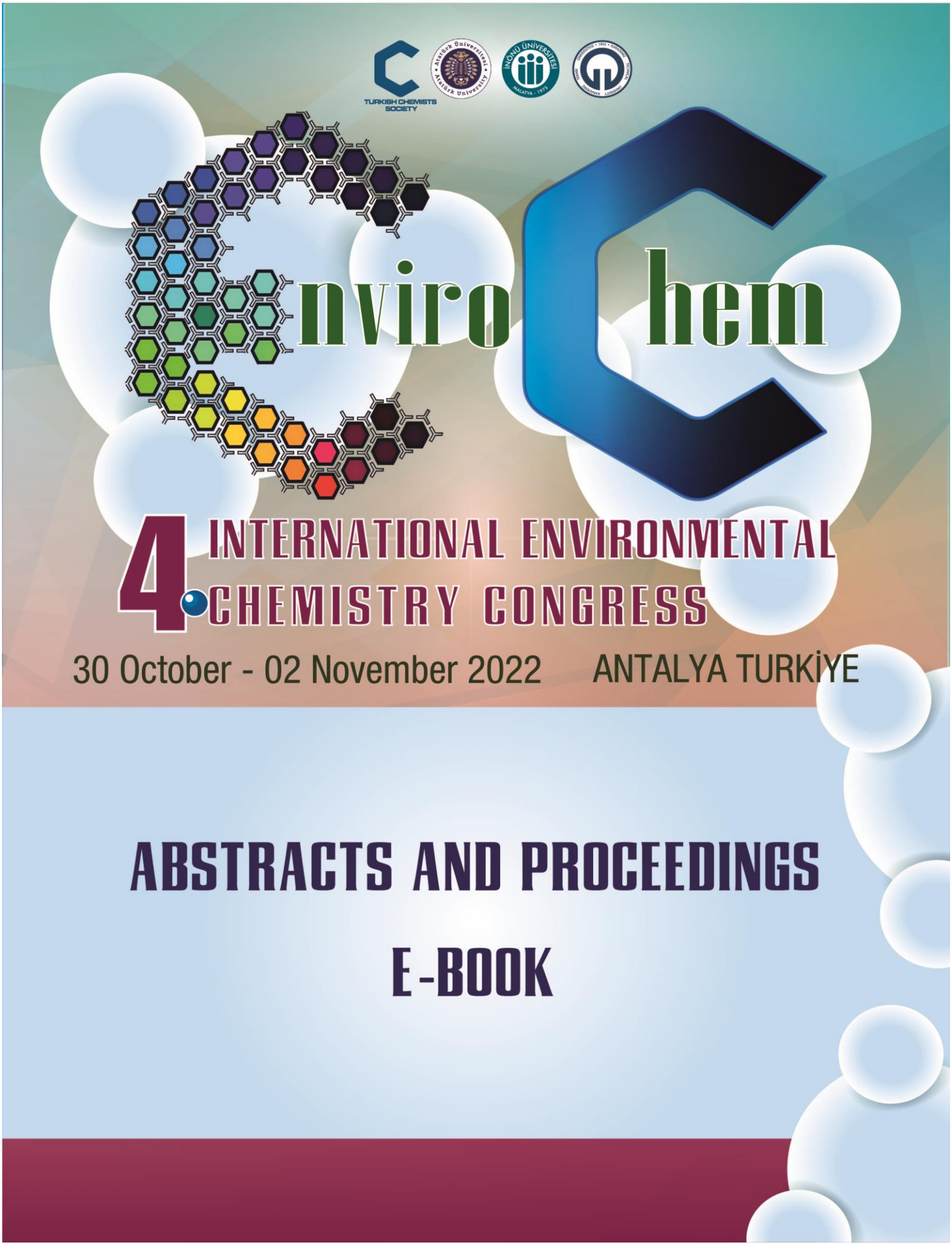


**EnviroChem 2022 was supported by The Scientific and Technological Research Council of Turkiye (TUBITAK) with 2223-B Support Programme for Scientific Activities**


## FOREWORD

### 4th International Environmental Chemistry (EnviroChem) Congress

Dear Participants and Colleques,

We are proud and glad to orginize our fourth congress by the ***Turkish Chemists Society*** under the title of Environmental Chemistry. As will be remembered, we organized our first congress with the title of 1st Eurasian Environmental Chemistry Congress in 2018, the second one with the title of 2nd International Environmental Chemistry Congress in 2019, and our third congress with the title of 3rd International Environmental Chemistry Congress in 2021. The fourth of our congress was held under the title of ***4th International Environmental Chemistry Congress (EnviroChem)*** from ***30 October- 02 November 2022*** in Antalya, Turkiye.

This congress was organised by Ataturk University, Inonu University and Karadeniz Technical University. The main aim of this congress was to contribute to the development of environmental chemistry sciences and applications and to bring together the members of the Environmental Chemistry community. This year we had participant from Algeria, Azerbaijan, Bulgaria, France, Greece, Latvia, Morocco, Pakistan, Palestine, Russia, Romania, Ukraine, Tunisia and Turkiye. Five invited speakers and more than a hundered researchers have contributed with their presentations, discussions and active participations about every part of the conference. We shared our own experiences and perspectives over the various topics via different findings and solutions.

On behalf of the organizing committee, we would like to thank you all for joining us and contributing to the success of the EnviroChem 2022. We also greatly acknowledge to Terra, SEM, Aquaren, Biotek Medikal, Ceyka, Özgül Medikal and ChromaScience for their very generous sponsorships and supports in the organisation of 4th International Environmental Chemistry Congress (EnviroChem).

Apart from these, we especially thank to Local Organizing Committee and our graduate students who have spent their energy for the success of this meeting. We want to thank Mirage Park Resort (Göynük, Kemer, Antalya/Turkey) for their excellent services.

Best wishes

Prof. Yucel KADIOGLU (Chair)

Prof. Hasan KUCUKBAY (Co-Chair)

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November 2022


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**IS – 1**

# Recent Trends in Environmental Chemistry Research

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Most actual world issues such as the Ukraine war, the COVID-19 lockdowns, climate change, public health and rising pollution are linked to research in environmental chemistry. The Ukraine war has revealed the urgency to replace fossil fuels by local and sustainable energies. The COVID-19 episodes has evidenced the sources of air pollution. Frequent climate alerts call for rapid carbon sequestration. Advanced treatments of water and wastewater are required to remove microplastics, antibiotics and other emerging pollutants. This conference will present key examples from the journal Environmental Chemistry Letters, published by Springer Nature.


**IS – 2**

# Littered Cigarette Butts: A Small-Sized Waste Creating Big-Sized Problems

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Cigarette butts are the most littered items in urban areas worldwide, accounting for 22-46% of visible litter.[1] Once disposed onto urban areas, they move through the storm drains to streams, into the ocean, and back onto the beaches. This makes cigarette butts the single most collected item in coastal environments each year, and the second most found item on beaches in the European Union.[2] Environmental awareness on the disposal of tobacco products mainly focuses on the inability of discarded filters to biodegrade. There is markedly less awareness on the potential of TPs to act as point sources and leach toxicants.[3,4] To this end, leachates from discarded cigarettes have been shown to be acutely toxic for different species such as marine bacteria (*Vibrio fischeri*), fish, snails and fish and frog embryos.[3]

The present contribution presents the inorganic and organic chemical components of environmental importance that are leached from used and unused tobacco products. Conventional cigarettes and the new generation Heat-not-Burn product are considered. The contribution of the different parts of tobacco products to the inorganic and organic content of leachates was assessed and compared to the total concentration of each chemical constituent initially present in the tobacco product. The organics were extracted using PDMS-based probes directly from the complex leachates. Cigarette leachates consist of highly complex mixtures of compounds across a wide concentration range, and such compounds typically elute as an unresolved complex mixture when subjected to one-dimensional gas chromatography (GC). Comprehensive two-dimensional gas chromatography time-of-flight mass spectrometry (GC×GC-TOFMS) with high-capacity sorptive extraction was therefore used for the exploratory profiling of leachates from used and unused cigarettes. Comparison of the results to those obtained from unused tobacco products allowed identifying the toxic compounds formed during the burning or heating process (depending on the product). All in all, the false perception that discarded tobacco products are the end point of a life cycle, points that there is still a way to go in addressing responsible disposal and post-consumer waste cleanup, to minimize the environmental hazards of discarded tobacco products.

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**IS – 3**

# Nanoflowers and Magnetic Nanoparticles in Analytical and Environmental Chemistry

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Nanoparticles have wide range application from analytical/environmental chemistry to medical, agricultural and pharmaceutical areas. In analytical chemistry, nanoparticles are commonly used in the extraction/preconcentration steps to detect low amount of analyte(s) found in complex sample matrices[1]. Due to the unique nature of nanoparticles such as large surface area, rapid extraction kinetic and high extraction yield, nanoparticles are indispensable adsorbents in modern solid phase extraction (SPE) methods[2]. These materials offer analytical methods with high selectivity and sensitivity thanks to great surface area and high affinity to the analyte(s) of interest[3]. Magnetic nanoparticles are one classification of nanoparticles having functionalizable surface, high adsorption capacity and good separation ability due to its manipulation by external magnetic field[4]. In addition to magnetic nanoparticles, nanoflowers have gained much attention in analytical and environmental sciences due to its flower like structure with many petals providing high adsorption capacity and high surface area[5]. Nanoparticle usage in environmental applications mainly includes water and effluent treatment strategies[6]. Pesticides, heavy metals, dyes, pharmaceuticals and bacteria are some examples to be removed from environmental bodies via nanoadsorbents[7]. As a result, nanoflowers and magnetic nanoparticles are promising materials in analytical and environmental chemistry.

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**IS – 4**

# POPs, and Synthetic Musks in indoor and ambient air of Turkey

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Persistent Organic Pollutants are compounds known for high hydrophobicity, low vapor pressure, and resistance to environmental degradation and they are subject to bioaccumulation. Due to their discharge or emissions from various sources in the past, unintentional sources currently, they can be found in indoor and outdoor environments. Partitioning is the main process in which form these compounds will be present in the environment. In this paper, their levels and fates in Turkey will be presented. Special attention will be given to synthetic musk compounds (fragrances) as emerging contaminants in indoors.


**IS – 5**

# Development of Methodologies and Implementation of Case Studies on the Assessment and Improvement of the Water Quality of Türkiye Watersheds

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In this study, some examples of basin-based studies in Türkiye conducted by TUBITAK MAM are presented. First of all, the Basin Protection Action Plans for 25 water basins of Türkiye, carried out between 2008 and 2013, are explained. Türkiye has come a long way in a short time with river basin-based studies within the scope of harmonization with Water Framework Directive. In this respect, Watershed Protection Action Plans formed the first and the most important basis for preparation and application of River Basin Management Plans.

As the second project, information on the Determination of Sensitive Areas and Water Quality Objectives for Turkish River Basin Districts, which is studied on the basis of water body in 25 water basins of our country, was given. The project is performed according to EU Directives of Nutrient-sensitive areas including areas designated as vulnerable zones under Directive 91/676/EEC and areas as sensitive under Directive 91/271/EEC. Water bodies and typologies were determined in national scale. 600 rivers and 73 lakes/reservoirs/dams were monitored 4 times a year in 2014 for all Türkiye. Biological indicators and conventional water quality parameters assessed. Sensitive areas and Nitrate Vulnerable Zones were determined.

The third project is called Identification of Receiving Water Body Based Discharge Limits Küçük Menderes River Basin Case Study. Aim of the project is, combine the approaches of Water Framework Directive (2000/60/EC) (WFD) and United States (US) for determination of discharge standards and present a modelling based method, which can be applied to river basins in Türkiye to determine discharge limits by using these different approaches. The study has been conducted for priority and specific pollutants that exceed the EQSs and the aim will be to achieve EQSs for the related pollutants. The Project aims to determine discharge limits for the pollutants exceeding EQS for the Küçük Menderes River Basin with the mathematical modelling based TMDL.

As a result of all these projects, the following results have been achieved.

- Watershed-based studies started in 2008 on the scale of Türkiye and continue today.
- In order to protect both the quality and quantity of surface water, action plans were prepared for the decision makers and guidance was provided.
- These works played an important role in eliminating the deficiencies in the environment chapter, which is Türkiye's obligation in the EU directives.
- After the studies that started on the whole Türkiye scale, these studies turned into detailed studies on the watershed basis, the monitoring started with conventional parameters, then biological parameters and hazardous substances were included and finally continued with priority and specific polluting parameters
- Studies on the assimilation capacities of the basins have been started and these studies have been completed in the case of Küçükmenderes. Suggestions to the decision makers and/or authorities have been made to determine the discharge limits based on the receiving environment in other basins.

- In Türkiye, watershed based studies led to the capacity development. The infrastructure of the laboratories has been strengthened, the number of analyzes of chemical and biological parameters has been increased, the measurement limits have been reduced, so there is no need to send samples abroad for analysis.
- These projects have led to the capacity development of technical experties in research institutions, universities and ministries. Thus, less need for bringing experts from abroad in the projects to be carried out on the scale of our country.
- Studies on a stream/lake have spread to the scale of our country, and the characteristics of the receiving water bodies in our country have been determined.

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**OP – 1**

# Simultaneous Determination of 29 Endocrine Disruptor Compounds-Pesticides in Rock, Soil, Water, Moss and Feces Samples from Antarctica Using Simple and Effective Fine Droplet Formation Based Spray Assisted Liquid Phase Microextraction Prior to Gas Chromatography-Mass Spectrometry

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The poles, which act as a compass about global environmental processes, are attracting more and more attention by scientists day by day. Especially in recent years, due to the increasing human activities, to prevent consequences such as environmental pollution, destruction of vegetation, and negative effects on living life, the determination of low levels of organic pollutants (POPs) that can be transported in the environment over long distances, is of vital importance[1,2]. In this study, simultaneous determinations of 29 different chemicals including endocrine disruptors and pesticides were performed by gas chromatography-mass spectrometry (GC-MS). In order to reduce the detection limit of the analytes in the conventional GC-MS system, a preconcentration method was applied to the aqueous mixture solution. The fine droplet formation based spray assisted liquid phase microextraction (FD-SA-LPME) method has been developed for simultaneous extraction/preconcentration of analytes. The developed method can reduce the experimental steps, does not require external dispersing solvent, and is suitable for easy and fast application[3]. For each variable affecting the extraction output, the optimum condition was determined by univariate optimization studies. According to the results obtained under the optimum conditions, the detection limit (LOD) values of the method, which offers wide linear range and low relative standard deviation for each analyte, were calculated between 0.0009 – 0.0066 mg $kg^{-1}$ (mass based sample/standard preparation). Accordingly, enhancements in detection powers were achieved in the range of 3.7 to 158.9 for analytes compared to the LOD values of the classical GC-MS system. In order to test the applicability of the method to real samples, spiked recovery experiments were carried out on different seawater, soil and moss samples. At last, by applying the developed method to the real samples collected from the 3rd National Polar Expedition (2020), the qualitative and quantitative determinations of the target analytes determined in the study were carried out in these samples.

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**OP – 2**

# Macro and Trace Elements Profile of Black Sea Bivalve Species *Mytilus galloprovincialis*, *Chamelea gallina* and *Donax trunculus*

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The marine bivalve's species such as mussel (*M.galloprovincialis*), striped venus clam (*C. gallina*), wedge clam (*D.trunculus* ) are among the ones most harvest in the world. Their capacity to absorb trace elements not only from the food but also from the surrounding waters makes them a good bioindicator for monitoring of heavy metals and integrating part of various monitoring programs worldwide.

This study is aimed to assess the macro- (Ca, Mg, Na and K) and trace element (Cd, Cr, Cu, Fe, Ni, Pb, and Zn) concentration in those three economically important bivalve species collected from the Bulgarian part of Black Sea. The highest average concentration for all samples were found for Fe (483.74 mg/kg w.w), followed by Zn (27.46 mg/kg w.w). The concentration of Cd in *C.gallina* (1.13 mg/kg w.w) exceed the maximum permissible level of Cd in bivalve mollusks of 1 mg/kg set by Commission Regulation (EC) No1881/2006. The results obtained for the elements in analyzed fish species were compared within acceptable limits for human consumption set by various health institutions.

**Acknowledgment:**

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**OP – 3**

# Effects of Chemical Agents on the Production of Adsorbents from Bituminous Coal for the Removal of Phenol from Wastewater

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## Abstract

In this study, KOH, HCl, $HNO_3$, $HClO_4$ and $H_2SO_4$ were tested as chemical agents for the production of adsorbent from bituminous coal. The removal efficiencies of the activated bituminous coal based adsorbents were investigated by performing batch adsorption experiments. According to the experimental results, the phenol adsorption capacity of the produced adsorbents was the highest (80%) when sulfuric acid was used as the chemical agent in activation. On other hand, the removal efficiency of phenol was only 5% when the chemical agent was KOH. The characterization of the studied adsorbents was performed by using FT-IR, SEM and gas adsorption techniques. The results show that when the activation process is applied with $H_2SO_4$, the pore structure of bituminous coal changes and increases in the surface area can be achieved.

## Introduction

Activated carbon is a widely used adsorbent for the adsorption of phenol due to its large specific surface area, superior porosity, high physicochemical-stability, and excellent surface reactivity.[1] However, recent studies have focused on new adsorbent materials with low cost and easily available because of costly regeneration of activated carbon.[2] Among various adsorbents available, such as clays, peanuts, sawdust etc., coal is one of the most effective adsorbents. However, the adsorption capacity of raw coal is low and it has been reported in literature that the adsorption capacity of raw coal can be increased by physical and chemical activation.[3] Low rank coal, coal reject, anthracite, bituminous coal and semi-coke were used as adsorbents for the removal of different pollutants from wastewater including phenol.[4-9] The bituminous coal originated from Zonguldak Province was chemically activated at high temperature in order to produce activated carbon for hydrogen removal.[10] Most of the activation studies in literature are carried out under high temperatures and the number of activation studies carried out at low temperature using bituminous coal is limited.

Phenol is an important chemical used as a raw material in many industries such as pharmaceuticals, petrochemicals, plastics, textiles, paints and pesticide production. Therefore, wastewater containing high amounts of phenol and phenolic compounds is generated in the industry. Phenol, which is on the priority pollutant list of EPA, has toxic, carcinogenic and mutagenic effects for humans, animals and aquatic organisms and these wastewaters must be treated before discharge.[11] Several methods are available in the literature for the treatment of phenol but adsorption process is the most widely used due to its relatively simple implementation and low operation cost.[2]

In this study, bituminous coal samples (Zonguldak, Turkey) were processed with KOH, HCl, $HNO_3$, $HClO_4$ and $H_2SO_4$ at relatively low temperature (30°C) in order to produce an adsorbent for the removal of phenol from wastewater. The physical and chemical properties of the produced adsorbents were characterized by nitrogen adsorption isotherm, Fourier transform infrared (FTIR) spectroscopy and scanning electron microscope (SEM). In addition, batch adsorption experiments were conducted in order to evaluate the removal efficiency of phenol for each adsorbents.

## Materials and Methods

### Raw Materials and Modified Coal Sample Preparation

Bituminous coal samples were collected from Zonguldak Province in Turkey. The coal samples were crushed and sieved at four different mesh size in the range of [1.7-0.212] mm. A base (KOH) and four acids (HCl, $HNO_3$, $HClO_4$, and $H_2SO_4$) were tested as chemical agents in the production of adsorbents from bituminous coal samples. In the activation process, the first step was washing the coal samples with distilled water and drying in the oven at 105°C for 48 h. Then, in the second step, the chemical agents were added to the coal samples at 30°C. When the chemical agent was KOH, coal samples were added to 5 mol/L KOH solution prepared by using deionized water keeping the solid/liquid ratio at 1:2. In the case of HCl, $HNO_3$, $HClO_4$ and $H_2SO_4$, 2 g coal samples was mixed with 4 mL of acids. The mixing of coal samples with chemical agents was performed in a thermoshaker at 30°C for 48 h. The speed of shaker was 150 rpm. In the final step of the activation process, the coal samples was thoroughly washed with deionized water until the filtrate reached a neutral pH and it was dried completely at 105°C for 24 h.

### Adsorption Experiments

The adsorption experiments were carried out in a 250 mL Erlenmeyer by mixing 1 g of modified coal with 100 mL of 50 mg/L phenol solution. The solution was shaken at 30°C and 150 rpm for 1 h. The supernatant solution was analyzed for the remaining concentration of phenol according to the standard method of 4-aminoantipirin using a UV–vis spectrophotometer (Shimadzu UV-1800).

### Characterization Methods

The surface morphologies were analyzed by a Scanning Electron Microscopy (Tescan GAIA3+Oxford XMax 150 EDS). The structural properties (specific surface area, average pore size, and total pore volume) were determined on the basis of nitrogen adsorption/desorption isotherms at 77 K, using Micromeritics, TriStar II Plus. The specific surface area was determined with the standard Brunauer–Emmett–Teller (BET) method. The surface functional groups were determined by Fourier-transform infrared (FT-IR) spectra (Thermo Fisher Nicolet iS50).

## Results and Discussion

### The parameters Analyzed in the Production of Adsorbent from Bituminous Coal

***Coal Size***

The coal samples were only washed with distilled water after sieving and then dried in an oven at 105°C. Then they were used as adsorbents in batch adsorption experiments. The effects of coal size on phenol removal efficiency are shown in "Table 1". According to the results, crushing causes a slight increase in phenol removal efficiency as a result of increases in surface area of the coal samples. The maximum phenol removal efficiency is only 7.04% when the coal size is between [0.25-0.212] mm.

**Table 1.** Adsorption performances of different sized coal samples.

| Coal Size (mm) | Removal Efficiency (%) |
|---|---|
| [1.7-1] | 0.82 |
| [1-0.5] | 4.01 |
| [0.5-0.25] | 5.87 |
| [0.25-0.212] | 7.04 |

***Chemical Agents***

KOH, HCl, $HNO_3$, $HClO_4$, and $H_2SO_4$ were tested as chemical agents at a ratio of 1:2 in the chemical activation of bituminous coal. The calculated removal efficiencies from batch adsorption experiments using the produced adsorbents are shown in "Fig 1".

