehensive Chromatographic Investigation of Anatolian Cyclotrichium niveum, significance of raw chromatographic data for chemometric discrimination of extracts
	OP26- Cemile YÜCEL- Optimization of on-line HS-SPME method combined with GC-MS/MS for determination of UV filters in wastewater
	OP27- Barış Gümüştaş- Authenticity Control of Sea Bream Products by Stable Carbon Isotope Ratio Analysis and Chemical Composition
	OP28- Elif Öztürk Er- Production, Characterization and Evaluation of Application Areas of Bi2S3, Al2O3, NiCO2O4 Nanoflowers using Microwave-Assisted Synthesis Method
	OP29- Efe Sinan Aydın- Flower-Shaped Ni(OH)2 Nanomaterial's Analytical Application in Preconcentration of Manganese Determination in Domestic Wastewater Samples by Flame Atomic Absorption Spectrometry
	OP30- Serdar ŞANLI- Qualitative Detection of Toxoplasma gondii with Graphene/Chitosan/Toxoplasma gondii Antibody-Modified Graphite Electrode

	OP31- Serhat Kocer- Evaluation of Chestnut Shell Bioactive Compounds and Color Properties Using Ultrasound Assisted Extraction
12:00-12:20	***Altium International*:Berk Müjde-**Rapid determination of five arsenic species in polished rice using HPLC-ICP-MS
12.20-14:00	**Lunch**
14.00-14.40	**Session 8-: Chairs: Prof. Dr. Malgorzata Grabarczyk– Prof. Dr. Dilşat Arıksoysal**
	Inv. 8: *Prof. Dr. Sezgin BAKIRDERE- Yildiz Tech. U.-TR*
	New Trends in Analytical Chemistry for the Determination of Organic Analytes
14:40-15:30	**OP32- Buse Tuğba Zaman-** Removal of Cadmium Ions from Synthetic Wastewater Samples by CuFe2O4 Magnetic Nanoparticle Assisted Batch Type Adsorption Based Removal Strategy
	OP33- Nursu Aylin Kasa- Sieve Conducted Two Syringe Based Mixing System Assisted Preconcentration and Determination of Nickel in Wastewater Samples by Slotted Quartz Tube Flame Atomic Absorption Spectrometry
	OP34- Ümit Can Erim- Deep Eutectic Solvent Based Preconcentration of Sulfonamides in Honey Samples and Their Determination by High Performance Liquid Chromatography
	OP35- Şeyda KIVRAK- Development Of Prototypes From Honey Bee (Apis Mellifera L.) Venom That Can Be Used In Cosmetic And Pharmacopuncture Applications
	OP36-in the first day
15:30-15:50	**Tea/Coffee break**
15:50-16:20	**Session 9-: Chairs: Prof. Dr. Slawomira Skrzypek - Prof. Dr. Hasan Ertaş**
	Inv. 9:* Prof. Dr. Mustafa Kemal SEZGINTÜRK- *Çanakkale 18 Mart U/TR
	A Potential Tool for Analytical and Bioanalytical Applications: Quartz Tuning Forks
16:20-16:50	***Inv. 10:* Dr. Mariola BRYCHT-*Lodz U/PL***
	Electrode materials based on boron-doped diamond: Factors influencing their properties and electrochemical performance
16:50-17:10	**OP37- Almira Ramanaviciene-** Gold Nanoparticles for the Sensitive Detection of Biomarkers using Surface Plasmon Resonance Immunosensors
	OP38- Gamze Emir Günay- Cathode Design with MWCNT-Mn3O4-PtNps Modified Pencil Graphite Electrode for Enzyme-Nanozyme Based Biofuel Cell.
17:10-18:10	**SEASIDE-WALKING**
	07 March, 2024
	Session 10- Chairs: Prof. Dr. Emad A. Khudaish -Assoc. Prof. Dr. Usama Alshana
09:00-09:30	***Inv. 11:* Prof. Dr. Yusuf DILGIN, *Canakkale 18 Mart U/TR***
	New approaches in CUPRAC method: Integration to enzyme-based optical biosensors, and use as a redox mediator in electrochemical sensing
09:30-10:30	**OP39- Muhammed Kaan Tonoz-** Spectroscopic Analysis of Curcumin Samples' Antioxidant Capacities
	OP40- Nagehan Kübra Zeytinci- Determination of Palladium at Trace Levels by Using CoS@ppy@Fe3O4 Nanocomposite Assisted Dispersive Solid Phase Extraction Procedure
	OP41- Melike Atakol-Determination of nickel at trace levels in Saint John's wort tea by using a solidification of floating organic droplet-based liquid-liquid microextraction method followed by matrix matching assisted slotted quartz tube-flame atomic absorption spectroscopy
	OP42- Arda Atakol- Simple and efficient adsorption of nickel from paper industry wastewater samples by utilizing magnesium ferrite nanoparticles synthesized by the sol-gel process
	OP43- Ayşe Hanbeyoğlu- Plant of Hypericum Lydium Boiss. in Human Health Role as Antioxidant
	OP44- Heshw Ali Omer-Determination of antiradar and thermal conductivities of reduced graphene oxide/epoxy nanocomposites
10:30-11:00	**Closing**

INVITED SPEAKERS (IS)

IS1- Selected Ion Flow Tube Mass Spectrometry, SIFT-MS, for analyses of volatile compounds: food flavour, storage and spoilage

Španěl Patrik[1*]
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The need for rapid and accurate measurement of trace concentrations of airborne compounds has prompted the development of specialised mass spectrometers, including Proton Transfer Reaction Mass Spectrometry (PTR-MS), Selected Ion Flow Tube Mass Spectrometry (SIFT-MS), Secondary Electrospray Ionization Mass Spectrometry (SESI-MS), and Dielectric Barrier Discharge Mass Spectrometry (DBD-MS). In comparison to Gas Chromatography-Mass Spectrometry (GC-MS), these techniques excel in sample throughput and real-time monitoring capabilities.

Whilst all these new techniques can analyse volatile organic compounds and reactive gases (ammonia, suplhane ...), SIFT-MS stands out due to its unique ability to provide absolute quantification. This is achieved by on gas phase ion-molecule reaction kinetics of H_3O^+, NO^+, and $O_2^{+\bullet}$ reagent cations together with $O^{-\bullet}$, OH^-, $O_2^{-\bullet}$, NO_2^- and NO_3^- reagent anions. 1,2 In SIFT-MS, these ion-molecule reactions occur within a flow tube during a precisely defined reaction time of 5 milliseconds, with the sample introduced at a known flow rate. Detection limits typically fall within the parts-per-trillion by volume (pptv) range. SIFT-MS finds applications in various fields, from real-time analysis of humid exhaled breath to industrial air quality monitoring.

Recent technological enhancements, including the use of nitrogen as a carrier gas instead of helium and the inclusion of reagent anions, have further improved the analytical capabilities of SIFT-MS.

This lecture will present recent applications of SIFT-MS in food science, highlighting examples such as flavour analyses of coffee, monitoring spoilage and freshness of leafy salads and fresh Atlantic salmon, as well as the characterization of regional origin and detection of adulteration. The outcomes will be elucidated in terms of specific analyte quantities and patterns, employing multivariate statistics for interpretation.

Keywords: Soft Chemical Ionisation Mass Spectrometry, Selected Ion Flow Tube Mass Spectrometry, Volatile Organic Compounds

References

1- Smith D., Španěl P., Demarais N., Langford V.S., McEwan M.J., Recent developments and applications of selected ion flow tube mass spectrometry, SIFT-MS, Mass Spectrom. Rev., (2023) e21835.
2- Dryahina K., Som S., Smith D., Španěl P., Characterization of spoilage-related volatile organic compounds in packaged leaf salads, Flavour Fragrance J., 35 (2020) 24-33.

Acknowledgment: Funding is acknowledged by the Czech Science Foundation (Grantová Agentura České Republiky, GACR; Project No. 21-25486S) and from the Praemium Academiae funding by the Czech Academy of Sciences.)

IS2- Mass spectrometry approaches for the monitoring of the environmental pollution by microplastics

Ryszard Lobinski, Javier Jimenez Lamana and Joanna Szpunar
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The discharge of plastic waste into the environment has led to an increase in the presence of small plastic particles known as microplastics or nanoplastics. These particles behave differently than larger plastics, raising concerns about their potential impacts on ecosystems, ecological consequences, human health, and the overall well-being of the planet. Consequently, there is a need for the development of analytical techniques to assess the extent of plastic pollution in various environmental compartments, exposure levels, and the potential transfer of micro- and nanoplastics along the food chain.
Inductively coupled plasma mass spectrometry (ICP-MS) in the single-particle mode (SP-ICP-MS), which offers exceptional detection capabilities, has been investigated as a potential candidate for analyzing micro- and nanoplastics. Different strategies, including monitoring natural plastic particles (such as the C13 carbon isotope and fluorine present in PTFE) and the use of metal tags, have been proposed. LC-MS and GC-MS were used to evaluate microplastics as carriers of environmental pollutants.
Advances in the analytical chemistry of microplastics will be illustrated through two case studies. An untargeted study of multiclass contaminants associated with microplastics (MPs) in the East Mediterranean was conducted. Deconvolution of accurate GC–MS scan data allowed the identification of >130 organic pollutants. Untargeted LC-MS demonstrated the persistence of several pesticides and pharmaceuticals. The second study focused on the development of SP-ICP-MS using the 13C isotope. Plastic microparticles up to 5 µm were completely volatilized and their components atomized, enabling the detection and measurement of particle size distribution. The developed method was applied to screen microplastics in personal care products and those released from food packaging.
The complementary features of different analytical methods for a comprehensive characterization of the risks posed by micro- and nanoplastics will be highlighted.

Keywords: microplastics, single-particle ICP MS, environmental pollution

IS3- Achievements and remaining challenges of electrochemical MIP-sensors

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Abstract The straightforward synthesis from one up to six functional monomers and the simple integration into a sensor are significant advantages of MIPs as compared with enzymes or antibodies. Furthermore, they can be synthesized against toxic substances and structures of low immunogenicity and allow multi-analyte measurements via multi-template synthesis. The affinity is sufficiently high for protein biomarkers, DNA, viruses and cells. However, the cross-reactivity of highly abundant proteins is still a challenge.
Keywords: MIP-sensors, artificial antibodies, biomarkers, cross-reactivity

1. Land Marks of Bio (mimetic) Sensors

Almost 100 years ago Warburg developed the NADH-based optical test for the investigation of the cell´s respiration. This principle of coupling a biological reaction with a physical readout device is the basis of the later enzyme test strips, enzyme electrodes, immunosensors and nucleic acid chips. Still in 1931 the Russian scientist Polyakov proposed the first polymer with molecular memory which mimicks the function of antibodies: Complementary binding sites resembling the "Lock and key principle" of
biomolecular recognition are formed by "templating" the target molecule in a polymer matrix. Initially, imprinted polymers were developed exclusively for low-molecular-weight substances, and these synthetic sorbents coined the name Molecular Imprinted Polymers (MIPs). Later imprinting of large biological molecules and viruses and cells has been accomplished[1].

2. Present Status of MIPs for Bio-macromolecules

MIPs using one up to eight monomers for low-molecular but also macromolecular targets exhibit
- K_d-values in the range from µM to pM i.e. the Free Enthalpy is between -30 and -60 kJoule and
- cross reactivities above two percent.

Since both exothermic and endothermic binding Enthalpies have been reported for MIPs the entropic term should be responsible for the driving force (overall negative Delta G). Because "real samples, e.g. blood serum", contain the analytes typically in the sub-nM range but potentially interfering constituents in in the mM-range the MIP should be saturated by the interferent.
Dilution cannot solve this problem.

3. Characterization, synthesis and application of (electrochemical) MIP-sensors

3.1 Direct vs. indirect electrochemical readout

All steps of MIP preparation, rebinding and regeneration have been characterized by the three approaches[2,3]:
The modulation of the diffusional **permeability of a redox marker** reflects the occupancy of the binding pockets by the target analyte but it is superimposed by all changes of the polymer film and non-specific adsorption. Indication of the **catalytic activity** sums up the action of all enzyme molecules bound to both the binding sites and on the non-imprinted polymer surface. The **electrochemical conversion** of electro-active analytes- including redox enzymes-involves exclusively the molecules which are in spatial contact with the electrode surface.

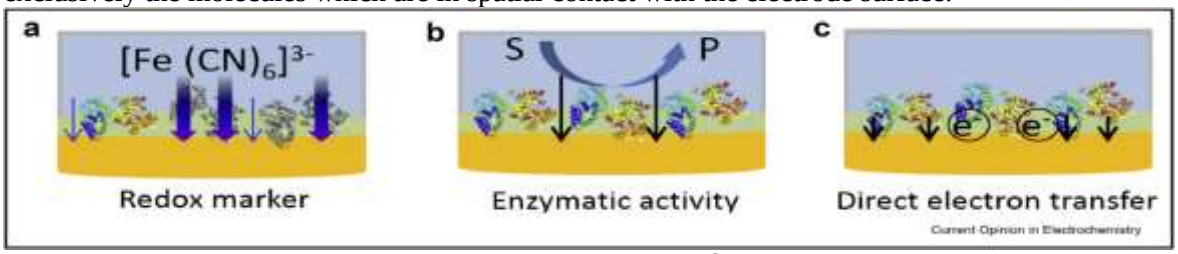

Figure 1: Principles of electrochemical readout of affinity sensors[2]

3.2 Random MIP *vs.* Hierarchical MIPs

Oriented fixation of the template on the electrode surface (via the terminal Cys) followed by the deposition of the polymer film around the template (hierarchical MIP) results in in higher affinities (Tab.2) as compared with random MIPs which are prepared by the polymerization of template-monomer mixtures (random MIP)[4].

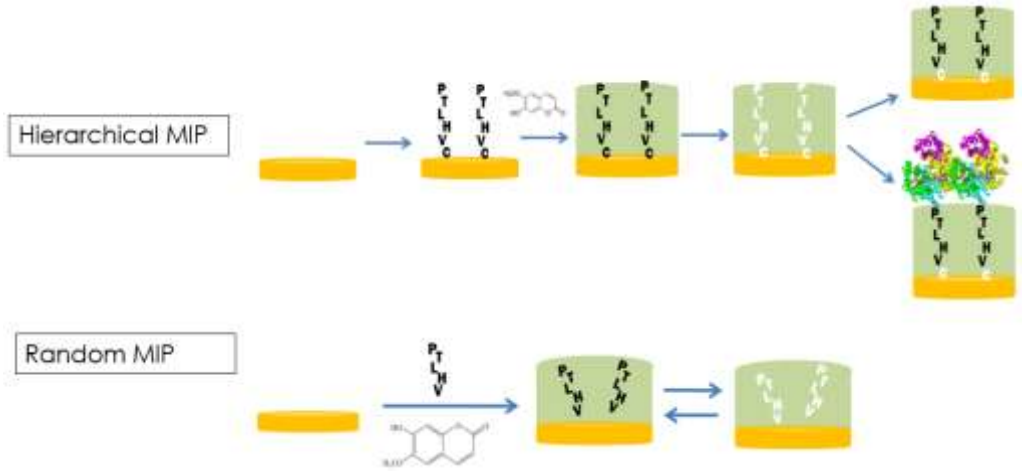

Figure 2: Schematic representation of two basic approaches to prepare HbA-MIPs.

Table 1 K_d values for binding of the target peptide and HbA to the hierarchical and mixture based MIPs.

MIP	**Target**	**K_d**
hierarchical MIP (**C**VHLTP template)	VHLTP	64.6 nM
	HbA	9.06 nM
Mixture-MIP (VHLTP template)	VHLTP	1.4 µM
	HbA	20.4 nM

3.3 Epitope imprinting *vs.* whole protein templating

MIP nanofilms prepared by electropolymerization of scopoletin for the protein biomarkers alpha-fetoprotein (AFP) and the receptor binding domain (RBD) of the spike protein of SARS-CoV-2 [5] using either a peptide (epitope-MIP) or the whole protein (protein-MIP) as the template showed comparable affinity towards the resp. protein target (Tab.2).

Table 2 Dissociation constants K_d and binding capacity B_{max} for AFP and RBD.

	AFP		**RBD**	
	Epitope-MIP	Protein-MIP	Epitope-MIP	Protein-MIP
K_d (nM)	13.2 ± 1.7	14.5 ± 3.0	14.7 ± 0.9	16.8 ± 1.1
B_{max} (%)	87.0 ± 2.5	98.5 ± 4.6	57.9 ± 0.9	92.5 ± 1.3

4. Conclusion

- Higher **SENSITIVITY** of hierachical MIPs is reflected by 10 to 20 fold lower K_d than for the respective „mixture MIPs“ (N-terminal Hb-peptid, StrepTagII).
- Epitope-MIPs and the respective whole protein-MIPs for AFP and RBD exhibit comparable **AFFINITIES** for the resp. target protein but lower **non-specific binding** and 50-fold lower **reagent costs** for epitope-MIPs.

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IS4- New Trends In Analytical Chemistry For The Determination Of Organic Analytes

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Organic contaminants, which are ubiquitous chemicals forming naturally or synthetically, contain the variety of chemical groups such as pesticides, surfactants, endocrine disruptor compounds, pharmaceuticals and the other compounds [1]. These chemicals threaten the health of human and environment, therefore; an accurate and sensitive analytical method is required to qualify and quantify these chemicals in several matrices at trace levels. Sample preparation have a crucial role in analytical chemistry before qualitative or quantitative determination to remove matrix interferences and decrease detection limits by preconcentrating the analyte(s) [2]. Extraction is a one of the sample preparation methods to eliminate possible matrix effects, however; conventional extraction methods have several tedious steps and require high-volume organic solvent consumption [3]. Nowadays, several solid phase (micro)extraction [4–7] and liquid phase microextraction methods [6,8,9], which are met the requirements of green analytical chemistry, have been introduced to the literature for the extraction and preconcentration of various organic analytes in several matrices. In most cases, chromatography based analytical instruments such as liquid chromatography and gas chromatography with suitable detectors have been employed to determine the organic contaminants in food, environmental and biological fluid samples. Although these analytical methods are advanced instruments, a sample preparation method should be used to form a suitable sample and eliminate possible matrix interferences in order to achieve more accurate results and low detection limits before sending the sample to the proper chromatographic system. Furthermore, several calibration strategies such as matrix matching calibration, standard addition and isotope dilution methods instead of external calibration method can be used to obtain more accurate analytical results.

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IS5- Analytical Chemistry and Artificial Intelligence

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The terms Artificial Intelligence (AI), Machine Learning (ML) and Deep Learning (DL) are used very often during the last decade. Artificial intelligence (AI) is the ability of a digital computer or computer-controlled robot to perform tasks commonly associated with intelligent beings. In simple words, Artificial Intelligence is a computer software that mimics aspects of human intelligence! Artificial Intelligence is supported by Machine Learning which uses algorithms to perform a variety of complex tasks and Deep Learning performs more complex tasks without any human intervention. Google maps, Text autocorrect programs, ChatGPT, self-driving cars and buses are some typical examples of Artificial Intelligence.

Food quality control and Cheminformatics have expanded tremendously by the support of Artificial Intelligence. Nevertheless, the advantages of Artificial Intelligence in Analytical Chemistry have not been fully developed.

Theoretically, Artificial Intelligence would be able to automate instrument calibration and diagnostics, optimize experimental conditions, perform multiple measurements, verify quantitative and qualitative results, and define unknown compounds from different types of spectra.

Chemometrics is an excellent example of application of Artificial Intelligence in Analytical Chemistry Spectroscopic and chromatographic data can be elaborated qualitatively and quantitatively successfully by Artificial Intelligence. A plethora of current examples of Artificial Intelligence in Analytical Chemistry will be presented and discussed.

The basic principles of Artificial Intelligence as well as current and possible future applications in Analytical Chemistry will be presented and briefly discussed during the lecture.

IS6- Electrode materials based on boron-doped diamond: Factors influencing their properties and electrochemical performance

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Boron-doped diamond (BDD) is a well-known electrode material with a range of exceptional properties, including a sufficiently wide potential window (especially in the anodic region), low and stable background current, chemical inertness resulting in lower susceptibility to adsorption and fouling, and biocompatibility[1-4]. Due to these properties, BDD-based materials have found broad applications in the electroanalysis of various biologically important organic compounds1. However, analytical performance of BDD may be further enhanced by enlarging the effective surface area. As a consequence, approaches aiming to fabricate nanostructured materials based on BDD have been emerging. Nanostructured materials based on BDD demonstrate better detection capability compared to conventional planar BDD[5,6].

The unique properties of BDD-based materials depend, among other factors, on the applied technique and conditions for depositing BDD from the gas phase, surface morphology, boron and sp2 carbon content, and surface functional groups. Understanding the role of these individual factors represents a significant challenge.

During the lecture, factors influencing the properties and electrochemical performance of electrode materials based on BDD will be discussed[2-6]. Examples will also be presented showcasing the use of thin BDD layers in the construction of electrochemical sensors and effective methods for detecting biologically significant organic compounds such as drugs1, pesticides3,4, and neurotransmitters[5,6].

Keywords: boron-doped diamond, surface morphology, surface termination, boron content, electroanalysis

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IS7- A Potential Tool for Analytical and Bioanalytical Applications: Quartz Tuning Forks

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QTF (Quartz Tuning Fork) sensors have become popular in recent years and can be used as temperature, humidity, pressure and, most importantly as biosensors. Since there are no commercially available devices that can use QTF sensors, their widespread use is limited. When the QTF crystal is excited with a signal with a frequency close to the fundamental resonant frequency, a signal with maximum amplitude is obtained[1]. Basically, AC signals are generated by scanning a certain frequency range with a signal generator. With these signals, the QTF crystal is excited. For each frequency value, the amplitude of the signal output from the excited QTF crystal is measured. The frequency value corresponding to the point where the highest amplitude read is determined as the resonance frequency of the QTF crystal.

Biosensors based on quartz tuning forks are often used in medical and environmental applications, such as in monitoring the stiffness of blood vessels, detecting changes in the viscosity of blood, or measuring the concentration of certain biomolecules in a sample. These sensors can provide highly sensitive and accurate measurements, and are often used in combination with other detection methods for improved sensitivity and specificity [2, 3].

QTF based biosensing systems have been developed for different biological relevant targets with all of their electronically devices by our research groups. In this work, these QTF based biosensing systems are presented with their fabrication steps and optimization and characterization parameters. Following this, the advantages and disadvantages of the system are discussed with the future perspectives.

Keywords: QTF, Quartz Tuning Forks, Biosensor, Immunosensor, Sensor

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IS8- New approaches in CUPRAC method: Integration to enzyme-based optical biosensors, and use as a redox mediator in electrochemical sensing

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The CUPRAC (Cupric Ion Reducing Antioxidant Capacity) method using bis-neocuproine copper (II) complex ([Cu(Nc)2]2+) was first developed by Reşat Apak et al., in 2004 for the determination of total antioxidant capacity1. The colorimetric CUPRAC method depends on the conversion of the slightly blue [Cu(Nc)2]2+ to yellow-orange bis-neocuproine copper (I) complex ([Cu(Nc)2]+) in the presence of a reductant (antioxidant), and many optical sensors were developed by monitoring the absorbance of cuprous-neocuproine chelate at 450 nm.

In our recent studies, two new approaches for this useful chromogenic oxidant have been proposed, namely the development of enzyme-based optical biosensors as well as electrochemical sensing based on its use as a redox mediator. In the first approach, the CUPRAC reagent was integrated into enzyme-based optical biosensors2. In the first step, enzymes such as oxidase, dehydrogenase, and acetylcholine esterase (AChE) were immobilized on the silanizated magnetite nanoparticles. In the second step, enzymatic reactions were accomplished between enzyme-immobilized nanoparticles and substrate (glucose, cholesterol, acetylthiocholine, etc.). In the final step, a colorimetric reaction was realized between enzymatically produced products (for example H2O2 from oxidase enzyme, NADH from dehydrogenase enzyme, thiocholine from AChE, etc.) and [Cu(Nc)2]2+. Thus, the optical biosensor was constructed based on measuring the absorbance of the yellow [Cu(Nc)2]+ product formed at 450 nm. Using this approach, glucose, cholesterol, and lactate biosensors based on their oxidase enzymes, a glucose biosensor based on glucose dehydrogenase enzyme, and a paraoxon ethyl biosensor based on the inhibition of AChE by the pesticide were developed.

In the second approach, modified electrodes have been prepared with CUPRAC-reagent for the electrochemical sensing of some compounds3. In the first step, bare electrodes such as glassy carbon, pencil graphite, and screen-printed carbon electrodes were modified with a negatively charged polymer or polyelectrolyte such as Nafion and sodium dodecyl sulfate. Then [Cu(Nc)2]2+ was modified on these electrodes through electrostatic interaction between negatively charged perfluorosulfonate groups and positively charged cupric complex. After this effective modification, the electrocatalytic activity of CUPRAC-modified electrodes was investigated for the oxidation of some compounds. Recorded cyclic voltammograms showed that the modified electrodes have a good redox couple at around formal potential of 400-500 mV vs Ag/AgCl. In the presence of a reducing analyte, the oxidation peak current of [Cu(Nc)2]2+ increased remarkably by the analyte, while the reduction peak current slightly decreased. Moreover, the oxidation potential of compounds studied at modified electrodes was remarkably shifted to more negative values compared to that at bare electrodes. These results indicate the typical electrocatalytic properties of the modified electrode. As a result, the CUPRAC reagent exhibits a good redox-mediator property. Using this approach, flow-injection-amperometric sensors were developed for the determination of H2O2, hydrazine, hydroxylamine, and total antioxidant capacity.

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IS9- The Role of Ceramide in Diabetic Dyslipidemia from a Biochemical Perspective

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Changes in the metabolism of sphingomyelins (SM), which are among the most abundant circulating lipids, have been associated with obesity. Patients with obese type 2 diabetes (T2DM) have increased plasma ceramides (CER), and this increase is associated with insulin resistance and the degree of inflammation. A persistent CER elevation results in excessive ceramide deposition in the muscles of obese individuals with T2DM. Ceramide accumulation in human tissues inhibits insulin action and subsequent glucose uptake through inactivation of protein kinase B (PKB), also known as Akt. The production of metabolites such as ceramide-1-phosphate (C1P), sphingosine and sphingosine-1-phosphate (S1P), which are important regulators of inflammation, have also been associated with an increase in CERs.
We have reported a significant decrease in serum levels of very-long-chain C24 SM, very-long-chain C22-C24 CERs, insulin resistance (HOMA IR) and C1P in obese patients following laparoscopic sleeve gastrectomy (LSG) after postoperative day 1 and day 30 compared to preoperative levels. At 30 days post-surgery, mean body mass index (BMI) was reduced by 11%, fasting triglycerides were significantly decreased, and insulin sensitivity was increased compared to presurgery values. A significant positive correlation was found between HOMA-IR and serum levels of C22-C24 CERs in LSG patients.
In a seperate study we have found that C16-C24 SM, C16-C24 CER and C16 CER-1P levels were significantly increased in T2DM patients with LDL-C above 160 mg/dL compared to those with LDL-C below 100 mg/dL. C16-C24 SM, C22-C24 CER, C16 CER and C16 CER-1P levels showed a statistically significant increase in T2DM patients with non HDL-C above 130 mg/dL compared to those with non HDL-C below 130 mg/dL. C24:C16 SM and C24:C16 CER ratio showed a significant correlation with both LDL-C and non HDL-C levels. Obese T2DM patients (BMI>30) had significantly higher circulating levels of C24 SM, C18-C24 CER compared to those with BMI<30.
In conclusion, plasma sphingomyelins and ceramides are elevated in obese dyslipidemic T2DM patients. Serum long chain CERs, C24:C16 SM, C24:C16 CER ratio may serve as prognostic and diagnostic markers in type 2 diabetic dyslipidemia.

Key words: sphingolipid, obesity, dyslipidemia, type 2 diabetes.

Introduction

The hallmarks of type 2 diabetes are hyperglycemia and insulin resistance (1-5). Insulin resistance contributes to the characteristic dyslipidemia associated with type 2 diabetes (5-7). The dyslipidemia associated with insulin resistance is also referred to as atherogenic dyslipidemia. Diabetic dyslipidemia is characterized by increased triglyceride, VLDL, IDL, apoB 100 levels; decreased HDL levels (8-10). Type 2 diabetic patients have small HDL particles which are triglyceride-rich (10-15). The aim of the presentation is to discuss levels of SMs and CERs in the circulation of obese patients undergoing LSG preoperatively, at day 1 and at day 30 after surgery. We also examined the relationship between plasma ceramide levels and insulin resistance. Circulating N-SMase activity, C1P and S1P levels were also measured at all time points.

Materials and Methods

Study groups included the sleeve gastrectomy group which was composed of 20 obese patients who were admitted to Antalya Research and Education Hospital, Surgery Clinic. The mean body mass index (BMI) of patients was 45,64 ± 6,10 kg/m2. The control group included 15 age and gender matched patients. Patients in the control group underwent laparoscopic abdominal surgery

for cholecystectomy. The mean body mass index of patients in the control group was 31,51 ± 6,21 kg/m2. Fasting blood samples were taken 3 times, before the operation, on the 1st day after the operation and at the 1st month after the operation. Exclusion criteria included coronary artery disease, arrhythmia, peripheral artery disease, kidney dysfunction, liver disease, thyroid dysfunction, and infectious diseases.

Results

Restoration of insulin sensitivity and reduction in secondary complications highlight one of the most important benefits of LSG. It is likely that the decreased long-chain CER levels following LSG contribute to the rapid reduction of insulin resistance seen after surgery. Ceramide levels may be an important mediator for insulin sensitivity and inflammation in peripheral tissues.

Discussion

Obese patients with type 2 diabetes mellitus (T2DM) show an altered lipid profile and a certain degree of insulin resistance, which might contribute to changes in both serum sphingolipidomic profile and high-density lipoprotein (HDL) subfractions. We aimed to investigate changes in serum sphingolipid levels and HDL subspecies in relation to low-density lipoprotein cholesterol (LDL-C), non-HDL-C and triglyceride (TG) levels in type 2 diabetic patients.

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IS10- Impedimetric measurements in analytical chemistry

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Electrochemical impedance spectroscopy (EIS) is a powerful technique based on the measurement of alternating current (AC) by implementing the alternating voltage depending on the applied frequency change of the voltage. The phase angle change can be extracted by changing the frequency of the alternating voltage beside the impedance. The key is the necessity that the output signal is linearly related to the input signal so that the behavior of the system is stable in measuring faradaic or non-faradaic impedance in EIS.

However, an equivalent circuit of an electrochemical system is defined by the major components such as resistance, capacitance, and inductance (R, C, L); the resistance corresponding to the charge transfer is extensively utilized to make a correlation between the signal and the analyte concentration in analytical chemistry. The measurement of the charge transfer resistance is very critical to obtaining qualified and reliable measurements for quantitative analysis.

EIS is often used to determine the macromolecules or larger particles such as proteins, bacteria, human cells, or extracellular vesicles because it allows label-free measurements despite of voltammetric and spectroscopic techniques used in their determinations. The other advantages are undoubtedly the non-destructive measuring capability of EIS for such sensitive materials and their nature with higher dielectric characteristics that has a role for increasing sensing capability of the surface.

Unfortunately, EIS is believed to be an avoided technique because of the difficulties in obtaining reliable and repeatable data as well as in the evolution process. To overcome these difficulties, in my opinion, should be chosen “simple surface design”. Briefly, the simplest is the best for EIS.

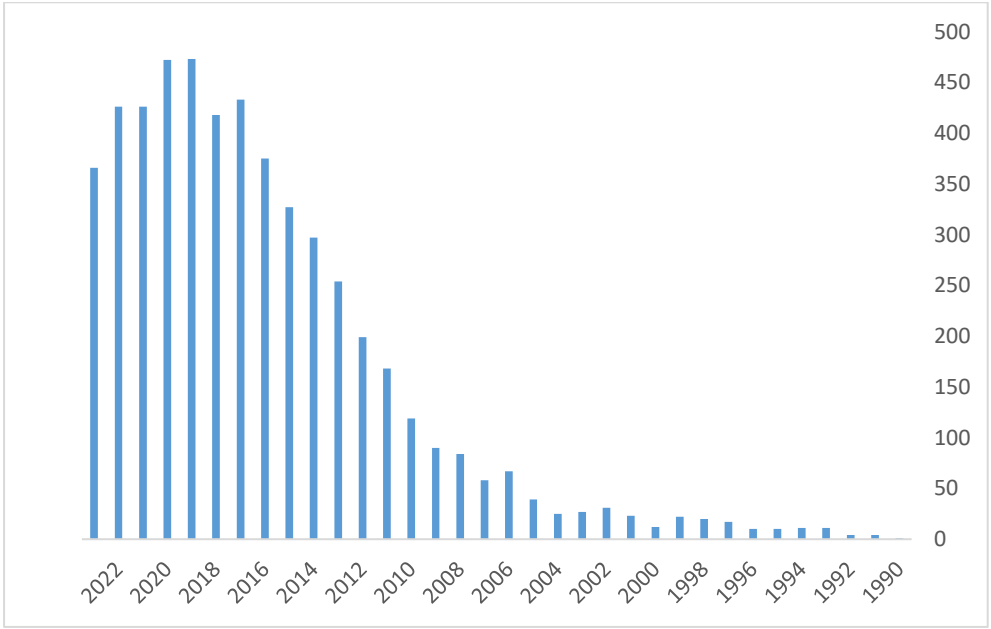

Figure. The number of published papers of “electrochemical impedance spectroscopy in analytical chemistry” by years. (from webofknowledge)

IS11- Affinity sensors for the diagnosis of COVID-19

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Various analytical systems are used for the diagnosis of SARS-CoV-2 virus induced COVID-19. Some of these analytical systems are based on the determination of SARS-CoV-2 virus pats or specific antibodies that are binding proteins of SARS-CoV-2 virus [1,2]. During our research we have designed several electrochemical sensors for the detection of antibodies against SARS-CoV-2 virus proteins based on gold electrode modified by self-assembled monolayer (SAM), which was used for the immobilization of SARS-CoV-2 proteins [3]. However, specific recognition exhibiting proteins such as antibodies are rather expensive, therefore, some alternative ways for the development of sensors selective towards SARS-CoV-2 proteins were designed. Therefore, in order to reduce these cots molecular imprinting of polypyrrole (Ppy) with SARS-CoV-2-S spike and nucleocapside proteins was applied in sensor design. The performance of molecularly imprinted polypyrrole based sensors was evaluated by cyclic voltammetry, pulsed amperometric detection and electrochemical impedance spectroscopy. According to the obtained results, a sensor based on MIP-Ppy is more sensitive to the SARS-CoV-2-S spike glycoprotein than a sensor based on NIP-Ppy. This proves that molecularly imprinted MIP-Ppy-based sensors might be applied for the detection of SARS-CoV-2 virus proteins. The design and application of molecularly imprinted polypyrrole based sensors for the determination of SARS-CoV-2-S spike and nucleocapside proteins will be discussed during the presentation.

Keywords: COVID-19, SARS-CoV-2, Nucleoprotein, Antibody, Cyclic Voltammetry, Differential Pulse Voltammetry, Pulsed Amperometric Detection.

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ORAL PRESENTATIONS (OP)

OP1- Testing of cosmetic products designed for various purposes - a brief overview of available analytical methods

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Cosmetic formulation is the final form of a cosmetic preparation created on the basis of a developed cosmetic formula, according to a specific technological process. *Regulation (EC) No 1223/2009 of the European Parliament and of the Council of 30 November 2009 on cosmetic products* indicates the determination of physicochemical properties and the assessment of the stability of a cosmetic product as necessary to be performed before placing any cosmetic on the market. In order to ensure the quality of cosmetic products manufactured, their physicochemical characterization is essential, including a number of parameters, such as pH, viscosity or product stability. In addition, manufacturers are required to prove the claimed properties of a cosmetic product, such as antioxidant properties or cosmetic's care effect. The presented research includes a review of available methods of physicochemical characterization and non-obligatory tests of cosmetic products, using the results of the research group of the Department of Applied Chemistry of the Adam Mickiewicz University as examples. The importance of the pH stability is presented on the example of cosmetic formulations containing aucubin, while the topic of viscosity is presented for car care products. The study of the stability of cosmetic emulsions containing retinol in the form of lipid nanoparticles and the application of a modern UV filter system based on raspberry seed oil are also discussed. An overview of methods for determining the antioxidant properties of cosmetic active substances is presented for costunolide, while the results of an exemplary *in vivo* study for hydrogels enriched with catalpol are mentioned.

Keywords: cosmetic analysis, stability, physicochemical properties of cosmetic products

Acknowledgment: This research was partially carried out within the project "Use of nanostructured lipid carriers (NLC) based on raspberry seed oil as an alternative to traditional UV filters in sunscreen products" funded by the National Science Centre (Miniatura 5 – 2021/05/X/ST5/00612).

OP2- HPLC-ICP MS and ESI MS – convenient analytical tools for the assessment of selenosugars, selenocysteine, and other selenometabolites expression in livers of animals fed with organic and inorganic selenium

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The Se requirement set for preventing overt Se deficiency disease in poultry was fixed on the bases of the research carried out over 50 years ago at 0.2 μg Se/g diet**.** Recent studies evaluating, among others, glutathione peroxidase activities concluded that the dietary Se requirement of the turkey poult should be raised[1] to values higher than currently permitted by FDA.
To characterize the metabolism of Se, animals were fed with graded levels of SeMet or selenite from 0 to 5 μg Se/g in a Se-deficient diet for 4 wk. The GPX1 and GPX4 activity were measured by the coupled assay procedure. The new procedures, using HPLC coupled with Se-specific (ICP MS) and molecule specific (ESI MS/MS) detection, allowing investigation of the fate and accumulation of liver Se were developed.
The study demonstrated that excess dietary Se ≤ 2 μg Se/g did not dramatically impair growth or alter selenoenzyme activity or selenoprotein transcript expression in the animals. There were not dramatic shifts in the metabolic flux of Se at different levels of high Se supplementation, once Se incorporation into Sec selenoproteins was maximized. Overall, selenosugars are the predominate form of Se that accumulates in liver as water soluble species in rats fed with either diet at high concentrations. SeMet and selenoglutathione species were only detected in rats suplemented with high SeMet whereas they were absent in selenite supplemented animals. Only at 2 and 5 μg Se/g, for both Se sources, the aqueous liver extracts contained an m/z 345 Se species that was far less abundant than the other selenosugar species. To the best of our knowledge, this m/z 345 species has not been reported so far.
The developed methodology for identification of the selenocompounds will allow understanding of metabolic processes and explain lack of apparent Se toxicity effects on transcription in case of administration of high doses of selenium.

Keywords selenite supplementation, selenium and sulphur speciation, selenosugars, ICP MS, ESI MS

Acknowledgment
NIFA, USDA, 1016808, Multistate NC-1170

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OP3 A novel sensor based on grafting a polymeric film with electrochemically reduced graphene oxide: Fabrication, characterization, and potential analysis on ephedrine.

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A catalytic surface was fabricated by incorporation of electrochemically reduced graphene oxide (rGO) on a poly(2,4,6-triaminopyrimidine) (PTAP) film modified glassy carbon electrode. The surface materials of the constructed electrode (rGO.PTAP/GCE) were characterized by means of electrochemical and surface scanning techniques including Cyclic Voltammetry (CV), Electrochemical Impedance Spectroscopy (EIS) and X-ray Photoelectrons Spectroscopy (XPS). Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) were carried out to identify the preparation of the graphene oxide (GO). The heterogeneous rate constant of the fabricated sensor (k_s = 0.149 cm. s^{-1}) was evaluated by Nicholson method. The synergistic effect of surface materials promotes the catalytic efficiency of the developed sensor for the detection of ephedrine (EPH) in pharmaceutical samples where the detection limit (DL3σ) of EPH was 1.8 µM (297 ppb). A thermodynamic parameter such as the diffusion coefficient of ephedrine was also evaluated using theoretical electrochemical approaches with a value of (2.55 × 10^{-5} $cm^2.s^{-1}$). The analytical performance of the sensor was successfully applied for real drug sample with acceptable analytical recovery percentage.

Key words:
Reduced graphene oxide; Poly(2,4,6-triaminopyrimidine); Ephedrine; Solid-state sensor.

Acknowledgement:
The author would like to thank Sultan Qaboos University (SQU), Sultanate of Oman, for supporting this work by the research grant number (IG/SCI/CHEM/22/05).

OP4- Analytical Method validation challenges in analyzing dissolution profile by Liquid Chromatography-Tandem mass spectrometry

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A dissolution procedure intended to be used as a routine control test for immediate release drug products should be robust, reproducible and discriminatory in order to assure a consistent product quality and to detect product quality attributes, which, if altered, may affect the in vivo performance.
The selection of a suitable dissolution medium (composition, volume) should be based on the physico-chemical characteristics of the active substance. In general, an aqueous medium should be used and the pH should first be evaluated in the physiological pH range. Rate of dissolution is a critical quality attribute of a pharmaceutical tablet. Tablet dissolution is typically studied by examining the form of the dissolution profile, which is the percentage of the tablet dissolved at various points in time. The complicating issue in the analysis of dissolution is that the response is a “profile” involving several data points rather than a single response metric. Several methods have been proposed to analyze dissolution profiles.
The medium and pH used in the analysis of dissolution profiles affect the analysis. Salts that provide the pH environment required for dissolution analysis affect the analysis, especially when a mass spectrometry detector is used. For this reason, various difficulties are encountered in the method to be developed and validated. In this study, dissolution profile analyzes of Cethyl pyridinium Pastille and Lubiprostone soft capsule products will be examined using the HPLC-MSMS analysis method.
Dissolution samples were taken in pH 6.8 phosphate buffer media for Cetyl pyridinium. in pH: 4.5 acetate buffer, pH: 6.8 phosphate buffer and 0.1 N HCl were used as dissolution medias for Lubiprostane and dissolution profiles were created.

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OP5- Multiple Headspace Extraction along with cryostatic cooling inlet - GCMSD for determination of volatile organic compounds in marine water and wastewater samples

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Volatile organic compounds (VOCs) are a vast group of chemicals that have highly volatile, low vapor pressure in ambient temperatures, lipophilic, impose negative effect on marine environment. Particularly, some of the chlorinated forms have toxic and carcinogenic effects on organisms[1]. The present study focused on, the mostly traced/quantified VOCs that are methylene chloride, benzene, 1,2 dichloroethane, trichloroethylene, tetrachloroethylene, *o/m/p* xylenes in the literature[2] based on monitoring and wastewater effluent samples among the diverse group of VOCs. Although the purge and trap (P&T) is a sensitive methodology approach stated in preliminary studies due to exhausting extraction method, the presence of some chemicals (such as anionic, cationic detergents etc.) could yield a foam formation during extraction; also, variety of sample pH and particulate matter are additional limitations for analysis of marine water and wastewater samples. Therefore, a multiple headspace extraction (MHE) coupled with cryostatic inlet technique was developed for determination VOCs in marine and wastewater samples in order to analyse at low levels (µg/L). The extractants were automatically desorbed from the inlet which is filled with Tenax, then detected in gas chromatography-mass spectrometry. The injection volume (6 ml), sample volume (10 ml), extraction temperature (70 ˚C), extraction time (45 min.), agitation speed (250 rpm), cryostatic inlet temperature (20 ˚C), and salt addition (5g) were optimized. The results showed that the temperature is the most effective on studied VOCs; particularly on the DCM due to boiling point. Additionally, salt addition enhances the compounds responses in wastewater, while this effect was negligible in marine water samples. The other challenge was the carry-over of benzene, however applying higher temperature on cooled injection could lead to overcome this issue. The analytical figure merit of the proposed methodology presents satisfied results in calibration range (0.1 – 5 µg/L) as recovery (71 ± 2 – 114 ± 3 %), limit of detection (0.001 – 0.01 µg/L), and repeatability (4.2 – 14 %). The finalized method was applied onto real marine water and wastewater which were sampled from Izmit bay.

Keywords: Multiple Headspace extraction (MHE), Volatile organic compounds (VOCs), marine surface water, CCI-GC/MSD.

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OP6- Simultaneous Determination of Pyridalyl and Metalaxyl by Gas Chromatography-Mass Spectrometry after Combination of QuEChERS and Orbital Shaker-Assisted Switchable Solvent Microextraction

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Pesticides have become an important component of the agricultural system as they allow a considerable uptick in food production[1]. Although they have benefits, pesticide residues in foods such as vegetables and fruits pose hazards to human health[2]. Therefore, developing an analytical method to detect these compounds at trace levels in complex food matrices with high accuracy and sensitivity is of great significance. In this study, Quick Easy Cheap Efficient Rugged and Safe (QuEChERS) and orbital shaker-assisted switchable solvent liquid phase microextraction (OS-SS-LPME) methods were combined to isolate and preconcentrate pyridalyl and metalaxyl pesticides from the sample matrix for quantification by GC-MS. After optimizing the SS-LPME variables, 0.50 mL of switchable solvent, 2.0 mL of tris buffer (pH 7.0), 1.5 mL of 2.0 M NaOH, and 360 s orbital shaking were determined as the optimum parameters. When comparing the detection limits under optimized OS-SS-LPME conditions with the detection limits obtained by direct GC-MS detection, big increase in the detection power of the analytes was observed. The combined QuEChERS and OS-SS-LPME method was applied to real samples, and a matrix matching technique was used to obtain high-accuracy recovery results.

Keywords: Pesticides, Switchable Solvent, Gas Chromatography-Mass Spectrometry, QuEChERS

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OP7- Aptasensor Development Based on Transition Metal Oxide for Diagnosis of Prostate Cancer

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Currently, prostate cancer (PCa) is one of the most lethal diseases in men worldwide, and the most common type is the adenocarcinoma which is difficult to diagnose since it does not show any early symptoms. Prostate-specific antigen (PSA) is the most used serum marker in the diagnosis, screening, and post-treatment follow-up of PCa[1]. Therefore, various types of immunosensors and aptasensors have been utilized for the detection of PSA in serum samples. Direct cellular analyses on the other hand, provide a reliable diagnosis.

Present study deals with the aptasensor development based on pulsed-deposited manganese oxide (PD-MnO_x) for selective determination of the PSA in cell culture. For this purpose, the amino-terminated aptamer specific to the PSA was attached to the pencil graphite electrode modified with PD-MnOx by the EDC/NHS mechanism as described in a previous study[2] and the charge transfer resistances (R_{ct}) of the electrode surface was monitored in the cell containing ferri/ferro-cyanide ions as a redox probe. The influence of the cell pH on the signal measured was also discussed and it was demonstrated for the first time that an adjustable response range with reverse bias can be obtained for PSA for different medium pHs due to the variation of the electrostatic environment with respect to the isoelectric point (pI) of the protein molecule. The method was applied to the PCa cells in vitro, suggesting a label-free, fast, specific, and inexpensive analysis to be achieved.

Keywords: prostate cancer, PSA, Pulsed deposition, electrochemical impedance spectrometry

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OP8- Carbon Quantum Dots Based Fluorescent Nanosensor Platforms for Detection of Gastric Cancer-Associated Genes

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Helicobacter pylori (*H. pylori*) is a spiral microanaerobic gram-negative bacterial pathogen that was first isolated in 1983 from gastric mucosa biopsy tissue of patients with chronic active gastritis. *H. pylori* infection is associated with chronic gastritis, ulcers, gastric lymphoma, and gastric cancer. The methods used today to detect the *H. pylori* pathogen are generally time-consuming, require expertise, and require the use of at least two methods together. Methods used other than PCR (Polymerase Chain Reaction), such as the ^{13}C-urea breath test and stool antigen test, have low accuracy [1]. In recent years, DNA-based biosensors have attracted great attention due to their high sensitivity and selectivity. DNA-based biosensors often rely on the principle of DNA hybridization, where the target analyte (e.g., a specific DNA sequence, RNA, or protein) binds to a complementary DNA probe attached to the sensor surface [2].
Carbon quantum dots (CQDs) are a small member of the nanomaterial family with a diameter of less than 10 nm. Notably, CQDs display distinctive physicochemical properties, including stable photoluminescence, adaptable surface chemistry, remarkable biocompatibility, low toxicity, and an environmentally friendly nature. CQDs are used in a lot of areas, such as bioimaging, optical sensors, solar cells, drug delivery, etc. In this study, carbon quantum dots-based fluorescent nanosensor platforms were developed using gastric cancer-associated genes. N-doped carbon quantum dots were synthesized using three different organic acids (citric acid, tartaric acid, and malic acid) and ethylenediamine by the microwave method. The photophysical and photochemical properties of the synthesized CQDs were investigated by UV-Vis, fluorescence, and FT-IR spectra. The surfaces of CQDs were modified by capture DNA, which is specific for gastric cancer. Ethidium bromide is a specific dye that increases fluorescence intensity when intercalated with DNA. The fluorescence intensity of CQDs at different wavelengths was quenched by ethidium bromide due to electron transfer processes. Upon the presence of target DNA, the fluorescence intensity of ethidium bromide was enhanced, and improved detection method for gastric cancer-associated genes. Performances of the nanosensors were compared to the synthesized three different CQDs.

Keywords: carbon quantum dots, gastric cancer, nanosensor, fluorescence sensors

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OP9- An electrochemical sensor based on molecularly imprinted polymer supported by ruthenium pincer complex and multi-walled carbon nanotube for the detection of anticancer drug Nintedanib

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Nintedanib is a multiple tyrosine kinase inhibitor effective in slowing down tumor growth. The drug is known for treating non-small cell lung cancer and pulmonary fibrosis[1]. Electrochemical sensing is a rapid, cost-effective, and sensitive analytical technique for the detection of a wide range of analytes, including pharmaceuticals, biomolecules, contaminants, additives, and so on. The electrochemical method provides the opportunity to integrate specific recognition elements as modifiers on the transducer surface for improving the sensitivity and selectivity of detection. Molecular imprinting polymers are synthetic materials with selective binding sites for specific target molecules. This helps to improve the sensitivity of detection. Multiwalled carbon nanotubes possess excellent conductivity and large surface area and are attractive candidates for nano-sensing applications[2]. Moreover, as redox-active molecules, immobilization of ruthenium complexes on electrode surfaces can assist the electron transfer between electrode and substrate. In this work, we have developed an electrochemical sensor following a molecular imprinting approach for the selective and sensitive determination of the anticancer drug, Nintedanib. The sensor was fabricated by modifying a glassy carbon electrode (GCE) with a ruthenium-pincer complex (Rubbz) and multi-walled carbon nanotubes (MWCNTs). Target-specific cavities were created on the electrode surface by electropolymerizing the monomer, *o*-phenylene diamine, in the presence of the drug Nintedanib, followed by the removal of the drug. 5 µL of MWCNTs followed by 2 µL of Rubbz was dropped onto the GCE. Then MIP surface was formed with the 40 cycles of *o*-phenylene diamine on the glassy carbon electrode (GCE/MWCNTs/Rubbz/MIP). Utilizing the GCE/MWCNTs/Rubbz/MIP nanosensor, Nintedanib was determined within the linear range of 1×10^{-10} to 5×10^{-9} with an LOD value of 1.83×10^{-11}. Finally, the fabricated sensor was successfully used to detect Nintedanib in pharmaceutical dosage forms.

Keywords: Electrochemical sensor, Nintedanib, molecularly imprinted polymer, metal complexes, multi-walled carbon nanotube.

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OP10- Electrochemical DNA Biosensor for the Investigation of Antioxidative Activity of Turmeric

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Species carrying unpaired electrons in their outer orbital are defined as "free radicals". The formation of free radicals in the living body occurs during normal metabolic processes or due to environmental influences (e.g., radiation exposure, stress, medications, smoking, exposure to chemicals, and certain foods). Approximately 2 x 10^4 DNA damaging events occur in every cell of the human body every day (1). This situation is tried to be prevented by the use of antioxidants. The evaluation of antioxidants is predominantly based on the detection of DNA damage. This approach utilizes DNA-based electrochemical biosensors, mirroring the response of antioxidant activity in biological systems. Typically, these biosensors simulate the damage induced by reactive oxygen species (ROS) in vivo. (2)

In consideration of these insights, in this study, three distinct types of turmeric samples were utilized to investigate their antioxidative activities. A disposable electrochemical DNA biosensor fabricated as a pencil graphite electrode (PGE) modified by a surface layer of the calf thymus double stranded (ds) DNA was used as a working electrode in combination with a silver/silver chloride reference electrode and a separate platinum auxiliary electrode. Samples were investigated using differential pulse voltammetry (DPV) technique based on the guanine signal obtained at about +1.0 V and cyclic voltammetry (CV) techniques. Bare and turmeric interactive electrodes were compared before and after the DNA immobilization onto the electrode surfaces. The optimum concentration and interaction time of turmeric samples were examined in experiments, and the results obtained with the first sample are that the active antioxidative components present in turmeric have a prominent signal-decreasing effect on DNA. The analysis of turmeric samples was also performed electrochemically in artificial body fluids such as gastric fluid, intestinal fluid, and saliva. The findings were also compared with those obtained by spectroscopic methods.

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OP11- Rapid and Sensitive Determination of I-Glutamate in Food Samples with Using Capillary Electrophoresis Coupled with Contactless Conductivity Detector

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Glutamic acid is one of the 20 amino acids which is used in protein synthesis. It is a non-essential amino acid. Glutamic acid is one of the most abundant neurotransmitters in the nervous system. Glutamate is the anionic form of glutamic acid, used as a flavor enhancer in food industry. It gives umami flavor which means "pleasant savory taste" in Japanese. Sodium salt of glutamate (MSG) is the most used flavor enhancer worldwide. For years, there has been debate about glutamate's health effects. It has been said to cause some symptoms namely, numbness, racing heart and weakness, but this has not been proven. However, some studies have found that MSG can damage the nervous system and cause obesity [1]. For this reason, it is important to determine the amount of glutamate in the foods.

Capillary electrophoresis (CE) is a separation and analysis technique based on the movement of particles at different speeds under an electric field. Separation is made by using the charge/size ratios of the particles. Advantages of CE are; short analysis time, small amount of sample and solvent consumption and high resolution. A more recently developed system of capacitively coupled contactless detection (C^4D) provides a simple and cost-effective analysis. CE-C^4D system, is applicable in detection of a wide range of analytes in food samples. The ionic species such as, organic acids have electrophoretic mobilities contradicting to the electroosmotic flow (EOF) in the capillary and causing longer analysis times. In order to reduce the migration times, EOF is usually reversed or suppressed by employing buffer additives such as cationic surfactants [2]. In this study, a novel, simple and sensitive CE-C^4D method was developed for the determination of glutamate in food samples within 3 minutes. The direction of the EOF was reversed and the sample was introduced into the capillary from the cathodic side. The developed method has been applied to various sauces and chips. The proposed method was successfully validated and satisfactory recovery results were found.

Keywords: Glutamate, capillary electrophoresis, contactless conductivity detection

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OP12- Efficient Synthesis of 5-Hydroxymethylfurfural from Glucose using Ionic Liquid MIL 101(Cr) Composite Catalysts: Towards Sustainable Biomass-Derived Chemicals

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The rapid depletion of petroleum-derived resources has led researchers to utilize renewable resources. The synthesis of 5-hydroxymethylfurfural (5-HMF), a key intermediate in the production of bio-based chemicals and fuels, from glucose has gained significance as a sustainable alternative to conventional petrochemical processes. This study investigates the efficient production of 5-HMF using a novel catalyst system based on ionic liquid-metal organic framework (IL-MOF, 1-methylimidazolium chloride-MIL-101(Cr)) composites. The research involves optimizing reaction conditions such as temperature, reaction time, and IL loading into the MOF to increase the yield of 5-HMF. Remarkably, the highest yield (210.4±2.3 mg 5-HMF/g glucose) was achieved with a 40% IL-MOF composite during a one-hour conversion reaction at 180°C. The experimental results highlight the effectiveness of the IL-MOF catalyst in promoting the conversion of glucose to 5-HMF, offering a promising path for the development of sustainable and environmentally friendly processes in the field of biomass-derived chemicals.