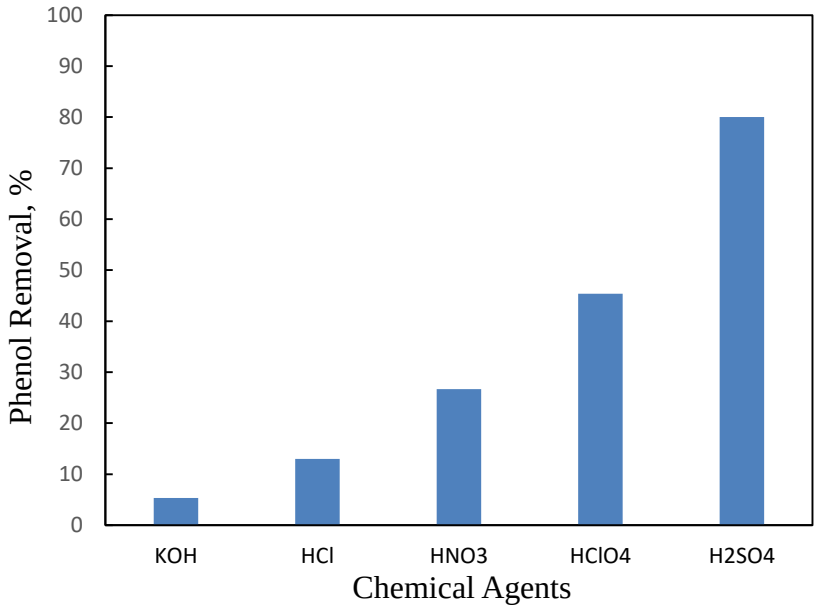


**Figure 1.** The phenol removal efficiencies of the activated bituminous coal samples depending on chemical agents.

The results show that the lowest phenol removal efficiency is obtained when the chemical agent is KOH, it is only 5%. On the other hand, the corresponding removal efficiencies for HCl, $HNO_3$, $HClO_4$, and $H_2SO_4$ are 13, 27, 45 and 80%, respectively. Many studies have revealed that KOH can react at high temperature values. In this study, the low temperatures might be the reason of low phenol removal efficiency for KOH.[12] The highest removal efficiency obtained at the use of $H_2SO_4$ can be due to the fact that this acid can disperse more strongly between layers of coal. Sulfuric acid basically allows the small pores in the coal to combine and they can be converted to the larger ones because of its strong breaking effect on chemical bonds.[13]

***Coal to Acid Ratio***

As it can be seen in "Fig 1", among the studied acids, the two highest phenol removal efficiencies (45% and 80%) are obtained when $HClO_4$ and $H_2SO_4$ are used. Therefore, the effect of coal to acid ratio on phenol removal efficiency are only investigated for these acids in the activation of bituminous coal. For both acid, when the coal to acid ratio is increased in the activation procedure, the phenol adsorption capacity of the bituminous coal derived adsorbent increases as well ("Table 2"). However, these increases are not linear. Considering the change in the adsorbent capacity with the change of coal to acid ratios, there is a slight increase in the phenol removal efficiencies when the coal to acid ratios is increased from 1:2 to 1:3. On the other hand, during experiments, it has been observed that when the coal to acid ratio is 1:3, the mixing of coal particles in acid solution was easier because of less agglomeration.

**Table 2.** The dependency of phenol removal efficiency on coal to acid ratio.

| *Acid* | *Coal/Acid (g/mL)* | *Removal Efficiency (%)* |
|---|---|---|
| **$HClO_4$** | 1:1 | 33.0 |
| | 1:2 | 48.8 |
| | 1:3 | 49.1 |
| **$H_2SO_4$** | 1:1 | 64.6 |
| | 1:2 | 72.8 |
| | 1:3 | 74.8 |

**The Characterization of Bituminous Coal Based Adsorbents**

Even though the characterization analysis of all activated coal samples were conducted, the porous structures, surface properties and functional groups of the activated coal samples with HCl and $H_2SO_4$, where the lowest and highest phenol removal efficiencies were obtained, respectively, are discussed in detail.

***Porous Structural Analysis***

According to the results of low temperature nitrogen adsorption, all isotherms of the studied adsorbents are a mixture of type I-IV isotherms depending on the IUPAC classification.[14] The structural properties (BET surface area, total pore volume and average pore diameter) of the bituminous coal activated by HCl and $H_2SO_4$ together with original coal as determined from the $N_2$ adsorption isotherms are presented in "Table

3". According to the results of BET analysis, transitional and micropores are the dominant porous structure of bituminous coal. The surface area and average pore diameter of the bituminous coal sample are evaluated as 0.5545 $m^2/g$ and 7.3 nm, respectively. When the bituminous coal sample was processed with $H_2SO_4$, the surface area increases to 2.6387 $m^2/g$ and the average pore diameter drops to 3.7 nm. In addition, the total pore volume of the bituminous coal sample is increased more than doubled (from 0.001 $cm^3/g$ to 0.0025 $cm^3/g$). The results of the pore size distribution show that as the micropores coalesce, new mesopores are formed. On the other hand, when HCl is used as a chemical agent in the activation of bituminous coal, the lowest surface area (0.3673 $m^2/g$) is obtained. In addition, the evaluated total pore volume is lower than the bituminous coal sample. However, it can be postulated that larger pores can form during HCl activation as a result of variations in micropores, hence the average pore diameter increases.

**Table 3.** BET analysis for the original and activated bituminous coal samples.

| Adsorbent | Surface Area ($m^2/g$) | Pore Volume ($cm^3/g$) | Average Pore Diameter (nm) |
|---|---|---|---|
| **Bituminous Coal** | 0.5545 | 0.00100 | 7.3 |
| **$H_2SO_4$-activated** | 2.6387 | 0.00250 | 3.7 |
| **HCl-activated** | 0.3673 | 0.00074 | 8.0 |

***Surface Morphology Analysis***

"Fig 2" shows SEM microscope images of the crushed and activated bituminous coal samples with HCl and $H_2SO_4$. Although the crushed bituminous coal has smooth surface with small and irregular pores, the acid treated coal samples are porous particles with rough surface. The results indicate that the acid treatment process can develop more voids and fragments on the coal surface which increases the available surface for adsorption. However, it can be observed from "Table 3" that the adsorption capacity of HCl-activated coal sample is low because of lower surface area.

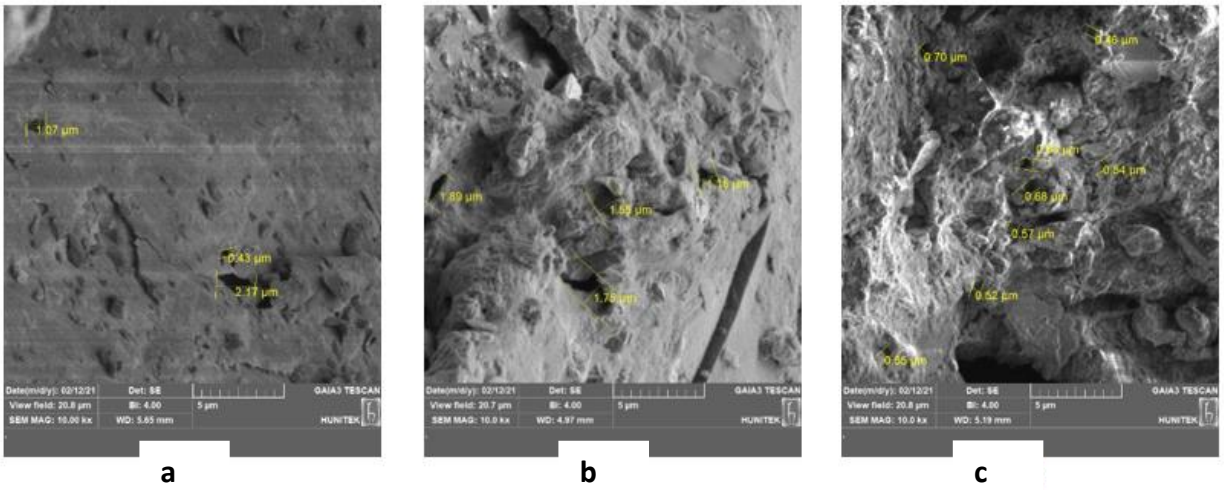


**Figure 2.** SEM microscope images of original and acid treated bituminous coal samples. (a: crushed, b: $H_2SO_4$-activated, c: HCl-activated)

***FT-IR Spectra***

The functional groups on the bituminous coal and the activated coals with $H_2SO_4$ and HCl were measured by FT-IR spectroscopy as depicted in "Fig 3". In the case of the production of adsorbent from bituminous coal, only applied process was crushing and sieving. During the activation process, as a first step, washing with the distilled water and drying were applied in addition to crushing and sieving before mixing with chemical agents. Therefore, the intense and sharp bands around 3600 $cm^{-1}$ is referred to the OH functional groups for the activated bituminous coal samples.

The spectra of the original and activated bituminous coals with HCl and $H_2SO_4$ indicate some differences depending on band and peak positions including intensities. The peaks occur at 2950 $cm^{-1}$ and 2870 $cm^{-1}$ can be assigned to CH stretching, suggesting that aliphatic hydrocarbons with long chains are dominant in the bituminous coals.[15]

The strong peaks at 1600 $cm^{-1}$ were observed in the HCl and $H_2SO_4$ activated coal samples. This peak probably belongs to aromatic ring stretching coupled to conjugated hydrogen-bonded carbonyl groups (C=O).

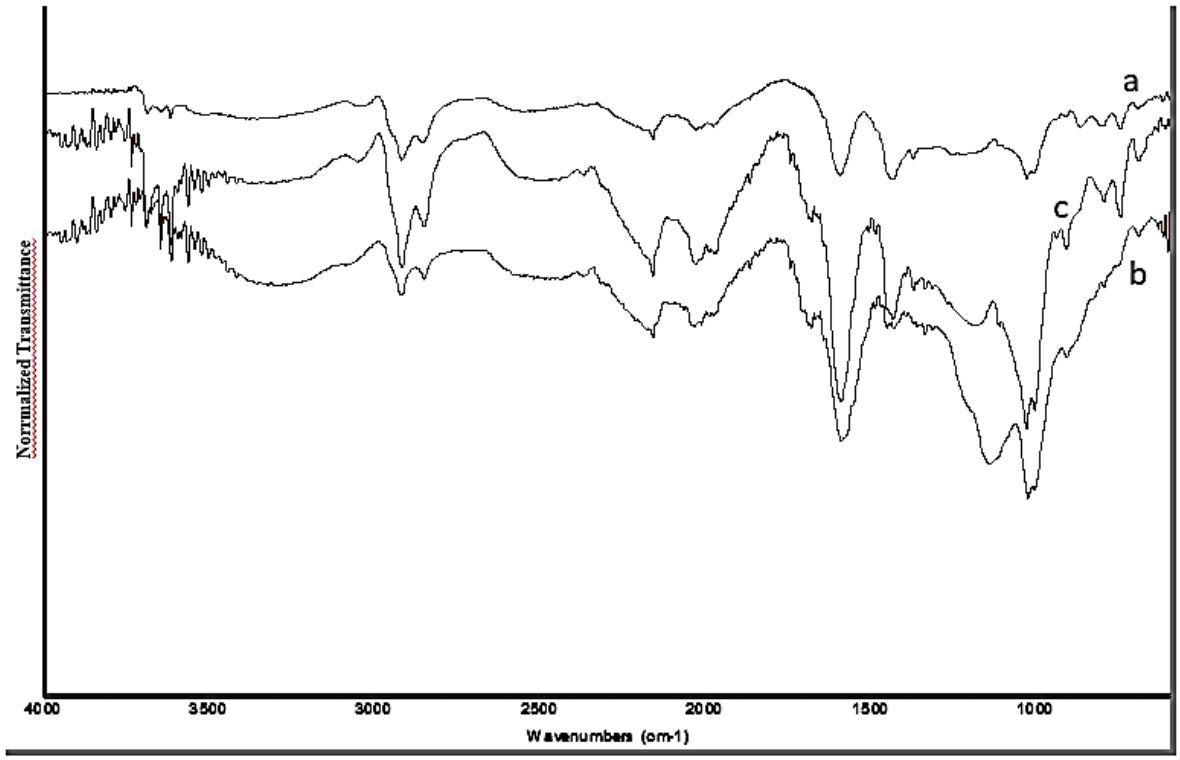


**Figure 3.** FT-IR spectra of the raw and acid treated bituminous coal samples. (a: crushed, b: $H_2SO_4$-activated, c: HCl-activated)

The peak at 1432 $cm^{-1}$ can be ascribed to in-plane C-H vibrations.[16] This peak is weak for $H_2SO_4$ activation.

The bands at 1300 – 1000 $cm^{-1}$ is referred to C-O-C or C-O stretching in phenols, alcohols and $H_2SO_4$ acid ($SO_3$ groups).[17]

## Conclusion

Bituminous coal can be successfully used as an adsorbent for phenol removal from wastewater when it is processed with $H_2SO_4$ even at moderately low temperature, i.e., at 30°C. The removal efficiency of phenol can reach up to 80% when the range of coal size is 0.25-0.212 mm. Among the studied chemical agents, $H_2SO_4$ has resulted in the highest phenol removal rate due to its strong dispersion property between the layers of coal. However, since the activation is applied at low temperature, only 5% phenol removal efficiency is achieved when KOH is used. SEM images show that the activation process with acids can develop more voids and fragments on the coal surface which increases the available surface for adsorption.

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**OP – 4**

# Development of a Switchable Solvent Based Liquid Phase Microextraction Method for the Simultaneous Determination of Selected Nervous System – Active Pharmaceutical Ingredients by Gas Chromatography Mass Spectrometry

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Due to their ecotoxicological and pharmacological effects on organisms, active pharmaceutical ingredients (APIs) have been identified as possible threats to the environment and human health. APIs can be detected in the aquatic environment at amounts ranging from nanograms to micrograms per liter due to both their extensive production and usage[1]. The switchable solvent-based liquid phase microextraction (SS-LPME) method is known to be a low-cost and non-hazardous preconcentration method that allows the extraction of analyte(s) without the necessity of any disperser solvents. For the synthesis of switchable solvent, $CO_2$ is used as an efficient phase transition trigger to change a non-ionic liquid into its ionic form having high solubility in aqueous matrix[2]. In this study, eleven nervous system - active pharmaceutical (antidepressant, antipsychotic, antiepileptic and anti-demantia) ingredients were preconcentrated from aqueous samples using SS-LPME method prior to determination by gas chromatography mass spectrometry (GC-MS). Important parameters in the microextraction method as volume of switchable solvent, concentration/volume of NaOH and period of vortexing were optimized to improve the extraction efficiency. The limit of detection (LOD) values for the analytes ranged between 0.20-8.0 µg $L^{-1}$ under the optimum conditions and percent relative standard deviation values for six replicate measurements were lower than 10%. The accuracy and applicability of the developed SS-LPME-GC-MS method was verified by satisfactory percent recovery results in complex matrices as synthetic domestic wastewater, municipal wastewater and lake water[3].

**Acknowledgements:** This work was supported by Yildiz Technical University Scientific Research Projects Coordination Unit (Project Number: FDK-2020–3842).

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**OP – 5**

# Magnetic Nanofluid Based Microextraction Strategy for Cadmium Determination in Rosemary and Eucalyptus Tea Extracts

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Cadmium (Cd) is a naturally occurring hazardous metal that has been demonstrated to have detrimental health effects in the general population; the International Agency for Research on Cancer classifies it as a Class I carcinogen[1]. Deep eutectic solvent (DES) based magnetic nanofluid (MNF) liquid phase microextraction method (LPME) was developed in this research for Cd preconcentration prior to determination in the slotted quartz tube flame atomic absorption spectrophotometry (SQT-FAAS) system. Deep eutectic solvent and magnetic nanoparticles (MNPs) constitute MNF. MNF generation and liquid phase extraction optimizations were performed to achieve high extraction efficiency. For this purpose, critical optimization experiments such as DES formation ratios (choline chloride:phenol), MNP and DES mixing ratios, mixing type and period were performed. The method developed under optimal conditions had limits of detection (LOD) and limits of quantification (LOQ) values of 0.25 ng $mL^{-1}$ and 0.84 ng $mL^{-1}$, respectively. The newly designed DES-MNF-LPME-SQT-FAAS approach boosted detection power by 330 times over the conventional FAAS system, as determined by a comparison of the LODs. The method was tested on rosemary and eucalyptus tea samples to determine its accuracy and applicability. The obtained recovery values were quite nearer to 100%, with a low standard deviation[2].

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**OP – 6**

# Evaluation of Heavy Metals and Microbiological Contamination of Selected Herbals from Palestine

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Herbal medicine is widely used for the prevention and treatment of diseases worldwide including Palestine and may require long term usage. The level of some heavy metals and microbial contaminants in some of these medicinal plants consumed by Palestinians were studied in order to evaluate their quality.

The level of metals including: Zinc, Cadmium, Lead and Copper were quantified by Atomic absorption spectroscopy (AAS). Moreover, the bacterial and fungal contaminations were tested for some of the selected plants in Palestine. The procedures of microbial and elemental testing of the plants followed USP.

The result of the heavy metals testing showed that copper and cadmium were above the allowable limits in all the tested plants. Zinc metal was above the allowable limit in 78.9% of the tested samples. The microbiological results of the tested plants showed that 63.2% of the tested plants were contaminated by bacteria and 89.5% were contaminated by yeast.

Herbal medicine used in the Palestinian markets doesn't meet the international requirement for heavy metal and microbiological limits. Therefore, urgent action has to be taken by the responsible authorities including the Ministry of health to implement importation and registration requirements and perform regular quality checks of sold and imported herbal medicines. Pharmacists as expert professionals must take an active role in selling and advising consumers about the quality and efficacy of the sold plants.


**OP – 7**

# Development of Gravimetric Nanosensor for Equilin Detection

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Natural water resources are contaminated by equilin and other estrogenic hormones that are naturally found in animal urine and are widely used in postmenopausal hormone supplements in women. These chemicals, which are applied directly to the soil or plants, are rapidly transported to groundwater.[1] US Environmental Protection Agency (EPA)'s health risk value of equilin in water is 0.35 µg/L.[2] Estrogens has been traditionally detected solid phase extraction systems, mass spectrometry and liquid chromatography.[3] The QCM sensor, which is a gravimetric sensor, has a wide potential of use for many applications and allows working with very small amounts of samples.[4] Therefore, unlike traditional QCM sensor applications, we have developed an affinity-based system that detects depending on the amount of mass moving away from the surface. Firstly, gold-coated AT cut QCM quartz crystals were modified with 11-MUA (11-Mercaptoundecanoic acid) molecules. Then, quartz crystals were incubated with amino acids (separately Phenylalanine, Tyrosine and Tryptophan) which adhere to 11-MUA with weak interactions. Finally, the equilin solution flowed through the sensor surface. Since the interaction between equilin and amino acids was stronger than the interaction between amino acids and 11-MUA, the amino acids attached to the quartz crystal were removed while the equilin solution flowed through the sensor. The resonance frequency shift is measured in real time. According to the results, the maximum mass changing in average 6 minutes were found as 147.44, 130.39 and 63.44 ng/cm$^2$ for Equ with Tyr, Trp and Phe modified QCM nanosensor, respectively. The lowest LOD (4.59 ng/L) and LOQ (15.31 ng/L) values were observed for the Tyr-modified QCM nanosensor for the detection of Equ.[5] It is thought that this gravimetric sensor system, developed for Equilin estrogenic hormone, will make a great contribution to the literature.

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**OP – 8**

# Electrochemical sensor based on molecular imprinted polymer/salinized graphene oxide composite for detection of 17 β Estradiol from wastewater samples

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In recent decades, endocrine disrupting chemicals (EDCs) have been considered as one of the environmental pollutants, widely exists in foods, river and soil [1]. Indeed, EDCs have gained more attention by the researchers in the field of environmental sciences because of their release in the environment without treatment. Among these EDCs, Estradiol, called also (17 β estradiol) is a natural steroid estrogen [2]. E2, one of the sex hormones, has the greatest physiological activity among naturally occurring estrogens [3]. It is primary female sex hormone (17-β-estradiol) and essential for the development and maintenance of female reproductive tissues such as the breasts, uterus, and vagina during puberty, adulthood, and pregnancy. The analyte (17-β-estradiol) is biosynthesized from cholesterol through a series of chemical intermediates. Levels of 17-β-estradiol in premenopausal women are highly variable throughout the menstrual cycle and the reference ranges widely vary from source to source [4]. In this study we develop the novel composite based on imprinted polymer on the surface of reduced graphene oxide for the selective detection of 17-β-estradiol. The two phase (organic and aquous phase) polymerization method was used to synthesized the imprinted molecule on to the surface of reduced graphene oxide. For this purpose, firstly the graphene oxide was synthesized by Hummer's method and then it was reduced by 3-(trimethyoxysilyl) propyl methacrylate (3-MPS). The molecular imprinted polymerization was done on the surface of reduced graphene oxide to achieve the large surface area for the recognition of analyte 17-β-estradiol. Different techniques were used to characterize the Molecular Imprinted polymer on selinized Graphene Oxide (MIP/SGO) composite including Fourier transform infrared spectroscopy (FTIR), Surface enhanced microscopy (SEM), and Raman spectroscopy. The results obtained from FTIR confirmed the successful synthesis of all the materials. Raman results further confirmed fabrication of single layered GO and polymeric composites. Differential pulse voltammetry (DPV) was used to quantify the amount of 17-β-estradiol. The essential parameters such as type of electrolyte, pH of electrolyte, scan rate and potential range were thoroughly optimized. 17-β-estradiol produced good DPV response using phosphate buffer of pH 6.5 at scan rate of 80 mV/s and potential range of 0.1 V to 0.7 V. The developed method was comprehensively validated and found to be linear in the range of 0.25 µg/mL to 0.81 µg/mL of 17-β-estradiol, with 0.08 µg/mL limit of detection and 0.25 µg/mL limit of quantification, respectively.

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**OP – 9**

# Source Identification of Polycyclic Aromatic Hydrocarbons (PAHs) in Surface Sediment Samples in The Golden Horn, İstanbul

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**Abstract**

The present study was conducted in Haliç (the Golden Horn) Estuary in İstanbul. Surface sediment samples were taken from upper, middle, and lower basins (3 different stations) of the Golden Horn in February 2013 and January 2014. After the ultrasonic extraction and clean-up stages, PAH analysis were done. Samples were taken into liquid phase by using gas chromatography Flame Ionization Detector (GC-FID). For 16 PAH congeners on surface sediment samples, T-PAH concentration were ranged between 29.3 and 43.1 ng/g for especially 2013 and 2014 winter seasons. Average T-PAH concentrations were considered according to the basins. The highest T-PAH concentration was detected in the Middle Basin in winter of 2014. During 2013 and 2014, PAH pollution source in the Golden Horn sediment was pyrogenic, in respect to Low Molecular Weight/High Molecular Weight and Phenanthrene/ Anthracene rates.

## Introduction

Located in the north of the Marmara Sea and in the southwest of Istanbul, Haliç (the Golden Horn) estuary has been a center of attraction for the city of İstanbul since its early years. In the past, it was reported that $3\times10^5$ $m^3$ of fresh water per day from Alibeyköy and Kağıthane streams flowed into the Golden Horn[1]. The discharges decreased considerably at the end of the 1990s[2]. In the following years, the only main fresh water source entering the Golden Horn became rain, and uncontrolled anthropogenic discharges[3]. The Golden Horn is one of the coastal plains that offers a wide range of services to the community. Until 1985, nearly 700 industrial facilities and more than 2000 workplaces were established in the region. The active operation of many shipyards, factories, and warehouses increased the pollution of the estuarine waters causing the reduction or extinction of fish species, accordingly an ecological degradation[4].

Polycyclic aromatic hydrocarbons (PAHs) are a large group of organic compounds formed by the fusion of two or more aromatic chains. Most PAHs are not easily soluble in the water; however, some evaporate easily into the air and most do not burn easily. The International Agency for Research on Cancer (IARC) has stated that a number of polycyclic aromatic hydrocarbons are 'probably carcinogenic to humans', while others are 'potentially carcinogenic in humans'[5].

PAHs occur naturally[6], as well as they are formed following incomplete combustion, especially in urban and industrial areas such as combustion of wood, coal, and petroleum derivatives (pyrogenic sources)[7,8]. Oil spill

after tanker accidents, unwanted leakages from petroleum pipelines etc. are other sources of PAH contamination (petrogenic sources). Also, petroleum refineries are important sources of PAHs pollution in marine ecosystems[9].

## Materials and Methods

The Golden Horn is an 8 km long branch of the İstanbul Strait with its 2.6 $km^2$ surface area. The Golden Horn consists of 3 different basins called as Upper Basin, Middle Basin, and Lower Basin. Its width varies between 293 m and 685 m and depth between 1 m and 40 m. The deepest part of the estuary reaches up to 60 m under the Galata Bridge [4,10](Fig 1).