Keywords: Catalyst, 5-HMF, biomass conversion, IL-MOF composite

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OP13- Smartphone digital image colorimetry combined with liquid–liquid- and solid-phase microextraction: A new analytical tool for chemical analysis

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Smartphone digital image colorimetry (SDIC) has emerged as a simple, rapid, cost-effective and green alternative technique for chemical analysis of a variety of samples. The image captured with a smartphone in a lab-made colorimetric box is split into red, green and blue (RGB) channels and the highest intensity at one of the channels is correlated to the analyte concentration. When combined with a suitable liquid–liquid- (LLME) and/or solid-phase microextraction (SPME) method, SDIC can be used to determine the concentration of the analyte in the final extract. In this study, SDIC was applied for the determination of aluminum in antiperspirant products[1]. It was combined with solidification of floating organic drop-dispersive LLME for the determination of iodate in table salt[2]. Supramolecular solvent-LLME followed by SDIC was used for the determination of curcumin in food samples[3]. It was hyphenated with reversed-phase switchable-hydrophilicity solvent LLME for the determination of copper in edible oil[4]. Switchable-hydrophilicity solvent LLME combined with SDIC was used for the determination of palladium in catalytic converters[5]. SDIC was paired with deep eutectic solvent-LLME for the determination of cobalt in milk and dairy products[6]. It was utilized with dispersive SPME for the determination of boron in food samples[7]. Ion-pairing dispersive LLME was applied prior to SDIC for the determination of tramadol in human urine samples (data not published). In each case, influential experimental parameters were optimized and applied to analyze real samples. Replacing the continuum light source, such as the white light-emitting diode, commonly used in SDIC, with a unicolored background from another smartphone screen significantly improved the sensitivity and selectivity when dealing with such complicated matrices. In all cases, good analytical performance, based on linearity, sensitivity, repeatability and accuracy was obtained. The low analysis cost, simplicity and minimum dependence on electricity and expertize make SDIC a good alternative to sophisticated techniques, particularly for low-income laboratories and researchers in developing countries. Adaptability to liquid, solid and semi-solid samples and compatibility of the final extract with the detection system are other advantages. With the rapid advancement of technology, especially high-resolution smartphone cameras and software, it is expected that the analytical performance of SDIC will be improved even further.

Keywords: Chemical analysis; liquid–liquid microextraction; smartphone digital image colorimetry; solid-phase microextraction

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OP14- Investigation of the Removal of Cadmium from Aquatic Medium Using Tin Oxide: Adsorption Kinetics and Isotherm Studies

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Processing and applications of metal elements are important in order to meet the needs of the developing industry and the increasing human population. Cadmium metal is known to be released from processes such as metal ore combustion, fossil fuel processing, waste burning, and even low concentrations of it is known to be harmful to the environment. With the increasing release of cadmium to environment day by day, environmental pollution poses harm to both terrestrial and aquatic habitats, including humans[1]. In recent years, many different methods and technologies have been developed for the removal of cadmium from polluted soil and water sources including precipitation, membrane filtration, electrodialysis and adsorption[2]. Therefore, the synthesis of nanoparticles involved in the application of many of these methods and the evaluation of their applicability in these systems are important. In the presented study, a nanoparticle-based adsorption method was developed to investigate the removal efficiency of cadmium in water matrices. Adsorption efficiency of synthesized adsorbents in cadmium removal were evaluated under constant conditions by application of the OFAT method. The efficiency of adsorption was significantly enhanced using tin oxide nanoparticles, as demonstrated by results indicating high interaction rates and removal efficiency. The chemometric approach was employed to optimize the surface interaction of cadmium with nanoparticles, thereby enhancing the treatment efficiency. Qualitative/quantitative determination of the target pollutant was carried out in the flame atomic absorption spectrometry system and calibration curves were constructed to calculate the removal efficiency prior to the method applicability. The adsorption treatment method's applicability in water samples was assessed under optimized conditions, removal efficiency of pollutant and adsorption capacity of adsorbent were analyzed for various pollutant concentrations. Adsorption isotherm models were applied to the results and conformity between the model predictions and experimental adsorption data were compared. Developed adsorption treatment method achieved high removal efficiencies for the target pollutant. It has been determined that the synthesized nanomaterial material can be used as an adsorbent and has high adsorption capacity values for target pollutant. It has been observed that the removal method applied in laboratory scale batch removal systems under the determined optimum conditions demonstrated high removal efficiency and produced satisfactory results regarding the reusability of the adsorbent. The method to be presented is prominent with its potential for application in removal of different pollutants from water samples and to be carried to an industrial scale.

Key Words: Heavy Metal, Tin Oxide, Adsorption, Adsorption kinetics

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OP15- Adsorptive Removal of Cadmium by Al2O3 Nanoparticles from Domestic Wastewater: Langmuir Isotherm Model Studies

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In recent times, there has been a growing use of the term 'heavy metal' to refer to metalloids and metallic chemical elements that have harmful effects on the environment and human health[1]. Considering the challenging and long-lasting presence of these metals in the environment, the removal of these substances from aquatic mediums remains a significant concern. Traditional removal strategies are not cost-effective for the removal of these hazardous substances, and they also generate a substantial quantity of dangerous chemical sludge[2]. The goal of this study was to develop a novel batch adsorption strategy for the removal of cadmium, which is known to be a heavy metal posing dangers to both human health and the environment, from wastewater. In this study, a microwave-based synthesis method was used to synthesize Al_2O_3 nanoparticles efficiently and environmentally. The nanomaterial was characterized by evaluating its chemical structure with X-ray diffraction spectrometry (XRD) and determining its morphological structure with scanning electron microscopy (SEM). The nanoparticles were employed as adsorbents for the adsorptive removal of cadmium from wastewater. Parameters impacting the analyte-adsorbent interaction, such as nanomaterial amount, pH/volume of buffer solution, and mixing type/period were optimized in order to improve the efficacy of the batch adsorption strategy. The equilibrium adsorption experiments were carried out on synthetic domestic wastewater. Cadmium concentrations in the treated wastewater were calculated by a matrix-matching calibration strategy, in which the calibration plot was developed in the wastewater. Moreover, the data was modeled with a commonly used adsorption isotherm, Langmuir, and the results confirmed that the simple and efficient adsorption strategy was successful in removing cadmium from synthetic domestic wastewater by Al_2O_3 nanoparticles.

Keywords: Adsorption, Al_2O_3 nanoparticles, Langmuir isotherm model, Synthetic domestic wastewater

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OP16- Electrochemical Study of Veterinary Drug – Danofloxacin – at Glassy Carbon Electrode and Electrified Liquid-Liquid Interface

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Danofloxacin (DANO) is a fluoroquinolone antibiotic with a broad spectrum of activity against Gram-negative and -positive bacteria. It is a synthetic drug specifically designed for veterinary applications and is primarily employed in the treatment of respiratory and gastrointestinal infections in animals

In this work, we have employed two electroanalytical platforms to study DANO. First, is based on the four electrode system with soft and electrified junction formed between water | oil interface (ITIES). The second is based on the traditional three electrode configuration with glassy carbon electrode (GCE) used as the working electrode. Using voltammetry, we effectively identified several electroanalytical and physicochemical parameters, which were subsequently compared (ITIES vs GCE) and assessed. Ion transfer voltammetry (ITV) was employed to examine DANO at the ITIES. Simultaneously, the electrochemical behavior of DANO on the GCE was explored through square wave voltammetry (SWV) and cyclic voltammetry (CV).

The results of these investigations are verified, and all analytical parameters such as limits of detection (LODs), limits of quantification (LOQs), linear dynamic ranges (LDRs), and detection sensitivities are presented comprehensively.

Keywords: Danofloxacin; fluoroquinolone antibiotic; ITIES; glassy carbon electrode.

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OP17-- Poly(thionine) Supported PtPd Bimetallic Catalyst for Electrochemical Oxidation of Ethanol

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Abstract
PtPd bimetallic nanocatalyst was prepared supported on poly(thionine) conducting polymer. Thionine was polymerized from its aqueous solution by cyclic voltammetry. Incorporation of Pt and Pd complexes were carried out via cyclic voltammetric scans from aqueous K_2PtCl_4 and K_2PdCl_4 solutions followed by reduction of the complexes in stirred 0.1 M hydrazinium hydrate solution. Experimental parameters were optimized according to the peak current values recorded in ethanol solution. Maximum performance was obtained when 60 cycles of Pt and 10 cycles of Pd was used as the experimental parameter.

Keywords: Poly(thionine), Platinum, Palladium, Ethanol, Electrocatalysis

Introduction

Due to the increasing need for energy and the decrease in existing energy resources, energy-related research has recently been focused on renewable energy sources. Fuel cells, one of the class of devices that use renewable energy sources depending on the type of fuel, have a significant potential as environmentally friendly systems[1]. In the design of a fuel cell, selection of the electrode material is crucial in order to increase the efficiency as well as reduction of the cost of a fuel cell system. The materials used for the construction of the catalyst layers contains precious (often Pt in PEMFCs) or non-precious metal particles which are generally supported on a suitable material[2]. Alloying Pt with another noble metal (co-catalyst) to prepare bimetallic heterogeneous catalysts may alter CO tolerance of the catalyst and hence increase the fuel cell performance. In addition, the electrocatalytic activity of an electrode material can be easily improved by incorporation of a conducting polymer as the supporting medium for immobilization of the metal nanoparticles[3].
Herein, we describe the preparation of PtPd particles-based bimetallic catalyst using poly(thionine) (PTH) conductive polymer as the support material and to test its effectiveness for the electrocatalytic oxidation of ethanol.

Materials and Method

Thionine acetate, H_2SO_4, K_2PtCl_4, K_2PdCl_4 and ethanol were obtained from Sigma-Aldrich and used as-received. Hydrazine solution was diluted from 80% hydrazine hydrate solution in water (Merck). All solutions were prepared using triple distilled water and purged with high purity nitrogen gas in order to remove the dissolved oxygen. All experiments were performed at ambient temperature. Electrochemical experiments were recorded with CHI 600E electrochemical workstation. A three-electrode system glass cell was used with a pencil graphite electrode (PGE, area = 6.48×10^{-2} cm^2) as the working electrode. The connector of the PGE (0.5 mm HB Tombow) was a Tombow pencil. A silver/silver chloride electrode (Ag/AgCl) was used as the reference electrode and a platinum (Pt) wire was used as the counter electrode.

Results and Discussion

PTH film was electrodeposited onto the PGE via cyclic voltametric scans from 0.05 M thionine acetate solution containing 0.5 M H_2SO_4. The potential window was between potentials -0.2 V and +0.8 V vs. Ag/AgCl (Figure 1a). In order to obtain the maximum performance from the catalyst for electrocatalytic oxidation of ethanol, ethanol CVs were recorded in acidic medium with PtPd decorated PTH film coated electrodes prepared using different number of cycles during electropolymerization of thionine. When the oxidation peaks of ethanol were compared with polymer films prepared with 5, 10, and 15 cycles in thionine solution, maximum performance was obtained with the film via 10 cyclic voltammetric scans. The polymer coating on the electrode can be seen when the CVs of the uncoated PGE and PTH coated PGE are overlayed (Figure 1b).

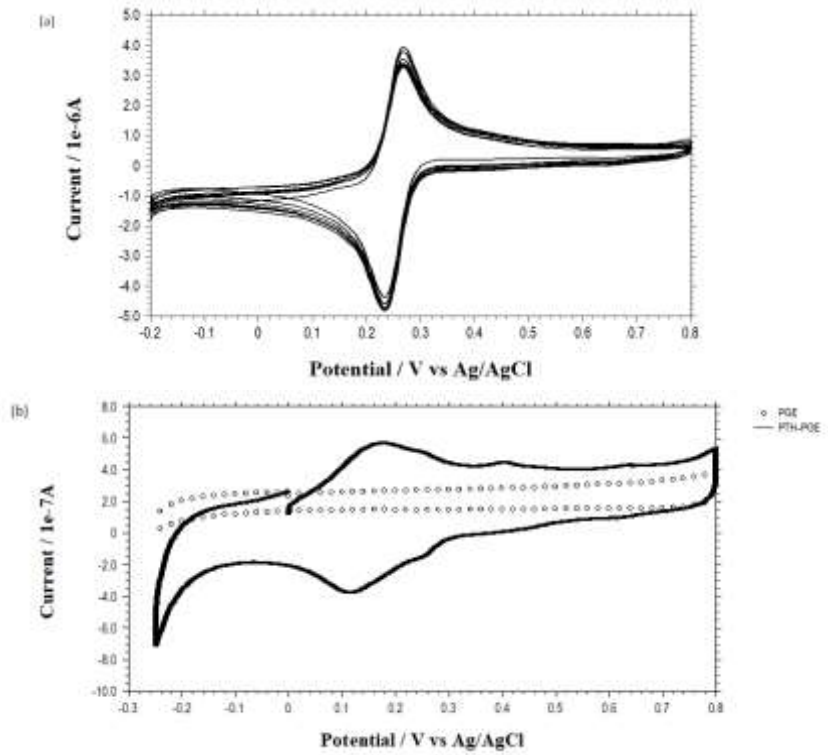

Figure 1 a) Polymerization of thionine from 0.5 M thionine acetate solution containing 0.5 M H_2SO_4. b) CVs recorded in 0.5 M H_2SO_4 with uncoated PGE (○○○) and PTH coated PGE (—). (Scan rate: 0.1 V s^{-1})

After coating the electrode surface with the polymer film, incorporation of Pt and Pd complexes were carried out via cyclic voltammetric scans from aqueous K_2PtCl_4 and K_2PdCl_4 solutions followed by reduction of the complexes in stirred 0.1 M hydrazinium hydrate solution. Ethanol CVs recorded using various combination of Pt and Pd are given in Figure 2. As clearly seen from the figure, maximum peak current was obtained when 60 cycles of Pt and 10 cycles of Pd was used as the experimental parameter.

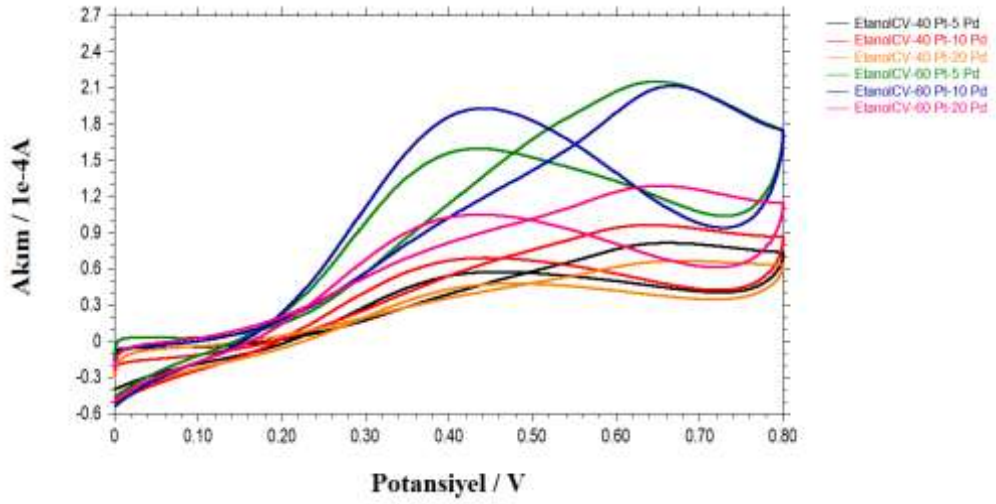

Figure 2 CVs of 0.5 M C_2H_5OH solution containing 0.5 M H_2SO_4 recorded with PtPd/PTH modified PGE with various Pt and Pd combinations. (Scan rate: 0.100 V s^{-1})

Conclusion

Electrocatalytic oxidation of ethanol was performed using PtPd/PGE modified electrode system in acidic medium. Experimental parameters were optimized according to the peak current values

recorded in ethanol solution. Maximum performance was obtained when 60 cycles of Pt and 10 cycles of Pd was used as the experimental parameter.

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OP18- Electrochemical Detection of one of the β-Blocker - Nebivolol at the Polarized Liquid-Liquid Interface

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Nebivolol (NBV) is a medication classified as a β-adrenergic blocker, primarily used in the treatment of arterial hypertension. Its mechanism of action involves blocking β-adrenergic receptors, resulting in a reduction of heart rate and vasodilation, ultimately lowering blood pressure. NBV plays a significant role in the management of cardiovascular conditions and continues to be an important therapeutic option in clinical practice[1,2]. Additionally, NBV is listed as a prohibited substance by the World Anti-Doping Agency (WADA) due to its potential to enhance athletic performance by affecting cardiovascular functions. The polarized liquid-liquid interface, known as the interface between two immiscible electrolyte solutions (ITIES), is a versatile platform for investigating the transfer of ionized molecules between two liquid phases. The electrochemical potentials of the two immiscible liquids create an electrical potential difference across the interface, which can be used to power various chemical and physical processes such as interfacial ion transfer and adsorption. ITIES can be sued to study chemical reactions that can not be studied in traditional electrochemical systems based on the solid electrodes, such as heterogenous and biphasic electron transfer reactions, redox processes, or catalysis[3–8]. This study focuses on the electrochemical detection of the β-blocker nebivolol at the polarized liquid-liquid interface. All electrochemical studies were performed with the cyclic voltammetry. All obtained results were validated and electroanalytical parameters such as limits of detection (LOD), limits of quantification (LOQ), and sensitivity were determined. We have also defined physicochemical parameters like diffusion coefficients (D), and formal Galvani potential differences of the ion transfer reaction. Finally, we determined the concentration of NBV in a pharmaceutical preparations using platforms based on electrified LLI.

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OP19- Synthesis of polyamide materials at the polarized liquid – liquid interface

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Interface Between Two Immiscible Electrolyte Solutions (ITIES) is a platform that under the influence of the applied interfacial potential difference allows the study of the charge transfer reactions happening within an interfacial region. Phenomena occurring at the interface of the hydrophilic and hydrophobic phases are purely ionic, and hence, are very different from the charge transfer eactions occuring at conventional solid electrodes. Experiments at the polarized liquid–liquid interfaces are usually performed in a classic macroscopic voltammetric cell with two Luggin capillaries and two sets of electrodes (platinum and Ag/AgCl used as counter and reference electrodes, respectively). Commonly, the aqueous phase is a solution of the hydrophilic salt, e.g. NaCl, LiCl; whereas the organic phase is a solution of highly hydrophobic salt dissolved e.g. in 1,2-dichloroethane, 1,4-dichlorobenzene ect.[1] In this work, we have synthesized the polyamide material at the miniaturized electrified liquid – liquid interface. Downscaling brings higher stability to soft junctions and significantly reduces the amounts of used chemicals. The micro-ITIES systems were made by embedding a silica capillary with a pore radius equal to 25 µm in the micropipette tip. The tip was filled with the organic phase and immersed in the aqueous phase.[2] Polyamide materials were formed at the ITIES as a result of the electrochemically controlled interfacial polycondensation reaction and characterized using SEM - EDX and Raman spectroscopy.

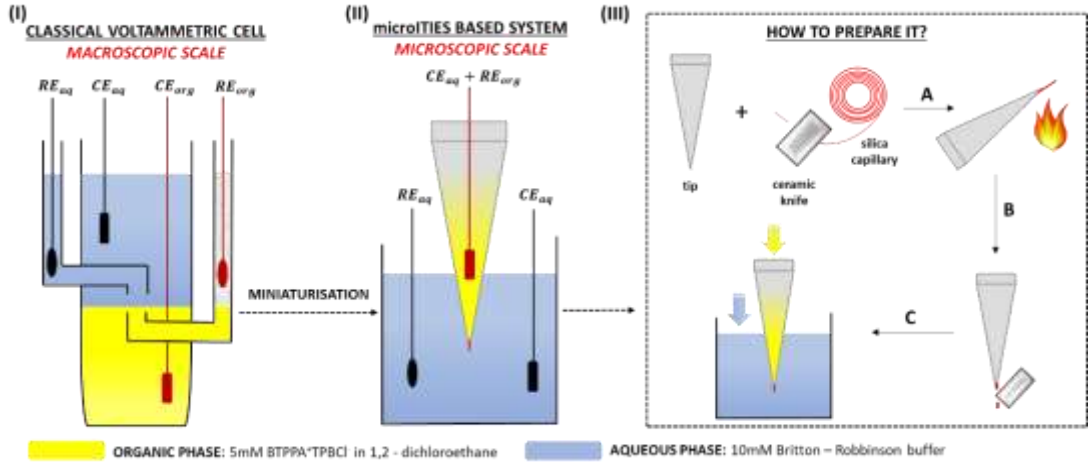

Fig 1. Classical voltammetric cell used to study macroscopic ITIES (left). MicroITIES supported with a silica capillary with an internal diameter of ~ 25 µm used as a support for the interfacially formed polyamides.

Keywords: polyamide materials, polarized liquid–liquid interface, ITIES, interfacial polymerization.

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OP20- Electrochemical Enzyme-Based Biosensors and Their Inhibition Applications

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Electrochemical enzyme-based biosensors are one of the most widespread and commercially effective biosensors. In the design of enzyme-based biosensors, enzyme immobilization has been shown to be an effective method to avoid the disadvantages of using free enzymes, thus improving the productivity and cost efficiency of the manufacturing process[1]. Immobilized enzymes offer great potential for developing sensors that can detect and characterize their respective targets. It is anticipated that substrate/analyte molecules will migrate from the medium to the immobilized enzymes in enzymatic biosensors. Enzyme inhibition by drugs is crucial for therapeutic interventions and regulating biochemical processes. It allows precise control of metabolic pathways, preventing overactivity or imbalance. Targeted inhibition can mitigate disease progression, offering effective treatments for various conditions. Understanding and manipulating enzyme inhibition aids in developing pharmaceuticals with enhanced efficacy and reduced side effects. Enzyme inhibition is integral to enzyme-based biosensors, influencing their sensitivity and selectivity. By strategically inhibiting or modulating specific enzymes, biosensors can detect and quantify target molecules with precision[2]. This synergy enables the development of biosensing platforms for diverse applications, including medical diagnostics, environmental monitoring, and food safety, enhancing analytical capabilities. Tyrosinase is a key enzyme in melanin synthesis, catalyzing the conversion of tyrosine to melanin. It plays a crucial role in skin pigmentation and is a target for cosmetic research. Its inhibition by several drugs was followed using novel electrochemical enzyme-based biosensors, and factors affecting the enzyme inhibition process were optimized. It is believed that these studies will shed light on the discovery of new drugs.

Keywords: Electrochemistry, Enzyme, Tyrosinase, Biosensor, Inhibition

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OP21- Chemometric Characterization of Kome with Pikola Hazelnut and Triangle-Shaped Mulberry Leather (Pestil): Exploring the Role of Proximate Analysis and Multi-Element Analysis

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Gumushane, a city in northeast Anatolia, Turkey, has been a centre of local desserts such as pestil and kome production around the world. Recently, the certificate of incorporation of pestil is taken by Gumushane city from the Republic of Turkey Ministry of Agriculture and Forestry. Pollution with heavy (toxic) metals is one of the most important problems for various environmental components and human health. In this context, the contamination of these new product desserts called as "triangle-shaped mulberry leather (pestil)" and "kome with pikola hazelnut" can threaten food security and public health both locally and globally.

In this study, chemical characterisation was carried out on 12 samples from six different companies for two types of new products. The following parameters were determined: Total solids % (m/m), moisture % (m/m), acidity (SSA cin.) % (m/m), pH, HMF(mg/kg), protein % (m/m), fat, total ash % (m/m), glucose % (m/m), fructose % (m/m), and sucrose % (m/m). The concentrations of elements were determined using inductively coupled plasma optical emission spectrometry (ICP-OES). The most abundant elements were Ca, K, Mg, Fe, Zn, and Na. The trace element mean contents ranged between 0.03 and 15.07 mg kg^{-1}. Chemometric methods such as principal component analysis (PCA) techniques were applied to classify these new dessert products according to mineral content. Chemometric analysis of the analytical data allowed the accurate classification of the pestil and kome dessert samples according to mineral analysis.

Keywords: Triangle-shaped mulberry leather (pestil), kome with pikola hazelnut, chemometrics

1. Introduction

Pestil and kome are a traditional snack that produced adding the nutritional flavoring ingredients to pulp which was made from many fruits species such as mulberry, grape, apple, or apricot. The physicochemical parameters of these natural desserts, such as moisture, sucrose, hydroxymethylfurfural (HMF) contents, protein content, insoluble matter, diastase, acidity and multi element analysis are strictly defined and constitute the quality indicators which characterise individual Mulberry pestil and pikola kome varieties. The measurement of these parameters is comparatively simple and they provide a good information value.

2. Materials and Method

2.1. Physicochemical analysis

Samples were analysed for dry matter, pH, acidity, ash content, fat, hydroxymethylfurfural (HMF), moisture content, glucose, fructose, sucrose and protein according to the standardised methods proposed by 'The Official Methods of Analysis of Association of Official Analytical Chemists'[1,2] . Samples were analysed during the same time period to ensure uniform conditions and comparability[1]. All chemicals and solvents used in our study were obtained from Merck (Darmstadt, Germany). Chemical standards were purchased from Sigma-Aldrich (St. Louis, MO, USA).

2.2.Multi-element Analyis

Preparation of the solutions was as follows: microwave digestion procedure was applied to the pestıl and kome samples. 0.5 g of each sample was digested with 9 ml of HNO_3 (65%) and 1 ml of H_2O_2 (30%) in a microwave digestion system and diluted to 5 ml with deionised water. A blank digest was carried out in the same way.The concentrations of elements were determined using inductively coupled plasma optical emission spectrometry (ICP-OES).

2.3. Statistical Analysis

Principal component analysis and the results were compared with commercially available programs XLSTAT. All results obtained by these methods are in agreement with each other. Principal component analysis is based on the concentration of the data variance into a small number of principal components (PCs) by means of mathematical transformation[3].Cluster analysis was performed to classify samples on the basis of the similarities of their physicochemical properties.

3. Results and Discussion

The detected physicochemical results and multi element analysis for 12 samples are shown in Table 1 and Table 2.

Table 1. Values of physicochemical analysis.

Sample	Acidity (Citric acid) (g/100g)	Protein (g/100g)	Moisture (g/100g)	Dry Matter (g/100g)	pH	Fat (g/100g)	HMF (mg/kg)	Fructose (g/100g)	Glucose (g/100g)	Sucrose (g/100g)	Total Sugar (g/100g	Total ash (g/100g)	Total Mineral (mg/kg)
KA	0.45^{j} ±0.01	9.92^{e} ±0.06	17.05^{h} ±0.18	82.94^{a} ±0.19	5.69^{bc} ±0.03	10.32^{b} ±0.10	12.75^{g} ±0.70	9.92^{e} ±1.05	9.76^{e} ±0.94	12.01^{a} ±0.71	31.69^{d} ±1.90	1.05^{c} ±0.03	1198.42^{d} ±0.18
KB	0.38^{h} ±0.02	8.24^{d} ±0.18	14.10^{f} ±0.86	84.96^{b} ±0.18	5.87^{def} ±0.01	10.61^{c} ±0.18	3.58^{c} ±0.49	5.74^{c} ±0.59	9.32^{e} ±0.83	6.80^{a} ±0.39	21.86^{abc} ±1.72	1.09^{cd} ±0.20	1015.43^{bc} ±0.09
KC	0.40^{i} ±0.02	10.89^{f} ±0.30	12.84^{e} ±0.25	87.15^{c} ±0.25	5.88^{def} ±0.11	10.46^{bc} ±0.14	2.85^{abc} ±0.22	5.57^{c} ±0.41	5.29^{b} ±1.02	8.31^{a} ±0.35	19.17^{ab} ±1.56	0.92^{bc} ±0.01	2161.96^{g} ±89.76
KD	0.28^{e} ±0.01	6.80^{b} ±0.10	15.18^{g} ±0.19	84.81 ±0.19	5.82^{cde} ±0.03	11.22^{d} ±0.19	5.67^{d} ±0.42	2.12^{a} ±0.27	3.22^{a} ±0.27	10.20^{ab} ±0.37	15.53^{a} ±0.59	0.75^{ab} ±0.04	974.40^{ab} ±0.31
KE	0.33^{g} ±0.02	6.54^{b} ±0.22	15.41^{g} ±0.13	84.58^{b} ±0.13	5.95^{efg} ±0.20	5.12^{a} ±0.07	1.98^{a} ±0.06	2.95^{a} ±0.30	3.28^{a} ±0.34	32.39^{d} ±7.96	38.61^{e} ±8.38	0.70^{ab} ±0.02	933.22^{a} ±0.42
KF	0.46^{k} ±0.02	7.13^{b} ±0.04	17.35^{h} ±0.04	82.65^{a} ±0.04	5.65^{b} ±0.08	16.65^{f} ±0.07	7.48^{f} ±0.03	2.02^{a} ±0.14	5.94^{bc} ±0.36	32.83^{d} ±0.18	40. 79^{e} ±0.33	0.79^{ab} ±0.04	1035.10^{c} ±1.83
PA	0.38^{h} ±0.01	8.15^{d} ±0.45	7.11^{b} ±0.09	92.65^{f} ±0.36	6.17^{h} ±0.03	28.68 ±0.03	27.07^{i} ±1.70	8.08^{d} ±2.62	4.17^{ab} ±0.24	13.08^{ab} ±6.99	25.34^{bcd} ±8.37	1.77^{f} ±0.06	3103.31^{h} ±1.38
PB	0.17^{a} ±0.02	5.60^{a} ±0.20	10.60^{d} ±0.18	89.40^{d} ±0.18	5.80^{cd} ±0.06	15.26^{e} ±0.07	6.05^{d} ±0.18	10.25^{e} ±0.67	8.87^{de} ±0.44	11.46^{ab} ±1.40	30.58^{d} ±1.57	0.57^{a} ±0.03	938.31^{a} ±0.43
PC	0.23^{c} ±0.02	10.23^{ef} ±0.61	6.37^{a} ±0.26	93.62^{g} ±0.26	5.80^{cd} ±0.05	36.20^{j} ±0.33	3.27^{bc} ±0.02	4.93^{c} ±0.29	9.27^{e} ±2.63	12.95^{bc} ±0.47	27.15^{cd} ±3.20	1.02^{c} ±0.16	925.79^{a} ±51.32
PD	0.20^{b} ±0.02	10.29^{ef} ±1.22	7.31^{b} ±0.29	92.68^{f} ±0.29	6.03^{g} ±0.02	26.25^{h} ±0.26	2.20^{ab} ±0.04	2.14^{a} ±0.53	4.87^{ab} ±1.32	16.39^{pd} ±0.87	23.41^{bc} ±0.69	1.40^{c} ±0.35	1481.75^{e} ±0.25
PE	0.31^{f} ±0.02	8.69^{d} ±0.36	8.22^{c} ±0.08	91.77^{e} ±0.08	5.49^{a} ±0.01	20.37^{g} ±0.03	22.65^{e} ±0.97	4.60^{bc} ±0.48	5.82^{bc} ±0.35	21.15^{c} ±0.80	31.57^{d} ±0.89	1.31^{de} ±0.02	1473.38^{e} ±1.89
PF	0.27^{d} ±0.02	7.80^{cd} ±0.87	6.27^{a} ±0.60	93.72^{g} ±0.60	5.97^{fg} ±0.04	26.17^{h} ±0.16	9.25^{f} ±0.12	3.15^{ab} ±0.02	7.32^{cd} ±0.82	15.53^{bc} ±0.19	26.01^{bcd} ±1.02	1.11^{cd} ±0.02	1546.71^{f} ±0.26

Table 2. Values of multi element analysis.