**Figure 1.** The Golden Horn and sampling stations (S1: Sirkeci, S2: Fener, S3: Sütlüce)

Field studies were carried out in February 2013 and January 2014 (Fig 1). Surface sediment samples were collected from the Upper, Middle, and Lower Basins of the Golden Horn (in this paper, the average of the T-PAH concentrations measured in the surface sediments collected from the basins was calculated and given as a single value over the years). Surface sediment samples were lyophilized in the laboratory, dry weight percentages were calculated and stored at -86 °C until the analysis. Sediment samples were concentrated after extraction and clean-up and the final volume decreased approximately 1 mL. PAH measurements were conducted using gas chromatography (GC) device equipped with a flame ionization detector (FID). The total of the 16 PAH congeners listed as priority pollutant PAHs by the US-EPA was determined as the T-PAH value. The rate of low molecular weight (2-3 ring) PAHs (LMW) to high molecular weight (4-6 ring) PAHs (HMW) was used to estimate the source of PAH contamination. If the LMW/HMW rate was lower than 1, PAHs were originated by pyrogenic. If the LMW/HMW rate was greater than 1, PAHs were originated by petrogenic[11,12]. In addition, PAHs were considered mainly pyrogenic PAHs if the Phenanthrene/Anthracene (Phe/Ant) rate < 10, while PAHs were considered to be of petrogenic origin if the Phe/Ant rate was > 10[13].

## Results and Discussion

Total mean PAH values in the surface sediment samples were 29.3 ng/g in Winter 2013 and 43.1 ng/g in Winter 2014 (Fig 2).

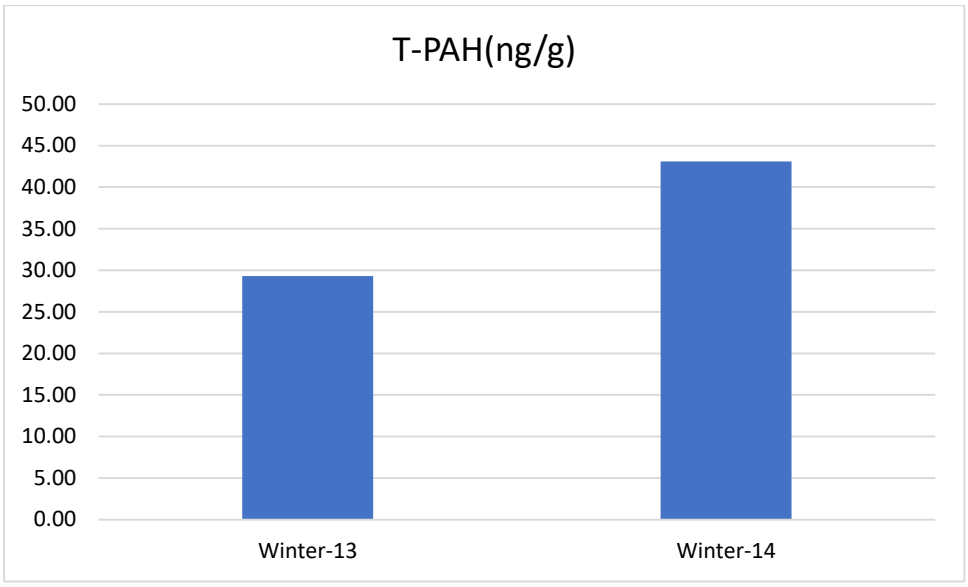


**Figure 2.** Mean T-PAH concentrations (ng/g dry weight) in the Golden Horn surface sediment samples in winter 2013 and winter 2014.

The average LMW/HMW rate was calculated as 0.09 in 2013 and 0.24 in 2014, while the Phe/Ant rate was 1.28 in 2013 and 0.9 in 2014 (Fig 3).

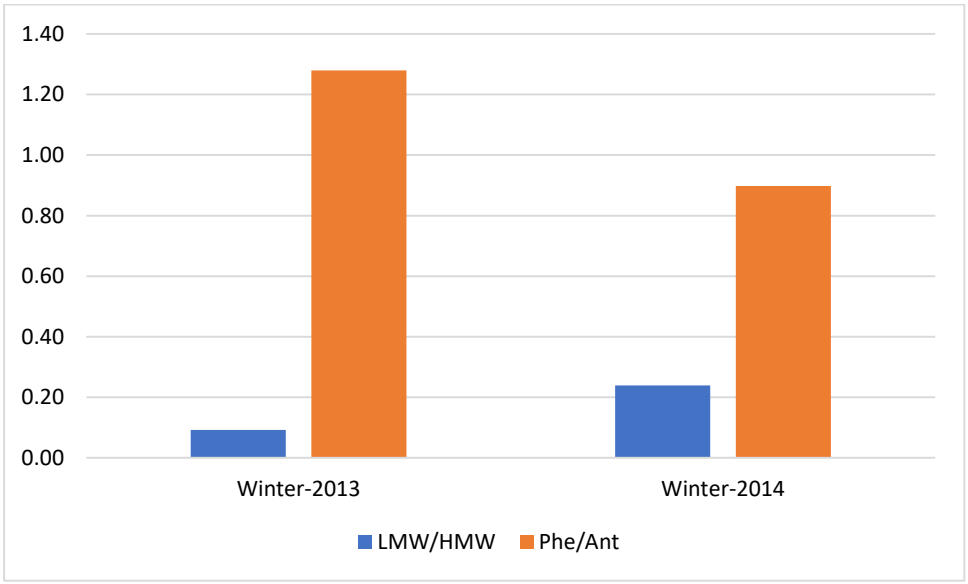


**Figure 3.** Calculated LMW/HMW and Phe/Ant rates for the surface sediment samples of the Golden Horn in winter 2013 and 2014.

Based on the data obtained, the source of PAH contamination was estimated. In 2013 and 2014, LMW/HMW rates are less than 1 and Phe/Ant rates are less than 10. According to the obtained data, the T-PAH contamination is of pyrogenic (combustion) origin in both years.

In the present study, the average T-PAH concentrations were higher in 2014 compared with the 2013 concentrations in the Golden Horn surface sediments. These data indicate that the pollution is especially resourced from combustion processes, and those discharges to the Golden Horn was higher in 2014 than in 2013. Also, their long-term half-lives[14] should be taken into consideration to assess their levels in the environmental samples. High concentrations of T-PAHs were measured in Winter 2014, especially in the Middle Basin, where the Golden Horn has hosted factories in the past. T-PAH source was pyrogenic both in 2013 and 2014 according to LMW/HMW and Phe/Ant rates. It can be explained by the presence of pyrogenic sources such as settlements, highways, and ship traffic in the Golden Horn[15]. According to a study conducted in the Golden Horn[15]in 2019, the PAH pollution in the Golden Horn sediment was found much higher (7893 µg/kg dw on average) than the T-PAH concentrations of the present study. In the same study, according to the LMW/HMW rate, the source of T-PAH is pyrogenic in accordance with the present study. On the other hand, the Phe/Ant rate indicated that the source of T-PAH was petrogenic, unlike the present study[15]. In petrogenic pollution, low molecular weight of PAHs in sediment are present in higher amounts than high molecular weight of PAHs[13]. Accordingly, in the present study, HMW PAHs were in higher concentrations than LMW PAHs in the sediments at all stations, supporting the pollution being pyrogenic.

According to the PAH pollution classification scale proposed by Baumard et all (1998), all three stations studied were slightly contaminated in both periods, with a total PAH concentration of 0–100 ng/g dry weight[16].

## Conclusion

Although the pollution in the Golden Horn sediment is classified as light contamination, contamination caused by combustion over time may pose a problem for the estuary ecosystem due to bioaccumulation. For this reason, inputs from domestic discharges, highways, and ship traffic to an important ecosystem such as the Golden Horn should be reduced as much as possible and the pollution over time should be supported by biomonitoring studies.

## Acknowledgments

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**OP – 10**

# Development an Analytical Method for the Determination of Silver in Metal Plating Wastewater by Magnetic Nanoparticle Based Dispersive Solid Phase Microextraction-Slotted Quartz Tube-Flame Atomic Absorption Spectrometry

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The recent development of industrial studies has increased the need for metals such as gold, silver, platinum, and palladium in industries where precious metals are used. The widespread use of these metals in many industrial areas due to their physical and chemical properties confirms this demand[1]. In this study, an analytical method is presented for the determination of silver, whose compounds carry possible risks in terms of human health and the environment, in the flame atomic absorption spectrophotometer system. Magnetic nanoparticles, whose surface area was increased by coating with stearic acid, were used as a sorbent for the preconcentration of silver. The slotted quartz tube was placed in the burner head to minimize the disadvantages of nebulization efficiency by increasing the analyte atoms' dwell time in the light path. After optimizing the parameters that play an important role in the extraction efficiency, the analytical performances of the system were determined. The applicability of the method developed was figured out in recovery experiments using metal plating wastewater[2].

**Acknowledgement:**

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**OP – 11**

# Anthropogenic and Biogenic Volatile Organic Compound Concentrations and Indoor Air Quality in Working Places in Bolu

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In this study, the indoor air quality of workplaces was evaluated using the concentrations of anthropogenic and biogenic volatile organic compounds. Samples were collected by Tenax-TA sampling tubes in a photocopy center, a painting class, two print-making classes, a tire shop, and a car painter by using the active and passive sampling strategy simultaneously. The outdoor samples were also collected. The sampling type and sampling time were optimized according to the characteristics of the working places. Volatile organic compounds were analyzed by TD-GC-MS. Totally, 217 samples were collected. Active sampling strategies were more suitable for the determination of air quality in the workplaces. The sampling time for active sampling varies between 15 minutes and 4 hours. Anthropogenic VOCs like isoprene, carbon tetrachloride, benzene, heptane, toluene, ethylbenzene, nonane, m+p-xylene, o-xylene, styrene, 1,2,3-trimethyl benzene, benzaldehyde, 1,4-cineole, 1,4-diethyl benzene, 1,3-diethyl benzene, phenol, acetophenone, dodecane, tridecane, tetradecane and biogenic VOCs like hexanal, heptanal, alpha-pinene, dl-limonene, p-cymene, m cymene, sabinene, ocimene, eucalyptol, dihydromyrcenol, 1-octanol, linalool were detected. Concentrations of benzene, toluene, ethyl benzene, and xylenes in the car-painter sampling point were found to be higher than other sampling points. The plant-derived resins used in the paints, parfumes, and detergents were the sources of the biogenic VOCs in indoor air. The usage of solvents and the polymers were the main sources of the anthropogenic VOCs in indoor air. The effect of cigarette smoking and emissions from trees were observed to be dominant in the outdoor environment.


**OP – 12**

# Preconcentration of Manganese by Magnetic Colloidal Gel based Dispersive Solid-Phase Extraction Method

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Manganese (Mn) is known to be an important element in the body fuction with the ability to optimize enzyme function and the transfer of enzymes through cell membranes. It is needed for a regular amino acid, protein, carbohydrate and lipid metabolism. This element is an essential micronutrient for different animal and plants growth. Furthermore, this element is greatly important in bone and brain development alongside with the functionating of the nervous system[1-4]. Manganese is found as in the ionic form of $Mn^{2+}$ in water samples that causes water pollution[5]. For these reasons, the high accurate Mn determination at trace levels is mandatory. In this study, a useful, simple and efficient analytical method was developed for the determination of Mn in flame atomic absorption spectrophotometry (FAAS) system. Regarding this aim, an extraction tool which consists of a plastic sieve fitted between two syringes was built to achieve dispersive solid phase extraction. The extraction of Mn was conducted by the usage of magnetic colloidal gel (MCG), which was a mixture of cobalt magnetic nanoparticles (Co-MNP) and deep eutectic solvent. All the experimental parameters of the developed methods was optimized to obtain the most efficient manganese extraction conditions. The method detection limit was found to be 4.0 ng/mL under the optimum conditions of the method. For the conformation of the applicability and the accuracy of the analytical method, the optimum extraction was applied to jasmine tea extracts samples[6].

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**OP – 13**

# The Influence of Both Elevated Carbon Dioxide and Drought on Plants Polyphenols

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Plants react differently to abiotic stress conditions such as temperature, drought, and flooding. In the last years, the non-physiological conditions currently increased in severity. Depending on the species, some plants can adjust and tolerate climate change [1]. A current example of prolonged stress is this year's drought which impacted most worldwide crops. This stress factor is affected by the increased level of atmospheric carbon dioxide.

Along with them could also be mentioned the increased temperatures and solar energy, or in some cases the floods. The research should focus on developing methods to decrease their impact, considering the trends in stress factors. The first steps were made [2, 3], but optimizing the strategies is based on studying different species in variable conditions. Significant information is determined when several aspects are studied simultaneously. The approach enables an analysis closer to natural conditions, where multiple disruptive factors coexist. Based on these considerations, our study's main objective was to determine the effect of two abiotic stress factors on *Brassicaceae* family plants (*Brassica oleracea* var. *capitata*, *B*. *oleracea* var. *botrytis*, and *Raphanus sativus*). The research focuses on the modification that emerged at the antioxidant level. They are an important marker of the plant's defensive reaction toward biotic and abiotic disruptive factors[4]. The simulated working conditions assumed plants grown at elevated carbon dioxide (800 and 1200 ppbv) are less tolerant to drought stress conditions. The results showed that the polyphenols concentration in the plants grown at high carbon dioxide decreased compared to those grown under normal conditions. On the other hand, the plants treated with high carbon dioxide concentration have lower polyphenols content after drought than the control plants.

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**OP – 14**

# High Accuracy Determination of Trace Levels of Metobromuron by Gas Chromatography Mass Spectrometry After its Preconcetration by Dispersive Liquid Phase Microextraction

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Metobromuron is known to be a phenylurea herbicide used for the control of annual meadow grass and broadleaf weeds in vegetables and grain crops[1,2]. This herbicide poses a risk to animals and humans due to its high toxicity and carcinogenicity and even cause death[3–5]. The determination of low concentrations of phenylurea herbicides in agricultural samples is of primary importance due to the risks to the environment and human health. For this reason, the determination of metobromuron at trace levels with high accuracy and sensitivity was performed in this study. For this aim, metobromuron was preconcentrated by the dispersive liquid-liquid microextraction (DLLME) method, prior to the determination of the gas chromatography-mass spectrometry (GC-MS) system. Optimum values for each variable that can affect the DLLME procedure efficiency were determined by performing one-step-time manner optimization studies. After the optimum conditions were determined, the analytical performance of the developed system was evaluated with the samples applied DLLME method. Additionally, the demonstration of accuracy/applicability of the method developed was performed by the recovery experiments carried out in food samples with high recovery results obtained.

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**OP – 15**

# Investigation of Purification of an Aqueous Solution Containing Salt and Boric Acid by Electrodialysis with Bipolar Membrane

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In this study, bipolar membrane electrodialysis (BMED) of synthetically prepared NaCl and $H_3BO_3$ (boric acid) feed solution was examined for different flow rate and initial boric acid concentration. The resistance of the membrane stack, the desalination of the feed solution, and the transport behavior of boric acid in the membrane stack were examined in BMED. The electrodialysis cell consists of a membrane stack consisting of five repeating cells with an anode, cathode and three compartments. Each cell in the membrane stuck was formed according to the sequence BM//KC//AC//BM. In the study, 5 different initial concentrations of boric acid as a parameter (0 - 30 $g.L^{-1}$) and four different flow rates (10 L/h - 25 L/h) were selected. The obtained data show that the increased initial concentration of boric acid in the feed solution does not affect the a membrane stack resistance and desalination too much. At the same time, with the increasing initial amount of boric acid in the feed and the increasing flow rate, it has been found that boric acid is transported more from the feed compartment to the acid, base and electrolyte compartments. In addition, with increasing flow rate, the feed solution was further desalinated and a purer solution of boric acid was obtained. The results show that the BMED process can simultaneously desalinate the feed solution, purify $H_3BO_3$, and achieve high purity of $H_3BO_3$.

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**OP – 16**

# Cyanide Detection with UV-Vis Spectrophotometer Based on the Colorimetric Detection by Using Silver Nanoparticles

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Cyanide which may accumulated in air, soil and water has been a critical risk to living organisms since the discharge of cyanide anions containing mainly from wastewater sources [1]. Therefore, in this study, an effective and sensitive analytical method was developed for the quantification of $CN^-$ ions with the synthesized AgNPs (silver nanoparticles) by UV-Vis spectrophotometer. Colloidal AgNPs were synthesized via chemical reduction method by reducing silver nitrate. For this purpose, Triton-X-100 as a stabilizing agent and sodium borohyride as a reducing agent were used in the NP preparation. The UV-Vis absorption spectra exhibited a significant decrease in the SPR (surface plasmon resonance) absorption (at 405 nm) indicating the formation of yellow-brown colored AgNPs that can be the visually observed. In order to check the stability of the synthesized AgNPs, characterization was performed by using Zeta Potential analysis. All the optimum parameters that can affect the sensitivity and the selectivity of the developed method such as mixing type and period, AgNP volume and buffer type and volume were examined. Under optimal conditions, the proposed colorimetric sensor showed great linearity between 0.5 and 3.50 mM. Finally, cyanide sensing colorimetric method was successfully performed on different water samples. The results showed satisfactory recovery values that were nearly 100% demonstrating the applicability of the developed colorimetric sensor for accurate and precise determination of cyanide as a proposed analytical method.

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**OP – 17**

# $CoSn(OH)_6$ Nanocube Based Dispersive Solid Phase Extraction for the Sensitive Determination of Lead Ions in Environmental Samples by Flame Atomic Absorption Spectrometry

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Heavy metals, known as conventional pollutants, are highly toxic, non-degradable, and bioaccumulative[1]. Lead, one of these heavy metals, is widely available in the environment. Generally, people are exposed to lead and its compounds through ambient air, foodstuffs, industrial materials, drinking water, battery recycling, grids, arm industry, pigments, printing of books, and consumer products[2,3]. Exposure to lead in humans can cause a variety of effects, either acute or chronic. These effects include cognitive deficits, disorders of intelligence, memory, and attention; depression, anxiety, anemia; central nervous system disorders; and neurotoxic effects. Therefore, inorganic lead is classified as a potential human carcinogen by the International Agency for Research on Cancer[4]. In this study, a dispersive solid phase extraction strategy based on $CoSn(OH)_6$ nanocubes (NC) was developed for the preconcentration and separation of lead ions in environmental samples. For the first time, the $CoSn(OH)_6$ NCs were used as a solid sorbent for quantitative measurement of lead ions before flame atomic absorption spectrometry. The cube-like $CoSn(OH)_6$ nanostructures were synthesized by a stoichiometric co-precipitation method. The morphology and structure of the prepared sorbent were confirmed by X-ray diffraction, scanning electron microscopy, and Fourier transform infrared spectroscopy. SEM results proved that the prepared nanostructures were cube-like in structure and uniform in size and shape. Various experimental parameters affecting the extraction conditions of lead ions were evaluated using a univariate optimization strategy. The extraction studies indicated that $CoSn(OH)_6$ NCs have good accuracy, selectivity, and stability for the target ions. The reliability and of the analytical approach applicability were investigated using spiked environmental samples. These findings indicate that the presented method has been practicable and reliable for sensitive determination of trace levels of lead ions in the selected environmental samples with good recoveries.

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**OP – 18**

# Poly(vinyl alcohol)-Magnetic Hydrogel Based Dispersive Solid Phase Extraction Method for the extraction/preconcentration of Cobalt from Chamomile Tea Samples Prior to Flame Atomic Absorption Spectrophotometry

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As an essential element, cobalt undertakes many functions in the human system, such as stimulating hemoglobin (HGB) synthesis, contributing to blood formation, and functioning various hormones, vitamins, and enzymes[1]. Cobalt, which is a part of vitamin B12, is approximately 1.0 mg in a healthy human body[2]. This metal is taken into the human body with solid and liquid nutrients and creates a carcinogenic effect in high doses. Therefore, it is crucial to detect this element at low amounts[2,3]. In this work, a simple and rapid analytical method was proposed for the determination of trace levels of cobalt with the flame atomic absorption spectrophotometry (FAAS) system after magnetic hydrogel-based dispersive solid phase extraction (DSPE). Poly(vinyl alcohol)-based magnetic hydrogels used as adsorbent for the DSPE method were synthesized cheap, quickly and easily in laboratory conditions. In order to obtain the highest enhancement in detection power, univariate optimization studies were applied on extraction parameters such as sample volume, buffer pH and volume, amount of sorbent material, mixing type/period and desorption solvent concentration/volume. Analytical performance evaluation studies were carried out for the poly(vinyl alcohol)-magnetic hydrogel based dispersive solid phase extraction- flame atomic absorption spectrophotometry (PVA-MH-DSPE-FAAS) system under the obtained optimum experimental/instrumental conditions. As a result of the studies, the linear range was obtained between 15 and 150 µg/L. Comparing the LOD of the FAAS system and the proposed method resulted in a 57.8-fold enhancement in the cobalt detection limit. In order to assess the applicability of the development system to real samples, recovery studies using chamomile tea samples were conducted. In this experiments, satisfactory recovery results indicating the accuracy of the proposed method were obtained.

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**OP – 19**

# Chromium Speciation in Soil, Water and Grass Samples by HPLC-ICP-OES

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Chromium contamination has gained substantial attention in recent years due to its high amount of presence caused by natural and anthropogenic sources[1]. It is commonly found in environmental bodies as trivalent and hexavalent states[2]. Hexavalent chromium is 100 times more toxic than its trivalent state[3,4]. It is well-known that hexavalent chromium can put human health at risk because of its teratogenic, mutagenic and carcinogenic effects on human bodies[1]. For this reason, chromium speciation is a need rather than total chromium determination due to the differences of chromium species in terms of their toxicity and biochemical properties. First, high performance liquid chromatography was coupled to inductively coupled plasma-optical emission spectrometry for the chromatographic separation and spectroscopic detection of trivalent and hexavalent chromium. Next, important parameters such as type of analytical column, pH and flow rate of mobile phase, injection volume were elaborately optimized by univariate approach. The optimized HPLC-ICP-OES method yielded limit of detection for trivalent and hexavalent chromium as 0.27 and 0.05 mg/kg, respectively. Accuracy and applicability of the developed method was tested by spiking experiments on soil, water and grass samples collected from Tuzla Organized Industrial Region (Tuzla, İstanbul). Percent recovery results were obtained between 88 and 104% by the strategy of matrix matching calibration for all selected samples[5].

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**OP – 20**

# Fate Analysis of Selected Pharmaceuticals in Erzurum Biological Wastewater Treatment Plant by Using Toxchem Modelling Method

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## Abstract

Mathematical modellings have become a widely used to evaluate the fate of new emerging micropollutants, especially pharmaceuticals, in the aquatic environment or in the wastewater treatment plants (WWTPs) in recent years. As one of most common used, the Toxchem offers several fate mechanisms for the micropollutants into process specific mass balance equations with their known physical, chemical and biological degradation properties. The mathematical basis of the micropollutants process simulation in the WWTPs is based on the mass transfer equations and the mass balances of the pollutants in each treatment unit, which include volatilization (including air stripping and surface evaporation), sorption processes and removal mechanisms by biodegradation (biodegradation) to sludge. Thus, the concentrations of some pharmaceuticals such as diclofenac, naproxen, sertraline, paracetamol and metformin were selected and used as target micropollutants in Erzurum Biological WWTP to determine the treatability and fate of the some micropollutants. Then, the WWTP was simulated by Toxchem software with these micropollutant concentrations and the WWTP operating parameters i.e., flow rate, inlet micropollutant concentration, temperature, and SS. Finally, the fate analysis of micropollutants diclofenac, naproxen, sertraline, paracetamol and metformin in the Erzurum Biological WWTP were modelled via the results of the Toxchem method. For this purpose, the pharmaceuticals were analyzed by LC-MS/MS method with liquid-liquid extraction in wastewater samples in one year period.

**Keywords:** Toxchem Modelling, WWTPs, Micropollutants, LC-MS/MS

## 1. Introduction

Micropollutants (MPs) consist of many organic or inorganic compounds in a wide range of natural compounds such as steroid hormones, which are caused by human activities and found in low concentrations in aquatic environments, and synthetic compounds such as pharmaceuticals, industrial chemicals. MPs emerged in aquatic environments for the first time in the 1970s-1980s when steroid hormones were detected in wastewater treatment plants and are generally present in trace concentrations ranging from ng/L to µg/L. However, due to the increasing diversity of MPs and intense production and consumption activities in recent years, their concentrations exceeding trace amounts have caused them to become a growing environmental threat on a global scale. The diversity and low concentrations of MPs not only complicate the relevant detection and analysis procedures, but also create difficulties in treatment for water and wastewater treatment processes. Therefore, there is different classification for (MPs); Pharmaceuticals and Personal Care

Products (PPCPs), sterodis and hormones, surfactants, detergents etc., industrial chemicals (BPAs, Phthalate) and pesticides (Luo et al. 2014; Tüzün 2017).

Although there have been many studies on MPs, they have been ignored for many years due to their low concentration in the aquatic environment and limited information about their toxicity. In addition, since conventional urban wastewater treatment plants are not designed for the treatment of micropollutants and monitoring and prevention actions are not established for these micropollutants, most of them can pass through wastewater treatment processes thanks to their persistence and/or their continuous entry into the plant. As a result, many of MPs enter the receiving water environment and thus become a threat to both the natural aquatic environment and drinking water. The presence of MPs in the receiving water environment is associated with many effects such as short and long-term toxicity, endocrine disrupting effects and increased antibiotic resistance of microorganisms. Information on the effects of long-term exposure to low concentration levels of MPs is still very limited since the effects on the environment and human health are still not fully determined (Fent et al. 2006; Rogowska et al. 2020).

Concerns about the increasing presence of MPs in aquatic environments as a result of their increasing concentrations and use around the world, along with the production of a large number of new molecules by the drug and chemical industries, has been the subject of many research in the last two decades. Data on the level of chemical concentration in different water sources are insufficient to investigate the risk to the environment and only provide specific information about potential threats to humans and ecology. As a result of the environmental risk assessment conducted to investigate the effects of micropollutants on plants, human health, ground/surface water quality and water species, a wide range of diseases that can be caused by exposure to micropollutants have been revealed. Studies show that micropollutants can cause significant long-term effects and cause irreversible mutations in human and natural life (Khan et al. 2021). American Environmental Protection Agency (USEPA) in 2006 stated that 7 active pharmaceutical ingredients and 12 personal care products were detected in fish tissues (EPA 2013). Drug consumption on a global scale varies greatly from country to country, and the high sales of over-the-counter drugs in many countries cause uncertainty about consumption estimates and usage rates. Cholesterol, diabetes, hypothyroidism, hypertension, cardiovascular drugs, respiratory tract drugs, stomach drugs, epilepsy-nervous system drugs and antibiotics are still on the list of the most widely used prescription medical drugs all over the world (Anonymous 2022a). Bioaccumulation studies also reveal potential toxic effects of pharmaceuticals to aquatic organisms. Because many pharmaceuticals exist at low concentrations in the aquatic environment for long periods, their toxic effects are more likely to be chronic than acute. The toxicity of pharmaceuticals in aquatic environments varies depending on the type of pollutant, exposure time, concentration, and developmental process to which organisms are exposed (Khan et al. 2020).

In this study, the fate of some selected pharmaceuticals (diclofenac, naproxen, sertraline, paracetamol and metformin) as one of most popular group of MPs in Erzurum Biological Wastewater Treatment Plants (BWWTP), the main source from which they discharged to the aquatic ecosystem, were analyzed. The target pharmaceuticals were analyzed using the liquid-liquid extraction via LC-MS/MS method in wastewater samples taken from the inlet and outlet points of the plant for 12 months between December 2020-November 2021. In order to determine the treatability and fate of the target micropollutants in Erzurum BWWTP, the plant was modeled with Toxchem software by using these pharmaceutical concentrations and the operating parameters of the plant by conducting sensitivity analysis.

## 2. Materials and Methods

The target micropollutants were analyzed using the liquid-liquid extraction and LC-MS/MS method in wastewater samples taken from certain points, especially the entrance-exit points of the plant for 12 months between December 2020-November 2021. The samples were taken by expert from BWWTP in accordance with the sampling conditions and quickly delivered to the laboratory. Samples that could not be analyzed immediately were stored at +4 $^{o}$C for a maximum of 48 hours. LC-MS/MS (Agilent Technology 6460 Triple Quad LC/MC) analyzes were made in Atatürk University Central Laboratories (DAYTAM-Eastern Anatolian High Technology Application and Research Center). Liquid-liquid extraction method was used to separate and collect the target compounds in the samples before LC-MS/MS analysis. In this study, considering the methods used in the literature, methylene chloride (DCM) and ethyl acetate (EtOAc) solvents were used for the recovery of target compounds (Duca et al. 2014; Issa et al. 2020). With the obtained data, the removal mechanism of each target pharmaceuticals in the plant general and in the biological treatment unit was predicted and modelled using Toxchem 4.0 software.

## 3. Results and Discussions

Table 1 represents the operating parameters which were used in the Toxchem model and, the inlet concentrations of selected pharmaceuticals. The inlet concentration of pharmaceuticals was analyzed for 12 months period between December 2020-November 2021 via LC-MC/MS method.

**Table 1.** The operating parameters and inlet pharmaceuticals concentrations of Erzurum BWWTP

| **Modelling Parameters** | **Unit** | **Range** | **MCs** | **Unit** | **Inlet Conc. Range** |
|---|---|---|---|---|---|
| **Flow Rate** | $m^3$/day | 65,000-93,000 | **Diclofenac** | µg/L | 0.16-140 |
| **TSS** | mg/L | 50-250 | **Paracetamol** | µg/L | 1.00-4,000 |
| **Temp.** | $^{o}$C | 10-20 | **Naproxen** | µg/L | 0 – 80.666 |
| | | | **Sertraline** | µg/L | 0.275 – 2.816 |
| | | | **Metformine** | µg/L | 0.828 – 398.4 |

Therefore, the treatability of pharmaceuticals in WWTPs varies in a wide range considered their specific chemical properties and removal pathway. Toxchem reveals the treatment pathway by defining the fate analysis in the WWTPs. Toxchem helps to find out the most effective treatment percentage for pharmaceuticals by changing plant operating parameters. Toxchem has been derived based on basic mass transfer equations and mass balances, which include evaporation (including air stripping and surface evaporation), sorption processes and removal mechanisms where transfer to sludge occurs by biodegradation (biodegradation). Volatilization and sorption occur as the transfer of micropollutant between

dissolved gas and dissolved-solid surfaces, while biodegradation occurs by removal of micropollutant from dissolved or solid matter. Therefore, Table 2 shows that this fate analysis of selected pharmaceuticals in the plant and the biological treatment unit. According to model results, metformin, sertraline and diclofenac are mostly absorbed to the sludge in the BWWTP; while naproxen is transferred to the water media to be able to biologically degrade in the biological treatment unit. Paracetamol is almost half, and half absorbed to the sludge and to the biological treatment unit. The air transfer of selected pharmaceuticals had very small percentage because, generally, evaporation and stripping of pharmaceuticals can be negligible because their $K_H$ is less than $10^{-5}$ atm-$m^3$/mol. $K_H$ is the Henry's law constant also called the air–water partition coefficient that is the ratio of a compound's partial pressure in air to the concentration of the compound in water at a given temperature (Rodrigues dos Santos, 2022).

**Table 2.** The Toxchem model results for Erzurum BWWTP

| MPs load, µg/L | To Air, % | To WW, % | To Sludge, % | To Oil, % | Air Treatment, % | Biodegraded, % |
|---|---|---|---|---|---|---|
| **Paracetamol** | 1.62 | 0.16 | 4.67 | 0 | 0 | 93.55 |
| **Diclofenac** | $4.88x10^{-4}$ | 34.78 | 62.73 | 0 | 0 | 2.49 |
| **Naproxen** | $1.1x10^{-3}$ | 57.15 | 23.74 | 0 | 0 | 19.11 |
| **Sertraline** | 0.07 | 9.30 | 90.34 | 0 | 0 | 0.29 |
| **Metformin** | 0.051 | 8.05 | 91.12 | 0 | 0 | 0.78 |

## 4. Conclusions

According to the rule of $K_{OW}$ coefficient, if logKow<2.5, it indicates low sorption potential, if logKow is in the range of 2.5-4, it indicates medium sorption potential and if logKow> 4, it indicates high sorption potential (Akkurt and Oğuz 2019). When the $\log_{KOW}$ coefficients of pharmaceuticals were examined; the lowest $\log K_{OW}$ belongs to paracetamol and is 0.46, so the lowest absorption rate to sludge was found to belong to paracetamol (4.67%). The $\log K_{OW}$ coefficients of diclofenac was 4.51 that indicates medium sorption ability. As a matter of fact, the sludge absorption of diclofenac was estimated as 62.73% with the Toxchem model. With the regard of the Toxchem model results, the percentage of pharmaceuticals to be absorbed into the sludge in the BWWTP was estimated in the order of metformin>sertraline>diclofenac>naproxen>paracetamol, respectively. While the percentage of leaching to air was negligible for all three compounds, the percentage of biodegradation of residual pharmaceuticals in the biological treatment unit was modeled as highest in paracetamol as 93.55%. While diclofenac, metformin and sertraline had almost non-biodegradability capacity.


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**OP – 21**

# The Use of Maquis as Bio-monitors in Air Pollution Monitoring: The Case Study of Antalya

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Biomonitoring is a viable method to monitor air pollution in many ways. Biomonitoring does not require expensive equipment, gives long-term results, is sustainable, and is environmentally friendly. This study was carried out in Antalya, Turkey. The maquis plant, unique to Antalya's vegetation, was chosen as a bioindicator.

In this study, maquis plants were collected monthly for five months (December 2021-April 2022) from four different areas to determine heavy metal pollution from the industry, traffic, urban background, and the public's impact on the natural environment.

The collected samples were analyzed with the ICP/MS device in the METU Central Laboratory. ICP/MS analyses were performed for the following heavy metals; Ni, Co, Zn, Cd, Cr, Cu, Pb, V, As, and Al.

The results were evaluated according to seasonal and geographic variations. In addition to this, the enrichment factor was applied to the results.


**OP – 22**

# Sensitive and Selective Spectrophotometric Determination of Cyanide by A New Benzothiazole Compound

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Among all the anions, cyanide is one of the most toxic and hazardous, causing serious clinical effects that are frequently fatal.[1] In spite of its toxicity, its application in various areas as raw material for synthetic fibers, resins, herbicides, metal, dye, drug, leather, textile and mining industries is unavoidable, which releases cyanide into the environment as a toxic contaminant.[2] The maximum cyanide concentration is 1.9 µmol $L^{-1}$ in drinking water permitted by the World Health Organization (WHO). In view of its toxicity and extensive applications in industries, the development of sensitive methods for detection of trace amounts of cyanide is of great significance.[3] Techniques like UV-Visible spectroscopy, fluorescence spectroscopy, atomic absorption spectroscopy, ion-exchange chromatography, etc. have been employed extensively to study the interaction between different molecules and cyanide ion in several types of medium. Among these, visible change of color is the most convenient method for the detection of cyanide ions.[4]

In this study, a benzothiazole compound was used for the determination of cyanide in water samples. The newly synthesized molecule was characterized by FT-IR, elemental analysis and $^1$H-NMR and the sensing properties were investigated by UV-Visible absorption spectroscopy. The molecule exhibited good selectivity to cyanide ions and its color changed from colorless to yellow in the presence of cyanide. From Job's plot, binding stoichiometry was found as 1:1. It was observed that the detection limit is lower than the maximum permissible level of cyanide in drinking water set by WHO.

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**OP – 23**

# New Type of Synthetic Graphite Synthesis Originated from Low-Grade Coal for Effective Removal of Nonylphenol Ethoxylates

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Nonylphenol ethoxylates (NPEs), a type of xenoestrogens, are non-ionic surfactants. NPEs can find place for themselves many different areas such as industrial, domestic, agricultural, and commercial applications due to their low cost and chemical properties. The use of NPEs result in the discharge of these pollutants to the environment via wastewater route and eventually reach to water bodies. The presence of NPEs in water bodies such as rivers, lakes, and groundwater may have a toxic and estrogenic effect on fish and aquatic organisms. Thus, NPEs and its degradation products are required to be treated to prevent their potential to create highly toxic effects on human and ecosystem before being released into the environment. Even NPEs reach to WWTPs, the conventional systems lack the capability of treating Endocrine disrupting chemicals. On the other hand, adsorption process has been applied in wastewater treatment for the removal of pollutants due to its simplicity of application, low cost, and high removal efficiencies obtained. This study investigated the potential for the treatment of NPEs using synthetic graphite originated from lignite as an absorbent.

A novel synthetic graphite originated from lignite (EGL) was synthesized by electrochemical methods with sulfuric acid treatment. The EGL was characterized by XRD, SEM, BET and FTIR and, then used as an adsorbent for batch removal of nonylphenol Ethoxylate (NPE) from aqueous solutions. The effective factors such as pH, contact time, initial concentration, temperature on the NPE removal by EGL was studied. The adsorption equilibrium data of NPE using EGL was best fitted to Langmuir isotherm providing with maximum adsorption capacity of 17.18 mg/g. Adsorption kinetics of NPE was followed to pseudo-second-order kinetic model. The NPE adsorption onto EGL was reached to 98% at 5 min of contact time indicating a rapidity of the process. The percent adsorption of NPE was found to be higher than 92% at wide pH values ranged between 2.0 and 10.0. The results clearly revealed that synthetic graphite originated from lignite has good potential for effective removal of NPE from aqueous solutions.


**OP – 24**

# Invasive Biofouling Marine Species in The Izmit Bay

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**Abstract**

For centuries, organisms being carried between far corners of the earth unintentionally by travelers, traders, and armies. These exotic species often found new habitats to settle and flourish. Some of them became too successful in adapting to new environments and became invasive species. Invasive species often harm the ecosystem and can be threatening to native species. The main transport routes for marine invasive species are artificial waterways (e.g. Suez Canal, Panama Canal) and international sea vessels.

Fouling marine organisms cover submerged surfaces of infrastructure and vessels causing both economic and ecologic problems. Due to their ability to adhere to the hulls of ships, fouling organisms are likely candidates to be transferred to new marine habitats. Also, ballast water is another possible source for their transport. Marine ecosystems with heavy marine traffic like the Marmara Sea are susceptible to exotic species more than other regions.

A total of 19 locations in the İzmit Bay (Marmara Sea) were surveyed by SCUBA divers between 2019 and 2022. Four different exotic biofouling species; *Magallana gigas* (Bivalvia), *Rapana venosa* (Gastropoda), *Amphibalanus improvisus* (Crustacea), and *Styela clava* (Tunicata) were observed on the submerged infrastructure of harbors and piers. These species are causing problems in the trophic system by competing with native species. Also, by adhering/boring to artificial structures, they cause corrosion in these structures and the release of unwanted chemicals like paint residues and metal oxides.

## Introduction

For centuries, organisms being carried between far corners of the earth unintentionally by travelers, traders, and armies. These exotic species often found new habitats to settle and flourish. Some of them became too successful in adapting to new environments and became invasive species. Invasive species often harm the ecosystem and can be threatening to native species. Also, some invasive species could have habitat-altering effects on the ecosystem. The main transport routes for marine invasive species are artificial waterways (e.g. Suez Canal, Panama Canal) and international sea vessels.[1,2]

Fouling marine organisms cover submerged surfaces of infrastructure and vessels causing both economic and ecologic problems. Due to their ability to adhere to the hulls of ships, fouling organisms are likely candidates to be transferred to new marine habitats. Also, ballast water is another possible source for their transport. Marine ecosystems with heavy marine traffic like the Marmara Sea are susceptible to exotic species more than other regions. [3,4]

Marmara region houses nearly 1/3 of Turkey's population and it's the most industrially developed region of the country. As the eastmost part of the Marmara Sea, İzmit Bay not only houses the largest freeports in Turkey but also is the main sea-to-land passage to Anatolia. Therefore, 19 locations along the shores of İzmit Bay were surveyed for some invasive biofouling species within the scope of this study.

**Materials and Methods**

Between 2019 and 2022, 19 locations were surveyed by SCUBA divers (Figure 1). All locations were chosen from industrial zones (harbors, piers, docks, and embarkments) along İzmit Bay. Surveyed location depths ranged between 6-30 m.


Figure 1 Surveyed locations along The İzmit Bay

Two SCUBA divers examined the seafloor and surface of man-made structures for *Magallana gigas* (Bivalvia), *Rapana venosa* (Gastropoda), *Amphibalanus improvisus* (Crustacea), and *Styela clava* (Tunicata) in each location. Target species were photographed with underwater cameras in situ and sampled if needed (Figure 2).

Figure 2 Underwater survey

## Results and Discussion

All four species were found to be widely spread along İzmit Bay. *R. venosa* was found in 18 locations as the most diverse species and *A. improvisus* was found in 15 locations as the least diverse species (Table 1).

Table 1 Distribution of invasive biofouling species in The İZmit Bay

| | | Biofouling Species | | | |
|---|---|---|---|---|---|
| | | ***Magallana gigas*** | ***Sytela clava*** | ***Rapana venosa*** | ***Amphibalanus improvisus*** |
| **Sampling Stations** | **A** | ● | | ● | ● |
| | **B** | ● | | ● | |
| | **C** | ● | ● | ● | ● |
| | **D** | ● | ● | ● | ● |
| | **E** | ● | ● | ● | |
| | **F** | ● | ● | ● | ● |
| | **G** | ● | ● | ● | ● |
| | **H** | ● | ● | ● | ● |
| | **I** | ● | ● | ● | ● |
| | **J** | | ● | ● | |
| | **K** | ● | ● | ● | ● |
| | **L** | | ● | | ● |
| | **M** | ● | ● | ● | ● |
| | **N** | ● | ● | ● | ● |
| | **O** | | | ● | |
| | **P** | ● | ● | ● | ● |
| | **Q** | ● | ● | ● | ● |
| | **R** | ● | ● | ● | ● |
| | **S** | ● | ● | ● | ● |

Sessile species (*M. gigas, S. clava, A. improvisus*) were mainly found on hard surfaces (Harbor infrastructure, concrete embankment blocks, etc.) while *R. venosa* was found mostly feeding on bivalve clusters and on the

seafloor, partially covered in sediment. Also, egg capsules of *R. venosa* were found on hard surfaces (Figure. 3-6)

Figure 3 *Sytela clava*

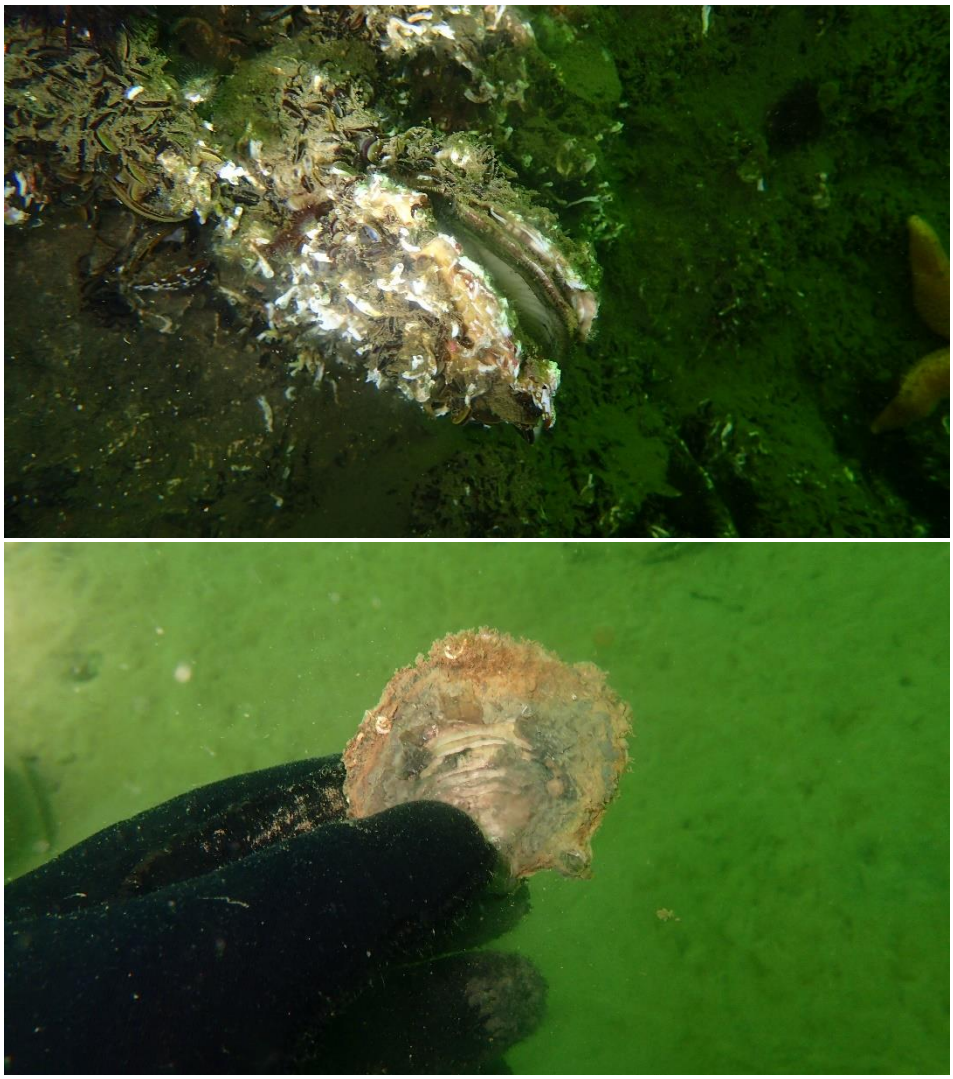

Figure 4 *Magallana gigas*

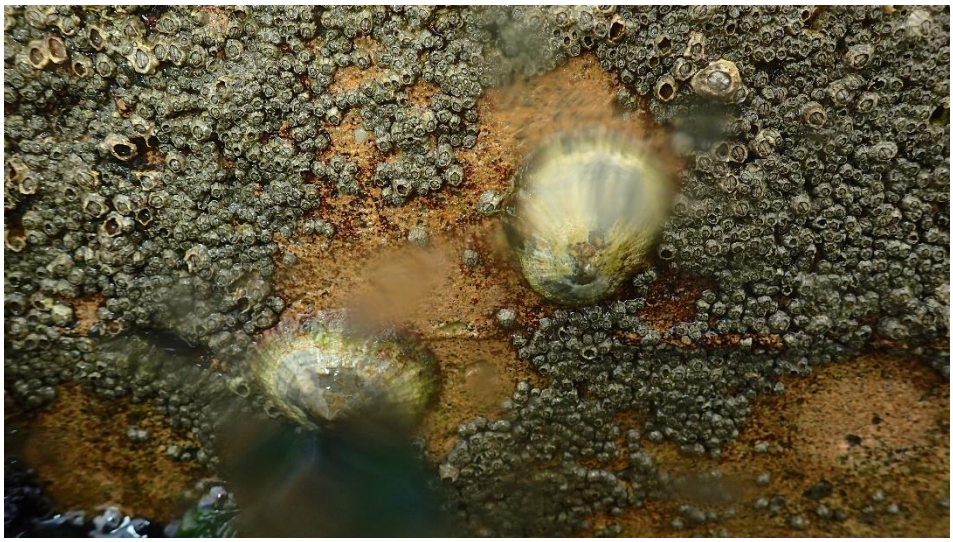

Figure 5 *Amphibalanus improvisus*

Figure 6 *Rapana venosa* and its egg capsules

## Conclusion

Biofouling species included in this study were widespread on the shores of İzmit Bay. In the survey of station "O" only *R. venosa* was observed. This location is different from other locations with its proximity to Hersek Lagoon. Low salinity and seasonal flow of fresh water in this location were expected. Also, only adult Rapana venosa was observed in this location. Sessile species could have difficulty to adapt seasonal differences in this area.