Sample	K (mg/kg)	Ca (mg/kg)	Mg (mg/kg	Na (mg/kg)	Fe (mg/kg)	Zn (mg/kg)	Cu (mg/kg)	Al (mg/kg)	Mn (mg/kg)	Cd (mg/kg)	Co (mg/kg)	Ni (mg/kg)	Pb (mg/kg)	Cr (mg/kg)
KA	0.35^{fg} 0.01	559.93^{g} 0.02	461.73^{i} 0.02	75.94^{b} 0.02	26.57^{i} 0.10	7.71^{j} 0.07	4.43^{h} 0.02	35.42^{f} 0.10	24.58^{i} 0.05	0.01^{a} 0.01	0.01^{a} 0.01	1.40^{g} 0.10	0.01^{a} 0.01	0.33^{a} 0.02
KB	0.24^{cde} 0.02	365.36^{a} 0.02	382.80^{d} 0.01	224.96^{e} 0.02	16.53^{c} 0.02	8.35^{k} 0.02	2.47^{c} 0.02	2.24^{ab} 0.03	11.06^{e} 0.02	0.01^{a} 0.01	0.01^{a} 0.01	1.03^{g} 0.06	0.01^{a} 0.01	0.36^{a} 0.01
KC	0.23^{cd} 0.01	1543.00^{k} 3.00	437.93^{h} 0.64	123.00^{c} 91.49	14.47^{g} 0.15	9.90^{l} 0.02	3.62^{f} 0.06	3.24^{cd} 0.02	25.05^{j} 0.04	0.01^{a} 0.01	0.01^{a} 0.01	1.13^{g} 0.02	0.01^{a} 0.01	0.36^{a} 0.02
KD	0.22^{c} 0.02	411.92^{d} 0.02	394.43^{f} 0.02	132.60^{cd} 0.28	13.39^{f} 0.03	5.91^{b} 0.03	2.04^{b} 0.02	2.21^{ab} 0.06	10.50^{d} 0.20	0.01^{a} 0.01	0.01^{a} 0.01	0.97^{f} 0.02	0.01^{a} 0.01	0.18^{a} 0.01
KE	0.22^{c} 0.01	404.44^{e} 0.02	321.62^{g} 0.10	173.66^{i} 0.09	9.66^{e} 0.10	6.30^{d} 0.06	3.30^{e} 0.08	3.92^{cd} 0.05	8.83^{c} 0.16	0.01^{a} 0.01	0.01^{a} 0.01	0.37^{a} 0.02	0.01^{a} 0.01	0.87b 0.02
KF	0.18^{b} 0.02	386.86^{f} 0.02	309.31^{b} 0.03	306.33^{f} 1.53	7.33^{a} 0.32	7.30^{h} 0.03	4.73^{i} 0.03	3.05^{bc} 0.01	8.34^{b} 0.02	0.01^{a} 0.01	0.01^{a} 0.01	0.27^{a} 0.01	0.01^{a} 0.01	0.33^{a} 0.02
PA	0.36^{h} 0.01	1647.56^{l} 1.16	464.24^{j} 0.04	939.65^{k} 0.05	15.84^{h} 0.16	5.34^{a} 0.05	2.85^{d} 0.03	12.70^{e} 0.10	12.82^{f} 0.01	0.01^{a} 0.01	0.01^{a} 0.01	1.45^{e} 0.02	0.03^{a} 0.01	0.45^{a} 0.01
PB	0.12^{a} 0.01	458.76^{f} 0.19	182.30^{a} 0.08	266.30^{df} 0.03	10.52^{d} 0.15	7.22^{g} 0.01	1.83^{a} 0.04	3.63^{cd} 0.03	6.85^{a} 0.02	0.01^{a} 0.01	0.01^{a} 0.01	0.53^{b} 0.06	0.01^{a} 0.01	0.23^{a} 0.02
PC	0.25^{de} 0.02	430.55^{e} 0.04	395.05^{f} 0.04	3.94^{a} 0.05	18.73^{j} 0.06	6.63^{f} 0.02	4.73^{i} 0.12	3.48^{cd} 1.86	30.43^{k} 0.02	0.01^{a} 0.01	0.01^{a} 0.01	0.31^{a} 0.12	0.01^{a} 0.01	0.34^{a} 0.03
PD	0.3f 0.02	823.22^{j} 0.08	544.29^{k} 0.07	40.47^{ab} 0.15	21.56^{k} 0.01	7.46^{i} 0.03	6.41^{j} 0.02	1.35^{a} 0.02	35.24^{l} 0.03	0.01^{a} 0.01	0.01^{a} 0.01	1.07^{f} 0.02	0.01^{a} 0.01	0.35^{a} 0.01
PE	0.33^{fg} 0.01	694.98^{i} 0.01	435.47^{g} 1.83	297.49^{f} 0.05	12.92^{e} 0.03	6.03^{c} 0.02	4.22^{g} 0.01	4.19^{d} 0.07	16.24^{g} 0.03	0.01^{a} 0.01	0.01^{a} 0.01	1.04^{g} 0.02	0.01^{a} 0.01	0.44^{a} 0.03
PF	0.26^{e} 0.01	674.25^{h} 0.01	384.41^{e} 0.22	438.57^{g} 0.06	10.04^{c} 0.02	6.54^{e} 0.04	9.27^{k} 0.15	3.34^{cd} 0.03	19.40^{h} 0.10	0.01^{a} 0.01	0.01^{a} 0.01	0.37^{a} 0.02	0.01^{a} 0.01	0.23^{a} 0.02

Different letters in the same column in the table indicate a significant statistical difference in pairwise comparison ($p<0.05$). n:3

All of the samples analysed had an HMF value lower than permitted limit in TS 12677 mulberry leather (Pestil) standards (at most 40 mg/kg) [4].The pH of samples are affected by conditions during extraction and storage, which also affects texture, stability and endurance. The analysed results for all pestil and kome showed acidic character. Their pH values ranged from 5.49 to 6.17. The monosaccharides glucose, sucrose and fructose are the major constituents of pestil and kome. In this study, the sucrose, glucose and fructose contents of mulberry pestil and pikola kome samples are ranged from 6.80 g/100 g to 42.95 g/100 g, 3.22 g/100 g to 9.76 g/100 g and 2,02g/100 to 10.25 g/100 g respectively. Correlation values of physicochemical and total mineral analyses are given in Table 3. According to multi element analysis the calcium was, quantitatively, the most abundant mineral having an average content of 1543 mg/kg and it showed a concentration.

Table 3. Correlation values of physicochemical and total mineral analyses.

	% Acidity (Citric acid) (g/100g)	Protein (g/100g)	Moisture (g/100g)	Dry Matter (g/100g)	pH	Fat (g/100g)	HMF (mg/kg)	Fructose (g/100g)	Glucose (g/100g)	Sucrose (g/100g)	Total Sugar (g/100g	Total ash (g/100g)	Total Mineral (mg/kg)
% Acidity (Citric acid) (g/100g)	1	0.161	0.626	-0.636	-0.176	-0.416	0.265	0.084	-0.026	0.127	0.229	0.097	0.252
Protein (g/100g)		1	-0.254	0.255	0.017	0.315	-0.032	0.022	0.172	-0.354	-0.306	0.468	0.299
Moisture (g/100g)			1	-0.996	-0.331	-0.844	-0.294	-0.006	-0.040	0.181	0.248	-0.545	-0.407
Dry Matter (g/100g)				1	0.318	0.847	0.294	-0.003	0.026	-0.159	-0.230	0.536	0.402
pH					1	0.216	-0.070	-0.020	-0.289	-0.126	-0.272	0.325	0.492
Fat (g/100g)						1	0.291	-0.064	0.148	-0.073	-0.097	0.550	0.309
HMF (mg/kg)							1	0.330	-0.104	-0.015	0.095	0.628	0.630
Fructose (g/100g)								1	0.513	-0.471	0.079	0.065	0.217
Glucose (g/100g)									1	-0.397	0.115	-0.072	-0.329
Sucrose (g/100g)										1	0.781	-0.166	-0.197
Total Sugar (g/100g											1	-0.193	-0.263
Total ash (g/100g)												1	0.706
Total Mineral (mg/kg)													1

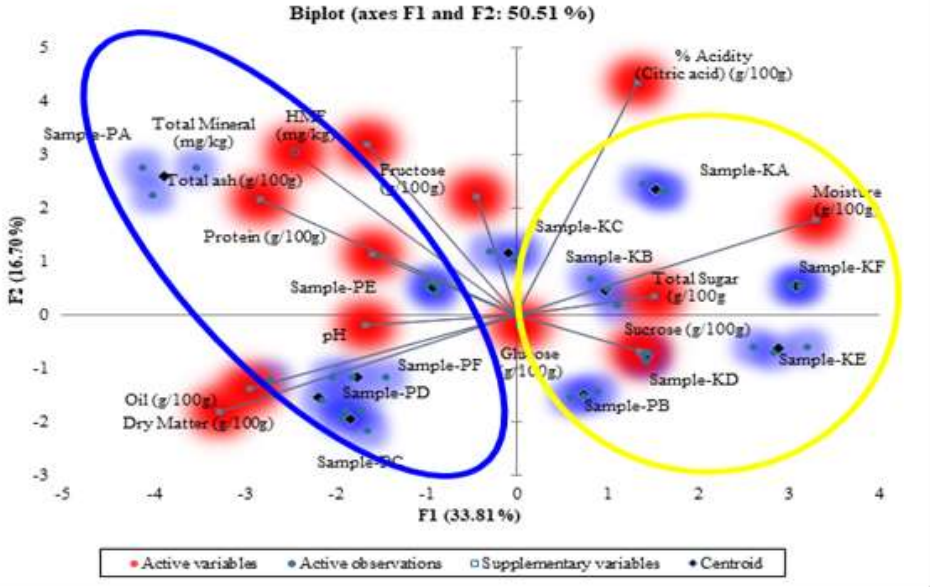

Figure 1. Principal component analysis (Scores and loadings plot).

Principal component analysis and Cluster analysis was applied to all samples examined to determine the differences between pestil and kome types. PCA was performed on standardised data to ensure that all physicochemical parameters and multi-element analysis values had equal weight in the results [5].

The principal component (PC) represented 50.51% shown in Figure 1.(PC1 33.81% and PC2 16.70%). In Figure 1, mulberry pestil and kome samples belonging to six different companies

have formed 2 groups in terms of physicochemical properties. Here KA, KB, KC, KD, KE, KF and PB show similar features. PA, PC, PD, PE and PF pestils have different properties. The score graph also shows a clear distinction between the samples of both groups at the operational level.

Cluster analysis was applied to the standardized data, the Euclidean distance was used to calculate the sample similarities and a hierarchical agglomerative procedure was employed to establish clusters [1,5]. The results obtained are shown as a dendogram in Fig. 2. Generally, at a similarity level of 60%, the samples clustered into two groups (C1 and C2) corresponding to each biochemical composition.

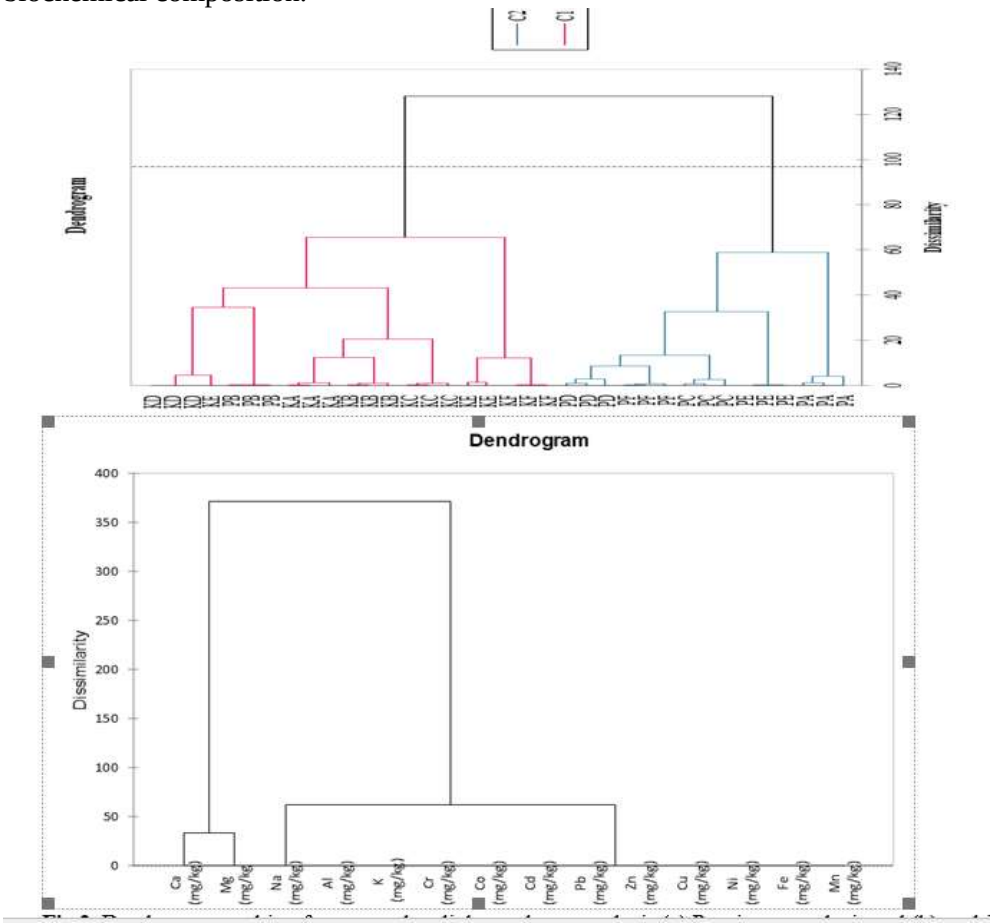

Fig 2. Dendogram resulting from complete linkage cluster analysis (a) Proximate analysis and (b) multi-element analysis.

The proximate analysis values and multi-element analysis were in the range of approved limits for all parameters. Chemometric methods such as principal component analysis and cluster analysis techniques were applied on both physicochemical data and multi-element data in order to differentiate Gumushane mulberry pestil and pikola kome. PCA explained 50.51% of the variance with the two PC variables. In the cluster analysis, moisture, sucrose and total sugar contents were high in the samples in the C1 setting, while dry matter, fat, total ash, protein, pH, HMF, Fructose and glucose analysis values were high in the samples in the C2 setting. In the cluster, there are examples of pikola kome samples within the PB sample kome, one of the triangle shaped pestils managed under a group. According to multi element results, although Ca, Na and Mg are most abundant elements in the samples, Na is in the same class with Al, K, Cr, Co, Cd and Pb. Na should be in the same class with other most abundant elements.

Conclusion

In this work, several physicochemical parameters have been determined in 12 samples of mulberry triangle-shaped pestil and kome with pikola hazelnut from different companies of Gumushane, in Turkey. The applications of these Chemometric methods aid in reducing the complexity of large data sets and offers better interpretation and understanding of the data

sets. The characterisation of these traditional desserts was carried out based on some common physicochemical parameters (pH, free acidity, ash content, fructose, sucrose, glucose and multi elements).The results showed that the use of chemometric methods on physicochemical parameters and multi-element values can be a useful tool to characterise different types of mulberry pestil and pikola kome.

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OP22- Development of a screen-printed electrochemical biosensor based on enzymatic inhibition for Anabaenopeptin B detection

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Cyanobacteria are photosynthetic microorganisms widely distributed in the world. They can inhibit several types of ecosystems, and some strains can produce toxins (cyanotoxins) and other metabolites that represent a huge danger to humans and animals, contaminating drinking water, water used in agricultural irrigation, recreational purposes, and cultivation. Among the most recurrent cyano-peptides encountered in the environment are anabaenopeptins (APs), a family of cyclic peptides containing six amino acid residues1 that demonstrated inhibitory activity towards phosphatases and proteases[1,2]. Considering that an electrochemical screen-printed biosensor is being developed for monitoring the inhibition of protein phosphatase enzyme (PP2) by using the Anabaenopeptin B (AP-B) as an inhibitor of the reaction between the enzyme and its common substrate, the 2sodium Phenyl Phosphate Dibasic Dihydrate (NaPPDD). The performance of the developed biosensor has been studied by cyclic voltammetry and chronoamperometry in phosphate buffer solution pH (7.4) as a working solution. The results were compared before and after the incubation of the inhibitor AP-B. Many conditions were optimized, such as the concentration of the enzyme and of the substrate, the incubation time of the inhibitor, the incubation time for the enzymatic reaction, and the concentration of the inhibitor. Preliminary results demonstrated the ability of these biosensors to be used as valuable tools to detect these kinds of species in the environmental field.

Keywords: Cyanotoxin, Inhibition, Electrochemistry, Screen printed electrodes, Biosensor

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OP23- Evaluation of the suitability of glassy carbon paste electrode modified with bismuth(III) oxide for electrochemical analysis of diphenoxuron

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Difenoxuron is a selective post-emergence pesticide. It can lead to disruption of cellular redox processes in the body. It shows toxicity to mammals and causes chronic health problems. Due to its rather high persistence in water, it is more toxic to fish, aquatic invertebrates and algae. Its use means that it can be found in different parts of the entire ecosystem, so it is very important to know its redox properties and to be able to monitor its concentration in environmental samples. This can be achieved by using electrochemical techniques.

The most important element in electrochemical studies are the working electrodes, which significantly affect all the redox processes taking place. Carbon Paste Electrodes (CPEs) are a sophisticated class of working electrodes. Various varieties of carbon are used in their construction. One such variety is powdered glassy carbon with specific particle sizes. An important advantage of CPEs is that they can be modified by adding nano- or micro-particles directly to the paste. CPEs modified with bismuth(III) oxide are an alternative to mercury electrodes due to their minimal toxicity, resistance to the presence of dissolved oxygen and sensitive electrochemical response.

The purpose of the study was to fabricate and characterize a paste electrode of powdered glassy carbon modified with bismuth(III) oxide (GCPE-Bi_2O_3) and test its suitability for electrochemical analysis of diphenoxuron.

Characterization of the GCPE-Bi_2O_3 electrode was carried out using atomic force microscopy, scanning electron microscopy and electrochemical techniques in solution of a standard redox system. The study showed that the best electrochemical response is achieved for an electrode consisting of carbon powder and binder in a 90:10 ratio additionally modified with 4% Bi_2O_3. The suitability for electrochemical analysis of diphenoxuron for the selected GCPE-Bi_2O_3 electrode was evaluated using the cyclic voltammetry (CV) technique in a basic electrolyte (Britton-Robinson buffer, BRB) over a wide pH range of 2.0-12.0. It was found that the optimal environment of the Britton-Robinson buffer is pH = 2. Determination of the nature of the electrode process of diphenoxuron on Bi-GCPE was performed at the optimal pH BRB using the CV technique, and the best conditions for conducting measurements to determine the standard straight line were determined.

Keywords:pesticides, electrochemical techniques, atomic force microscopy, scanning electron microscopy

OP24- Incorporation of bismuth(III) oxide nanoparticles into carbon ceramic electrode for improved electroanalytical performance in 4-chloro-3-methylphenol determination

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Phenolic compounds have been classified as pollutants of priority concern by both the United States Environmental Protection Agency and the European Union. This classification is attributed to the toxicity of these chemicals, which have severe short- and long-term effects on both humans and animals. The presence of phenolic compounds in the aquatic environment is not only objectionable and undesirable but also poses a danger to human health and wildlife. Consequently, various wastewater treatment techniques have been developed and used for the removal of phenolic compounds from industrial, domestic, and municipal wastewaters before their disposal into water bodies[1]. Moreover, numerous procedures, such as chromatographic and electroanalytical methods, have been developed to quantify phenolic compounds in water. One notable phenolic priority environmental pollutants is 4-chloro-3-methyl-phenol (PCMC), used as a medicinal or non-medicinal ingredient in final pharmaceuticals, disinfectants, veterinary drug products, cosmetics, and as an active ingredient in registered pest control products. Due to the wide use of PCMC, it is necessary to monitor its content in the aquatic environment to avoid its destructive effect on both human and aquatic lives.

Carbon-based electrodes play a prevalent role in numerous electroanalytical applications. An interesting but underestimated type of carbon-based electrodes is carbon ceramic electrode (CCE). The development of CCEs began in 1990s, and they are produced in a sol-gel route, involving hydrolysis and condensation processes of silica precursors[2]. The resulting silica matrix acts as a binder for the conductive carbon (graphite) powder. CCEs, similarly to carbon paste electrodes, offer the advantage of bulk-modification, which proves to be more beneficial than surface modification. Our previous studies on the partial replacement of graphite with various carbon and non-carbon materials such as reduced graphene oxide, carbon black, and ferrierite, have demonstrated improved electroanalytical performance of CCEs. This replacement not only enhances the reproducibility of the results but also simplifies the electrode surface refreshing process through a straightforward polishing procedure between measurement series.

In this study, a CCE with improved electroanalytical performance was developed by bulk-modifying it with bismuth(III) oxide nanoparticles (Bi_2O_3NPs) (Bi-CCE). Characterization of the Bi-CCE was conducted employing atomic force microscopy and cyclic voltammetry (CV). Comparative analysis was conducted using an unmodified CCE. The findings proved that the incorporation of Bi_2O_3NPs into the CCE significantly altered the topography of the ceramic composite and improved the electrochemical properties of CCE. The developed Bi-CCE was effectively employed to explore the electrochemical behavior and quantify PCMC using CV and square-wave voltammetry (SWV), respectively. Notably, the developed SWV procedure utilizing Bi-CCE exhibited significantly enhanced sensitivity, an extended linear range, and a lower limit of detection compared to the unmodified CCE. Furthermore, the Bi-CCE was utilized effectively for the detection of PCMC in a river water sample intentionally spiked with the compound, and the selectivity towards PCMC determination was confirmed. In summary, all analyzes performed confirmed the positive effect of Bi_2O_3NPs on the electroanalytical performance of the CCE. Consequently, the Bi-CCE emerges as a promising material for various electroanalytical applications, including phenolic pollutants determination.

Keywords: bulk modification; electrochemical characterization; surface topography; effective surface area; pollutant determination,

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OP25- Comprehensive Chromatographic Investigation of Anatolian *Cyclotrichium niveum*, significance of raw chromatographic data for chemometric discrimination of extracts

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Abstract
Cyclotrichium niveum (Boiss.) Manden. & Scheng. is an endemic species to Türkiy,e with various pharmaceutical usages[1] Air-dried and powdered aerial parts of C. niveum collected from Mt. Nemrut (Adiyaman), were extracted with n-hexane, EtOAc, and MeOH, sequentially, and also total EtOH (ultrasound assisted-UA and soxhlet) extracts and infusion were prepared. Chlorogenic acid, p-coumaric acid, ferulic acid, rosmarinic acid, rutin, luteolin and apigenin compositions of the extracts were determined by HPLC-PDA. HPLC method displayed linearity in 1.0-25.0µg/mL concentration range for the entire standards. Corresponding LOD and LOQ (µg/mL) for phenolic compounds were calculated. Voltammetric antioxidant capacity determinations, based on superoxide scavenging mechanism, revealed Total antioxidant compound contents and AC of the extracts are mainly in harmony with each other. Additionally, chemoinformatics based on PCA and CA were performed to discriminate the solvent effect on extracts and various solvent varieties were successfully discriminated based on raw HPLC-PDA data.
Keywords: Cyclotrichium niveum, HPLC-PDA, polyphenols, chemoinformatics

Introduction

Cyclotrichium niveum (Boiss.) Manden. & Scheng. is an endemic species to Türkiye, with various pharmaceutical usages[1]. Its essential oil contains pulegone and isomenthone as major metabolites, and phytochemical studies have generally revealed the presence of flavanoids and terpenic compounds[2]. Baytop[3] reported that various members of this genus, especially C. niveum and C. origanifolium (also known as “nane ruhu” in Turkish), commonly have been used to flavor soups and salads in Türkiye.

In the current study, our aim was to determine antioxidant activities and phenolic contents (PCs) of the n-hexane, ethyl acetate, ethanol, methanol and aqueous extracts of Cyclotrichium niveum. Chlorogenic acid, p-coumaric acid, ferulic acid, rosmarinic acid, rutin, luteolin and apigenin compositions were quantified for determining PCs by using our developed gradient HPLC-PDA method. The antioxidant activities was assessed by a fast and simple differential pulse voltammetry (DPV) method described by Korotkova et al[4]. Additionally chemometric studies were carried out by principle component analysis (PCA) and cluster analysis (CA) to evaluate the significance of raw chromatographic data for chemometric discrimination of the extracts.

Materials and Methods

Air-dried and powdered aerial parts of *C. niveum* collected from Mt. Nemrut (Adiyaman), were extracted with *n*-hexane, EtOAc, and MeOH, sequentially, and also total EtOH (ultrasound assisted-UA and soxhlet) extracts and infusion were prepared. The phytochemical compositions of the extracts were determined by HPLC analysis with Thermo Dionex Ultimate 3000 HPLC-PDA. Chlorogenic acid, p-coumaric acid, ferulic acid, rosmarinic acid, rutin, luteolin and apigenin were the polyphenolic standards analyzed. Chemoinformatics based on PCA and CA was performed by Minitab 17 software to discriminate the solvent effect on extracts.

Mobile phases A, B and C were acetonitrile, aqueous 2% CH3COOH and methanol, respectively. Absorbance was sampled at 330nm while 3D spectrum of 200-700nm range for a run time of 20min. Autosampler was kept at 15°C while column at 25°C. Mobile phases were pumped at 1.0mL/min flow rate with a gradient program of; 0min (A:10, B:90), 2min (A:10, B:90), 8min

(A:35, B:50, C:15), 12min (A:45, B:40, C:15), 16min (A:10, B:90), 20min (A:10, B:90). Iinjection volume was kept at 10µL. Analytical column was ACE5 C18 (250x4,6mm-RP-5µm), no guard column was necessary.

DPV was employed for ACA, which was carried out in 50-50% (v/v) methanol-50mM PBS buffer (pH 7.4). Potential was scanned in 0.0 to -2.0V (vs Ag/AgCl) potential range with 20 mV/sec scan rate and response current was acquised. Entire samples were freshly prepared and had a final concentration of 5mg/10mL. Extracts were dissolved in the solvent system under sonication for 10 minutes and filtered through 0.45µm injector type membrane filter.

Results and Discussion

Related retention times were 7.14, 9.02, 9.53, 9.82, 10.29, 11.84 and 13.04 minutes for chlorogenic acid, p-coumaric acid, ferulic acid, rosmarinic acid, rutin, luteolin and apigenin, respectively (**Fig.1**). HPLC method displayed linearity in 1.0-25.0µg/mL concentration range for the entire standards. Corresponding LOD (µg/mL) and LOQ (µg/mL) for chlorogenic acid, p-coumaric acid, ferulic acid, rosmarinic acid, rutin, luteolin and apigenin were calculated as 0.016-0.05, 0.008-0.023, 0.005-0.017, 0.011-0.034, 0.009-0.026 and 0.011-0.032, respectively. The lowest and highest amounts (µg/mL) and the corresponding solvents for the very same standards were 3.89 (EtOH-soxhlet)-10.96 (H_2O-infusion), 0.51 (MeOH)-0.68 (H_2O-infusion), 0.09 (EtOAc)-6.12 (MeOH), 2.39 (EtOH-soxhlet)-6.61 (MeOH), 2.12 (EtOH-soxhlet)-3.62 (EtOH-UA), 0.20 (EtOAc)-0.77 (EtOH-UA) and 0.36 (H_2O-infusion)-23.43 (EtOAc), respectively.

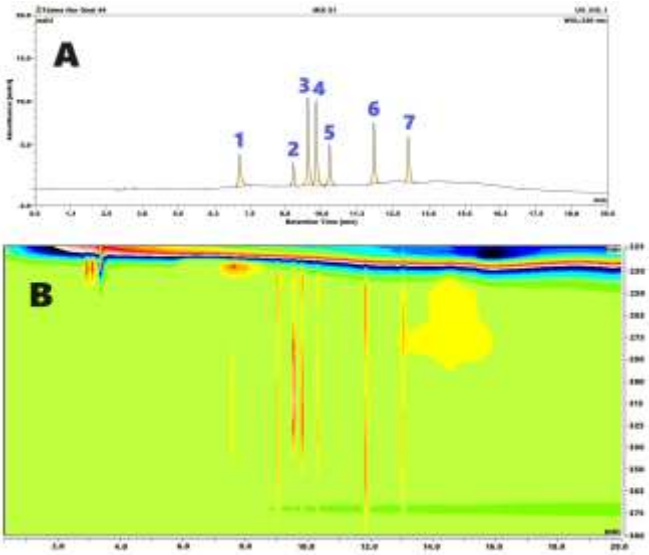

Fig.1. Typical 2D(A) and 3D(B) HPLC-PDA chromatograms of chlorogenic acid (1), rutin (2), p-coumaric acid (3), ferulic acid (4), rosmarinic acid (5), luteolin (6) and apigenin (7).

Voltammetric antioxidant capacity determinations, based on superoxide scavenging mechanism, revealed that antioxidant capacities (AC), in descending order, were as 0.844, 0.464, 0.441, 0.373, 0.327 and 0.076 for the extraction methods of EtOH/UA, EtOH/Soxhlet, H_2O/infusion, MeOH/LLE, EtOAc/LLE and n-Hexane/LLE, respectively. Based on the superoxide scavenging pathway, the extracts prepared by ultrasonic assisted extraction of ethanol (EtOH/UA) and liquid-liquid extraction with n-hexane (n-Hexane/LLE) own the highest and lowest calculated antioxidant capacities as 0.844 and 0.076, respectively. Calculated entire results are in harmony

with the concept that major antioxidant compounds own higher solubility in polar solvents than apolar ones.

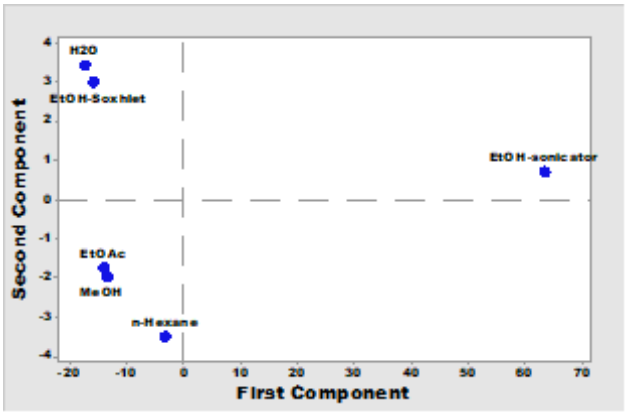

Fig.2. PCA score plot based on HPLC-PDA raw data, (n of comp: 6, type of matrix: covariance)

Regarding chemoinformatics (**Fig.2**), extracts of various solvent varieties were successfully discriminated based on PCA performed using raw PDA data.

Conclusion

Chromatographic analysis performed on related extracts of different solvent varieties. ACA, revealed that antioxidant capacities were relatively found to be higher in extracts of polar solvents. PCs and ACA of the extracts are mainly in harmony with each other. Chemometric discrimination was also successfully achieved based on raw chromatographic data.

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OP26- Optimization of on-line HS-SPME method combined with GC-MS/MS for determination of UV filters in wastewater

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UV filters are a group of chemicals added to sunscreens that can absorb ultraviolet light and therefore protect the skin from the harmful effects of UV rays. Their levels increase in wastewater and surface water due to their widespread use in the plastic, rubber, automobile and paint industries. It was reported that organic UV filters accumulate in biota and display an endocrine disrupting effect. The priority UV filters ranked according to no observed effect concentration (NOEC) values obtained from toxicological tests which is typically at sub-ppb concentration[1]. Therefore, sensitive, and selective methods for their trace residues in environmental samples is of great importance. Although a variety of techniques have been reported for their detection in surface waters[2], the studies on wastewater are quite limited in number probably due to the complex matrix of the sample.

In the current study, a gas chromatographic method coupled with head space solid phase microextraction (HS/SPME-GC-MS/MS) was developed for the extraction of nine UV filter compounds belonging to different chemical groups. Considering the chemical properties of the UV filters selected, PA fiber was preferred for the extraction due to its matching polarities with the targeted analytes. Experimental conditions namely, sample volume, extraction time and temperature, desorption time and temperature along with the ionic strength of sample solutions were scanned through Placket-Burman Design. Then, the featured parameters were optimized by using Central Composite Design. Under optimal conditions, the method performance was tested in real wastewater samples. The limit of detections were obtained in ppt regions while the recovery values were in between 89.4–115% for spiked samples.

Keywords: UV filter, wastewater, HS-SPME, GC-MS/MS

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OP27- Authenticity Control of Sea Bream Products by Stable Carbon Isotope Ratio Analysis and Chemical Composition

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Animal origin foodstuff play significant roles in human nutrition but, food fraud is a major problem due to the potential risks to public health and negative impacts on the economy[1]. Therefore, the development of analytical techniques to detect the fraud and verify the authenticity of such products is of vital importance[2].

In this study, it was aimed to compare various types of fish grown in fish farms with the same type of seafood in terms of isotopic carbon ratio and chemical composition. For this purpose, wild Sea Bream samples from different locations of Aegean Region collected in the past two years have been subjected to the stable carbon isotope ratio analysis by the IRMS. In addition, the amino acid contents were determined by the HPLC-FLD method, and the fatty acid profile was constructed by the GC-FID method.

Basic statistical analysis of the results revealed that all data obtained considerably differ for the wild and farmed samples as well as which part of fish samples was subjected to the analysis. Advanced analysis was made by using principal component analysis and it was revealed that wild and farmed Sea Bream samples can be discriminated based on the data with a total effect of 84.4.

Keywords: Food fraud, fish, isotope ratio, amino acid, PCA

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OP28- Production, Characterization and Evaluation of Application Areas of Bi2S3, Al2O3, NiCO2O4 Nanoflowers using Microwave-Assisted Synthesis Method

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In nanomaterial production, the size, particle diameter, and shape of the nanomaterial are the most important parameters that determine the properties and application area of the nanomaterial. Especially for applications requiring advanced technology, the design of nanomaterials consisting of metal and semi-metal cores that offer a very large surface area in a 3D structure comes to the fore. Nanoflowers, which are included in various nanomaterial structures such as nanorods, nanoribbons, nanowires, nanotubes, and nanoparticles, are recently developed nanomaterials with a size of 100-500 nm and a morphology resembling a flower[1]. Solvothermal, sol-gel, hydrothermal, co-precipitation, chemical vapor precipitation, microwave-assisted synthesis, sonochemical, electrodeposition, and biosynthesis methods can be used for the synthesis of nanoflowers[2]. Among these, the microwave-assisted hydrothermal synthesis method offers advantages such as rapid heating, energy saving, high reaction rate that provides synthesis in size-controlled synthesis in a very short time period. In this study, microwave-assisted synthesis approaches for the transformation of Bi_2S_3, Al_2O_3, and $NiCo_2O_4$ nanostructures into nanoflowers with economical, rapid, and controlled growth were introduced. Characterizations of the produced nanoflowers were carried out using Scanning Electron Microscopy, Fourier Transform Infrared Spectroscopy, and X-Ray Diffraction systems. Additionally, the potential application areas of these nanoflowers such as the preconcentration of analyte(s) and the removal of pollutants from water matrices, were evaluated. With the proposed method, flower-shaped Bi_2S_3, Al_2O_3, $NiCo_2O_4$ nanostructures can be synthesized easily in a single step. It is anticipated that these nanostructures will be potential nanomaterial candidates for future analytical applications.

Keywords: Nanoflowers, Microwave assisted synthesis, Nanomaterials, Characterization

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OP29- Flower-Shaped Ni(OH)2 Nanomaterial's Analytical Application in Preconcentration of Manganese Determination in Domestic Wastewater Samples by Flame Atomic Absorption Spectrometry

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Manganese (Mn) is a silvery-white metal which is fragile and can be found in the environment in a considerable amount[1]. This heavy metal is engaged in metabolic processes in the human body because of its role for many enzymes and cellular reactions. Mn is mostly stored in kidneys and liver in the human body. Despite of its essential effects, the excess take of Mn can be resulted in poisoning which can concluded as disruption of the nervous system while deficiency can cause growth and reproduction problems[2]. In this study, a new method of Mn determination was suggested by spectroscopy with low standard deviation between samples and high sensitivity in conclusion. To achieve these standards, a system of spectroscopy combined with flame atomic absorption spectrometry was used. Before that, a preconcentration method was applied to the samples to preconcentrate Mn. For this aim $Ni(OH)_2$ nanoparticles with a flower shape were synthesized. To reach the optimum conditions for this extraction of Mn; all experimental factors including nanoflower amount, mixing type and sample volume. Limit of detection was found as 2.2 µg L^{-1} by using the optimum experimental conditions and enhancement in detection power was calculated as 41. The viability of the suggested method in the real samples was assessed in domestic wastewater. For this aim, recovery experiments were conducted by applying developed extraction strategy in domestic wastewater. This recovery experiments were concluded in the percent recovery results of Mn at 10, 20, 40, and 60 µg/L as 114.3±4.6%, 120.1±19.4%, 95.1±7.3%, and 95.6±5.0%, respectively. These results verified the sensitivity and accuracy of this newly proposed Mn determination method at trace levels.

Keywords: Domestic wastewater; Manganese; nanoflowers; flame atomic absorption spectrometry

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OP30- Qualitative Detection of Toxoplasma gondii with Graphene/Chitosan/Toxoplasma gondii Antibody-Modified Graphite Electrode

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Toxoplasma gondii (T. gondii) is a parasite with cats as its primary host and is widely prevalent worldwide[1]. This parasite can cause lethal harm, particularly to fetuses and individuals with weakened immune systems[2]. Therefore, early detection of the parasite before infection is crucial. In this study, graphite electrodes were modified with a graphene-chitosan composite to create diagnostic surfaces and *T. gondii* antibodies were immobilized on the surface of the electrode. Additionally, T. gondii/gold nanoparticle (Ab_T/AuNP) bioconjugate was prepared as a signal probe. The *T. gondii* surface proteins and signal probe were sequentially dropped onto the recognition surface, and 20-minute incubation periods were waited for each. Qualitative tests were successfully conducted using differential pulse voltammetry signals of Ab_T/AuNP. In this study, a sensor prototype of quality that could serve as a foundation for future research endeavors was obtained.

Keywords: Toxoplasma gondii, qualitative test, immunosensor, biosensor, electrochemistry.

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OP31- Evaluation of Chestnut Shell Bioactive Compounds and Color Properties Using Ultrasound Assisted Extraction

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The whole chestnut processing technology generates a large amount of waste, mostly in the form of burrs, leaves, and shells.[1] Chestnut shells, which have valuable bioactive compounds that can be re-processed, come as a waste for the candied chestnut production.[2] Approximately, shells arise as of the 20-30 % weight of chestnut[3] resulting one of the reasons of enviromental pollution and economical loss.[4] Therefore, it is important to valorize the chestnut shell bioactive compounds.

In this study, chestnut shells were obtained from chestnut candy production unit. Chestnuts used in the production are in the family of *Castanea Sativa* Mill. and Sarıaşlama cv. which is a native cultivar grown in Bursa City. The residue of chestnut fruits was removed, and then inner (integument) and outer (pericarp) shells were grounded, sieved, and stored at 18 ˚C until analysed. Moisture content of the powdered shells was determined as 11.10% (w/w) of total chestnut shell weight. Ultrasonic assisted extraction was performed through an ultrasonic bath (Elma S 300H) operated at 1500 W power at 37 khz frequency. Optimization was performed within the range of: temperature 30-70 ˚C, duration 15-75 min, solvent (NaOH) concentration 0-0.2 M.

By using a central composite design in ultrasonic assisted extraction, optimum condition was obtained as 30 min at 60 °C and 0.15 mol/L solvent concentration. For the optimized condition, color value, total phenolic content, total antioxidant capacity with DPPH and CUPRAC methods were determined as 17.52, 190.50 mg GAE/g dm (Folin Ciocalteu method), 211.15 mg TE /g dm and 700.38 mg TE /g dm respectively. Additionally the most abundant individual phenolics were found as Gallic acid (93.45) , Ellagic acid (62.81), Protocatechuic acid (56.85) as mg/kg dry extract and the most plentiful flavanoids were determined as Catechin (16.53) and Epicatechin (11.53) as mg/kg dry extract.

Ultrasound assisted extraction of chestnut shells with alkaline solvent (NaOH) possessed strong bioactive potential and brown color properties while it reduced the extraction time. Besides it can be used as a promising extraction technique to valorize chestnut shell bioactive compounds.

Key words: Chestnut Shell; Bioactive Compounds; Ultrasound Assisted Extraction, Alkaline Solvent,

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OP32- Removal of Cadmium Ions from Synthetic Wastewater Samples by CuFe2O4 Magnetic Nanoparticle Assisted Batch Type Adsorption Based Removal Strategy

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Industrial production can lead to the release of a high diversity of noxious substances, encompassing both organic and inorganic components. Insufficient treatment and discharge of these pollutants into aquatic environments will result detrimental results[1]. Cd (cadmium) is a toxic element that is not *'essential'* for the body's functioning and can be found in various places in the environment. Anthropogenic and geogenic sources can boost Cd concentrations in soils and groundwater, which are vital for supplying healthy food and safe drinking water[2]. This study devised the development of a novel procedure employing nano-sized adsorbents to remove cadmium ions from wastewater by batch type adsorption based process. For the aim of the study, $CuFe_2O_4$ nanoparticles with high magnetic properties were synthesized by using a co-precipitation process with the intention of using them as an adsorbent in a rapid and efficient removal strategy. The characterization of the synthesized magnetic $CuFe_2O_4$ nanoparticles was performed by X-Ray diffraction (XRD), field emission scanning electron microscope (FESEM) and Brunauer-Emmett-Teller analysis (BET) systems. The method variables were examined through univariate experiments to attain the most suitable state for the interaction between cadmium ions and the adsorbent. The assessment of removal efficiency (%RE) was conducted using synthetic wastewater samples. The method's efficacy in removing cadmium ions from synthetic wastewater was found to be high within the linear operating range of flame atomic absorption spectrophotometry (FAAS). Furthermore, the Langmuir isotherm model was applied to elucidate the developed adsorption process. The results showed that this model accurately suited the experimental data. The verified models demonstrated the effective adsorption of cadmium from synthetic wastewater by the magnetic $CuFe_2O_4$ nanoparticles.

Keywords: Adsorption, Cadmium, Magnetic nanoparticles, Removal strategy, Wastewater

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OP33- Sieve Conducted Two Syringe Based Mixing System Assisted Preconcentration and Determination of Nickel in Antarctic Lake Water Samples by Slotted Quartz Tube Flame Atomic Absorption Spectrometry

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Compared to other transition metals, nickel (Ni) is a moderately toxic element. Nevertheless, it is established that the act of breathing in Ni and its compounds can result in severe complications, such as the development of cancer in the respiratory system [1]. Hence, it is crucial to determine the presence of Ni at low quantities. The emergence of novel approaches in sample preparation is a developing tendency that aims to diminish the usage of organic solvents, streamline procedural steps, and enhance the consistency of repeated processes. The sieve employed a dual syringe pressurized liquid-liquid microextraction system to enhance the extraction solvent's surface area by utilizing the automated reciprocating motion of two syringes for pressurized mixing [2]. Within the scope of this study, the complex formation parameters of Ni metal and the mixing system parameters containing a double syringe were optimized. Several parameters include pH and volume of the buffer solution, concentration of the ligand, type and volume of the extraction solvent, duration of mixing, and concentration of the eluent. After calibration studies carried out under optimized conditions, the developed SQT-FAAS method with dual syringe mixing system showed more than 20-folds improvement in detection power compared to the traditional FAAS method. The applicability of the method to real samples is supported by recovery studies in Antarctic lake water samples with satisfactory results.

Keywords: Nickel, liquid-liquid microextraction, Antarctic lake water, sieve conducted to syringe mixing.

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OP34- Deep Eutectic Solvent Based Preconcentration of Sulfonamides in Honey Samples and Their Determination by High Performance Liquid Chromatography

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A category of synthetic antibiotics, sulfonamides (SAs) are a broad term for a group of medications that contain p-aminobenzenesulfonamide structures [1]. The exposure of SAs, particularly in livestock, and their subsequent release into the environment can result in the development of microbial resistance to specific strains and pose detrimental health hazards [2]. Deep eutectic solvent (DES) has gained significant interest as an environmentally friendly extractant in various fields in recent years. The advantages of DES include its affordability, simple preparation, minimal evaporation, and ability to biodegrade. Because of these advantages, DES has been extensively employed as a solvent for extraction purposes [1]. This study utilized a High-Performance Liquid Chromatography (HPLC) system to determine four active substances from the sulfonamide antibiotic group in honey samples. Prior to analysis, the substances were concentrated using DES. In pursuit of developing an eco-friendly extraction technique, the DES species utilized in this investigation were synthesized by combining numerous hydrogen bond donor (HBD) and hydrogen bond acceptor (HBA). To achieve this purpose, the parameters for both DES and HPLC system were optimized. The parameters that can be listed include the type and volume of the DES, mixing time, pH and volume of the buffer solution, type and volume of the eluent, pH of the mobile phase, column temperature, and injection volume. The recovery studies conducted to evaluate the developed method's accuracy and applicability utilized honey samples; satisfactory results were obtained.

Keywords: Antibiotics, sulfonamides, deep eutectic solvents, high performance liquid chromatography.

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OP35- Development Of Prototypes From Honey Bee (Apis Mellifera L.) Venom That Can Be Used In Cosmetic And Pharmacopuncture Applications

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Abstract
The production of pine honey, which is the main beekeeping product of Muğla Province, has come to a standstill due to the major fires that happed in the summer months of 2021, drought and the increasing effects of climate change. The disappearance of the source of income of families, whose only source of livelihood was beekeeping, has caused these get stuck to continuing their beekeeping profession. At this point, it has been detected that bee venom can be produced during periods when honey production cannot be made and that the beekeeping sector can be saved from this predicament with bee venom production. The increasing need for bee venom, the demand for high quality products from medicine to cosmetics, and the lack of a domestic product that can be used in these sectors, the reasons such as the lack of a standardized method for purification of bee venom, the lack of consistency of purification techniques and with their results in the literature, and the lack of anticancer effect studies with melittin purified from bee venom constitute the basis of the presented project.
Bee venom is a protein and peptide-dominated secretion product produced by the honeybee (*Apis mellifera* L.) for use in colony defense. The composition of bee venom consists of melittin, apamin, MCD-peptide, dopamine, phospholipase-A2 and hyaluronidase enzymes. Due to its rich chemical content, bee venom (Apitoxin) is widely used as an anti-aging in Traditional and Complementary Medicine Centers (GETAT) and in the cosmetics industry. Bee venom has been detected to be efficient on arthritis, Multiple sclerosis (MS), Parkinson's, Alzheimer's, low back pain, stroke, skin diseases, neuroprotective actions and immunological diseases. Bee venom has been also used to treat cancer and to boost the immune system. Studies have shown that pure melittin, a powerful anticancer peptide, is a better choice than the use of raw bee venom.
The use of bee venom in apitherapy applications is increasing gradually. Pharmacopuncture is an unique treatment in oriental medicine that combines traditional acupuncture with chemical stimulation. Pharmacopuncture is an alternative treatment for incurable diseases. Therefore, within the scope of this presented study, it is aimed to determine and standardize the procedures for obtaining the active ingredients used in **bee venom pharmacopuncture (BVP)**, which is a very effective technique in oriental medicine, from raw bee venom.
Raw bee venom to be used within the scope of the project will be procured from our contracted beekeepers operating in our region. The percentage component values of bee venoms, brought into the laboratory, can be determined by High Performance Liquid Chromatography (HPLC/VWD). Three different techniques can be used to purify the raw bee venom by separating it into fractions. **The first of these;** Centrifugal Partition Chromatography (CPC), **the second** is Flash-Prep Chromatography and **the third** is open column chromatography where different stationary phases can be used. Three chromatography techniques can be applied in the isolation of the prepared poison solutions using a gradual gradient solvent system (Water-Ethanol, Water-Propanol). Among these three chromatographic methods used, the technique with the highest melittin yield and percentage can be determined. During these processes, it is also anticipated that allergenic proteins can be effectively removed. The melittin content of fractionated and purified bee venom can be determined using HPLC/VWD.
Within the scope of the study, two prototype products can be obtained. **The first prototype** is a purified and standardized powdered bee venom with 100% water solubility from raw bee venom. This product can be used by drug and cosmetic companies. **The second prototype is** a product obtained by separating raw bee venom into its fractions, with a melittin content of at least 90%, 100% soluble in physiological saline, its content standardized and bottled. This product can have standards that can be used in GETAT centers.
As a result of this study, both the purified raw bee venom and its main active component (melittin) were increased, allergens were removed from the environment to a certain extent, it could be preserved under room conditions, its content and purification process were standardized with the recommended methods, it could be used in bee venom pharmacopuncture, its anticancer effects were determined, it was safe and effective. It is aimed to develop stable prototypes.
Keywords: Apitherapy, pharmacopuncture, bee venom, melittin, anticancer, HPLC/WVD, UPLC/MS-MS

INTRODUCTİON

Bee Venom and Apitherapy

Arthritis is a very old human disease, and bee stings are thought to be the first apitherapy discovered by humans to treat arthritis. This is because that Homo sapiens are assumed to find relief after bee stings. Austrian Doctor Philip Terc, the father of modern Apitherapy, suffered from rheumatism and treated himself using bees. In his Terc hypothesis, he assumed that the stronger the form of rheumatism, the stronger the doses of bee venom should be. Healing occurs in three stages, in the first stage; in the second stage, patients develop a pathological immunity that reacts very weakly to bee stings; he suggested that

with the development of a local painful reaction, sensitivity to BV occurs as much as normal people, and healing begins at this stage, and healing is completed in the third stage. Terc treated 660 patients in total and stated that he used 1 to 50 bees per session. It was reported that 544 of these patients recovered completely, 99 recovered, and the remaining 17 did not respond to treatment [1-3].

Melittin is a biologically active peptide found between 40% and 65% in bee venom. It reduces and stabilizes the surface tension of membranes. It has an anti-inflammatory effect in very small doses and stimulates smooth muscles. It activates the pituitary and adrenal glands. It increases capillary permeability and blood circulation. It lowers blood pressure, reduces blood clotting, and stimulates the immune system. It affects the central nervous system. It has anticancer, antibacterial, antifungal, antiviral, antibacterial, antiatherosclerosis and endosomolytic effects. Higher doses are inflammatory and hemolytic [3,4,5]. Bee venom acupuncture has also been used to treat autoimmune disorders. Favorable immunological responses to bee venom acupuncture were detected in a mouse model of trimellitic anhydride-induced atopic dermatitis [6].

Bee Venom Ingredient Studies

Teoh et al. (2017) examined the specific effects of different buffers and pH values on the purification of melittin. In general, purification processes have been carried out by using cation exchange and size exclusion chromatography in a series of purification stages [7]. In another study, Lee et al. (2018) conducted studies on bee venom content using a C18 column (5µm, 4.6 x 150mm) using high-performance liquid chromatography (HPLC) and a Waters Alliance UV detector [8].

Chmielewska et al. used high-performance liquid chromatography (HLPC) and routine tests to separate, identify and quantify the main components of honeybee venom (*Apis mellifera* L.). Method development studies were carried out by. They tested different variables in an effort to develop an HPLC method to test the main venom components. These; These are chromatographic columns with different pore size (100, 180 and 300 A), separation temperature (25, 30, 35 °C), flow rate (1.0, 1.5 and 2.0 ml/min), different gradient elution conditions and C18 filling materials [9].

METHOD

The raw bee venom used within the scope of the study was supplied from our contracted beekeepers operating in our region. Three active substance screenings were performed on the samples. The active ingredients are Apamin (Sigma-A1289), Phospholipase-A2 (Sigma-P9279) and Melittin (Sigma M2272).

First, additive-residue analyzes (Pesticides and Antibiotics) were performed on bee venom samples by Ultra Performance Liquid Chromatography-Tandem Mass Spectrometry (UPLC-MS/MS) and the samples that passed this test were taken to purification and fractionation processes. The percentage component values of the poisons that passed additive and residue analyzes were determined by High Performance Liquid Chromatography (HPLC/VWD). Three different techniques were used to purify raw bee venom by separating it into its fractions. **The first of these**; Centrifugal Partition Chromatography (CPC), **the second**; Flash-Prep Chromatography and **the third** is open column chromatography in which different stationary phases will be used. With these methods, it is aimed to significantly increase the melittin yield. During these processes, it is also envisaged that allergenic proteins will be effectively removed. Melittin content of fractionated and purified bee venom was determined using HPLC/VWD.

Within the scope of the study, two prototype products were obtained. The first prototype is powdered bee venom, purified and standardized from raw bee venom with 100% solubility in water. This product can be used by cosmetic companies. The second prototype is; It is a product obtained by separating raw bee venom into its fractions, with melittin content of at least 90%, 100% soluble in physiological saline, its content standardized and bottled. This product has standards that can be used in GETAT centers.

1. Bee Venom Composition

Approximately 88% of the bee venom composition is water, and the rest consists of peptides, polypeptides, enzymes, amines, lipids, sugars and amino acids. Melittin is a major bee protein and is a peptide consisting of 26 amino acids, with a molecular weight of approximately 3 kDa and constituting 50-60% of the dry weight of bee venom. Bee venom contains the enzymes Phospholipase A2 (10-13%), hyaluronidase (2%), apamin (2-3%), MCD peptide (2-3%), secapin (0.5-2%), dopamine (1 -3%) forms peptides. To date, various chromatographic methods have been developed for bee venom characterization, including thin layer chromatography, capillary electrophoresis, high pressure liquid chromatography (HPLC), ultra performance liquid chromatography (UPLC), and MALDI-TOF techniques [3,7].

2. Determination of Pesticide Residues by UPLC-MS/MS

First, 0.1g (100 mg) of homogenized raw bee venom sample was weighed into 15 mL falcon tubes. Then, 10mL of MeCN (acetonitrile) containing 1% Acetic acid (CH3COOH) was added and vortexed. After it was completely dissolved, 1.5g of anhydrous Sodium acetate (CH3COONa) was added and vortexed again. Then, 6g of anhydrous Magnesium sulfate (MgSO4) was added and 150µl of Internal standard (ISTD)

solution was added. The tubes were then shaken vigorously for 1.5 min and then centrifuged at 4000 rpm for 5 min [10].
4mL extract from the upper part of the tube coming out of the centrifuge was transferred to a new 15 mL falcon tube, and 200mg Primary Secondary Amine (PSA) and 600mg anhydrous Magnesium sulfate were added to the solution and vortexed again and centrifuged at 4000rpm for 5 minutes. After the clean-up step, 1mL of extract was transferred to 2mL vials using 0.20 µm PTFE filters and the mobile phase containing 300µL 2mM Ammonium Formate (HCOONH4) was added. The vial was analyzed by UPLC-MS/MS [10].