*M. gigas* is a reef-building bivalve with a thick hard shell. It can cause damage to infrastructure by altering its features (surface shape, depth, etc.). Also, surveys show that *M. gigas* is a rival for native bivalve species (Eg. *Mytilus galloprovincialis*) by sharing the same environment. On the other hand, M. gigas has economical value as seafood.

*S. clava* has a higher tolerance of environmental factors in comparison with native tunicate species. Also as a filter feeder, it in competition with other native species, sharing the same diet. *S. clava* was seen on hard surfaces during underwater surveys.

*A. improvisus* is found in abundance on the surface level of concrete infrastructure and ship hulls. It can rapidly take over living space of native species such as *Patella* sp. (Figure 5)

*R. venosa* is a voracious predator of the bivalve species. Many native bivalve populations were affected by R. venosa. Also, R. venosa lays its egg capsules on hard surfaces like ship hulls, therefore can transfer over shipping routes.

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**OP – 25**

# Evaluation of chemical composition and source apportionment of fractional rain samples collected in urban and semi-urban sites of Bolu

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In this study, volume-based wet precipitation samples were fractionally collected in May and June 2020 during COVID-19 lockdown period. A total of 167 samples were taken from Bolu city center and the Campus of Bolu Abant İzzet Baysal University, which are the urban and semi-urban areas of Bolu. One hundred twenty-one samples were obtained from the city center of Bolu whereas forty-six samples were obtained from the Bolu Abant Izzet Baysal University campus. Major ions ($F^-$, $Cl^-$, $NO_3^-$, $SO_4^{2-}$, $Na^+$, $NH_4^+$, $K^+$, $Mg^{2+}$, $Ca^{2+}$) and elements (Ag, Co, As, Cr, Pb, Ga, V, Cd, Mn, Fe, Ni, Cu, Zn, Al, Ba, Mo, Sb, Sn, Sr, Mg, Cs, Bi, and Li) were determined by using IC and ICP-MS, respectively. The pH range of the collected rain samples varies between 4.66 and 7.74 in the urban area and between 5.37 and 8.37 in the semi-urban area. Only 3 rain samples had a pH of less than 5.6. For this reason, acid rain was observed at the sampling point, rarely. The VWM concentrations of anions and cations were 148.2 µeq/L and 154.8 µeq/L for the urban area, and 379.8 µeq/L and 571.6 µeq/L for the semi-urban area, respectively. Furthermore, the total VWM concentrations of elements were 44.3 mg/L and 70.0 mg/L for the urban and semi-urban areas, respectively. Source apportionment studies were performed for two sampling points by using PMF version 5.0. Six sources for urban sites and five sources for semi-urban areas were identified. The major sources affecting the urban area were iron-steel factories, Bolu cement factory, biomass burning, transportation from Anatolia, coal burning, and vehicular emission. Likewise, the major sources affecting the semi-urban area were long-range transportation, nitrate factor, iron-steel factory, biomass burning, and the mixture of vehicular emission and coal burning. By evaluating the daily variations of G scores, the back trajectories of the days with the highest associated G scores were examined.


**OP – 26**

# Determination of steroid hormones in artificial serum samples by coupling $Fe_3O_4$/reduced graphene oxide nanocomposites based dispersive solid phase microextraction to LC-MS/MS

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Steroid hormones are endogenous molecules that participate in important physiological processes such as reproduction, development and metabolism in the endocrine system[1-3]. Because of the abnormal production of steroid hormones and their responsibilities in the development of endocrine disorders, accurate measurement is critical in clinical diagnosis/treatment[4-6]. For this purpose, steroid hormones detection limits are great important for biological samples. The aim of this study was to develop a $Fe_3O_4$/reduced graphene oxide ($Fe_3O_4$/rGO) nanocomposite based dispersive solid phase micro-extraction (DSPME) method for the practical and effective extraction of steroid hormones from serum samples. After the $Fe_3O_4$/rGO nanocomposite was synthesized and characterized by Fourier Transform Infrared Spectrometer (FTIR) and Scanning Electron Microscopy (SEM), DSPME optimization studies were completed. Trace amounts of five steroid hormones (aldosterone, progesterone, estrone, testosterone and 17-hydroxyprogesterone) in the serum sample were determined by liquid chromatography combined with tandem mass spectrometry (LC-MS/MS) using the developed method. Very low detection limits for 17-hydroxyprogesterone, aldosterone, progesterone, testosterone, estrone were obtained with the optimized $Fe_3O_4$/rGO based DSPME method. In order to test the feasibility and accuracy of the developed method, a recovery study was carried out using an artificial serum sample. High percent recovery results were obtained from two different spiked concentrations.

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**OP – 27**

# Efficient Removal of Acetaminophen (Paracetamol) and Diclofenac from Aqueous Solutions by Adsorption onto Activated Carbon Cloth

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Chemical contaminants such as salts, bleaches, nitrogen, dyes, pesticides, metals and human or animal drugs pollute underground and surface water resources[1]. Consumption of water that has been exposed to pollutants can cause chronic health problems. Therefore, it is necessary to decontaminate drinking water before consumption by utilization many techniques. Adsorption is the most preferred techniques due to low cost, low energy consumption, high efficiency and simplicity of application[2]. In this study, removal of active pharmaceutical ingredients such as acetaminophen (also known as paracetamol) and diclofenac (commercially known as voltaren) from their aqueous solutions was investigated by adsorption onto activated carbon cloth (ACC). The commercial ACC is used in this study was Spectracarb 2225 (Spectra Corp., Shelton, USA). The adsorption process was followed on-line by in-situ UV-vis spectroscopy via specially designed adsorption cell. Excellent removal efficiencies were achieved for acetaminophen (99%) and diclofenac (97%) end of 6 hours adsorption studies. Kinetic data were treated according to pseudo-first order and pseudo-second order models. Pseudo-second order model was found to fit best in representing the experimental kinetic data. Adsorption isotherms were derived at 25 °C on the basis of batch analysis. Isotherm data were examined according to Langmuir and Freundlich models. Adsorption isotherm data fitted to Langmuir isotherm better than Freundlich model.

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**OP – 28**

# Research of Amino Triazole Chitosan-Based Biopolymers' Platinum Group Metals (PGMs) Recovery Capacity

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Due to the world's finite freshwater supply and the alarming rate at which the human population is growing, there is an urgent need for water filtration. The large number of chemicals that promise to enhance the comfort of agriculture, medical procedures, industry, and domestic living circumstances have recently caused grave worries about the possible severe ecological and human health effects1. But as technology has advanced, it has become increasingly important to recover electronic waste since it contains precious metals, which has increased dramatically. The systematized recovery technique should, however, be the most affordable and efficient option in terms of environmental, technological, social, and economic factors[1].

Chitosan, a bio-derived polysaccharide species, has sparked widespread and significant interest over the last two decades. Because it is non-toxic in polymer synthesis and is used very efficiently, chitosan is biocompatible, biodegradable, and low in cost and maintenance. Chitosan has been shown in numerous published studies to act as a scaffold for gene and drug delivery[2].

In this study, the synthesis of tetrazole derivatives began with the preparation of diazonium salt from chitosan, $NaN_3$, which was then heated in DMF with $NH_4Cl$ and malona nitrile. Including using spectroscopic techniques including FTIR, TGA, and elemental analysis, their structures were explained. The recovery of platinum group metals from the environment was then studied at various pH levels and room temperature. Acquisitions pertaining to amino triazole substitution chitosan biopolymer adsorption capacities for platinum metal recovery from synthetic wastewater at pH 2.0, 3.0, 4.0, 5.0, and 6.0. In these tests, a biopolymer concentration of 10 ppm was recovered to at least 75% of its initial amount in 45 minutes at pH 2.0 and 20 minutes at pH 3.0. This shows that using such biopolymers to remove precious metals may be beneficial.

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**OP – 29**

# Preconcentration of Endocrine Disruptor Phenolic Compounds using Switchable Solvent based Microextraction for the Determination by GC-MS and Assessment of Green Profile

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Alkylphenols and bispenol A (APs and BPA) are widely used in making of industrial and commercial products such as paints, textiles, compact discs, detergents, dental sealants, automobile parts, bottles and several plastic products. Both BPA and APs have the potential to disrupt the normal function of the endocrine system of humans and cause severe effects in other organisms, especially in aquatic habitats[1,2]. Even though they occur at very low levels in the environment, BPA and APs are capable of accumulating over long periods and eventually cause severe disorders. It is therefore relevant to develop analytical protocols that are sensitive for the identification and quantification of contaminants at trace levels. Thus, a simple and efficient microextraction approach based on switchable solvent was used to preconcentrate three APs (4-n-octylphenol, 4-n-nonylphenol and 4-tert-octylphenol) and BPA for determination by a gas chromatograph coupled to a mass spectrometer. Parameters of the microextraction method were optimized using a Box-Behnken experimental design. Optimum conditions of the method derived from the model predictor were applied to aqueous standards and very low detection limits ranging from 0.13 to 0.54 ng/mL were calculated for four analytes. Calibration plots of the analytes recorded high linearities that covered broad concentration ranges. Very low percent relative standard deviations validated the precision of the method. Tap water and municipal wastewater spiked at different concentrations recorded satisfactory recovery results (87–106%). Bisphenol A concentrations ranging between 4.0 and 14 ng/mL were recorded for plastic samples subjected to harsh sample pretreatment conditions[1]. The penalty points of the proposed method were low and this validated the greenness of the method with respect to green analytical chemistry.

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**OP – 30**

# Determination of Cadmium in Lake Samples Using an Effervescence Tablet Assisted Dispersive Solid Phase Exctraction

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Heavy-metal pollution has been a serious concern for the environment, aquatic life, animals, and humans that continues to grow rapidly with industrialization[1]. Cadmium is a non-essential heavy metal known to have no biological function in plants and animals. Besides, it causes significant health effects even at very low amounts[2]. Hence, it is of utmost importance to develop highly accurate and sensitive analytical methods to detect and determine this pollutant to control its adverse effects on public health and the environment. In this study, an effective, rapid and green microextraction method called effervescence tablet-assisted magnetic $Fe_3O_4$ nanoparticle-based dispersive solid-phase microextraction (EA-MNP-dSPME) was developed for the cadmium determination in lake water samples taken from Horseshoe Island. Magnetic effervescent tablets obtained using magnetic nanoparticles ($Fe_3O_4$), 1-undecanol (solidification agent), effervescent salts and tablet strips were used for the homogenous dispersion of sorbent throughout the sample solution. Comprehensive optimization of the significant parameters of the developed microextraction method was carried out. The developed method recorded a 135-fold enhancement in the detection power of flame atomic absorption spectrophotometry. Matrix matching strategy was utilized for the recovery experiments to validate the method's accuracy to real samples and the percent recoveries obtained were satisfactory ranged to between 99.9 – 104.3% [3].

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**OP – 31**

# Removal of Gaseous Styrene by Biotrickling Filter

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The study aims to remove gaseous styrene by using mixed and selected microorganism cultures in the pilot-scale biotrickling filters. A research project was conducted with the cooperation between Subor Boru San. Tic. A.Ş. and Kocaeli University Technology Transfer Office to get an economically and environmentally feasible system for removal of the gaseous styrene emitted during the production of glass fiber reinforced plastic pipes. As the biofiltration with specially selected microorganism cultures presents a good solution according to the related literature[1,2]; three pilot-scale biotrickling filters were designed and installed in the Design Center of Subor Boru San. Tic. A.Ş. Mixed culture was taken from the organic wastes of poultry farm, while pure culture studies were conducted by using the bacterial strain of *Pseudomonas putida* based on the literature[3,4]. A microbiology laboratory was installed to reproduce *Pseudomonas putida* culture to be used in the filters. A mixture of wooden chips, polypropylene Raschig rings, polyester resin pipe chips, and polyethylene chips was used as the filter medium to provide a sufficient surface area for the formation of bacterial films. Gaseous styrene fed to the filters was synthetically produced by air-stripping of liquid styrene. Styrene measurements in the inlet and outlet of the reactor were performed using dragger-based gas detector tubes according to the Method ASTM D 4490-96. The removal rate of the styrene was observed during the tests lasting about 1-2 months. Results indicated that gaseous styrene could be removed in a biotrickling filter with removal rates up to 90% by using both mixed and pure culture. Adoption of the mixed culture to styrene as the carbon source was faster, but rapid the growth of the microorganisms led to some operational problems such as clogging and foaming. Use of the *Pseudomonas putida* indicated gradually increasing removal rates with time, but it offered more controlled and proper operation of the filter. Consequently, the study indicated that biotrickling filters with specially selected microorganism cultures may present an economically and environmentally feasible way of removing gaseous styrene if they are properly designed and operated.

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**OP – 32**

# Sensitive and Accurate Determination of Oil Soluble and Water Soluble Organosulphur Compounds in Garlic Matrix Using Reversed Phase-High Performance Liquid Chromatography

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Garlic (Allium sativum) is a universal plant of the Liliaceae family that has been used for centuries for its biological and medicinal properties. These vegetables contain high amounts of organosulphur compounds (OSCs)[1,2,3]. This study aimed to develop a simple, sensitive, and accurate measuring method that uses reversed phase-high performance liquid chromatography (RP-HPLC) to determine the concentrations of selected organosulfur compounds (OSCs) in garlic bulbs. In order to extract oil-soluble and water-soluble OSCs from garlic matrix, acetonitrile and deionized water were used. The OSCs were separated on a Phenomenex C18 column (250 mm, 4 mm, and 5 mm) and monitored by a UV detector at a wavelength of 220 nm. The mobile phase used in isocratic elution was 0.10 M trifluoroacetic acid (TFA) in 85% acetonitrile (ACN) and 0.10 M TFA in distilled water (DW) (90:10, % v/v). In optimal experimental conditions, the limit of detection (LOD) was calculated in the range of 0.09 - 0.17 mg/kg. Dialyl sulfide (DAS), diallyl disulfide (DADS), and diallyl trisulfide (DATS) were detected in garlic samples under the developed instrumental system in concentration ranges of 8.0-32.5 mg/kg, 20.4-67.3 mg/kg, and 60.7-356.6 mg/kg, respectively. The accuracy and applicability of this method were confirmed by spiked samples experiments conducted on garlic samples. For the garlic samples extracted in deionized water, recovery values ranged from 39.0 to 90.9%. This strategy is appropriate for the determination of OSCs because it is precise, accurate, reliable, and time-effective.

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**OP – 33**

# A Preliminary Study on Microplastic Pollution in Freshwater Streams of the Marmara Basin

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## Abstract

Microplastics are small plastic pieces with less than 5 mm size.[1] In the modern world, microplastics became an environmental threat nowadays, and spreads into the environment through different pathways including wastewater treatment plants, industrial discharges, and domestic wastes.[2] Marmara region is the most industrialized region of Turkey and hosts about one third of the country's population. Biga stream-west (Çanakkale), Biga stream-east (Çanakkale), Kırandere stream (Bursa), Karsak stream (Bursa), Bahçıvan stream (Kırklareli), Istranca-Terkos outflow (İstanbul), Sarıçay stream (Çanakkale), Kavak stream (Çanakkale), Göksü stream (İstanbul), Istranca stream (Kırklareli), Gönen stream (Çanakkale-Balıkesir) stations were chosen for microplastic samplings along the Marmara freshwater basin. Ten minutes samplings were carried out using a 300 µm mesh-sized sampling net from flowing water in all stations. Collected particles were kept in an amber glass for immediate transportation to the laboratory. Samples were dried at 55 °C and treated with 10% hydrogen peroxide. Then, the samples were re-dried and ZnCl2 solution (d: 1.6 g/cm3) added for gravitational separation. The floating matter was collected and dyed with Nile red for fluorescence microscope examination. Later, samples were analyzed by an FT-IR Spectrophotometer for characterization.

Microplastics were observed in 10 of the 12 stream samples. The highest number of microplastics were detected in the Istranca Stream, while the lowest number was in the Gönen stream. In Bahçıvandere and Bulanıkdere Stream samples, there were no microplastic particles. Samples with the highest to the lowest abundance of microplastic particles were Istranca stream, Korsak stream, Göksu stream, Sarıçay stream, Biga stream-west, Istranca-Terkos outflow, Kavak stream, Kırandere stream, Biga stream-east, and Gönen stream. The types of plastic particles were determined using FT-IR analysis as High-Density Polyethylene (HDPE), Polypropylene, Polystyrene, and polyethylene Terephthalate. The most abundant type was HDPE at 64%.

The present study is preliminary work on the microplastic presence in the streams of the Marmara Basin. The threat of microplastics in river basins should be considered seriously as they can be transferred to marine environments and various trophic chains easily.[3]

## Introduction

Microplastics are complex plastic particles that could differ in properties like shape, color, size, chemical composition, and density. Microplastic use and production increase day by day and became a global environmental problem.[4] Microplastic pollution can originate from the breaking down of larger plastic wastes by wind, waves, UV light, etc. (secondary microplastics) or micron-sized plastic products like exfoliating creams and toothpaste additives (primary microplastics).[3,5]

The damages of microplastic pollution on aquatic ecosystems are documented in several studies.[6-9] Main entryways of microplastic pollution into marine ecosystems are rivers and streams that carry urban and industrial waste. In the context of this study, 12 sampling points were chosen from the freshwater basin of the Marmara Region which is by far the most populated region of Turkey.

## Materials and Methods

12 streams in the Marmara freshwater basin were sampled for microplastic pollution. These are; Bahçivandere Stream, Biga Stream (East)(Çanakkale), Biga Stream (West)(Çanakkale), Bulanıkdere Stream (Kırklareli), Göksu Stream (İstanbul), Gönen Stream (Balıkesir), Istranca Stream (Kırklareli), Istranca-Terkos Outflow (İstanbul), Karsak Stream (Bursa), Kavak Stream (Çanakkale), Kırandere Stream (Bursa), and Sarıçay Stream (Çanakkale) (Figure 1).


**Figure 7** Sampling stations (A:Bulanıkdere, B: Bahçıvandere, C: Istırancadere, D: Istıranca-Terkos, E: Göksu, F: Kırandere, G: Karsak, H: Gönen, I: Biga(E), J: Biga(W), K: Sarıçay, L: Kavak)

Water samples were taken with a hand net (mesh size 300µm). Composite samplings were made from different parts of the stream. Total samplin time were between 15-25 minutes. Non-plastic equipment was used for sampling. After transferring to the laboratory, samples were wet sieved from a 500 µm mesh-sized sieve using distilled water. Sieved samples were then dried and treated with 10% $H_2O_2$ to remove organic residue. Samples were then dispersed gravimetrically inside a concentrated $ZnCl_2$ solution. Samples were then washed with distilled water and dried out. Dried samples were dyed with Nile Red examined under a microscope and then sent to FTIR analysis to determine polymer types.

## Results and Discussion

No microplastic particles were encountered in samples from Bulanıkdere Stream and Bahçıvandere Stream. In the remaining samples, a total of 28 microplastic particles from 4 different types (High-Density Polyethylene (HDPE), Polypropylene (PP), Polystyrene (PS), and Polyethylene Terephthalate (PET)) were found (Table 1). HDPE was the most abundant microplastic type with 18 total particles (64%).

Tablo 1 Microplastic particle quantity from streams of Marmara Basin

| Samplin Station | Flow Rate ($m^3/s$) | Sampling Time (m) | Sampled Microplastic Quantity | Estimated Microplastic Count For Stream (1h) |
|---|---|---|---|---|
| Bahçivandere Stream | 0,001 | 15 | 0 | 0 |
| Biga Stream (East) | 2,62 | 20 | 1 | 1.080 |
| Biga Stream (West) | 15,2 | 20 | 3 | 41.400 |
| Bulanıkdere Stream | 0,039 | 20 | 0 | 0 |
| Göksu Stream | 0,159 | 20 | 4 | 828 |
| Gönen Stream | 5,33 | 20 | 1 | 1.740 |
| Istranca Stream | 0,04 | 20 | 5 | 456 |
| Istranca-Terkos Outflow | 0,002 | 20 | 3 | 12.900 |
| Karsak Stream | 0,449 | 20 | 4 | 660 |
| Kavak Stream | 1,57 | 20 | 2 | 660 |
| Kırandere Stream | 0,327 | 20 | 2 | 60 |
| Sarıçay Stream | 0,002 | 20 | 3 | 2.115 |

After calculating expected microplastic load of the sampled stations, Biga Stream (West) satation found to have largest microplastic load (ie. 41.400 particles per hour)

After visual examination, microplastic particles were divided into three shape categories (ie. Film, fragment, and filament.) (Figure 2).

These results show that microplastic pollution is a serious issue in the freshwater bodies of the Marmara Region. By housing about one-third of the population of Turkey, and being the most industrially developed region of the country, the Marmara region is a great source of both industrial and urban waste. There are several studies on microplastic contamination in marine biota and sediment of The Marmara Sea.[10,11] Rivers and streams are highways for low-density pollutants like microplastics to larger ecosystems like lakes and marine habitats.