3. Determination of Antibiotic Residues by UPLC-MS/MS

The homogenized 0.1g (100 mg) raw bee venom sample was weighed into 10 mL volumetric flasks. 5mL of pH: 3.2 pure water containing 1% Acetic acid (CH3COOH) was added and the final volume was completed to 10mL. The volumetric flasks were shaken vigorously and vortexed for 3 minutes. Flasks were subjected to ultrasonic extraction in an ultrasonic bath at 50°C for 1 hour in Sweep Mode. The samples were transferred to 15 mL falcon tubes and centrifuged at 4000 rpm for 4 minutes. After centrifugation, 1 mL of extract was filtered through 0.20µm PTFE filters and transferred to vials. Finally, it was analyzed by UPLC-MS/MS [11].
Mass transfer information for antibiotic groups and retention time information for the compounds are given in the table below.

Table 1. Mass transfers and retention time for antibiotic groups

	Parent ion (*m/z*)	1° daughter ion (*m/z*)	Cone voltage (V)	2° daughter ion (*m/z*)	3° daughter ion (*m/z*)	Collision (V)	ESI	RT (min)
Amphenicols								
Chloramphenicol	305.0	165.0	25	258.0	275.0	20,20,12	+	5.41
Tetracyclines								
Tetracycline	445.4	154.0	22	410.2	427.0	26,20,18	+	4.66
Epichlortetracycline	479.2	444.2	31	462.2		22,15	+	5.01
Epioxytetracycline	461.3	426.2	19	444.2		19,16	+	4.56
Epitetracycline	445.3	410.2	25	427.2		19,15	+	4.38
Doxycycline	445.2	154.0	25	428.2		28,20	+	5.73
Chlortetracycline	479.3	444.2	27	462.2		20,18	+	5.31
Oxytetracycline	461.2	426.2	22	443.1		19,13	+	4.72
Methacycline	443.0	201.0	28	381.0	426.0	25,20,16	+	5.55
Sulfonamides								
Sulfadimethoxine	311.1	92.0	28	156.0	245.0	32,20,12	+	5.44
Sulfamethazine	279.1	92.0	30	124.0	186.0	28,20,16	+	4.58
Sulfamerazine	265.0	92.0	26	156.0	172.0	28,15,17	+	4.22
Sulfamethoxipyridazine	281.1	92.0	27	126.0	156.0	30,18,15	+	4.61
Sulfadoxine	311.0	92.1	27	108.0	156.0	32,28,15	+	4.95
Sulfathiazole	256.0	92.1	23	108.0	156.0	25,23,15	+	3.96
Sulfameter	281.0	156.0	20	215.0		26,26	+	4.43
Sulfacetamide	215.0	92.0	17	108.0	156.0	22,18,12	+	3.18
Sulfadiazine	251.0	92.0	25	156.0		27,15	+	3.77
Sulfamethoxazole	254.0	92.0	25	156.0		26,16	+	4.78
Sulfisoxazole	268.0	92.0	22	156.0		28,13	+	4.93
Sulfamethizole	271.0	92.0	19	156.0		30,15	+	4.47
Sulfabenzamide	277.0	108.0	13	156.0		22,15	+	5.10
Sulfachloropyridazine	285.1	92.0	22	156.0		28,15	+	4.72

4. Determination of Bee Venom Content by HPLC-WVD

Three components were screened in bee venom samples. These; The active ingredients are Apamin (Sigma-A1289), Phospholipase A2 (Sigma-P9279) and Melittin (Sigma M2272). 5-point calibration curves for these standards are also created. Poison analysis was performed with an Agilent 1260 series HPLC device. 20 mg of bee venom is taken and dissolved with Mobile Phase A (0.1% Trifluoroacetic acid-TFA) in a 20mL volumetric flask. The extraction process is performed in an ultrasonic water bath in sweep mode for 30 minutes. The injection volume is 30 µL and the column temperature is 10°C [12, 13].
Using VWD as the detector, the components in the bee venom were separated with the help of a Poroshell C18 column and the chemical content amounts were determined qualitatively and quantitatively. During the preliminary method validation process, the optimum separation temperature was 20°C and the column flow rate was 0.5ml/min. It has been determined that. Water (A) containing 0.1% TFA and acetonitrile containing 0.1% TFA were used as mobile phases. The gradient program was optimized according to peak retention times, and the detection of analytes was performed using the Agilent 1260 HPLC-VWD device at 220nm [12, 13].

5. Purification of Bee Venom and Isolation of Active Components

Raw bee venom was brought into a 10% solution with pure water and a homogenizer. This 10% diluted sample was fractionated with the help of gradient ethanol solution (0%-80%) using Flash-Prep chromatography and open column chromatography. In the CPC technique, fractionation was performed by using 50% water-proponol solutions and eluting the sample. If necessary, additional isolation processes such as thin layer and repeat column chromatography can be applied for the resulting fractions. Three techniques described below could be used in isolation processes.

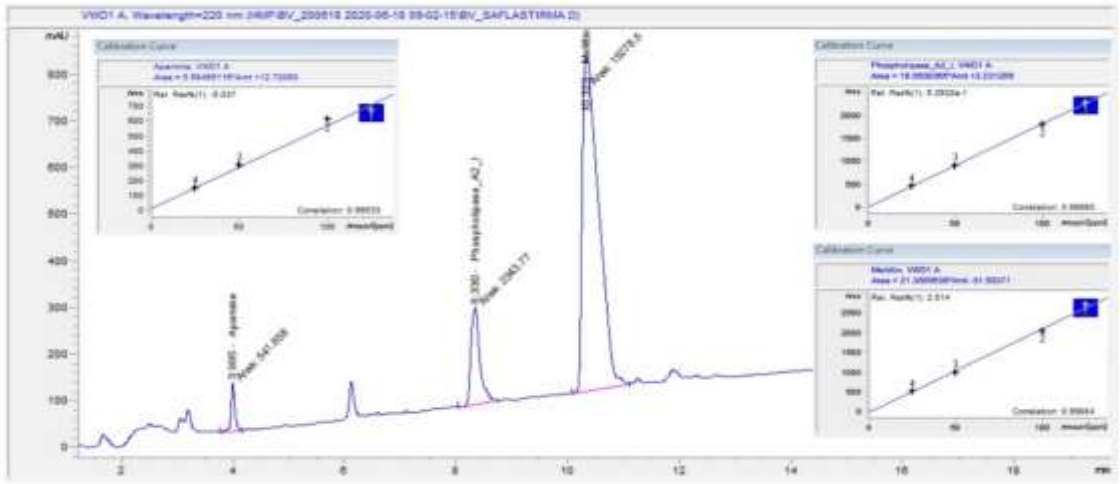

Figure 1. Chromatogram of the active ingredients contained in bee venom obtained by HPLC-VWD device and calibration curves of components

- **Centrifugal Partition Chromatography, CPC)**

In the CPC technique, choosing the solvent system is like choosing the column and eluent in HPLC. The chromatographic effect in CPC is based on the distribution constant (Kd). This is the equilibrium constant for the distribution of an analyte in two immiscible solvents. For a given compound, Kd is equal to the ratio of its molar concentration in the stationary phase to its molar concentration in the mobile phase according to the equation $[C_{Stationary\ phase}] / [C_{Mobile\ phase}]$. A Kd of 1 unit represents an equal partition between the mobile and stationary phase. To achieve a CPC separation, the relevant analysis Kd must be between 0.5-5. If Kd is too low (below 0.5) the analyte is retained in the mobile phase and no separation occurs, if it is too high (above 5) the analyte is retained in the stationary phase. The solvent system can then be determined based on the partition coefficients of all molecules that need to be separated**.**

- **Flash-Prep Chromatography**

Isolation of pure compounds from extracts of natural samples is one of the challenges of chromatography that has been going on for years. While we used only open column techniques before 2000, today we can perform preparative fractionation with Flash-Prep Chromatography with less time, solvent and cost. Flash chromatography is a system combined with a high-pressure pump, resizable cartridges, fraction collectors, ELSD and UV detectors. Samples can be loaded into the system with solid and liquid loading apparatus, and very efficient elutions can be made by adjusting the mobile phase components and percentages on the system.

When separating bee venom into its components, C18 cartridges and ethyl alcohol solution (range 0 - 80%) could be used as the mobile phase. The reason for choosing these solvents is; This is because melittin is better soluble in ethyl alcohol, while other components such as Phospholipase A2 and Apamin have a better solubility in water.

- **Column Chromatography**

Column chromatography is widely used for the purification of biomolecules. First, the sample to be separated is added to the column, usually glass, with the stationary phase attached to it, and then the mobile phase, which will provide the elution of the analytes, is added. Analytes interact with the stationary phase and are separated from each other by the influence of the mobile phase and collected at different times. The resulting fractions are checked by thin layer chromatography and the process is continued until the analytes in the sample are completely separated from each other.

6. Lyophilization and Vialing

For the Purified Bee Venom (PBV) prototype intended to be used in the cosmetics industry, first 3g of raw bee venom was dissolved in 1000mL (1L) of cold pure water. Then it was filtered through 0.20 µm polyester

membrane filters with the help of vacuum. The purified bee venom obtained or the fractions obtained by chromatography techniques were pulverized with the help of Christ Beta 2-8 LSCPlus after HPLC analysis. Vialing process was also carried out with the vial closing feature within the lyophilizer. In this way, a purified bee venom (PBV) prototype was obtained from raw bee venom, which was a strategic product.

CONCLUSION

Three components were screened in bee venom samples. These; The active ingredients are Apamin (Sigma-A1289), Phospholipase A2 (Sigma-P9279) and Melittin (Sigma M2272). 1000ppm main stock of these standards and intermediate stock solutions were prepared for later use.

Additive-residue analyzes of poison samples could be performed with UPLC-MS/MS device. Before starting the purification process, pesticide and antibiotic residue analyzes must be performed on all samples to be studied. Samples that do not comply with the directives or are outside the MRL (maximum residue limit) limits will not be evaluated. In this context, bee venom samples should not contain pesticide and antibiotic residues.

For this analysis, the pesticide method developed and modified in our laboratory was used. Although it is a disadvantage that bee venom samples are small in volume and weight, this problem has been overcome with the methods we have pre-tested in the laboratory. In this context, samples that did not comply with the directives or were outside the MRL limits were not evaluated.

As a result of the analysis, it was determined that there were no pesticide and antibiotic residues in the bee venom samples produced with the contracted beekeeping model. However, in the samples taken from outside, residues of Sulfamethazine and Tetracycline group antibiotics were found, which can be seen in the chromatogram below.

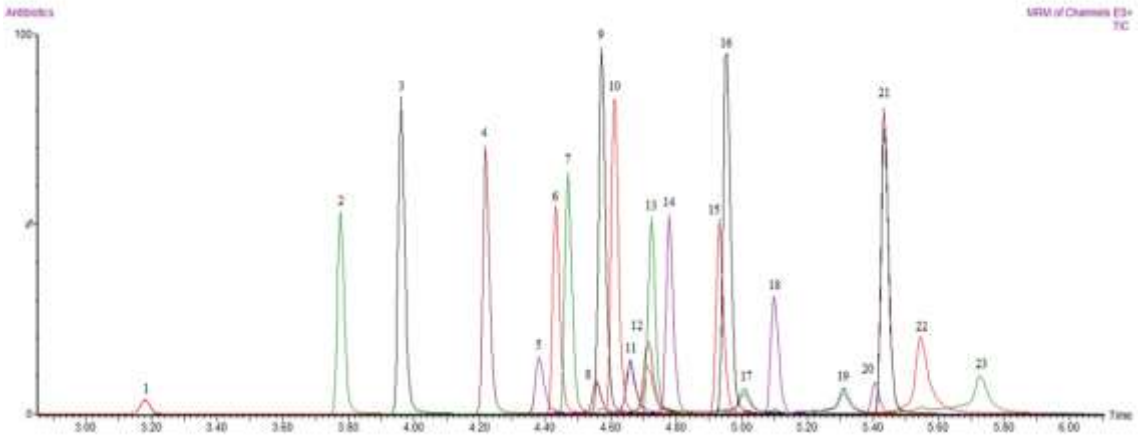

Figure 2. Total Ion Chromatograms (TIC) and chromatogram numbers of antibiotic compounds: (1) Sulfacetamide, (2) Sulfadiazine, (3) Sulfathiazole, (4) Sulfamerazine, (5) Epitetracycline, (6) Sulfameter, (7) Sulfamethizole, (8) Epioxytetracycline, (9) Sulfamethazine, (10) Sulfamethoxipyridazine, (11) Tetracycline, (12) Dxytetracycline, (13) Sulfachloropyridazine, (14) Sulfamethoxazole, (15) Sulfisoxazole, (16) Sulfadoxine, (17) Epichlortetracycline, (18) Sulfabenzamide, (19) Chlortetracycline, (20) Chloramphenicol, (21) Sulfadimethoxine, (22) Methacycline, (23) Doxycycline.

Determination of sample contents and % composition using HPLC-VWD device

Determination of sample contents using HPLC-VWD device was made with Agilent 1260 series HPLC device. VWD was used as the detector, and the components in the bee venom were separated with the help of a Poroshell C18 column and the chemical content amounts were determined qualitatively and quantitatively. In the chromatogram given below, it can be seen that the 3 active ingredients in bee venom can be easily separated from each other and can be analyzed by HPLC-VWD.

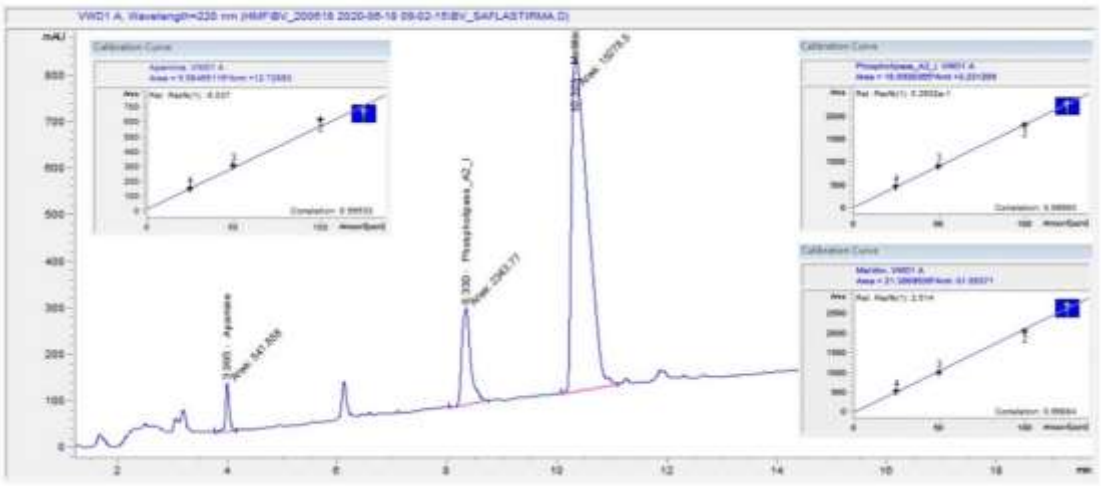

Figure 3. Chromatogram of three active ingredients in bee venom

It has been determined in laboratory studies that peptides in bee venom solution can be separated by CPC chromatography under the following conditions. It has been determined that melittin from bee venom can be fractionated at a rate of 89% by CPC. Below are the device conditions and output.

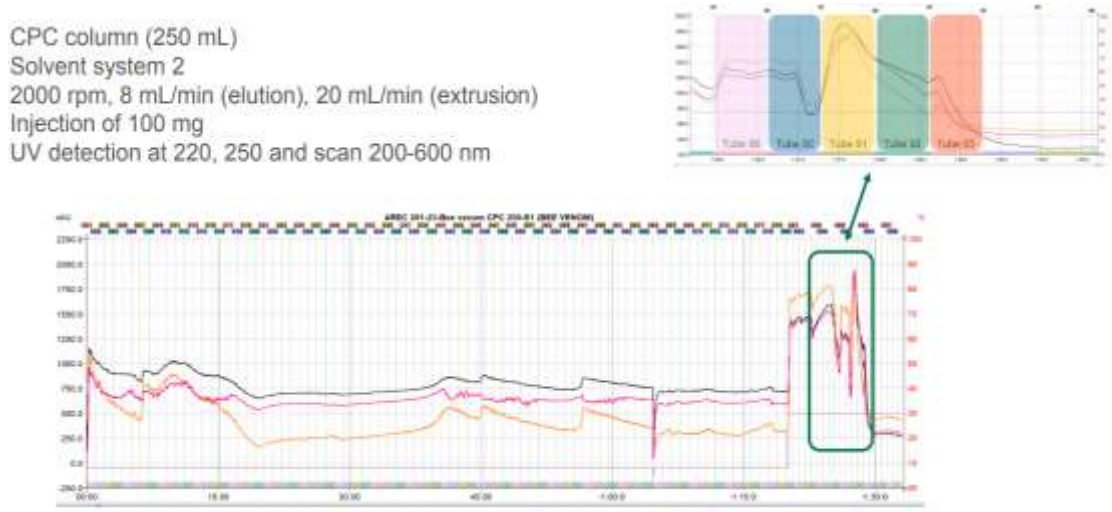

Figure 4. CPC chromatography of bee venom

Powdering of suitable fractions with the help of a lyophilizer and carrying out vialization processes

Solvents that may have been present in the components that met the percentage purity criteria and were separated into fractions were removed with the help of a lyophilizer and the vialization process was carried out.

In conclusion; Within the scope of the study, raw poison was processed; Two commercial prototypes have been developed, purified bee venom (PBV) and melittin purified from bee venom (MPBV), with all production steps standardized.

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OP36- A New Approach to Immobilization Porcine Pancreatic Lipase Using Copper-Based MOFs: Mechanochemical Encapsulation

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Abstract

Metal-organic frames (MOFs) are crystalline porous materials made up of metal ions/metal clusters and coordination-bridged multivalent organic ligands[1,2]. Cu-MOF, a copper-based MOF, has excellent physicochemical properties and its synergistic effect is highly significant in creating a high-performance catalyst[3,4].

Porcine pancreatic lipase (PPL) is cheaper, easily accessible, and most widely used in reactions such as biotransformation compared to other commercial microbial and animal lipases[5]. Although lipases are widely used enzymes in industry, the use of techniques such as immobilization to improve their stability is gaining importance[6]. In immobilization technique, it is also important to select the appropriate support material and method. MOFs are thought as ideal support materials for enzyme immobilization due to their unique properties[2].

In this study, Cu-MOF, a copper-based MOF synthesized by mechanochemical method, was used for the immobilization of *Porcine Pancreatic* lipase (PPL). The prepared Cu-MOF and immobilized lipase (Cu-MOF@PPL) were characterized by FT-IR, SEM and XRD. Cu-MOF@PPL's pH, thermal, and storage stabilities were examined and contrasted with those of free lipase (PPL). The enantioselective hydrolysis of R/S naproxen methyl ester was used to determine the catalytic ability of Cu-MOF@PPL.

The maximal activity of Cu-MOF@PPL was at pH 6, whereas the maximum activity of free lipase (PPL) was at pH 5. When incubated at 60 °C for 2 h to examine their thermal stability, it was found that free lipase (PPL) retained 25% and Cu-MOF@PPL 46% of its activity. When the storage stability of Cu-MOF@PPL for 10 days was examined, it was determined that 74% of the Cu-MOF@PPL still retained its activity. Furthermore, Cu-MOF@PPL obtained by mechanochemical method was found to have higher stereoselectivity and conversion than free lipase.

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Keywords: *Cu-MOF, enzyme, immobilization, lipase, PPL.*

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OP37- Gold Nanoparticles for the Sensitive Detection of Biomarkers using Surface Plasmon Resonance Immunosensors

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Researchers are consistently investigating innovative technological approaches to create immunosensors that are ultra-sensitive, selective, rapid, affordable, and precise. Surface plasmon resonance (SPR) immunosensors stand out as the most prevalent optical immunosensors, primarily selected for their unique ability to achieve high sensitivity and perform real-time monitoring of biomolecule binding events. However, the sensitivity is not always sufficient for the detection of extremely low quantities of the targeted molecules. Nanotechnology and nanobiotechnology have a high impact in various fields of research, particularly in the development of immunosensors. An efficient approach to increase the sensitivity of SPR immunosensors can be achieved by employing functionalized gold nanoparticles or magnetic core/gold shell nanoparticles for the preconcentration of biomolecules or for the amplification of the analytical signal using these nanoparticles as labels[1-3].

In this research magnetic core/gold shell nanoparticles were synthesized and modified with monoclonal antibodies. The challenges in the selection of the best method for antibody immobilization and optimization of antibody surface concentration will be discussed. Furthermore, a signal amplification strategy using magnetic core/gold shell nanoparticle-based SPR immunosensor and sandwich immunoassay format for ultrasensitive (femtomolar) quantification of the biomarker will be presented. The obtained results indicate that the proposed signal amplification strategy offers promising potential to significantly increase the sensitivity of SPR immunosensor.

Keywords: nanoparticles; antibody immobilization; optical immunosensors; signal amplification strategy.

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This research was funded by a grant (No. S-MIP-22-46) from the Research Council of Lithuania.

OP38- Cathode Design with MWCNT-Mn3O4-PtNps Modified Pencil Graphite Electrode for Enzyme-Nanozyme Based Biofuel Cell

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Enzymatic biofuel cells (EBFCs) using enzymes as biocatalysts are highly efficient devices that convert the chemical energy stored in fuel directly into electricity and have been widely studied due to their many benefits[1]. In this process, fuel is oxidized at the anode by enzymatic reactions while oxygen is typically reduced at the cathode[2]. In this study, we presented an EBFC that contains the glucose oxidase (GOx) based composite anode and multiwalled carbon nanotube (MWCNT), manganite (Mn_3O_4) and metal nanoparticles (MNps) composite-based cathode. The anode was modified with MWCNT and ferrocene (Fc) as a conductive layer and the enzyme GOx as a sensitive detection layer for glucose. On the other hand, the cathode consisted of MWCNT-Mn_3O_4-PtNps composite modified pencil graphite electrode (PGE) for oxygen reduction reaction because of their good conductivity and electron transfer rate. The best results were obtained with the anode containing MWCNT (5.0 mg/mL), Fc (25 mM), and GOx (20 mg/mL) modified glassy carbon electrode and for the cathode containing MWCNT (1.0 mg/mL), Mn_3O_4 (1.0 mg/mL), and PtNps (5.0 mM) modified PGE. The performance of the EBFC was investigated using a potentiostat/galvanostat. It was found that the EBFCs produced an open circuit voltage of 0.305 V and a maximum power density of 42.05 μW cm^2. The proposed EBFC is a highly promising candidate for detecting glucose while simultaneously harvesting power from various glucose samples.

Keywords: Enzymatic biofuel cell, glucose detection, glucose oxidase.

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This study supported the Scientific Research Projects Coordination Unit at Canakkale Onsekiz Mart University. Project ID: FDK-2022-4073

OP39- Spectroscopic Analysis of Curcumin Samples' Antioxidant Capacities

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Antioxidants are essential for the human bodies, they help neutralize free radicals and protect cells from oxidative stress. Curcumin, polyphenol found in turmeric, has been shown to exhibit strong antioxidant activity due to the presence of functional groups. In this study, the antioxidant capacity of curcumin was examined using three methods: Total phenolic content, CUPRAC and DPPH test.

In the study, different types of curcumin samples, including two pharmacy preparations, curcumin powder from local bazaar in Uzbekistan and 95% pure curcumin from Zade Global are used. The antioxidant capacity of curcumin was determined in water, ethanol, artificial saliva, artificial gastric fluid and artificial intestinal fluid.

Total phenolics determined by using the Folin-Ciocalteau reagent. The total phenolic content test measures the total phenolic compounds in a sample and is based on the measurement of absorbance according to the color change during the reaction. The results of total phenolic content test showed that the highest phenolic antioxidant content was observed when curcumin powder from Uzbekistan was dissolved in artificial intestinal fluid. In contrast, the lowest phenolic antioxidant content was found when one of pharmacy preparation was dissolved in distilled water.

CUPRAC (Cupric Reducing Antioxidant Capacity) method is based on the reduction of Cu (+2) to Cu (+1) by antioxidants. This method has advantages over other methods, such as fast response to certain antioxidants, good reproducibility and workable at physiological pH. (1) For the CUPRAC method is based on the absorbance measurement of Cu-Neocuproine, the highest absorbance was observed when one of pharmacy preparation was dissolved in artificial intestinal fluid. However, since precipitation occurred in the environment where curcumin extracts were present during the experiment, no data could be obtained with the DPPH test. Thus, according to the results, it was determined that the most suitable body fluid (solvent medium) for the determination of curcumin was artificial intestinal fluid. This study was also supported by electrochemical studies to determine the active antioxidative activity of curcumin.

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OP40- Determination of Palladium at Trace Levels by Using CoS@ppy@Fe3O4 Nanocomposite Assisted Dispersive Solid Phase Extraction Procedure

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Palladium is a precious silver-white metal that is frequently used in industry such as dentistry, cars catalytic converters, electronics, and in jewelry to make white gold[1]. Palladium is not as toxic as other heavy metals. Despite the fact that palladium may have some toxicity for human health, especially intravenous injection of palladium chloride solution resulted in rapid following death[2]. In this study, the CoS@PPy@Fe_3O_4 nanocomposite-assisted preconcetration method was used for the determination of palladium in environmental samples. A new and innovative method was used for the synthesis of CoS@PPy@Fe_3O_4 nanocomposite. Environmental samples were preconcentrated before the determination of palladium by FAAS (flame atomic absorption spectrometry). Parameters affecting the extraction and desorption efficiency were optimized with a univariate approach. The analytical performance of the system was determined by using the optimized conditions as a result of the optimization experiments. With the method presented in this study, a new method with higher extraction efficiency and less chemical usage has been developed.

Keywords: Dispersive Solid Phase Extraction, CoS@PPy@Fe_3O_4, FAAS, Magnetic-nanocomposite

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OP41- Determination of nickel at trace levels in Saint John's wort tea by using a solidification of floating organic droplet-based liquid-liquid microextraction method followed by matrix matching assisted slotted quartz tube-flame atomic absorption spectroscopy

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Exposure to high levels of nickel by water and food intake has been known to cause potentially fatal health problems including the lung fibrosis, cancer, contact dermatitis, kidney failure, and cardiovascular disease at high levels[1]. Herbal products with widespread medicinal use are potential carriers for the excessive nickel in the surface-level soil into the human systems because plants are known to readily absorb Ni ions from the soil. While being all-natural and unprocessed products, they have been misleadingly labeled as safe by people without further thought[2]. This study puts forward an easily available, eco-friendly analytical method with high sensitivity for the determination of nickel in St. John's wort tea samples. The extraction and enrichment of Ni were established by first forming a Schiff base complex with bis-N,N′-(salicylidene)-1,3-propanediamine, then using 1-decanol as the extraction solvent, which was solidified in a freezer and easily removed from the medium for detection by using a FAAS (flame atomic absorption spectrometer) equipped with a SQT (slotted quartz tube). Methanol was utilized as a dispersive solvent in the process to increase the interaction of 1-decanol droplets with the analyte complex. A univariate optimization strategy was employed to investigate the effects of different experimental parameters on complex formation and enrichment steps. The resulting method demonstrated a 26.5-fold enhancement in detection power compared to direct measurements by flame atomic absorption spectrometer. The limits of detection and quantification were 24 and 79 µg/kg, respectively, within a dynamic linear working range between 50 and 500 µg/kg. This proposed method was applied to two St. John's wort tea samples acquired from different local suppliers, and spiked recovery experiments were conducted, yielding results in the range of 89.3–121.4% by adopting matrix matching calibration for further elimination of the matrix interferences. The results indicate that the developed method was appliable and accurate while offering a user-friendly, quick, and sensitive means to determine Ni in herbal tea samples, utilizing an easy-to-prepare ligand and highly available laboratory equipment.