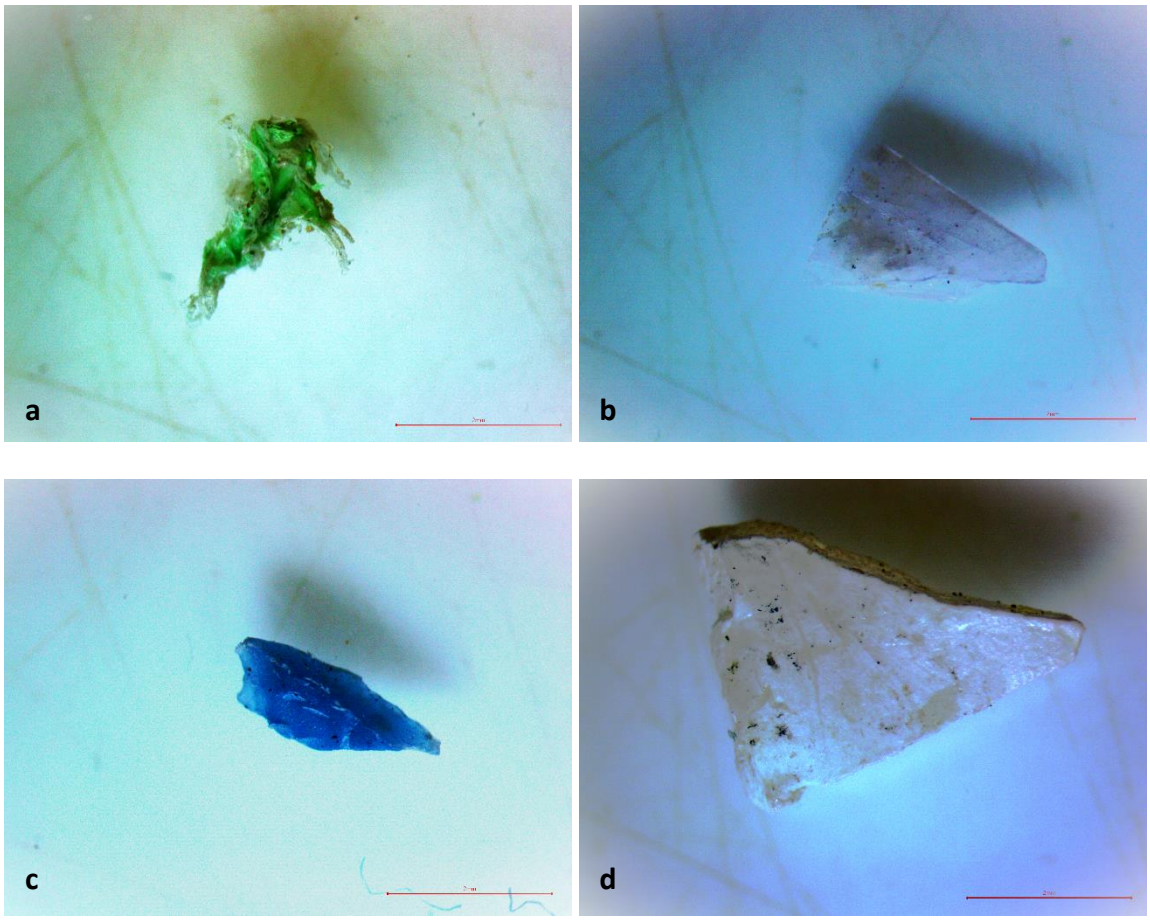


**Figure 8** Microplastic particles (a: HDPE, b: PET, c: PP, d: PS)

## Conclusion

Four different microplastic types were identified from 10 of 12 stations among freshwater streams of The Marmara Basin. These streams flow into marine and lake ecosystems and microplastic pollution carried by these streams ends up in the trophic chain. After entering the trophic chain, microplastics can easily climb up to human consumption.

Even though the direct hazards of microplastics on humans are still being investigated intensively, their damage to aquatic life has been very well documented.

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**SOP – 1**

# Microplastics in Patients with Colorectal Adenocarcinoma

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Investigation of the relationship of microplastics with various diseases and cancer is a current issue and studies are limited in the literature[1]. In this study, we examined microplastics in the colon tisssue samples of patients with colorectal adenocarcinoma (TCT: tumorous colon tissues and N-TCT: non tumorous colon tissues groups) and in the colon tissue samples of patients without colorectal cancer (C group). Microplastics were extracted from the tissues and various methods including microscope imaging, the attenuated total reflection-Fourier-transform infrared (ATR-FTIR) and Raman spectroscopies were used to identify the types and number of microplastics. Mean age of patients (N=16) was 60.81±10.72 years, and mean age of subjects without colorectal cancer (N=15) was 65.93±15.875 years, and there was no significant difference in terms of age between groups (*P* =0.299). The number of microplastic particles in per 1 gr colon tissue were 702.68±504.26 in TCT group, 207.78±154.12 in N-TCT group, and 218.28±213.05 in C group. Comparisons of groups in terms of number of microplastics showed that number of microplastics in per 1 gr colon tissue was significantly higher in TCT group compared to N-TCT group (*P* =0.001). However, the number of microplastics were not significantly different in N-TCT group and the C group (*P*=0.876). Our results show that there may be a link between colorectal cancer and microplastics. Further studies in larger patient groups are needed.

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**SOP – 2**

# Application of Central Composite Design to Optimize Moxifloxacin Antibiotic Removal by Carbon Black Coated Fabric

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Moxifloxacin, one of the fluoroquinolones group, is a broad-spectrum antibiotic used in atypical pneumonia.[1] Fluoroquinones have been shown to be effective against viruses, including SARS-CoV-2, in many in vitro experiments, and were among the antibiotics preferred in the treatment of Covid-19 in the pandemic.[2] Carbon black (CB), a family of small particle size carbon pigments, is theoretically a potential candidate as a high-performance adsorbent because it has a large specific surface area[3]. CB can be added to the textile fabric surface as a coating or directly to the textile fabric[4]. The removal of Moxifloxacin from aqueous solutions by CB coated fabric were optimized by using central composite experimental design (CCD) involving response surface methodology (RSM) using with three significant independent factors including pH, ionic strength and antibiotic initial concentration. It is modeled as polynomial between the independent variable (absorption capacity) and the dependent variables mentioned above. The absorbed moxifloxacin was determined by HPLC method. The chromatographic separation was achieved on the reversed-phase Zorbax C18 column with the mobile phase consisting of phosphate buffer (25 mM, pH 2.5) and acetonitrile at 290 nm. The statistical significance of the quadratic regression equation demonstrated that the regression is statistically significant with $P<0.01$ obtained from the ANOVA for response surface quadratic model. In this case pH, ionic strength and antibiotic initial concentration are significant model terms and significantly affects the absorption capacity of Moxifloxacin. The best conditions, predicted by the model, for the removal of the Moxifloxacin (168.04 µg/cm$^2$) is obtained at pH values of 9.06, ionic strength of 0.39 M and initial concentration of 4430 µg/L. In addition, Langmuir's classical isotherm model (at 0.39 M ionic strength and pH 9.06) was applied, and it was found to be in good agreement with the CCD model, with an estimated maximum absorption capacity of 161.3 µg/cm$^2$.

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**SOP - 3**

# Removal of Summifix Yellow EXF Reactive Azo Dye by Electro-Fenton Method

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Reactive dyes can be found large quantities in textile industry wastewater due to their widespread use for dyeing cotton fabrics and their durable nature. In the treatment of wastewater containing dyestuffs in this class, advanced treatment methods has become necessary due to the inadequacy of conventional treatment methods and their some disadvantages.[1] For this reason, electro-Fenton which is an electrochemical advanced oxidation method, has become a strong alternative as a treatment technology that provides complete disintegration of dye molecules. In this study, the treatment of model wastewater containing the reactive azo dye Summifix Yellow EXF using the electro-Fenton method was investigated. The electro-Fenton method is based on in stu production of one or both of the Fenton reagents, $Fe^{2+}$ and $H_2O_2$, in an electrochemical cell through electrode reactions. In the electrochemical cell used in this study, carbon fiber was used as the cathode and iron was used as the anode. Experiments were carried out at room temperature is an undivided and cylindrical 250 mL glass beaker. While $Fe^{2+}$ ion is produced at the anode, $H_2O_2$ is added to the cell externally. Experiments were carried out by changing the voltage(5-10 V), $H_2O_2$ concentration(9-74 mM), $Na_2SO_4$ amount(0,1775-0,71 g/200 mL) and pH(3-5) in order to provide the highest dyestuff removal and the effect of these parameters on dye removal and energy consumption were investigated. It was found that for the best dye removal, voltage is 7,5 V, the $H_2O_2$ concentration is 74 mM, the $Na_2SO_4$ addition is 0,71 g and the optimum pH value is 4. At these values, 98.14% removal at 30 minutes was achieved with an energy consumption of 7.98 Wh/L.

**Keywords:** Electro-Fenton; advanced oxidation; dye removal; Summifix Yellow EXF; reactive azo dye

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**SOP - 4**

# Chemometric Assisted Spectrophotometric Method for Simultaneous Determination of Desloratadın and Montelukast Sodium in Combined Pharmaceutical

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Allergic rhinitis is defined as chronic inflammation of the nasal mucosa characterized by nasal discharge, sneezing, itching and congestion, often conjunctival injection, and itchy eyes. Recent studies conducted in different regions show that the incidence of allergic rhinitis is increasing[5-8]. As a result of global warming, increasing environmental pollution and COVID-19 pandemic, studies on allergic rhinitis gain importance. Montelukast sodium and Desloratadine are combined preparations used in the treatment of allergic rhinitis under various generic names. The aim of this study was to develop and validate a Spectrophotometric Method in which a spectral separation is carried out with the Chemometric PLS and PCR calibration methods for simultaneous determine Desloratadine and Montelukast Sodium in the combined pharmaceutical preparation. The procedure, based on have been evaluated the multivariate analysis by the PLS and PCR methods of spectral data in the 225−400 nm region at an interval of 1 nm. The experimental calibration and validation matrixes were constructed with 25 and 8 samples, respectively. The concentration range considered was 20-80 µg/mL for both the drugs. The correlation coefficients for the relationship between actual and predicted values of both drugs for calibration and validation matrixes were higher than 0.9997 both PLS and PCR indicating good accuracy. The percent relative standard deviation (RSD%) for precision of methods were found to be ≤ 2.59 % for PLS and ≤ 2.47 % for PCR. The proposed PLS and PCR calibration methods were successfully applied for the determination of Montelukast sodium and Desloratadine in Pharmaceutical Dosage form, with no interference from excipients by being found to be in good agreement with the label claim.

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**SOP - 5**

# Bioremediation of Pharmaceuticals from Contaminated Water by Microalgae Culture

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The demand for pharmacological products is increasing day by day with the remaining importance given to the treatment of diseases and human health. These pharmaceuticals and their metabolites, which are released into the environment with the domestic, hospital, and pharmaceutical industry wastewater, pose a serious threat to the aquatic ecosystem and public health.[1] Even if high-efficiency wastewater treatment is accomplished with conventional wastewater treatment technologies, the total removal of organic micropollutants cannot be achieved.[2] Microalgae are known to have an important role in the removal of pharmaceutical compounds from wastewater.[3]

In this study, the removal of paracetamol and diclofenac pharmaceuticals under photoautotrophic conditions using *Scenedesmus sp.* was investigated. The microalgae strain was illuminated for 12 hours per day with a light intensity of 100 µmol-photon/$m^2$·s at 25 ± 1 °C. At the end of the 8-day experiment period, 69% and 91% removal efficiencies were obtained for paracetamol and diclofenac, respectively. These results show that *Scenedesmus sp.* is a suitable microalgae species for bioremediation of wastewater containing paracetamol and diclofenac.

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**PP – 1**

# Accumulation and Human Health Risks of Potentially Toxic and Essential Elements in *Donax Trunculus* from Black Sea (Bulgaria)

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The Bulgarian coast of Black Sea is an ecosystem of great ecological importance and has been degraded as a result of industrial and anthropogenic activities. The aim of this study was to determine the total concentrations of some potentially toxic (Cd, Pb and Ni) and essential (Cr, Cu, Fe and Zn) elements in wedge clam species (*Donax Trunculus*) collected from the Bulgarian shore of Black Sea. The highest average concentration for the samples were measured for Fe (274.86 mg/kg w.w), followed by Cu (34.12 mg/kg w.w). Human health risk assessments were performed to determine the risks to the environment and population's health based on of target hazard quintet (THQ), target risk (TR), and hazard (HI) indexes. Additionally, the estimated daily intake (EDI) were calculated. The results indicate that EDI values were below the published RfD values. THQ and HI were below 1 so it could be concluded that consumption of wedge clam species (*Donax Trunculus*) sampled from different Black Sea localities, did not pose any risk for the health of adult people as copper, iron, lead, zinc, cadmium, chromium and nickel were concerned.

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**PP – 2**

# Investigation of Electrodialysis of an Aqueous Solution Containing Salt and Boric Acid by Electrodialysis with Bipolar Membrane

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In this study, desalination, acid and base production were examined simultaneously with bipolar membrane electrodialysis from boric acid and salt-containing solution. The experiments were carried out in the electrodialysis cell in the batch flow mode. The electrodialysis cell consists of a membrane stack consisting of five repeating cells with an anode, cathode and three compartments. Each cell in the membrane stack was formed according to the sequence BM//KC//AC//BM. Purification of aqueous solution in terms of boric acid and conversion of salt to acid and base were determined as process performance criteria. Initial salt concentration, initial acid-base concentration, current and volumetric flow rates of solutions were also selected as parameters that could affect process performance. In the obtained data, it was found that the initial salt concentration was the most effective parameter for acid and base formation, and the concentration of acids and bases produced in the process increased with increasing initial salt concentration. However, it was observed that the desalination in saline solution was between 95-99%. In addition, it was observed that increased current values also make a positive contribution to the formation of acids and bases, increased current increases the difference in electrical potential and makes an important contribution to shortening the processing time. It was observed that the increased flow rate has a negative effect on salt conversion. The optimum operating conditions were determined taking into account the product concentration; the initial salt concentration was 160 g.L-1, the initial acid-base concentration is 0.2 M, the current/voltage value is 6.2A/20V, the flow rate is 10 L.h-1. As a result of these optimal conditions, the amount of acid and base produced was found to be between 2-2.2 M, and the conversion of salt-based products was about 80-95%.

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**PP – 3**

# Antioxidant Activities of Some Microwave-Assisted Synthesized Dipeptide-Indole Conjugates

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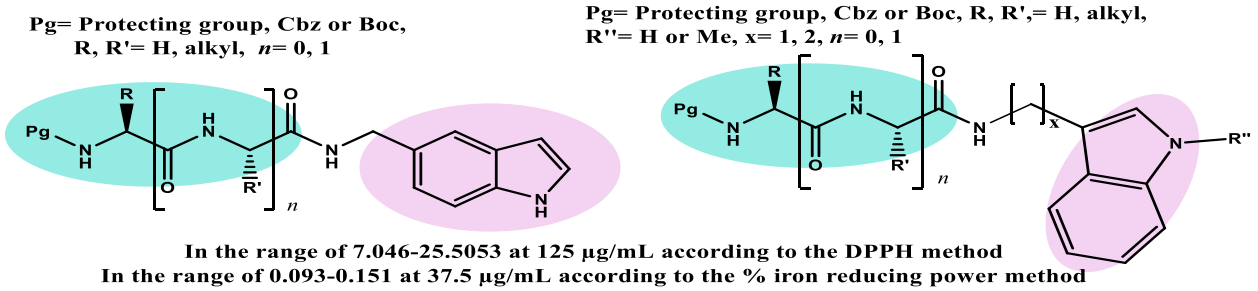
Indole, a heterocyclic compound, is among the active ingredients of many medicinal plants and has various biological activities such as anti-tumor, anti-inflammatory, antibacterial and anti-fibrotic.[1] Synthetic and natural peptides have many important therapeutic effects. Peptides are of great interest in pharmaceutical research and development due to their highly selective and biologically compatible nature. Due to these properties, more than 100 peptide-based drug candidates are currently in clinical trials[2] and many drugs also approved by the FDA (U.S. Food and Drug Administration), such as sumatripan, melatonin, osimertinib and tegaserod, also contain an indole ring. In this study, the antioxidant properties of a series of indole-peptide conjugates obtained by conjugation of biocompatible peptides with indole, an important heterocyclic structure, by microwave irradiation, one of the green reaction synthesis methods, were investigated. The general structures and antioxidant ranges of the compounds whose antioxidant activities were tested are shown below.




**In the range of 7.046-25.5053 at 125 µg/mL according to the DPPH method**
**In the range of 0.093-0.151 at 37.5 µg/mL according to the % iron reducing power method**

**Acknowledgements:** We thank İnönü University, Turkey (BAPB-Grand No-FOA-2020-2203) for financial support.

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**PP – 4**

# Optical Characteristics and Chemical Composition of Some *Spirulina* from the Market

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We enjoy food for its taste, aroma, and pleasure, but "What's in it?". The aggregate of various chemical substances beneficial for the functioning of the body in it are known as nutrients. Spirulina has been present in human nutrition since ancient times. In this study, we will pay attention to some of them and the optical properties they possess in Spirulina, purchased from four different producers - Bulgaria, China, Belgium, and Hawaii. ATR-IR was utilized to assess the significant constituents in each sample. The main components in the analyzed Spirulina samples are proteins (1 657 and 1 537 cm-1) and carbohydrates (1 069 and 1054 cm-1), which showed distinct fingerprint characteristics of IR spectra. Significant differences between the IR spectra of the analyzed samples were not observed. Magnesium (Mg), iron (Fe), zinc (Zn), copper (Cu), and manganese (Mn) are among the vitally important and necessary trace elements for the body. After their preliminary acid digestion with nitric acid, FAAS analysis was used to determine the content of the listed elements in the four investigated Spirulina samples. The concentrations of the ingredients in the models vary depending on the producers; Mg and Fe are the highest for Spirulina from Hawaii, while the Bulgarian Spirulina has the highest content of Zn and Cu. All analyzed samples follow a trend: Mg>Fe>Mn>Zn>Cu.

Using an Avantes reflectance fluorimeter, the fluorescence spectra were obtained for the investigated *Spirulina platensis* samples at an excitation light wavelength of 385 nm. Since there is no significant difference in the location of the peaks, the relative fluorescence intensity will be considered the averaged fluorescence spectrum of the cyanobacterium *Spirulina platensis*. Four fluorescence maxima are observed in it:

At 420 nm due to phenolic acids[1]; at 464 nm due to nicotine amide dinucleotide phosphate[2]; at 673 nm due to chlorophyll *a*[3]; at 720 nm due to the similar pigments such as chlorophyll;

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**PP – 5**

# Synthesis of New Surface-Active Catanionic Salt for Removing Thin Petroleum Films from Water Surface

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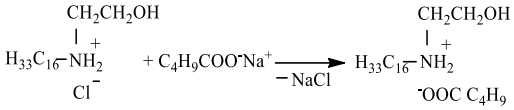
Pollution of water by petroleum iaeynes a great problem facing our ailing environment. This problem has a significant impact on the marine environment and also on both birds and animals. Sources of such pollution include production operations and oil exploration, natural seeps, tanker accidents, industrial discharge, atmospheric input, and urban run-off. Surfactants are widely used for collecting and dispersing thin films of petroleum on water surface[1]. Many types of surfactants were synthesized and their usage as petroleum-collecting and dispersing agents were studied. Such types include catanionic surfactants which exhibit a good efficiency of removal of oil slicks from the water surface. Moreover, that type salts show a high activity in collecting and dispersing the spilled crude oil[2]. The main aim of the presented work is to produce a new, ecologically-safe and efficient oil slick-collecting agent based on a novel catanionic surface-active salt. It was synthesized by interaction of hexadecylethylolammonium chloride with Na-pentanoate salt:



It is a yellow wax, well-soluble in ethanol and water accompanied with intensive foam formation. Composition and structure of this reagent have been identified by IR- and UV- spectroscopy methods. Furthermore, by the method of dynamic light scattering the size distribution profile of small particles in suspension and in solution at different concentrations was determined. Surface tension at the water–air interface in the presence of the synthesised surfactants was determined by a Du Nouy ring tensiometer. A high surface activity of aqueous solutions of the synthesized product was revealed (at 0.2% -35.6 mN/m; 0.5%-31.5 mN/m; 0.7%-28.2 mN/m; without surfactant 72.0 mN/m). The specific electroconductivity of the surfactant solutions was measured using a conductometer. By the electroconductometric method it was found that the specific electrical conductivity (□, in μS/cm) of aqueous solutions of this surfactant rises as the concentration (% by weight) of the solution increases (21°C): 0.025% 47.9; 0.075% 102.2; 0.1% 153.1; 0.5% 215.3; 0.7% 230.8.The petrocollecting effectiveness of the surfactant was studied using an unthinned reagent and its 5 wt.% aqueous solution (or dispersion). The tests were carried out in three types of water having various degrees of mineralization (fresh, Caspiansea and distilled waters) using thin (thickness: 0.17 mm) layers of Pirallahy petroleum (from the oil field near Baku, Azerbaijan). This surfactant has a high petrocollecting capacity. When it is applied in unthinned form and used as a 5% aqueous dispersion, high values of petrocollecting coefficient (K), which characterizes a ratio of surface area of initial petroleum slick and petroleum spot formed by a surfactant action are observed. Maximum value of K equals 80.2, the time of the reagent action exceeding 192 hours.

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**PP – 6**

# Determination of Thermodynamic Parameters of Titanium Lanthanides by Measuring the Emf of Concentration Chains

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Alkali metal titanates are of interest for magneto-optical, ion-conducting and spintronic devices, ion batteries, for the creation of new highly efficient catalysts and functional nanoparticles.

$Li_2TiO_3$ is used in the cathode of some lithium-ion batteries together with an aqueous binder and a conductive agent. $Li_2$ TiO3 is used because it is able to stabilize high-capacity cathode conducting agents; $LiMO_2$ (M = Fe, Mn, Cr, Ni). $Li_2TiO_3$ and conducting agents ($LiMO_2$) are layered to create a cathode material. These layers allow lithium diffusion.

Of lithium titanates, thermodynamic data are available only for the $Li_2TiO_3$ compound [1]. To determine the thermodynamic parameters of titanium lanthanides the EMF of concentration chains was measured:

$$Pt|Li_2O|\ ZrO_2 + 10\ mas.\%\ Y_2O_3,\ lithium\ glass\ |(Li_2O)x(TiO_2)_{1-x}|Pt \quad (1)$$

The experimental technique described in [2] was used. Solid electrolyte of zirconium dioxide $ZrO_2$, stabilized by yttrium oxide $Y_2O_3$, has sufficient ionic conductivity, which is due to the migration of $O^{2-}$oxygen through anionic vacancies under the action of an electric field at temperatures above 800C.

The measurements were carried out in the temperature range T = 1000-1200K and concentrations 0.35–0.95mol fraction of $TiO_2$. The EMF values were reproduced with an accuracy of ± 5 mV. The temperature dependences of the EMF were approximated by linear equations E = (a ± Δa) + (b ± Δb) T using the OriginPro2021 computer program**:**

$E, mV = (961\pm10) - (3.106\pm0.03)10^{-2}T$, phase region $Li_4Ti_5O_{12} + TiO_2$ (2)

$E, mV = (430\pm4) - (1.520\pm0.02)\cdot10^{-2}T$, phase region $Li_{1.92}Ti_{1.04}O_{3.04} + Li_4Ti_5O_{12}$ (3)

$E,mV = (382\pm4) - (1.431\pm0.02)\cdot10^{-2}T$, homogeneous phase $Li_2TiO_3$ (4)

$E, mV = (134\pm2) - (1.380\pm0.02)\cdot10^{-2}T$, phase region $Li_4TiO_4 + Li_{2.12}Ti_{0.94}O_{2.92}$ (5)

According to the Gibbs-Helmholtz equation, the parameters of the linear equations are related to thermodynamic characteristics with the following relations:

$$\Delta G = -zFE,\ \Delta H = -zFa,\ \Delta S = zFb \quad (6)$$

where z = 2 (oxygen charge in one mole of $Li_2O$), Faraday number F = 96.5 J / mV.

**Keywords:** Titanium lanthanides, EMF method, free energies, enthalpy, standard entropy

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**PP – 7**

# Quantitative Analysis of PAHs in the 7 Areas of the Caspian Sea

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Caspian Sea borders with 5 countries and for this reason, monitoring is carried out frequently and the necessary measures are taken. Pollution of aquatic ecosystems is one of the main global environmental problems. In addition, the chemical composition of sea water is an important condition for flora and fauna. From this point of view, we took samples from different parts of the Caspian Sea in Azerbaijan, analyzed them and studied their suitability as seawater[1]. Water samples were taken and analyzed from 7 different regions of the Caspian Sea in which we analyzed mainly toxic organic compounds -PAHs.The analyzes were carried out using extremely sensitive devices a GC-MSD gas chromatograph 6890N with a highly efficient mass-selective detector-Agilent 5975[2].It has been established that the investigated toxic organic compounds in the waters of the Caspian Sea coast exceed the permissible norm.