Keywords: Solidification of floating organic droplet, Nickel, St. John's wort, Flame atomic absorption spectrometry, Slotted quartz tube

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OP42- Simple and efficient adsorption of nickel from paper industry wastewater samples by utilizing magnesium ferrite nanoparticles synthesized by the sol-gel process

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Nickel (Ni) is rightfully considered among many environmental pollutants with crucial and undesired impacts all around the ecosystem. Although Ni naturally occurs in the environment, increased industrial activities have been causing the flow of alarming amounts into natural resources. Nickel can elusively get into the body via inhalation, ingestion, or absorption through the skin to cause various adverse health effects, including but not limited to allergies, lung fibrosis, and respiratory tract cancers[1]. Magnetic ferrite nanoparticles have attracted interest for their potential use as effective removal agents for heavy metal pollutants from water since they possess the required high surface area and chemical inertness, in addition to their ease of collection thanks to their ferromagnetism and simple synthetic modification possibilities[2]. In this study, the adsorption of Ni^{2+} ions from aqueous media was achieved by utilizing magnesium ferrite ($MgFe_2O_4$) nanoparticles as dispersive solid-phase adsorbents. $MgFe_2O_4$ particles were synthesized by using a conventional sol-gel technique at mild temperatures, followed by auto-combustion of the gelatinous adduct and annealing of the product at 500 °C. The characterization of the material was carried out by SEM (scanning electron microscopy). and XRD (X-ray diffraction). Consequently, the experimental parameters that majorly affect the removal efficiency, such as the pH buffer type/volume, the amount of nanosorbent, and the duration of mixing, were evaluated by following a univariate optimization strategy. The direct reusability of the nanoparticles after recycling was also investigated in the process.

Keywords: Nickel, Magnesium ferrite nanoparticles, Adsorption, Industrial wastewater

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OP43- Plant of Hypericum Lydium Boiss. in Human Health Role as Antioxidant

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Plants of the genus Hypericum are used in traditional medicine for the treatment of many diseases. In traditional medicine, it is used to treat menstrual problems, stomach aches, gastric ulcers, hemorrhoids, colitis, and wounds. The wide range of uses of Hypericum genus in folk medicine and more recent studies suggest that it is a medicinal plant with a great deal of potential.[1,2] The present study aimed to investigate the total phenolic content, total flavonoid content, total antioxidant capacity, free radical scavenging activity, and hydrogen peroxidase scavenging activity of methanol extract of Hypericum lydium Boiss (HLB). The methanol extract of HLB was found to have total phenolic content of 62.27±1.63 mg gallic acid/g extract, total flavonoid content of 3.15±0.24 µg quercetin/g extract, and total antioxidant capacity of 24,90±2,29 mg trolox/g extract. Furthermore, it was also found that the free radical scavenging activity of 200 µg/ml of the HLB extract (48.30±1.77%) was higher than that of BHT (32,23±0,024 %) calculated by the galvinoxyl method. The hydrogen peroxide radical scavenging activities were similar for extract (5,95±0,14 %) and BHT (5,56±0,86 %). Consequently, the data obtained show that HLB methanolic extracts have promising antioxidant capacity. Hence can be regarded as antioxidants and raw materials in the food, cosmetics, and pharmaceutical industries.

Keywords: Hypericum Lydium Boiss., antioxidant, CUPRAC, galvinoxyl

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OP44- Determination of antiradar and thermal conductivities of Reduced graphene oxide/epoxy nanocomposites

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In this study, the nanocomposites of the rGO/cured epoxy were studied by ultrasonication and without ultrasonication. The dielectric and thermal conductivities of the rGO/cured epoxy composites were investigated based on the filler load and frequency. It was found that the applied frequency and the filler concentrations affected the dielectric properties of the rGO/cured epoxy composites. The rGO was obtained from Doruk Grafen (Anbiokim Doruk, Turkey) and characterized by X-ray diffraction analysis, Raman spectroscopy and High resolution Transmission Electron Microscopy (HRTEM). In order to investigate the intrinsic reasons for the electromagnetic interference (EMI) shielding effectiveness of the composites, the dielectric properties of the cured epoxy and the rGO/cured epoxy composites at different percentages of rGO loading at the frequency range of 1–4 KHz were measured. The results showed that the introduction of rGO particles to the composites increased their dielectric properties and thermal conductivities smoothly.

Keywords: Reduced graphene oxide, epoxy, nanocomposites, antiradar.

Introduction

Efforts to reduce pollution of an electromagnetic nature have been attracted the use of Electromagnetic Interference Shielding (EMI) and Microwave-Absorbing materials (1). Signals are attenuated by such materials through absorption and/or reflection of the radiation power. On the other hand, modern electronic devices with increasing power densities require ultrahigh heat dissipation materials to dissipate the temperature of thermosensitive components. Carbon allotropes and their derivatives including graphene, reduced graphene oxide (rGO), carbon nanotubes (CNT) have highly potential to use for this purpose due to their properties such as high thermal conductivity, flexibility, as well as high conducticity, lightweight and excellent corrosion resistance (1-2). Among graphene derivatives, the rGO shows an improvement in microwave absorption when compared to carbon nanotubes and graphite, is expected to present superior absorption to high-quality graphene, and exhibits promise as microwave absorbing material.
This aim of this study is to investigate the dielectrical and thermoelectrical properties of rGO/epoxy cured-nanocomposites that obtained by a facile and low-cost method.

Materials and method

The rGO was obtained from Doruk Grafen (Anbiokim Doruk, Turkey) and characterized by X-ray diffraction analysis, Raman spectroscopy, and High-resolution-Transmission Electron Microscopy (HRTEM). To obtain the nanocomposite, the rGO was dispersed in ethanol using ultrasonic bath for 10 min. and then added to epoxy monomer. After shaking wery well, the sample was put in vacuum oven to evaporate ethanol. Afterthat, the hardner was added to this mixture and shaked again very well. The mixture was transfered to the container special for this purpose and dried in room for a few days.
The dielectrical properties were measured by using a QuadTech 7600 precision LRC meter impedance analyzer range from 50 Hz to 2 MHz. To determine thermal conductivity, TLS-100 device was used.

Results and Discussion

Electromagnetic interference (EMI) shielding is defined as the attenuation of electromagnetic radiation by reflection and/or absorption of the incident power. The obtained results related to the dielectrical constant were given in Figures 1-2.

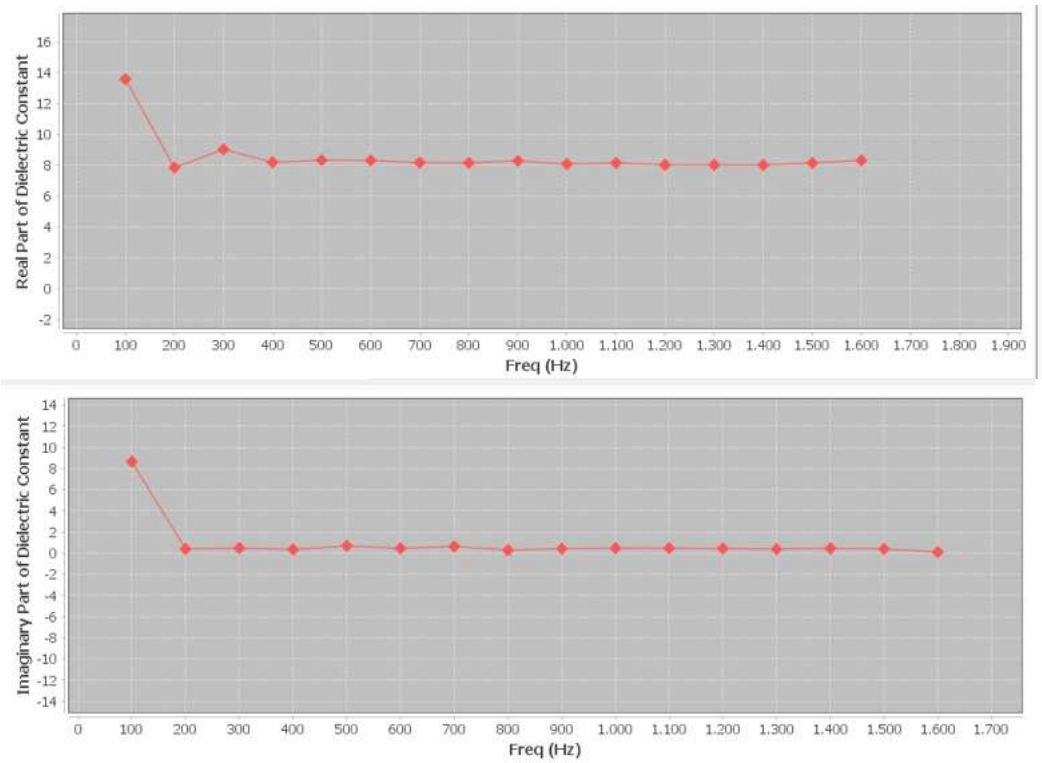

Figure 1. Dielectrical constant of neat epoxy cured.

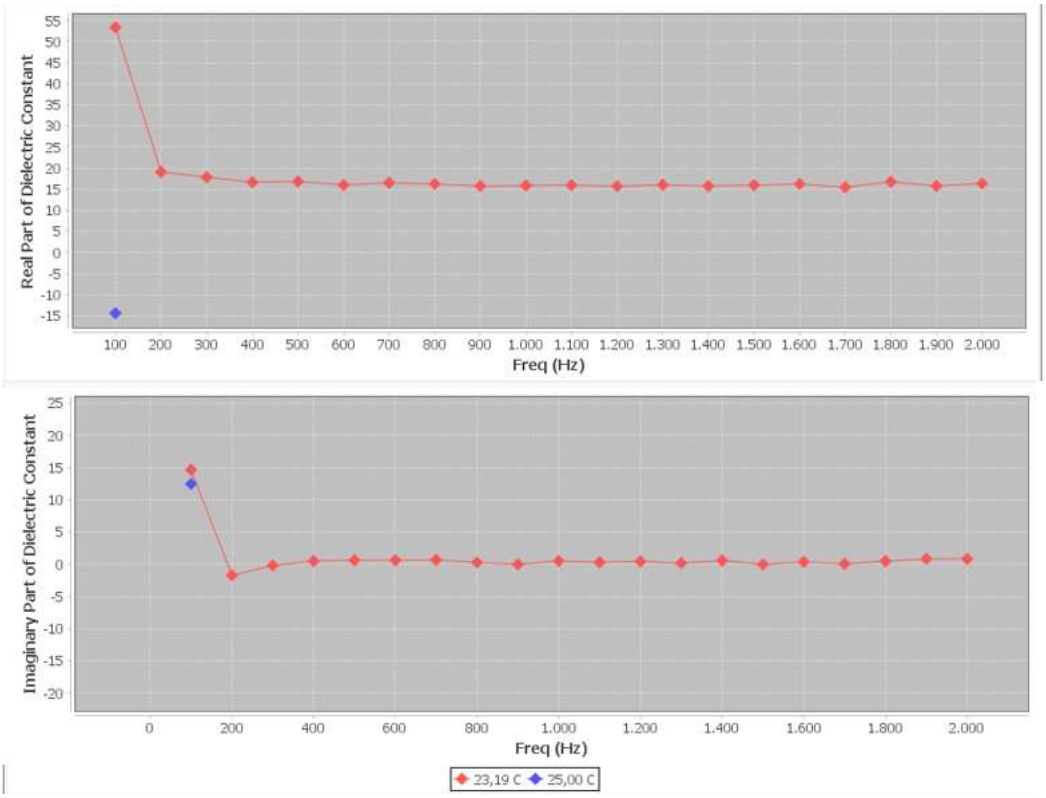

Figure 2. Dielectrical constant of nanocomposite of rGO/epoxy cured.

The rGO-epoxy nanocomposite (rGO 3%) presented a 87% higher dielectrical constant than neat epoxy.
The thermal conductivities tend to increase with increasing rGO content at room temperature. The maximum thermal conductivity recorded was 0.210 W/mK for the 3 wt% rGO/epoxy composite. The corresponding thermal conductivity of the neat epoxy polymer is 0.170 W/mK

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POSTER PRESENTATIONS (PP)

PP1- Synthesis of MXenes Nanostructures for the Application in the Design of Optical Sensors

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MXenes is a recently (2011) discovered 2D nanomaterial with unique optical and electrical properties[1]. It is known that MXenes have a complex surface chemistry, which is suitable for the design of optical sensors[2]. In this study, the $Ti_3C_2T_x$ MXenes were synthesized from its precursor Ti_3AlC_2 by selective etching in acidic solutions. Multi-layered and single-layered MXenes were synthesized to test the morphology and surface chemistry effect on the nanostructures' properties. Multi-layered MXenes were used for the design of SERS-based sensors for the detection of drug metabolites. Specifically, this type of sensor shows promising results for detecting salicylic acid, a metabolite of acetylsalicylic acid, also called Aspirin. The single-layered nanostructures were applied in colourimetric sensors for the detection of metal ions. During the experiments, it was determined that MXenes could be applicable in the design of optical sensors for the detection of analytes in low concentrations.

Keywords: 2D materials; MXenes; Optical sensors

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This project has received funding from the Research Council of Lithuania (LMTLT), agreement No S- PD- 22- 155.

PP2- Development of Analytical Methodology for Determination of free N=C=O Assay with FT-IR MULTI ATR

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Free N=C=O is well known as can react with moisture rapidly, so analysis cycle time and conditions are very important for determination to assay of free N=C=O. General method refers to wet chemistry but sometimes wet chemistry is not fast for the analysis of free N=C=O content.

In this study, assay of free N=C=O is determined by FT-IR MULTI ATR. This method is supported with linerity. Calibration curve include many point and sufficient r^2 value.

As a results, studied analytical data has been examined. For determination of free N=C=O, there are specific regions on FT-IR Spectrum for selecting and this selected region give more accurate results. Examined results are positive.

As a conclusion study showed that in some cases, FT-IR with MULTI ATR is more usefull than wet chemistry for quick response and efficiency,

Keywords: free N=C=O, FT-IR, MULTI ATR

PP3- Simultaneous voltammetric determination of selected fungicides on a boron-doped diamond electrode

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Fungicides are chemical compounds that penetrate plant tissues and spread throughout the plant, preventing the development of fungal pathogens[1]. The representative fungicides are bixafen, fluopyram, and prothioconazole which are the components of the ASCRA Xpro formulation. Bixafen[2] and fluopyram[3] are succinate dehydrogenase inhibitors, while prothioconazole[4] is a sterol biosynthesis (demethylation) inhibitor. AscraXpro delivers broad-spectrum disease control, including more curative activity against Septoria than any other fungicide formulations and the highest yields.

The main objective of this work was to evaluate the possibility of the simultaneous voltammetric determination of three fungicides, *i.e.*, bixafen, prothioconazole, and fluopyram on a boron-doped diamond electrode (BDDE). Detailed studies of the electrochemical activity of each fungicide were carried out using cyclic voltammetry in a supporting electrolyte (Britton-Robinson buffer (BRB)) within a wide pH range of 2.0–12.0. It was found that prothioconazole and bixafen are being oxidized on the BDDE in a wide pH range from 2.0 to 12.0, while fluopyram has no electrochemical activity on the BDDE. In the next step, the effect of the pH of the BRB on the separation of the square-wave voltammetric oxidation signals of prothioconazole and bixafen was investigated. As the optimal medium for the simultaneous determination of both pesticides, the BRB of pH 3.0 was selected. It was found that the simultaneous voltammetric determination of prothioconazole and bixafen on the BDDE was possible in the range of 5.0–80.0 μmol L^{-1} (LOD = 1.53 μmol L^{-1} for prothioconazole and LOD = 1.54 μmol L^{-1} for bixafen).

Keywords: boron-doped diamond electrode, square-wave voltammetry, bixafen, fluopyram, prothioconazole

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PP4- Authenticity Control of Free-Range and Market Chicken Samples by Stable Carbon Isotope Ratio Analysis

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Chicken is one of the most consumed meat products all over the world due to its high protein content[1]. According to the related regulations of EU, during the sale of poultry, the chickens must be fed with nutrients containing maize during the majority of their feeding period, in order to enable the inclusion of the phrase Fed with Maize. Therefore, development of a reliable method to verify this claim is important. The isotope ratio mass spectrometry is a primary method for the authencity studies more than two decades.

In the present study, it is aimed to compare naturally raised chickens with commercial chicken brand products studied by measuring the $\delta^{13}C$ values. The chicken samples were analyzed by the EA-IRMS method and the results were evaluated statistically. It can be concluded that mean values calculated for organically grown chicken samples (-23.98±0.40) was clearly less than those obtained for market chicken samples (-21.10±0.61). These results are in agreement with a previous study[2].

Keywords: Food fraud, chicken, isotope ratio

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Acknowledgment: This study was supported by Ege University Scientific Research Council for the financial support (project no 22647).

PP5- Stripping voltammetry using bismuth film electrodes as a responsive tool in the chemical analysis of selected metal ions in environmental water samples

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One of the more sensitive methods used in analytical chemistry is stripping voltammetry. Measurements can be carried out by anodic stripping voltammetry (ASV), adsorption stripping voltammetry (AdSV) or cathodic stripping voltammetry (CSV), depending on how the analyte to be determined is accumulated and the subsequent recording of the analytical signal. As the key reaction takes place at the working electrode, it is very important to select it appropriately, taking into account the type of analyte being determined. For many years, mercury electrodes have been the main choice for voltammetric analysis of metal ions. Recently, safety and environmental protection considerations have limited their use and encouraged the search for alternative materials that are more environmentally friendly. These include bismuth electrodes, which have been used in our research group to develop of analytical procedures to determine trace amounts of metal ions. The analytical procedures we developed used bismuth film electrodes formed in situ on a glassy carbon electrode. For this purpose, bismuth ions were added directly to the sample to be analysed and the bismuth film formation step occurred simultaneously with the accumulation of the labelled ions, either by their reduction to metallic form (ASV procedures) or by adsorption of their complexes with cupferron (AdSV procedures). The following detection limits were obtained using bismuth film electrodes for selected ions: 6.2×10^{-10} M for Cd(II), 1×10^{-10} M for Ga(III), 1.6×10^{-9} M for In(III) and 2.5×10^{-10} M for V(V). All developed procedures have been successfully used for the determination in aqueous environmental samples such as river water, lake water or waste water [1,2].

Keywords: stripping voltammetry, bismuth film electrodes, metal ions, environmental water samples

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PP6- Development of Electrochemical Biosensor for Detection of Kidney Injury Molecule (KIM-1)

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KIM-1, a biomarker also known as Kidney Injury Molecule-1, plays a crucial role in the early detection and monitoring of kidney damage and certain kidney diseases[1]. A sensitive and rapid method is essential for the detection of the KIM-1 protein. The developed biosensor system detects KIM-1 protein by electrochemical methods using ITO-PET (indium tin oxide - polyethylene terephthalate) electrode. The designed biosensor holds the advantage of being highly cost-effective, disposable, and user-friendly. Optimization studies were carried out in which numerous critical parameters, including biosensor, CTES (carboxyethylsilanetriol) concentration, EDC/NHS (1-Ethyl-3-(3-dimethylaminopropyl)carbodiimide / *N*-hydroxysuccinimide) concentration, antibody concentration, and incubation times for EDC/NHS, antibody, and antigen, were comprehensively examined. Following the completion of the optimization steps, comprehensive characterization studies were conducted to assess the biosensor's performance. Repeatability, reproducibility, regeneration, and storage life studies were performed to evaluate the durability and suitability of the proposed biosensor. Electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV) techniques were employed in conducting experimental investigations. The proposed immunosensor exhibited a wide linear detection range spanning from 0.01 ng/mL to 2.5 ng/mL.

Keywords
KIM-1, CTES, Immunosensor, ITO-PET electrode, EDC/NHS,

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PP7- An HPLC Method Development for Guanfacine HCl

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Guanfacine (GNF) is a derivative of guanidine, a nucleic acid, has the chemical name N-amidino-2-(2,6-dichlorophenyl)-acetamide. GNF has been used to treat high blood pressure since 1975. Pharmacologically, hydrochloride salt form of guanfacine (GNF.HCl) (Fig.1) affects the brain and acts as an agonist on α2-adrenergic receptors in the stalk and prefrontal cortex. This drug causes a decrease in the sympathetic release of neurotransmitters which is directly related to hypotensive effects. This relationship between α2-adrenergic receptors may contribute to guanfacine attention deficit hyperactivity disorder. It makes guanfacine it effective in the treatment of (ADHD).

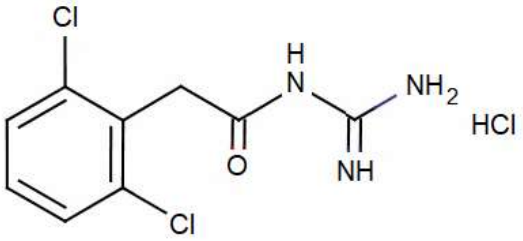

Fig 1. Chemical structure of Guanfacine hydrochloride

For more than half a century, ADHD has been successfully treated with stimulant drugs such as methylphenidate and amphetamine. However, not all patients respond to such drugs, which have a stimulating and addictive effect on the central nervous system and cannot tolerate the drug due to some side effects. In some cases, it may not be preferred by families because it is sold with a controlled prescription. For this reason, non-stimulant alternatives have been investigated in the treatment of ADHD. Guanfacine was developed as one of these drugs and has been approved by the United States Food and Drug Administration (FDA).

In this study, it was aimed to develop a simple, precise, and fast analytical LC procedure that will serve as a quantification analysis method for Guanfacine hydrochloride and its impurities. While developing the method, elution buffer and type of organic phase, the pH of the solution, the use of ion pair reagent have been optimized. In addition, instrumental parameters such as flow rate of the mobile phase and the wavelength used for measurement were taken into consideration. Here, the results of preliminary studies were carried out for this purpose have been represented.

Keywords: Guanfacine hydrochloride, impurity, chromatography, HPLC-PDA

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Acknowledgment: This study was supported by Ege University Scientific Research Council (Project no 31898) for financial support.

PP8- An Amperometric Biosensor for Detection of Paraoxon Ethyl based on Inhibition of Acetylcholinesterase Enzyme Using SPCE Modified with Cupric Neocuproine Complex

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Paraoxon ethyl (POE) is an organophosphorus pesticide, commonly used substance in biochemical and toxicological studies. An amperometric biosensor was developed using CUPRAC-reagent ($[Cu(Nc)_2]^{2+}$ complex[1]) modified Screen Printed Carbon Electrode (SPCE) for the sensitive and selective detection of POE. The detection procedure is based on the inhibitory effect of pesticide on acetylcholine esterase (AChE) enzyme. CUPRAC-reagent was used as a redox mediator for the electrocatalytic oxidation of thiocholine (TCh), which is produced through the enzymatic reaction of Acetylthiocholine (substrate) with AChE [2].
The modified electrode was prepared via three steps. In the first step, 6 µL of a suspension prepared by mixing Nafion-multiwalled carbon nanotube (%0.5 Nf – 2 mg MWCNT in 2 mL DMF) and Chitosan (%0.5 in dissolved 0.1 M CH_3COOH) in 1:1 volume ratio was dropped on the SPCE and dried. In the second step, 20 µL of $[Cu(Nc)_2]^{2+}$ complex solution, prepared by mixing 0.40 mM $CuCl_2$ and 0.80 mM neocuproine (Nc) was dropped on this electrode. After drying, 5.0 µL of 1 mg/mL AChE was immobilized onto the electrode using the crosslinking function of chitosan in the final step. Then the cyclic voltammograms of AChE/$[Cu(Nc)_2]^{2+/}$Nf-MWCNT/SPCE were recorded at pH 8.0 Britton Robbinson Buffer solution in the absence and the presence of Acetylthiocholinechloride (as a substrate, ATChCl) in the concentration range from 1 to 10 mM. The results show that enzymatically produced TCh is oxidized at about +400 mV vs. Ag/AgCl and peak current increased by increasing of ATChCl concentration. However, the enzymatically produced TCh was oxidized at AChE/Nf-MWCNT/SPCE around +500 mV with wider and lower peak than that at the CUPRAC-reagent-modified electrode. These results showed that the CUPRAC-reagent exhibits good electrocatalytic activity toward the oxidation of TCh. When POE was added to a supporting electrolyte including 10 mM ATChCl, the peak currents obtained from CVs of TCh at AChE/$[Cu(Nc)_2]^{2+}$/Nf-MWCNT/SPCE were decreased proportionally with the concentration of POE due to its inhibitory effect on AChE. Thus, using this approach, an electrochemical pesticide biosensor based on the use of the CUPRAC-reagent was designed for the first time. The fabricated electrochemical biosensor exhibits promising potential for the accurate and rapid detection of POE, suggesting applications in environmental monitoring and chemical safety assessments.

Acknowledgements: *This study was conducted with the financial support of the Scientific and Technological Research Council of Turkey (TUBITAK). The authors are grateful for this support. (Project number 120Z963)*

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PP9- The influence of water-organic electrolyte on the kinetics and mechanism of electroreduction of Bi(III) ions in the presence of cationic surfactant; “cap-pair” effect

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Due to its low toxicity and unique properties, bismuth finds broad applications in various industrial sectors, often replacing harmful lead. Consequently, there is a need to develop new analytical techniques enabling precise detection and determination of this element in samples, such as biological or environmental ones[1]. In the context of the electroreduction of Bi(III) ions, there are significant aspects influencing the kinetics and mechanism of this process. One of the factors is the presence of organic substances, such as the cationic surfactant CTAB (cetyltrimethylammonium bromide) used in the study, which accelerates the electrode process in accordance with the "cap-pair" rule[2].

As literature suggests, research on accelerating electrode processes in the context of the "cap-pair" rule has mainly focused on aqueous solutions, with limited references to the use of water-organic solvent mixtures. This opens up a significant field for research, allowing a better understanding of the impact of these electrolytes on the kinetics and mechanism of metal ion electroreduction. In the conducted experiments, a methanol-chlorate(VII) solution in appropriate volume ratios was used as the base electrolyte. The concentration of bismuth ions in the studied solutions was consistently $1 \cdot 10^{-3}$ mol/dm^3. The choice of methanol is crucial as it serves as a protic polar solvent, influencing the adsorption equilibrium of organic substances and depolarizer on the electrode surface[3].

The obtained results indicate a change in the catalytic action dynamics of CTAB on the electrode process, in relation to the change in the volume ratio of methanol to perchlorates(VII) in the base electrolyte solutions. The choice of the appropriate solvent and control of electrode processes are crucial in understanding the mechanism of Bi(III) ion electroreduction and the potential applications of this knowledge in practice.

Keywords: Bi(III) electroreduction, “cap – pair” effect, CTAB, kinetic parameters

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PP10- All solid state nitrate ion-selective electrodes based on different electrode substrate -a comparative study

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Ion-selective electrodes (ISEs) without inner electrolite solution are becoming more and more popular due to their many advantages, such as simple construction, low production cost and small size, as well as the ability to work in any position. Performances of such ISEs depend not only on the properties of ion sensitive membrane but also on the type of internal electrode and intermediate layer between this electrode and the membrane[1].
In this paper properties of nitrate ion-selective electrodes depending on kind of substrate electrode were studied. Three kinds of inner electrode, glassy carbon disc, gold disc and gold microelectrode array, were used for sensor construction. The simple coated disc electrodes as well as electrodes with intermediate layer composed with multiwalled carbon were prepared and studied. Effect of inner electrode and its modification was evaluated on the basis on potentiometric, chronopotentiometric and electrochemical impedance spectroscopy measurements.
Depending on the type of internal electrode, ISEs differed in stability and potential reversibility. The best results were obtained for electrodes obtained on the basis of gold microelectrode array. For these electrodes (even without modification with carbon nanotubes) good measurement results and reproducible course of calibration curves were obtained, regardless of the direction of concentration changes or solution mixing.
The obtained results confirm that the applied new electrode substrate in the form of a gold microelectrode array is a promising substrate in the construction of all solid state ISEs.

Keywords: all solid state ion-selective electrode; electrode substrate; gold microelectrode array (between 3 to 5)

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PP11- Study of the removal of rhodamine B by a carbonaceous adsorbents

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Water pollution has become a global problem as it has harmful effects on ecosystems and thus contributes to climate change.Activated carbons are among the most effective and promising carbon-based adsorbents used for wastewater treatment [1,2]. The adsorbents used in adsorption processes should be environmentally friendly, efficient and derived from as few reagents as possible. The study presents the synthesis of adsorbents from low-rank coals from the collieries Labin and Spitsbergen. Activated carbons were obtained through the chemical activation of low-rank coals using sodium hydroxide. The prepared activated carbons were studied using various techniques to determine the surface morphology, structure, elemental composition, porosity and acid-base properties of all the activated carbons prepared. The kinetics and adsorption mechanism of rhodamine B from an aqueous solution were investigated. The effect of reaction parameters such as temperature and pH changes, reaction time, dye concentration, adsorbent mass and sample shaking rate on the dye removal efficiency of the obtained adsorbents was evaluated. The results showed that the obtained adsorbents exhibited a mesoporous texture and the acid-basic character of the synthesised adsorbents depended on the starting material. The pseudo-second-order kinetic model provided the best fit to the experimental results, indicating that the adsorption process followed a chemical process. On the other hand, the Langmuir adsorption isotherm showed the best fit, indicating monolayer adsorption. The Langmuir adsorption isotherm showed a high maximum adsorption capacity, which was 155 mg/g and 73 mg/g, respectively. Negative Gibbs free energy values indicated that the removal of rhodamine B could be thermodynamically favourable due to the spontaneous nature of the adsorption. In the future, the stability of the studied adsorbents and the possibility of their reuse in real wastewater should be tested.

Keywords: activated carbon, chemical activation, rhodamine B, adsorption, desorption

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PP12- Specific determination of non-metabolized selenium in Se-enriched yeast feed supplements

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Yeast fermentation, converting selenite or selenate into selenomethionine (SeMet), is the basis of biotechnology, producing an attractive supplementary nutritional source of selenium. The total conversion of Se into organic forms is considered an important parameter of the quality of the supplement. However, the analysie of the marketed Se-rich yeast do not account for more than 90% of selenium, leaving doubt regarding the speciation of the remaining 10%.
Several sequential extraction procedures targeting different classes of selenium species have been developed. The precise determination of the total Se in the fractions and its quantitative speciation analysis have been carried out by ICP MS and HPLC-ICP MS, respectively. Dedicated derivatization procedures using iodoacetamide were developed to target the inorganic forms of selenium (selenate, selenite, and Se^0).
Several commercial selenized-yeast products, as well as the standard reference material SELM-1 (NRCC), were analyzed. The analysis of the non-metabolized selenite/selenate by the method of standard additions turned out to be impossible. The added spikes bound strongly and randomly to the different constituents of yeast and could not be recovered by water extraction, as demonstrated by experiments with enriched ^{78}Se. Inorganic forms of selenium were found to react quantitatively with iodoacetamide, producing a derivative that could be readily quantified by HPLC-ICPMS.
Consequently, all the selenium species present could be accounted for, as demonstrated by the sum of the concentrations of the individual species matching the total content of selenium. In all the samples tested, the inorganic selenium content accounted for more than several percent.
The study demonstrated that the process of yeast enrichment in selenium leaves considerable amounts of unmetabolized inorganic selenium largely exceeding the 2% threshold fixed by the regulatory agencies. This selenium is potentially comprised of residual inorganic salts used as an enrichment source and Se^0 nanoparticles."

Keywords: selenium supplementation, selenium speciation, selenized yeast, ICP MS, HPLC-ICP MS

PP13- Ion pairing-dispersive liquid–liquid microextraction combined with smartphone digital image colorimetry for the determination of tramadol in human urine

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Tramadol (TH) is an opioid pain medicine and a serotonin-norepinephrine reuptake inhibitor used to treat moderate to severe pain. In several countries, the drug is categorized as a "controlled substance" that requires medical prescription. When used at high doses, it can have similar effects to heroin. TH levels can be measured in the body fluids to monitor for abuse, confirm a poisoning diagnosis, or aid with the forensic investigations of a sudden death[1]. Most commercial opiate immunoassay screening tests do not cross-react appreciably with TH or its main metabolites, hence chromatographic techniques with spectrometric detectors are preferable for the detection and quantification of TH[2]. Smartphone digital image colorimetry (SDIC) has recently emerged as a complementary analytical technique for chemical analysis owing to the rapid advancement of mobile technology[3]. This technique can reduce analysis costs and time, increase access to instrumentation systems, widen and simplify the analysis process, and ensure a high degree of adaptability for on-site analysis. Nonetheless, for complex samples like urine, effective sample cleanup and preconcentration methods are required to maximize selectivity and sensitivity. In this study, ion pairing-dispersive liquid–liquid microextraction (IP-DLLME) was used prior to SDIC for the determination of TH. Images of the coloured extracts were captured in a laboratory-made black colorimetric box, which were then split into their red-green-blue channels and the most intense channel (i.e., blue) was used to quantify TH. Optimum ion pairing conditions were achieved using 1.50 mL of 120.0 μg mL^{-1} Eriochrome Black T as the ion-pairing agent, sample pH adjusted to 4.50 with 0.200 M acetate buffer within 10 s reaction time. Optimum IP-DLLME were obtained with 500 μL chloroform as the extraction solvent, 2.50 mL of acetontrile as the disperser solvent, and 25.0 mL of sample solution within 30 s extraction time. Optimum SDIC conditions were attained at a distance of 12.00 cm from the detection camera, a region of interest of 1600 px^2, a detection wavelength of 480 nm and 30.0% brightness of the light source. Under these conditions, the limits of detection and quantitation were found as 1.20 and 4.10 μg mL^{-1}, respectively. A good linearity was achieved with an average coefficient of determination of 0.9951. The proposed method was applied for the determination of TH in human urine samples with percentage relative recoveries of 89.0–110.8% and percentage relative standard deviations below 8.8%. Accuracy of the proposed method was checked with UV/Vis and the results were statistically in a good agreement.

Keywords: Human urine; ion pairing; liquid–liquid microextraction; smartphone digital image colorimetry; tramadol.

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PP14- Deep Eutectic Solvent Extraction of Phenolic Compounds from Agri-Food Waste Nut Shells Using LC-q-TOF/MS

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The agri-food sector generates a large amount of waste every year worldwide that could be an excellent source of bioactive compounds.[1] Their utilization is therefore economically and environmentally important. To recover these valuable molecules, conventional extraction technologies use organic solvents. Therefore, the use of alternative green solvents to reduce the environmental impact in the extraction of phenolic compounds is becoming increasingly important.[2] Therefore, in this study, deep eutectic solvents (DESs) were compared with methanol which is one of the conventional solvent for the extraction of phenolic compounds from nut shells (pecan nut, walnut, hazelnut, chestnut) which are among the wastes of the agri-food sector. Choline chloride (ChCl) based DESs of formic acid, acetic acid (AA), propionic acid, butyric acid were prepared in different ratios and tested. According to the results obtained, ChCl-AA exhibited the highest extraction yield for compounds. Therefore, ChCl-AA DESs were prepared at different ratios and the recovery was evaluated. Since ChCl:AA (1:4) DES showed the highest recovery, this DES was used in the sample application. By performing a recovery study with rutin and apigenin spiked samples, the accuracy of the method was demonstrated with high recovery. The quantification and qualification of phenolic compounds were performed using LC-q-TOF/MS. The advantage of DES in the high recovery of phenolic compounds in addition to their low toxicity, recyclability, tunability and environmental friendliness has been demonstrated in this study. The results showed that DESs can be an alternative to conventional solvents in the extraction of phenolic compounds from agri-food waste.

Keywords: Deep Eutectic Solvents, liquid extraction, bioactive compounds, agri-food by products.

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PP15- Determination of Sudan Dyes in spices, beverages, sauces and water samples with switchable solvent-based liquid phase microextraction using HPLC

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Sudan dyes belong to a class of industrial azo dyes that consist of a range of red colours. It is widely used instead of natural dyes in various industries such as leather, cosmetics, textiles and plastics to reduce production costs, as well as in the food industry to improve the color, taste and appearance of foods. However, due to their potential carcinogenic properties, there are concerns about potential health risks associated with their use.[1] Although they have negative effects on human health, the control and monitoring of these dyes, which are still used in food and other industries, is necessary for food safety and the environment. In this study, environmentally friendly switchable hydrophilicity solvents (SHS) which is hydrophilic N, N-dimethylbenzylamine carbonate compound in two forms, polar and non-polar,[2] were used in LLME before HPLC-DAD analysis to separate and concentrate Sudan (I, II, III and IV) dyes from spices, beverages, sauces and water samples. The optimization of the microextraction process was performed by studing the effects of various parameters such as pH, SHS volume, phase transition triggering agent volume, sonicitaion and centrifugation time. In this study, where 300 µL SHS was used, LPME total analysis time was performed in 5 minutes with high recovery at pH 8. The pre-concentration factor of the SHS-LPME procedure is 50. This SHS-LPME method offers an environmentally friendly option for the extraction of Sudan dyes from spices, beverages, sauces and water samples.

Keywords: Switchable Solvents, liquid phase micro extraction, Sudan dyes, liquid chromatography.

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PP16- Miniaturized electrochemical platforms in the study of Rhodamine B

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The analyte studied in this research was Rhodamine B (RhB), which is a synthetic compound from the group of xanthene dyes. RhB has a pink color when dissolved in water and it is used to dye various types of fabrics, as well as colorant in the food industry. Because RhB is an electrochemically active compound, we were able to investigate it using electrochemistry at the interface between two immiscible electrolyte solutions (ITIES). For this purpose, we have employed the ion transfer voltammetry (ITV) technique applied to the microscopic ITIES-system (μITIES).

Miniaturization was done with a silica capillary having a diameter of 25 μm. During the measurements the capillary was filled up with 1,2-dichloroethane solution and served as the organic part of the liquid/liquid interface[1,2]. The interfacial and electrochemical behavior of RhD was studied by changing its concentration in the aqueous phase. Subsequently, calibration curves for positive and negative currents were calculated and plotted. Based on the obtained results, significant physicochemical and analytical parameters are determined (LOD - the limit of detection, LOQ - the limit of quantification). The key advantages of the applied method include high sensitivity, reproducibility, and repeatability of the obtained results, low equipment costs, minimal consumption of toxic organic solvents, and simplicity of measurement.

Keywords: Rhodamine B, micro-ITIES, Electrochemistry

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PP17- Fabrication of plastic membranes as potential electrochemical sensors in the study of biogenic amines

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This research aimed to fabricate plastic membranes to be used as electrochemical sensors when employed as the support of the interface formed between two immiscible electrolyte solutions (ITIES)[1]. Prepared membranes served as the ITIES miniaturization protocol. The analyte studied in this research was phenylethylamine (PEA), one of the biogenic amines that is mainly found in chocolate, but also other types of food. In addition, PEA has a stimulating effect that leads to the release of neurotransmitters, resulting in feelings of pleasure and improved well-being[2].
This study was carried out with the ion transfer voltammetry (ITV) that was used to follow ion transfer process happening across the interface between two immiscible liquids (water | 1,2-dichloroethane). The membranes were made in a heat-sealable foil (for lamination) after preparing the aperture with a sharp needle.
The experiments carried out using heat-sealed film enabled the detection of electrochemical signals that were attributed to the transfer of ionized analyte molecules across the liquid-liquid interface. Based on the results obtained limits of detection (LOD) and limits of quantification (LOQ) for the PEA detection were calucalted.
Keywords: interface between two immiscible electrolyte solutions, ion transfer voltammetry, miniaturisation, phenylethylamine, biogenic amine.

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PP18- A Novel Electrochemical Approach of Anticancer Drug Vandetanib on Glassy Carbon Electrode

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Pharmacokinetic studies of anti-cancer drugs in cancer patients demonstrate that dose selection is highly crucial, as are dosing intervals in clinical applications. Rapid and accurate analysis, facilitated by numerous electrochemical nanosensor-based technologies[1], plays a key role in monitoring potential overdoses. These advancements also address the quality control aspects of pharmaceutical production and drug administration. Vandetanib (VAN) is an antineoplastic drug belonging to the class of tyrosine kinase inhibitors, characterized by its inhibitory action on vascular endothelial growth factor receptor-2 (VEGFR-2) and epidermal growth factor receptor[2]. This study represents the electrochemical investigation demonstrating that a bare glassy carbon electrode (GCE) can be utilized for the quantification and electrochemical characterization of VAN. Cyclic voltammograms and differential pulse voltammograms are employed to analyze Vandetanib, providing insights into the kinetics of charge transfer during the redox reactions of these compounds. Using the relationship between the logarithm of peak currents vs. the logarithm of scan rate, the electrochemical mechanism of the drug was enlightened. The mechanism was found to be as adsorption-controlled since the slope of the logarithm of peak currents vs. the logarithm of scan rate was found to be 1.027. Since the mechanism is adsorption-controlled, accumulation potential and time were further optimized. Accumulation potential (0.8 V) and accumulation time (90 s) were used for VAN analysis. Using optimized conditions, sensitivity, limit of detection, repeatability, reproducibility, and accuracy parameters were meticulously studied.

Keywords: Vandetanib; Glassy Carbon Electrode; Differential pulse voltammetry; Cyclic Voltammetry.

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PP19- Authentication of the Herbs and Spices with Emerging Spectroscopic Techniques: A Review

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The history of herbs and spices back to as far as history of humanity. Use of the spices were expended by Romans in too many fields (as colorant, flavored and presevative of foods, cosmotics, parfumes and folk medicine). The spices trade have been crucial due to economical value via trade routes. Thanks to the developing trade networks in recent years, it has become possible to access things with more monetary value. At this point, in order to meet the demand of herbs and spices increasingly, especially dried forms, become susceptible to adulteration as difficult to detect with human eye. The adulteration factors deteroation the originality and unique form of the spices. In this state, caused to negative effect on food safety, quality, and geogrephical characteristcs, as well as misleading and fraud the customer. There are too many resources in the literature about applying targeted and non-targeted authentication techniques combine with chemometric methods in order to identification unique chemical composition and distinguish based on geographical location with qualitative and quantitative approach, detection and seperation foreign component from standart profile of the spices. It is important that the methods used provide verifiable and repeatable results quickly, minimal damage to the environment, easy to use and integrated into production areas. In this review, discusses the merits and demerits of the authentication some herbs and spices (red pepper, saffron, and ginger) with FT-IR (Fourier Transform Infrared) and NIR (Near Infrared) spectroscopy.

Key words: Herbs; Spices; Authentication; FT-IR; NIR

PP20- Quality Determination in Fruits and Vegetables through Novel Analytical Techniques

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Fruits and vegetables play a crucial role in meeting our daily nutritional requirements. Regarding these essential food sources, the quality of fruits and vegetables is vital for consumers. Evaluating fruit quality involves considering a range of internal and external attributes. Internally, the quality of fruits and vegetables is mainly determined by various factors, including aroma, flavor, taste, texture, and nutritional composition, encompassing parameters such as soluble solids content, sugar content and composition, organic acids, carotenoids, total flavonoids, total phenolics, antioxidant activity, and starch content. Factors like flesh firmness, the presence of any diseases, and potential chemical residues contribute to the overall internal quality assessment. On the other hand, the quality can be determined externally by factors such as appearance, color, size, and the presence of bruises. Internal and external quality measures ensure a comprehensive evaluation of the overall fruit quality, enhancing the consumer's experience and satisfaction. The food industry consistently explores practical methods for assessing quality to meet consumer expectations. Currently, many existing analytical methods remain labor- and time-intensive, often involving destructive procedures. Furthermore, these traditional analytical techniques demand extensive sample preparation, along with the use of expensive instruments and chemicals. On the other hand, as the demand for real-time fruit and vegetable quality detection grows, there has been significant progress in developing novel analytical evaluation methods. This review critically examines recent advancements in analytical techniques for evaluating fruits and vegetables' internal and external quality attributes. We include the latest methodologies and innovative analytical tools for characterizing and quantifying these traits.

Key words: Quality, Fruits and Vegetables, Innovative Analytical Tools

PP21- Determination Of Fatty Acid Contents Of Seed Oils by GC/MSD

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Lipids are found in all parts of plants but mostly dominant in seeds [1]. The edible oils are a main substance in human diet, and they are comprised of mostly of triacylglycerols (TAGs) and other minor constituents such as free fatty acids (FFAs), phospholipids, sterols, vitamins, and monoacylglycerols and diacylglycerols. Edible oils are necessary for the appropriate development of human tissues [2-3]. In addtion, edible oils are generally utilised as food components, lipid components in cosmetics, and also as a carrier in certain pharmaceutical formulations such as suspension and emulsion in cream and lotion [4]. The lipophilic character of polyunsaturated fatty acids (PUFAs) mostly are an applicable carrier to increase therapeutic efficacy of anticancer drugs. There is an approach to substitute synthetic antioxidants, due to their toxic side effects, with naturally occurring antioxidants by the use of natural compounds like free fatty acids (FFAs) [1-3].

100 mg of seed oil was mixed with 10 mL of n-hexane and 100 µL of 2 N methanolic potassium hydroxide. The sample was then centrifuged at 4000 rpm for 10 min. After transferring the upper layer to a new tube, it was filtered using a disposable 0.20-µm Macherey-Nagel Chromafil Xtra PTFE-20/25 LC filter disk. Fatty acid methyl esters (FAMEs) were analyzed by gas chromatography mass spectrometry (GC/MS) (Agilent 7890 GC 5975C inert MSD; Agilent Technologies, Wilmington, DE, USA).
The obtained results show these agricultural products, twenty vegetable oils, to be abundant in free fatty acids (FFAs) as well as confirming the important contribution of saturated (SFA), monounsaturated (MUFA), polyunsaturated (PUFA) fatty acids, omega 3 and omega 6. In this study, a comprehensive profiling of FFAs in edible seed oils was investigated by GC-MSD, including the detecting of most FFAs. According to principal component analysis, MUFA components displayed different behaviour then other components. The analysis of free fatty acids (FFAs) in edible seed oils can provide important information for quality control and oil authentication.

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PP22- Determination of European Grapevine Moth Pheromone by Headspace Sampling Coupled with Gas Chromatography

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Present study focus on the development of a gas chromatographic method for the determination of sexual pheromone used for biotechnical control studies against the *Lobesia botrana Den. Schiff.*, which is known to be the most important harmful species in the vineyards of Turkey[1]. For sensitive analyses, the volatile components are collected on porous adsorbents and then analyzed after treatment with a suitable solvent[2]. Although these methods provide results with the desired sensitivity, there is an increased trend towards more environmentally friendly and efficient extraction techniques.

Headspace (HS) analysis technique provide sensitive gas chromatographic (GC) determination for the volatile components without using harmful solvents. A sample taken in a certain volume from the headspace of the samples in equilibrium under certain temperature and pressure conditions in the vial were injected into the GC-MS system. Here, the main parameters such as equilibrium time and temperature, initial inlet temperature and vent flow time, and other instrumental parameters have been optimized. Under optimized conditions, reproducible results have been obtained (RSD<%5) and the calibration curve has been constructed in ng/mL concentration ranges. A sensitive and selective method has been developed by this means.

Keywords *Lobesia botrana*, sexual pheromone, gas chromatography, head space analysis

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PP23- Supramolecular Solvent-Based Liquid-Liquid Microextraction Coupled with High-Performance Liquid Chromatography for the Determination of Organophosphorus Pesticides in Tea Beverages

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Supramolecular solvents (SUPRASs) are nanostructured liquids produced by self-assembly and coacervation in colloidal suspensions of amphiphiles [1-2]. A new environmentally friendly SUPRAS-based liquid-liquid microextraction method has been utilized for the determination of organophosphorus pesticides in tea beverages. To demonstrate the feasibility of the method fenitrothion, phosalone, and chlorpyrifos were selected as target analytes. The method was performed by extracting analytes into the spontaneously formed SUPRAS phase within a mixture of di-(2-ethylhexyl) phosphoric acid, tetrahydrofuran and the aqueous sample. The SUPRAS was separated from water by centrifugation due to its lower density and easily transferred to a vial for analysis using high-performance liquid chromatography-ultraviolet detection (HPLC-UV). The study thoroughly examined various factors influencing the performance of the extraction method. These parameters include the volume of di-(2-ethylhexyl) phosphoric acid, the volume of tetrahydrofuran, ionic strength, and extraction time. Extraction efficiencies in the range of 77-98% were obtained under optimized conditions. Calibration curves were linear over the 25-1000 µg/L concentration range with coefficients of determination better than 0.9994 for all analytes. Furthermore, low detection limits in the range of 6-10 µg/L and good reproducibility of less than 9.5% were attained for the target analytes. The proposed method was applied to analyze various tea beverages samples. The recoveries of organophosphorus pesticides from the spiked samples varied between 82% and 106%.

Keywords: Tea beverage, organophosphorus pesticides, supramolecular solvent, high performance liquid chromatography

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PP24- Preconcentration of antidepressant residues in wastewater by applying green microextraction strategy prior to high performance liquid chromatography determination

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The utilization of antidepressants has increased at an exponential rate over the past two decades. Numerous antidepressants function by impeding the cerebral reuptake of norepinephrine and serotine [1]. Nearly one quarter of patients exhibit no response to existing treatments, while approximately 33% fail to achieve full recovery. Despite their medicinal use, these medications are frequently linked to unintentional fatalities and suicides [2]. Fluoxetine (FLU), chemically known as N-methyl-3-phenyl-3-[4-(trifluoromethyl)phenoxy]propane-1-amine, was the initial selective serotonin reuptake inhibitor (SSRI) to be developed and sold as Prozac® by the pharmaceutical corporation Eli Lilly. Citalopram (CIT) is a second-generation antidepressant medicine that works by selectively inhibiting the reuptake of serotonin. Its chemical name is 1-[3-(dimethylamino)propyl]-1-(4-fluorophenyl)-1,3-dihydro-5-isobenzofurancarbonitrile. The effectiveness of this treatment is like tricyclic antidepressants, but it is better tolerated and has a lesser likelihood of causing negative side effects [2]. Optimizations such as DES type/volume, buffer type/volume and mixing type/period were performed to determine antidepressants using the DES method with HPLC method. During the optimizations, satisfactory preconcentration factors were obtained for FLU and CIT analytes.

Keywords: Antidepressant, deep eutectic solvent, high performance liquid chromatography.

Acknowledgment: This study supported by the Scientific and Technological Research Council of Turkey (TUBITAK) within the scope of 2209-A - Research Project Support Programme for Undergraduate Students (Project No:1919B012304353).

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PP25- Differential Pulse Voltammetric Determination of Paraoxon Ethyl at Disposable Graphite Pencil Electrode

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Paraoxoon ethyl (PEL) is one of the important organophosphate pesticides (OPs) with a high insecticidal effect. Due to this feature, i is often used in agriculture to increase productivity and control pests. Despite these beneficial properties, humans, animals, and the environment are seriously and negatively affected due to excessive and careless use of OPs. Due to their remarkable inhibition of the acetylcholinesterase enzyme, these OPs cause neurotoxicity in animals and humans, resulting in various diseases such as Alzheimer's, and Parkinson's[1]. Therefore, the determination of OPs is of great importance, and different analytical methods such as chromatography, electrochemistry, and spectroscopy have been developed for the sensitive, selective determination of PEL. Among them, recent electroanalytical methods have found great attention in the determination of pharmaceutical compounds, because they offer several advantages, such as high selectivity, simplicity, low cost, and fast response[2].
The aim of this study is to perform selective and highly sensitive determinations of PEL based on an easily available, disposable, highly electrochemically reactive, and very advantageous graphite pencil electrode (GPE). A reversible redox couple (Ox_I/Ind_I) at about -200 mV and an irreversible reduction (Ind_{II}) peak at -600 mV were observed in the cyclic voltammograms (CVs) of PEL recorded at GPE in pH 10.0 Britton Robinson Buffer (BRT) solutions containing 0.10 M KCl. The irreversible reduction peak and redox couple are attributed to the reduction of the aromatic nitro group (Ar-NO_2) to Ar-N-hydroxylamine (Ar-NHOH) and the reversible oxidation of the formed Ar-NHOH to the nitroso group (Ar=NO). CVs show that both its oxidation and reduction of PEL at GPE are performed by an adsorption-controlled process, because both peak currents increased linearly based on the scan-rate. Analytical performance studies were performed by recording differential pulse voltammograms (DPVs) of PEL in pH 10.0 BRT containing 0.10 M KCl under optimized conditions. The calibration curve for the Ind_I and Ox_I peaks in the reversible redox couple was found to be linear between 0.1 and 10 µM and between 0.5 and 75 µM, respectively. For the irreversible Ind_{II} peak, the concentration versus peak current varied linearly between 0.1 and 10 µM. Relative standard deviation values found below 5% for oxidation and reductions of PEL indicate that the proposed disposable, low-cost GPE has acceptable intra-day and inter-day precisions. Finally, the proposed method was successfully applied to determine PEL in water samples

Keywords: Paraoxon ethly, pencil graphite electrode, pesticide analysis, voltammetry.
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PP26- Investigation of Phenolic Contents of Artichoke Vinegars

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Vinegar is a product which worldwide used as a seasoning or food preserving agent, contributing to sensory characteristics of food. They contain bioactive compounds and are reported to have several effects such as anti-infective, anti-tumor, antimicrobial, and blood glucose and lipids regulation activities. These effects of the vinegar depend on the raw materials[1].
Artichoke (*Cynara scolymus* L.) is a perennial plant belonging to the Asteraceae family and native to the Mediterranean region. The edible portion of the plant, the flower head, and part of the stem are highly appreciated by consumers for their organoleptic properties and nutritional value. Artichoke has considerable protein and mineral contents, a low percentage of lipids, and a high proportion of dietary fiber, minerals, and bioactive phenolic compounds. In addition, artichoke is also an herbal medicine and has been recognized since ancient times for its beneficial and therapeutic effects. Various studies, including in vitro and in vivo, have demonstrated pharmacological activities, such as hepatoprotective, anticarcinogenic, and hypocholesterolemic effects [2,3].
The present study focused on the phenolic contents (chlorogenic acid, cynarin, luteolin, and apigenin) evaluation of artichoke vinegar by HPLC-DAD system. In artichoke vinegar, chlorogenic acid was found in the range of 7.37-11.4 mg/l, cynarin in the range of 1.72-1.42 mg/l, and apigenin in the range of 0.58-0.83 mg/l. Luteolin was not detected in the samples.

Keywords: Artichoke vinegar, polyphenols, HPLC

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PP27- Investigation Of Voltametric Behavior Of Dopamine Imprinted Polymer-Based Sensors

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In this study, new type all solid-state voltametric molecule selective microsensors were developed based on based imprinted polymer of dopamine. Dopamine as target molecules, as functional monomer and ethylene glycol dimethacrylate as crosslinker were used for the polymer synthesis. The synthesized molecule imprinted polymers were used as ionophores to achieve a selective response in the polyvinylchloride membrane structure towards the main molecule[1]. The microsensors were successfully used for determinations of dopamine in pharmaceutical drug samples. Voltammetric performance characteristics of molecule selective microsensors were investigated. The linear working range of the dopamine electrode was determined as 0.1-10 μM. Using microsensors, dopamine determinations in pharmaceutical drug samples have been successfully performed.

Keywords : Sensor, Molecular Imprinted Polymer, Voltammetry Dopamine

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PP28- Electrochemıcal MIP Sensors For Captopril, Imatinib, and Glutathione

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As early as 1931, the Russian scientist Polyakov developed the first synthetic polymer with a "molecular memory," which later gave rise to "Molecularly Imprinted Polymers (MIPs)." The template is incorporated into the polymer network during the polymerization of monomers and subsequently removed, which creates cavities with a molecular memory mirroring the size and shape of the template (Scheme. 1). The development of imprinted polymers was significantly advanced by Wulff and Mosbach [1]. While MIPs were initially limited to small molecules, today, imprinted polymers can be produced for ions, metabolic products, pharmaceuticals, environmental toxins, biomacromolecules, cells, viruses, and synthetic nanoparticles [2]. MIPs for small molecule analytes dominate in the approximately 1,300 publications per year, but the proportion of protein MIPs is already over ten percent [2].In contrast to antibodies comprising 20 amino acids, MIPs are produced from one to five monomers and possibly a cross-linker. This simplification of complexity represents a significant technological breakthrough. In addition, MIPs are characterized by higher stability under extreme conditions such as pH, temperature, and organic solvents. They are also easier to regenerate than antibodies[1-3].
Herein, we present the fabrication and characterization of electrochemical MIP sensors for the detection of captopril, an antihypertensive drug [4], imatinib, an anticancer drug [5], and the tripeptide glutathione known for its antioxidant properties using a redox marker [6]. The captopril MIP sensor, prepared through the electropolymerization of o-PD on the GCE surface, exhibited a linear measuring range of up to 50 pM and a remarkable selectivity for captopril, effectively distinguishing it from paracetamol (4.6-fold higher suppression), ascorbic acid (almost no binding), and L-Proline (1.6-fold higher suppression). The imatinib MIP sensor was also synthesized via electropolymerization on the GCE surface, utilizing o-PD as a monomer. In contrast, the glutathione MIP sensor was prepared by electropolymerizing scopoletin monomers on an Au electrode. Following electropolymerization, the ferricyanide signal was suppressed entirely. Removal of the template resulted in a significant increase in the ferricyanide signal, which was subsequently suppressed upon rebinding.

Keywords: Molecularly imprinted polymers, biomimetic sensors, captopril, imatinib, and glutathione

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