**Table.** PAHs in 7 water samples taken from the Caspian Sea

| Polysilic aromatic hydrocarbons, mg/ l | Guneshli | Hovsan | Shikov | Boulevard | 28 may | Sahil | Bilgah |
|---|---|---|---|---|---|---|---|
| Naphthalene | 0.09 | 0.19 | 0.07 | 0.04 | 0.07 | 0.02 | 0.01 |
| Achenthylene | <0.01 | <0.01 | <0.01 | <0.01 | 0.03 | 0.01 | 0.01 |
| Acenaften | 0.01 | 0.01 | <0.01 | 0.05 | 0.04 | 0.03 | 0.02 |
| Fluoren | 0.04 | 0.07 | 0.04 | 0.07 | 0.24 | 0.02 | 0.02 |
| Fenantren | 0.09 | 0.17 | 0.06 | 0.17 | 0.29 | 0.04 | 0.03 |
| Anthracene | 0.01 | 0.01 | <0.01 | 0.01 | 0.04 | 0.04 | 0.02 |
| Fluoranten | 0.01 | 0.01 | <0.01 | 0.01 | 0.03 | 0.01 | 0.01 |
| Piren | 0.01 | 0.01 | <0.01 | 0.01 | 0.05 | 0.01 | 0.00 |
| Benz (a) anthracene | <0.01 | 0.00 | <0.01 | 0.00 | 0.01 | 0.00 | 0.00 |
| Chrezen | 0.02 | 0.01 | <0.01 | 0.01 | 0.02 | 0.01 | 0.01 |
| Benz (b + j + k) fluorantene | 0.01 | 0.03 | 0.03 | 0.03 | <0.01 | 0.02 | 0.02 |
| Benz (a) pyrene | 0.01 | 0.01 | <0.01 | 0.01 | <0.01 | 0.01 | 0.01 |
| Inden (1,2,3-cd) pyrene | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | 0.01 |
| Benz (ghi) perilen | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | 0.01 | 0.01 |
| Dibenz (ah) antracen | <0.01 | <0.01 | <0.01 | <0.01 | 0.02 | 0.02 | 0.01 |
| Σ 15 Individual PAH | 0.35 | 0.56 | 0.31 | 0.46 | 0.88 | 0.28 | 0.19 |

Naphthalene belongs to a class of high-risk substances and is considered the most hazardous among PAHs. Therefore the main focus was on which areas of the water samples had the highest levels of naphthalene. As you can see this indicator is mainly found in Hovsan. This is due to the fact that there is an industrial plant in the area located near Hovsan and the water in Hovsan can be considered more polluted due to the constant discharge of wastewater.

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**PP – 8**

# Analysis of Phenolic Compounds in the Industrial Wastewaters

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Wastewater pollution with organic toxic substances leads to many environmental problems. Industrial wastewater discharges pollute many water bodies which leads to serious damage to the flora and fauna of the aquatic ecosystem. At the catalytic cracking unit of the oil refining industry 3 water samples were taken and 13 phenolic compounds were analyzed in the samples. All GC-MS analyzes were performed on a high-sensitivity Agilent 5975 mass detector equipped with an Agilent 6890N gas chromatograph.

Table. Amount of phenol and its derivatives in 3 water samples taken from the Oil Refinery

| Compounds (mg/l) | 1 | 2 | 3 |
|---|---|---|---|
| phenol | 5.34 | 5.65 | 1.44 |
| o-cresol | 0.76 | 0.26 | 0.16 |
| 2-nitrophenol | 0.59 | 0.48 | 0.08 |
| 2,4-dimethylphenol | 31.9 | 8.71 | 0.20 |
| m,p-cresol | 20.6 | 3.81 | 0.85 |
| 2,6-dichlorphenol | 1.14 | 0.67 | 1.80 |
| 4-chloro-3-methylphenol | 0.63 | 0.43 | 0.69 |
| 2,4,5-TCP | 0.48 | 0.28 | 0.18 |
| 2,4,6-TCP | 0.14 | 0.04 | 0.25 |
| 2,3,4,6-tetrachlorophenol | 0.27 | 0.18 | 0.002 |
| 2-methyl-4,6-dinitrophenol | <0.04 | <0.04 | <0.04 |
| pentachlorphenol | 0.27 | 0.25 | 0.06 |
| 2-sec-butyl-4.6-dinitrophenol | <0.08 | <0.08 | <0.08 |

As can be seen from Table 1 the amount of phenol in the water samples was higher. This can be explained by the fact that phenol is well soluble in water and chemical stable[1]. The permissible limit for chlorinated derivatives is smaller and is 0.001 mg/l. Chlorine compounds of phenol are formed in water which is 250 times more dangerous for living things than phenol itself [2].

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**PP – 9**

# Comparison of Adsorption Performances of Various Multiwalled Carbon Nanotube-Based Adsorbent Materials for the Removal of Diquat Dibromide Herbicide from Water

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Herbicides used in agricultural applications not only prevent the growth of weeds or kill them, but also increase product yield.[1] On the other hand, they may reach the water sources in different ways and cause the environmental contamination. Adsorption is one of the most effective techniques in the removal of pollutants from water.[2]

In this study, oxidized multi-walled carbon nanotube (OMWCNT), magnetic OMWCNT/$Fe_3O_4$ and magnetic OMWCNT/κ-carrageenan/$Fe_3O_4$ materials with different structural and surface properties were prepared, characterized and their adsorption performances in the removal of toxic diquat dibromide (DQ) herbicide from water were examined by UV spectroscopic method. The effect of initial herbicide concentration, contact time and temperature onto the adsorption process was determined. The surface and morphologic properties of adsorbent materials were investigated by SEM and TEM analyses. Adsorbent materials were characterized BET, FTIR and XRD measurements.

For all adsorbents, the amount of DQ adsorbed by carbon nanotube-based adsorbents increased with the increase in the initial herbicide concentration, contact time and solution temperature. All the adsorption systems reached equilibrium at the end of 300 minutes. In all adsorption systems, experimental kinetic data fitted perfectly with pseudo-second-order kinetic model. At 25 °C, maximum DQ adsorption capacities of OMWCNT, OMWCNT-$Fe_3O_4$ and OMWCNT-κ-carrageenan-$Fe_3O_4$ adsorbents were found to be $16.09 \times 10^{-5}$ mol.$g^{-1}$, $5.784 \times 10^{-5}$ mol.$g^{-1}$ and $2.954 \times 10^{-5}$ mol.$g^{-1}$, respectively. In addition, thermodynamic studies displayed that all adsorption processes are favorable, spontaneous and endothermic.

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**PP – 10**

# Removal of Methylene Blue from Water with Halloysite Nanotube and Surface-Activated Halloysite Nanotube: Kinetic Study

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Halloysite nanotubes (HNTs) take place in kaolin class. They are abundant in nature and have different crystal structures and valuable properties such as tubular nanostructure, high specific surface area, large aspect ratio, high dispersion in water, biocompatibility, active surface chemical groups and high mechanical strength, etc.[1-3] Methylene Blue (MB) is frequently found in wastewater and used in the textile, paper, leather and plastic industries.[4] MB must be removed from water owing to its potential toxicity and nonbiodegradability. HNTs with a negative surface charge are very suitable for adsorption via electrostatic interactions with cationic dyes such as MB. Among the many different techniques developed for the removal of dyes from wastewater, adsorption is the most used technique. In this study, surface-activated HNT (A-HNT) with NaOH treatment was prepared from the original HNT (HNT). Kinetic studies were performed for the adsorptive removal of MB onto HNT and A-HNT adsorbents from water polluted with dye and the effect of the contact time and the initial concentration of MB solution on the adsorption process were investigated. In adsorption process, the concentrations of MB solutions were followed by UV-visible spectroscopic technique. SEM and TEM analysis was performed to investigate the changes in HNT and A-HNT morphology. FTIR analysis was carried out to see the changes in functional groups on HNT surface after surface activation of the original HNT. The fitting of the obtained kinetic data with pseudo first-order, pseudo second-order and Elovich kinetic models was investigated. It was found that the kinetic data of MB adsorption on HNT and A-HNT adsorbents followed the pseudo-second order kinetic model. When the initial dye concentration was increased from 125 $mg.L^{-1}$ to 175 $mg.L^{-1}$, the initial adsorption rate showed a decreased from 51.61 $mg.g^{-1}.min^{-1}$ to 29.80 $mg.g^{-1}.min^{-1}$ for HNT and from 17.48 $mg.g^{-1}.min^{-1}$ to 15.05 $mg.g^{-1}.min^{-1}$ for A-HNT, respectively. The adsorption amount of MB onto HNT and A-HNT increased with increasing initial dye concentration. For each initial MB concentration, the amount of MB adsorbed onto the original HNT was higher than that of A-HNT.

**Acknowledgments:** This study was supported by the Scientific Research Projects Coordination Unit of Akdeniz University with the project number FDK-2021-5840.

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**PP – 11**

# Determination of Pb(II) Ion in Bovine Liver

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Lead has been used by man since ancient times. The symptoms of lead poisoning-saturnism have been known for a long time. In the 20th century, the prevalence of lead increased dramatically, and a lot of work was done on the toxic effects of this metal on the human body. Ca3(PO4)2 in lead phosphate replaces calcium and accumulates in bones. Like some other heavy metals, lead (in the form of ions) blocks the activity of some enzymes. For these reasons, it is important to develop new methods for the determination of lead in environmental objects. In the literature, many works have been done for the determination of lead[1,2].

After determining the optimal conditions of sorption and desorption equilibrium of Pb(II) ion with new sorbents under static conditions, the sorption and desorption equilibrium of the studied metal ion was studied under dynamic conditions. Thus, during the analytical determination of elements, condensation is mainly used under dynamic conditions (in mini calonca). The effect of eluent concentration, sample and eluent delivery rate, sample volume, and matrix components on the sorption and desorption equilibrium of Pb(II) ion with the obtained sorbents under dynamic conditions was studied and the optimal conditions of solidification were determined. After determining the optimal conditions of solidification, the methods of determination of Pb(II) ion in liver, kidney, stomach and intestinal capacity were developed. For example, 0.697 mg/kg of Pb(II) ion was determined in the liver sample during concentration with the new sorbent. The correctness of the developed methods was confirmed by the addition method. Determination of lead ion in animal liver. The methodology developed during the course of the analysis was applied to determine the lead ion in the animal's liver. It is known that lead is slowly removed from the body. Two liver samples weighing 24.6387 g (I sample) and 28.2116 g (II sample) were taken for analysis. Pb(CH3COO)2 solution was injected into the first sample and then dissolved in a graphite crucible by heating in 55ml HNO3 acid solution. Then the second sample is dissolved in a graphite crucible by heating it in 55 ml HNO3 acid solution. Dissolving the liver in HNO3 acid continued for one hour. Then the ash of the first and second sample is obtained by burning the mixture in a muffle furnace. After burning, the weight of the ash of the first sample was 5.1320 g, and the weight of the ash of the second sample was 5.6511 g. We dissolve the obtained ash in tsar vodka and dissolve it in distilled water and pass the insoluble part through a filter. Pour the obtained mixture into a 100 ml flask and add pH (pH = 5). We add pH up to the line and mix. The density of the lead ion is determined by thickening according to the used methodology. The calculation results of 100% of the assigned ion are listed in the table. Results of the analysis. (Sample volume 100 ml, eluent volume 5 ml, msorb=100 mg, P=0.95, N=5)

| Bovine liver samples | found,Pb(II)<br>X± $t_pS/\sqrt{n}$, mq/kq | |
|---|---|---|
| Sample I<br>Sample II | 1,534±0,087 | 0,697±0,0086 |

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**PP – 12**

# Determination of Copper(II) in Different Water Samples

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Copper is considered the most important trace element for the body, as it protects our body from osteoporosis, increases immunity, affects brain activity, and at the same time stops the development of bacteria. Lack of copper in the body leads to memory loss, attention deficit disorder, and often diseases associated with colds. Copper is also involved in the synthesis of hemoglobin. The activity of the brain directly depends on the amount of this element in the body. Copper protects the body from free radicals. This is the most important function of copper. However, the excess of copper in the body also causes many complications. Copper enters the human body mainly through food. Most often found in seafood, legumes, corn, spinach, etc. contained in the products. The excess amount of this element in the environment, especially in waste water, causes its accumulation in some agricultural products. Therefore, the determination of microquantities of this element in environmental objects is of great importance.

For this purpose, the synthesis of new reagents and the development of copper determination methods are relevant. A new reagent based on acetyl acetone: sodium 2-(2-(4,4-dimethyl-2,6-dioxocyclohexylidene) hydrazinyl) terephthalate was synthesized. The complexation of this reagent with Cu(II) has been studied. Methods for the determination of copper in different water samples: sea water and waste water have been developed.

Sea water: 2 l of sea water (Turkan settlement, Caspian Sea) is evaporated until it boils. The obtained residue is dissolved in water, treated with 5 ml of HNO3 and heated at 50-60°C until HNO3 completely evaporates. The obtained solution is transferred to a 50 ml flask and diluted with water up to the line. Transfer 1 ml of the obtained solution to a 25 ml flask, add 3 ml of 1•10-3 M R, 1 ml of 1•10-2 SPCl and rinse (pH 3). Optical density 5 min. then they measure at λ-400 nm in KFK-2 in a l=1 cm cuvette. It was found by photometric method (3.30±0.02)•10-5%, by atomic-absorption method (3.31±0.01)•10-5% [1].

Determination of copper in wastewater:

Proportion 1 to 1 of waste water ("Azerneftyag") is taken for analysis. They evaporate the water without boiling until a precipitate is obtained. The obtained precipitate is dissolved in 5 ml of HNO3, transferred to a 50 ml flask and diluted to the line with distilled water. To determine copper, a 1 ml aliquot is taken and transferred to a 25 ml flask, and 2 ml of 1•10-3 M R, 1 ml of 1•10-3 SPCl are added to it and diluted with pH=3 buffer solution to the line. The optical density of the solution is measured in KFK-2 at λ=490 nm (l=1 cm) after 5 minutes. By photometric method (5.04±0.01)•10-5%, by atomic-absorption method (5.02±0.02)•10-5% copper was found[2].

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**PP – 13**

# A New Pillar[5]Arene Derivative Including Five Quinoline Fragments

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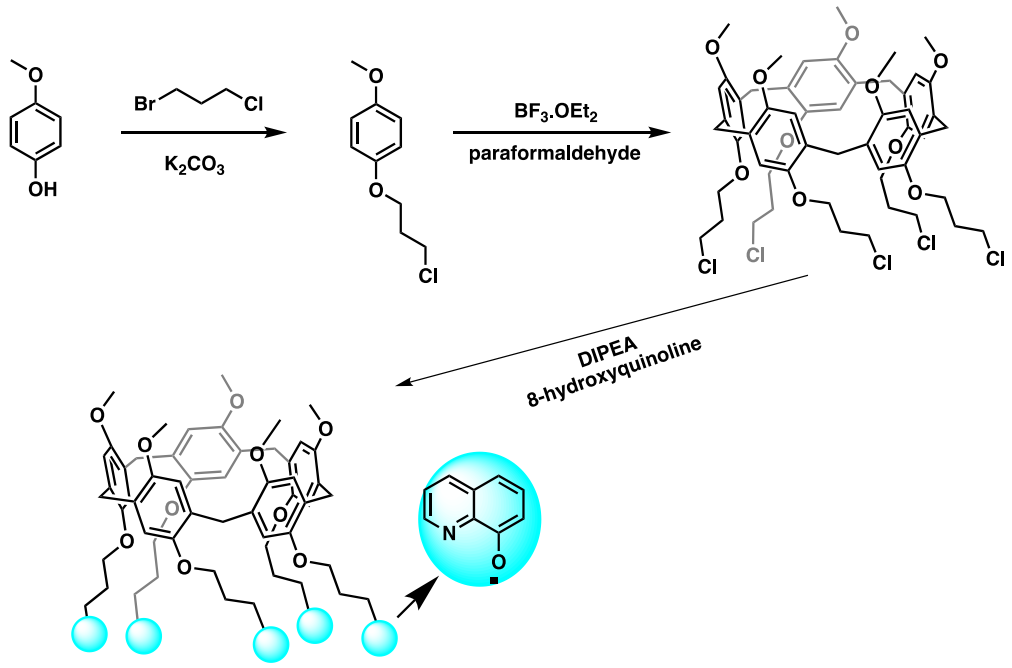
Pillar[n]arene macrorings were introduced in supramolecular chemistry after other macrocycles such as calix[n]arenes, cyclodextrins, cucurbit[n]urils. Pillar[n]arene macrocycles can be presented as analogues of calixarene derivatives that are different in hydroquinone groups since they are bridged in para-positions by methylene linkages forming pillar-like conformation while meta-bridged calixarenes are in basket conformation [1,2]. The sensor applications of macrocyclic compounds are useful in biochemistry and material chemistry. The similarity between pillararenes and calixarenes compounds assume that the latter can be employed for design of the complexometric interactions with diverse applications. A new Pillar[5]arene derivative including five quinoline fragments was obtained from 1-(3-chloropropoxy)-4-methoxybenzene prepared with 8-hydroxyquinoline. Paraformaldehyde and $BF_3OEt_2$ were used for the synthesis of Pillar[5]arene under appropriate conditions.

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**PP – 14**

# Investigation of Interaction with Lead Using Naproxen as Waste Drug by Fluorimetric Method

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Most of the wastes and wastewater generated after industrial activities contain heavy metals. Heavy metals have become an important issue in recent years due to their mixing with surface and underground waters and the potential risks they pose on living things. Heavy metals are not biodegradable and tend to accumulate in living organisms. Although some of the heavy metals are necessary at certain concentrations for vital activities, they show various toxic properties at high concentrations. Therefore, the development of an inexpensive, applicable and reliable method for the determination and removal of heavy metals in wastewater is of great importance in terms of protecting human health and ecological balance. It is thought that it will be an economical method to investigate the use of waste drugs for the determination of heavy and toxic metals that can be found in wastewater, especially in industrial wastewater, and to develop a method for both the evaluation of wastes and the determination of toxic heavy metals such as lead in wastewater.[1,2] For this purpose, naproxen, a non-steroidal anti-inflammatory drug, was used as a waste drug (expired), due to its molecular structure suitable for forming complexes with metal ions. The metal binding properties of Naproxen (NPRX), one of the NSAIDs, were investigated using the spectrofluorimetric method. The interaction of Pb (II) ion with NPRX and optimal experimental conditions were determined by a fluorimetric study. The excitation and emission wavelengths of the NPRX-Pb(II) complex were determined as $\lambda_{ex}$= 295 nm and $\lambda_{em}$= 359 nm in pH 3.0 and water media, respectively. Fluorescence intensity values were measured from emission spectra taken after the solutions were left for 15 minutes for complex formation. Under the specified experimental conditions, the calibration graph ($[Pb^{2+}]$ - F graph) was plotted and under optimal conditions the linear study range for the NPRX-Pb (II) complex was determined. Limit of detection (LOD) and limit of quantification (LOQ) were calculated. The fluorimetric method based on the complex of NPRX with Pb (II) ions was applied to industrial waste water.

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**PP – 15**

# Wastewater Pollution Prevention for Volatile Organic Compounds (Benzene, Toluene, Ethylbenzene, and Xylene) Using Cloud Point Extraction and Regeneration of Surfactant by Evaporation.

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Preserving and protecting the environment have become of prime importance. Due to the increase of human population, industrial developments and human activities, there are higher levels of environmental pollutants. Water, one of the most valuable resources, is polluted by contaminants such as volatile organic compounds (VOCs): for instance, the flammable aromatic hydrocarbons, benzene, toluene, ethylbenzene and p-xylene (BTEX), important components of fossil fuels and refinery products, and widely used as industrial solvents in the petrochemical and fine chemicals industries, are likely to create significant nuisances [1-2]. Several conventional separation techniques for the removal of BTEX from water have been proposed. However, it is important to note that, despite the availability of different methods, the efficient removal of BTEX is still an important problem possibly attributed to significant drawbacks associated with these processes, such as limited applicability, extended treatment time or higher costs of operation[3] ) . In this work we have opted for an intense, revolutionized and practical technique, namely the cloud point extraction process by nonionic surfactant readily biodegradable. The results obtained for each parameter were represented on three-dimensional diagrams using response surface methodology (RSM). The optimal tradeoff between the parameters which govern the efficiency of the extraction according to the initial concentration of surfactant and the temperature was infestigated. E% increases with surfactant concentration and hydrocarbon alkylation degree in the following order: B < T < E < X, with respective maximum values: 75, 83, 90, and 95%. On the other hand, Surfactant recycling and solute recovery were performed for the four pollutants, this step has a substantial role in cost reduction of the CPE.

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**PP – 16**

# Development of a Method for The Analysis of Co-Produced Gas Obtained in The Used Tyres Pyrolysis Process for The Determination of Tar

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The biggest problem with discarding old tyres is that they contain chemicals and heavy metals that leach into the environment as the tyres break down. Some of these chemicals are carcinogenic and mutagenic. One of the methods for solving the problem is pyrolysis of old tyres. Pyrolysis dissolves waste and also produces useful by-products. In this case, gas, liquid and solid phases are formed. Gas mixture containing tar, among other components. Traditional methods for tar sampling are based on cold solvent-trapping coupled with solvent absorption in impingers. The present work focuses on a solid-phase adsorption (SPA) method for determining the concentration of tar compounds. A modified sampling device consisting of 500 mg of amino-phase sorbent and 100 mg of activated coconut charcoal was chosen as optimal for sampling tar (including its volatile organic compounds) in gas produced in tyre pyrolysis. For research in a real life context, the double-layered fixed-bed reactor situated in south-eastern Latvia (Daugavpils region) was used. Varying volumes of pyrolytic gas were drawn through the adsorbents, and the total amount of tar was then compared to the number of its individual component compounds.

Tar was sampled at the pyrolytic gas temperature of 250°C. It was drawn through the adsorbent cartridges at the flow rate of 100 mL $min^{-1}$ for various periods of time, namely 1, 2, and 3 minutes, resulting in 100, 200, and 300 mL of the pyrolytic gas being drawn through the adsorbents respectively. When 100 mL of pyrolytic gas was passed through the adsorbents, the total tar concentration was 7348±228 mg $m^{-3}$; when 200 mL was collected, the concentration was 7412±302 mg $m^{-3}$; and at 300 mL the concentration was 7395±269 mg $m^{-3}$. When 100 mL of pyrolytic gas was passed through the adsorbents, about 60% of benzene and 75% of toluene were collected in the amino-phase adsorbent. Conversely, when 300 mL of pyrolytic gas was passed through the adsorbents, practically all of the benzene and about 70% of the toluene passed through the amino-phase adsorbent and collected on the activated coconut carbon adsorbent.

Testing the device consisting of 500 mg of the amino-phase adsorbent and 100 mg of activated coconut charcoal in real life conditions, along with varying volumes of the pyrolytic gas drawn through the adsorbents, gave results that were comparable in the total amounts of both tar and its individual component compounds. However, with an increase of the volume of pyrolytic gas drawn through the adsorbents, greater amounts of benzene, toluene, and other light compounds pass through the amino-phase adsorbent and are collected on the activated coconut charcoal. An increased volume of pyrolytic gas leads to a growing number of compounds detected and identified on the amino-phase adsorbent. It appears reasonable to take into account the concentration of tar in the pyrolytic gas while selecting the volume of gas for sampling, as well as whether it is necessary to detect those individual tar compounds whose concentration is very small.


**PP – 17**

# Temperature Dependence of Biogas Output Obtained from Aquaculture Waste

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The study investigates the possibilities of biogas production from aquaculture waste. Samples of sludge from fish farming basins were taken at a fish farm located in Nagļu parish, Rēzekne district (Latvia). Prior to experimental fermentation and biogas extraction, samples were analyzed for moisture and organic matter content. To increase the organic matter content available for fermentation, the sludge was mixed with crushed reeds. Biogas was obtained from the samples in different ratios of sludge and reed residue. During the experiments, 2.75 liters of biogas were obtained from the mixture of 1,200 g of aquaculture sludge from the fish farming basin and 100 g of crushed reeds. The results of experimental studies show that aquaculture residues can be used for biogas production.

For the implementation of experimental research bioreactor EDF-5.4_2 manufactured by “Biotehniskais centrs” (Latvia) was used. The aforementioned bioreactor has a compact, ergonomic and robust design fully customized for experimental research on biomethane production. Glass cylinder vessel is mounted between the upper lid and the metallic jacketed bottom. The design of the bioreactor is easy to maintain and apply basic operations and preparatory processes, in particular, washing and autoclaving.

95% of the total biogas produced during the fermentation period (60 days) was produced between days 10 and 55. The optimal retention times are approximately 36 days. The total volume of biogas produced during the fermentation period is approximately 2.75 liters - it corresponds to 2,155 mL $kg^{-1}$ waste. The optimal storage time is approximately 35 days at 40°C, 38 days at 37°C and 33 days at 43°C. The feed mixing mode during the experiment prevents the formation of dry and inactive flotation layers and can affect the optimal retention time. In this study, more than 95% of biogas can be produced in less than two months.

For three temperatures, the average cumulative biogas production in liters was measured and recorded daily. The influence of the temperature on cumulative biogas production is substantial. The temperature affects bacterial and archaeal community structure, diversity of microbiota and the high complexity of their interactions that mediate biogas production. Hence a detailed understanding of the temperature impact on microbiota is essential for the overall stability and performance of the anaerobic digestion process.

The yield of biogas during the bioprocess depends on the effect of temperature, the best results in our study were obtained at 40°C. During the experiment, 2.75 liters of biogas with an average methane content of 37.3% were obtained from a mixture of bog sludge and crushed reeds at 40°C. The highest proportion of methane in biogas, i.e., 40.16% was at 43°C, but at this temperature regime the total amount of biogas turned out to be about 15% lower. The worst results were obtained at 37°C – both in terms of biogas volume and methane content.


**PP – 18**

# UV Curable Polyesters Used in Preparation of Polymeric Networks Containing Pendant Acid Groups to Remove Cationic Dyes from the Water

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Because petroleum-derived polymers harm the environment and human health and are more expensive as a result of rising oil costs, bio-based polymers are now being employed as an alternative. In biotechnological and agricultural applications, as well as in researches aimed at preventing environmental pollution, the use of bio-based and biodegradable polymers is significant.

Environmental contaminants that impair both human health and ecosystems include heavy metals, dyes, pesticides, and surfactants [1]. Nowadays, there are various ways to remove dyes from the environment, such as adsorption, chemical precipitation, electrochemistry, ion exchange, nano-filtration, reverse osmosis and solvent extraction. Adsorption is commonly favored among these techniques because to its great effectiveness, low cost, and simplicity of usage [2].

In this study, bio-based oligomers that are UV-curable were developed. These oligomers are used to create bio-based polymeric adsorbents, which are used to remove pollutants. Using PEG 400 and itaconic acid, UV-curable polyester oligomers with unsaturated double bonds and different acid values were produced.

With the use of FTIR, GPC, and 1 H NMR, the polyester oligomers were characterized. Using a UV-Vis spectrophotometer, the adsorbents' adsorption capacities were determined. The ability of the synthesized adsorbents to adsorb dye were found to be enhanced by increasing the acid value in their structural composition.

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**PP – 19**

# Metal Adsorption Studies in Aqueous Solutions Using Polymeric Network Structures Derived from Dendrimers and Bio-based Chemicals

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Water is an essential resource for human health and development. Water pollution has become a serious environmental problem as a result of the rapid increase in population over the years. Metal pollutants that pollute the environment and are not biodegradable cause health and environmental issues. As a result, metal removal studies have become an important research topic.[1]

Research has increased in recent years to expand the use of bio-based chemicals as an alternative to petroleum derivatives in polymers due to the fact that they are environmentally friendly, sustainable, and do not harm human health or the environment. Dendritic polymers, also known as dendrimers, are the fourth most significant class of polymers after cross-linked, linear, and branched polymers. These macromolecules have three dimensions and have extremely smooth, branching architectures that extend from a multifunctional center. They have particularly intriguing physical and chemical characteristics as a result of their structural characteristics. Dendrimers can develop exponentially by symmetrically adding new groups from the core, and the utilization of cores, endgroups, and void spaces determines how well these structures work.[2]

In this study, bio-based phytic acid and ethylene diamine cored PAMAM type dendrimers (G1) were synthesized and modified with glycidyl metacrylate (GMA). FTIR-ATR (Attenuated Total Reflection-Fourier Transform Infrared Spectrometer) and 1H-NMR(Proton Nuclear Magnetic Resonance) were used to characterize the acquired products. These two products were subjected to UV curing to create a polymeric network as an adsorbent. Metal removal investigations from aqueous solutions were conducted using this absorbent. Studies were carried out utilizing the univariate approach, and UV-Visible Spectrophotometer was used for analysis.

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**PP – 20**

# Determination of Copper at Trace Levels by Employing DES/Dithizone Based Dual Detection Methods

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The chemical and biological processes in the environment are known to be strongly impacted by copper. Although, the presence of this metal is vital for environmental cycles, in high concentrations, can cause a toxic threat to the same cycles and processes[1,2]. Therefore, it is so critical to determine and control their levels in real samples. In this study, an analytical method was developed for the determination of copper with the FAAS system with high accuracy/sensitivity. In this developed method, copper was determined by increasing the detection power of the flame atomic absorption spectrophotometry (FAAS) system after pre-concentration of copper. For this purpose, dithizone (DZ) was used to form Cu-Dithizone (Cu-DZ) complex and a colorimetric probe (a mixture of DZ and a deep eutectic solvent (DES)) was created for the separation of copper ions from aqueous solution. Thanks to the developed probe, the complexation and extraction steps were carried out simultaneously, thus, reducing the experimental steps. As a result, a simple application and relatively low error was achieved for trace copper determination. The experimental parameters were optimized by univariate optimization steps. Detection power of the FAAS system in the determination of copper (according to the LOD value of the FAAS system) was enhanced as 24 times. Eucalyptus tea samples were used to determine the accuracy of the method developed in the study. The results proved that the developed extraction method is suitable for the precise and accurate copper determination. In addition, color intensity changes are measured depending on the concentration of Cu-DZ complexes using DES/DZ, which was used as the colorimetric probe in the method, and a digital image-based colorimetric detection (DIC) system designed with a simple styrofoam box. Thus, the relationship between the change in the intensity of the red color in a linear RGB scale and the copper concentration is determined.

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**PP – 21**

# Comprehensive and Accurate Method for The Treatment of Hormones in Wastewater Samples with the UV-assisted Fenton Digestion

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Estrogens, such as estradiol, estrone, and estriol, are important female hormones necessary to protect breasts, reproductive tissues, skin, and brain health[1]. These hormones are primarily excreted by humans and animals, and also can be released into the aquatic environment[2]. For this reason, advanced treatment methods are necessary to remove hormones having the potential to exhibit endocrine-disrupting properties such as estrone from the aquatic environment. In this study, a UV-assisted Fenton digestion treatment method was developed. β-estradiol and estrone hormones were simultaneously determined by high-performance liquid chromatography (HPLC) system. The chromatographic conditions such as injection volume mobile phase composition, sample flow rate, and wavelength were optimized. β-estradiol and estrone hormones in the aqueous sample solution were removed by UV-assisted Fenton digestion reaction and determined by high-performance liquid chromatography with UV detector (HPLC-UV) system. For this aim, a UV-light assisted digestion system designed by our research group was used[3]. Under the optimum conditions for both chromatographic and digestion systems, the removal efficiency of the UV-assisted Fenton digestion reaction was evaluated. The developed method was applied to wastewater samples and satisfactory removal efficiencies were recorded.

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**PP – 22**

# Analytical Methods for Quality Control of Bioproducts – Bulgarian Wine

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Wine is one of the Bulgarians' symbol and is famous all over the world. In the recent years the consumers are interest mostly to bio-products and want to be sure on their quality. Many wineries in Bulgaria which produced different traditional wines (monovariety and bouquet) claimed their wines as organic wines. For the production of organic wine, the whole technology needs to be certified organic, according to Amended EU Regulation No. 848/2018 on authorized oenological practices and treatments in organic winemaking. However, transfer of chemical elements in the system soil/grape/wine is important factor defining quality of wine and its authenticity. In the present research 34 monovariety Bulgarian wines with proofed geographical and botanical origin were analyzed for 44 chemical elements. The content and levels of chemical elements in the wines are related to their levels in soils and hence in the grapes, their content in possible fertilizers with which the soil was cultivated and their content in the irrigation water and also possible contamination from the vessels for wine storage. Regarding to this the selected chemical elements content is determined also in the soil extracts (acetate and EDTA as potential bio accessible fraction), vine leaves and must samples in accordance with each final wine sample. From the results obtained the statistically significant correlation coefficients (>0.5) were calculated for the system soil extract acetate/EDTA/leaves/must/wine. It was observed that relatively low values for coefficients were obtained as well for essential, as well for potentially toxic elements. This leads to conclusion that the vine controls the bio assimilation of essential elements and also the vine has a mechanism to reduce the intake of toxic elements, regardless of their content in soils. It was found correlation between the ratios of lithophile elements presented as macroelements in analyzed wines (Rb/Sr; K/Fe; Na/Mn). Also correlation was observed between microelements As/Cd; As/Sb As/Co; Ni/Co. In addition, good correlation between Li (macroelement) with Sr and Rb, and also Co (microelement) with K and Al were calculated. The elements as Li, Rb, Sr have higher capacity to identify the geographical origin of wines.

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**PP – 23**

# Determination of Chlorfenson in Green Tea Samples using Solid Phase Microextraction Strategy by High Performance-Liquid Chromatography-Ultraviolet Detection

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The chemicals known as pesticides are used to protect plants from pests or other harmful species[1]. Pesticides can contaminate the environment if they are used excessively without control, which poses a major risk to both human health and the environment[2]. This leads to a critical demand for the ability to identify and determine the amount of pesticides in various environmental compartments. In this research, a vortex assisted dispersive micro solid-phase extraction (VA-DMSPE) method based on reduced graphene oxide- magnetite nanocomposites (rGO-$Fe_3O_4$-NC) was developed for chlorfenson detection as pesticide by HPLC with UV detector. In order to extract chlorfenson, rGO-$Fe_3O_4$-NC was synthesized and used as an adsorbent. To achieve the maximum chlorfenson detection power in the high-performance liquid chromatography-ultraviolet detection system, the extraction parameters including the buffer volume, buffer pH, eluent volume, sorbent amount were optimized. Limits of detection (LOD) and quantification (LOQ) were calculated under the optimal conditions. The proposed method's accuracy was verified on green tea samples, where high percent recovery values for two different green tea samples were obtained.

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**PP – 24**

# Investigation of Electrochemical Behavior of Salophen Based Nitro Group on Solid Electrode Surface

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Salophen and its derivatives are considered excellent ligands in coordination chemistry due to their many properties (easy synthesis and structural diversity). The complexes can be used in a variety of applications (biological and technological), with high stability and structural versatility. [1-4]

In this study, the electrochemical behaviour of salophen was investigated on the glassy carbon electrode surface by electrochemical and spectroscopic methods. The glassy carbon electrode was modified with salophen based nitro group to prepare a new surface. Electrochemical experiments were carried out in different pH environments at different scan rates and number of cycles to determine the optimum conditions. Metal determination studies were carried out using the prepared electrode in order to examining the impact on the environment. As a result, it was determined in this study that salofene was sensitive to metal ions because it was a Schiff base at different pH and concentrations.

**Keywords:** Salophen, schiff base, electrode modification.

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**PP – 25**

# Investigation of Humic Substances in Leonardite Mineral Using Analytical Methods

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Leonardite, which is a completely natural organic material that has not reached the coal level, containing high levels of humic acid (HA) and fulvic acid (FA), is brown coal that has not been fully lignified.[1] Leonardite owes its soil regulating positive properties to phenolic substances containing humic components. Leonardite contains 25-85% HA, while soils contain only 1–5% HA on average. Alkaline conditions are required for the extraction of humic substances from leonardite. HA and FA, which become soluble in alkaline medium (0.25 M NaOH), are separated from insoluble humin residues. In this study, FA+HA, FA and HA extracts were obtained from commercially available solid and liquid leonardite mineral samples by applying alkaline and acidic solubilization steps, respectively. UV-Visible spectra of the extracts obtained from solid and liquid leonardite samples were monitored. Since the antioxidant properties of HA and FA have been reported in many studies, the total antioxidant capacity (TAC) and total phenolic contents (TPC) of the obtained HA, FA and HA+FA extracts were evaluated by CUPRAC and Folin methods. In addition, the molar absorption coefficients were determined by drawing calibration graphs with the CUPRAC and Folin method of the HA standard. By chromatographic analysis of solid and liquid leonardite minerals, the peaks of HA and FA components and the PDA spectra of each peak were displayed. In this study, the QUENCHER method, applied directly to the solid sample without extraction step, was successfully adapted to the CUPRAC method for determining the total antioxidant capacity of solid leonardite minerals.[2]

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**PP – 26**

# Synthesis of Bismuth Nanoflowers with a Microwave Assisted Hydrothermal Method and Its Application for the Removal of Copper in Tap Water Samples

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Copper, which has been widely used in many areas, is considered as a heavy metal. Heavy metals are found in wastewater generated after industrial activities[1,2]. As a result of the discharge of copper waste water, which is formed as a result of industrial activities, it has a harmful effect on the environment and therefore on people. Different methods such as chemical precipitation, coagulation - flocculation, ion exchange and solvent extraction are used for the removal of copper in wastewater[1,3]. These methods have disadvantages such as incomplete removal of heavy metals, high energy costs and high labor[3,4]. In this study, bismuth sulphide based nanoflowers were used to remove copper contaminant in wastewater samples. For this aim, bismuth sulfide nanoflowers are synthesized with a microwave assisted hydrothermal method by modifying the reported synthesis procedure[5]. The use of microwave has outstanding features such as reducing the long synthesis process and increase the yield by obtaining more products in a short time comparing to the conventional hydrothermal method. In the study, univariate optimization studies were performed to achieve the highest removal efficiency by determining optimum experimental conditions. After the optimum removal conditions were determined, the removal efficiency was calculated by using tap water samples. High removal efficiency indicated that the developed method can be used for copper removal from the water samples.

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**PP – 27**

# Features of the Cemical Composition of the Soil of the Ribbon Forest and its Restoration After Fires

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The most important feature of the Siberian forests of Russia is the preservation of natural plantations in large areas, relatively weakly affected by the anthropogenic impact of people. However, in many regions of Siberia, undesirable phenomena are observed, such as catastrophic natural fires dying in the fire, which is due to their high flammability and extremely slow recovery.

Fires cause a lot of harm to both humans and the environment. The upper fertile soil layer is especially exposed to harmful effects. After burning, the soil is subject to erosion. The soil becomes loose and prone to movement. Landslides are a frequent problem in burnt forest areas. Also, due to the destruction of the soil, swamping of the territory is possible, there is a risk of flooding. It takes several decades for the full restoration of the forest massif. The land, deprived of its fertile layer, does not accept vegetation.

In order for the land subjected to fire to begin to bear fruit again, it is necessary to thoroughly study this topic and use the most successful experience in restoring the soil cover.

The purpose of this work was to study the effect of forest fires on the physical and chemical properties of the soil and use the experience of the processes of its successful restoration. The paper presents an analysis of the literature on this issue, a comparison of soil restoration methods, and identification of the most effective methods in order to use them in practice.

**Keywords:** Physical and chemical composition of the soil, forest fires, soil restoration


**PP – 28**

# Smart Materials for Speciation Analysis

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Hyphenated methods combining chromatographic separation with highly sensitive ICP-MS detection are well known and preferable tool for speciation analysis. However, in most cases determination of only one chemical species of element is required for sample toxicity profile assessment. Smart materials incorporated in selective sensing procedure or in selective separation procedure are accepted as an efficient nonchromatographic fit-to-purpose approach in such cases. Selective solid phase extraction is widely used method for separation and determination of toxic analytes, however any simplification of analytical procedure is highly preferable. The application of membranes is useful approach for retention of analytes thus avoiding long centrifugation of in most cases very small particles. Several types of membranes were synthesized and applied for: multielement enrichment (chitosan, poly(vynil alcohol membrane loaded with noble metals nanoparticles), speciation of Cr (membrane based on Cr(III) imprinted poly(vynil alcohol)/sodium alginate/AuNPs) and for simultaneous speciation of Cr and Mn (hybrid nano-sorbent of membrane type based on poly(vynil alcohol-polyethylene oxide-tetraetoxysilane-Au@Starch NPs). Careful optimization of synthesis procedures ensured membranes with high mechanical stability, easy for manipulation. All attempts to find suitable elution agent failed and instead membrane dissolution is proposed for analytes recovery. In this way the membranes were not reusable but for each sorption/desorption cycle new membrane with high efficiency was used. Experiments performed with membranes from different batches showed good reproducibility of synthesis procedure – relative standard deviation for degree of sorption was between 4 and 8%. Extraction efficiency of membranes was tested with model solutions and real samples. Recoveries achieved varied between 95-98% for all studied analytes and toxic species. Electrothermal atomic absorption spectrometry or mass spectrometry with inductively coupled plasma were used as measurement methods. Simple and fast analytical procedures were developed and applied for analysis of surface waters, haemodialysis solutions and textile. Determination limits achieved depend on the instrumental method used but, in all cases, they fulfilled the requirements of national and EU legislation. Relative standard deviation is good due to simplicity of analytical steps and possibility to perform whole procedure in one vessel avoiding analyte losses or contamination.


**PP – 29**

# Manganese Ferrite Magnetic Nanoparticles Based Dispersive Solid Phase Extraction Before Flame Atomic Absorption Spectrometry for The Determination of Trace Level Cadmium in Lake Water

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In this research, trace level cadmium was determined by flame atomic absorption (FAAS) which method assisted by manganese ferrite magnetic nanoparticles based dispersive solid phase extraction (MF-MNP-DSPE). As solid phase extraction sorbent material magnetic manganese ferrite nanoparticles were synthesized and used. All parameters in the method were optimized with one variable optimization. After the calculations obtained with operating scientific data, improvement result was recorded that the MF-MNP-DSPE-FAAS strategy has low LOD value than the conventional FAAS technique. With lake water spiked at various concentrations, recovery strategies were carried out to evaluate the accuracy and application of the developed approach. It has been demonstrated that the method created with percentage recovery values utilizing a matrix matching calibration strategy can accurately and successfully determine the presence of cadmium in lake water samples.

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**PP – 30**

# Macro and Microelement Compositions of Persimmon *(Diospyros L.*)

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**ABSTRACT**

During the research, a comparative analysis of the content of macro- and microelements in the fruits of persimmon (Diospyros L.) samples belonging to varieties of local folk selection common in the Shaki-Zagatala economic region was carried out. Varieties of folk selection persimmons with the best biochemical parameters, which determine the nutritional and pharmacological value of persimmons, have been identified.

**Keywords:** persimmon, microelements, macroelements, minerals.

## 1. Introduction

Oriental persimmon (Diospyros kaki), which is a world industrial crop, occupies a special place among subtropical fruit crops. Oriental persimmon and other species of the genus Diospyros L. are valuable ornamental and most frost-resistant plants among all subtropical fruit crops. Despite the origin of D. kaki from the regions of East Asia, the modern area of cultivation covers almost all tropical and subtropical regions of the Earth. According to the FAO, the leading positions in the production of persimmons in the world are occupied by Asian countries - 88.9% (5.1 million tons); Europe - 7.9% (0.46 million tons); America - 3.2% (0.18 million tons); Oceania - 0.1% (0.03 million tons) (FAOSTAT, 2017).

In Azerbaijan, the territory of persimmon cultivation is concentrated in Balakan, Zagatala, Shaki, and Gakh regions. Traditionally, most varieties were bred in China, Japan and Korea. However, breeding work is also underway in Russia, Turkey, Spain, etc. (Sato et al., 2016). World selection of persimmons aims to obtain new varieties with different periods of vegetation and flowering, lack of astringency in fruits and their increased keeping quality, quality (with a high content of biologically active substances) and yield. Breeding is also underway for resistance to biotic and abiotic environmental factors, optimal plant habits, and others. One of the important economically valuable traits in persimmon breeding is the lack of fruit astringency. It is well known that tannins, in particular tannins, are the source of the astringency of persimmon fruits. Persimmon fruits, during their development, accumulate a large number of proanthocyanidins (P.A.), also known as condensed tannins, causing dryness and astringency due to their astringency. Based on the degree of manifestation of astringency, persimmon varieties are divided into several groups. It has been found that persimmon varieties from a particularly valuable group of constantly pollinated and non-astringent varieties lose astringency as the fruit ripens (Chen et al., 2017). Persimmon fruits are rich in biologically active substances. Persimmon fruits contain 25% sugar, 1.16-1.61% protein, 2.96-53.32% vitamin C, 20% carotene,

and 0.3-0.85% fat. The presence of potassium, sodium, calcium, magnesium and phosphorus are essential indicators of the nutritional value of persimmons. Potassium, sodium, magnesium and phosphorus are contained in fruits in the form of salts of inorganic acids, calcium - in water-soluble, acid-soluble and adsorbed forms. Calcium is involved in implementing the processes of nervous excitability, muscle contraction, blood clotting, and bone tissue formation. According to the degree of iodine accumulation in the fruits, persimmon is second only to feijoa. Iodine is involved in the formation of thyroxine and the regulation of metabolism. (Abdurakhmanov et al. 2004).

## 2. Materials and Methods

The studies were carried out at the National Center for Nuclear Research of Azerbaijan on an X-ray fluorescent apparatus OMEGA-4000 (INNOV-x, USA). Fully ripened persimmon fruits collected in 5 districts of the Sheki-Zakatala economic region were taken as the study material. Fruit picking was carried out in early December 2021. The relief of the region is divided into highlands and foothills. The zone has a temperate climate. The hottest months of the year are July and August when the air temperature can exceed 30 $^{o}$C.

Statistical data processing was performed using the SPSS 12.0 software package for Windows.

## 3. Results and Discussions

To maintain efficiency and good health, a person needs minerals that play an important role in metabolic processes. Macro- and microelements are essential for the prevention and treatment of the cardiovascular, digestive and nervous systems, and the prevention of immunodeficiency. The mineral composition of the fruits of the studied persimmon varieties was studied for the presence of potassium, calcium, sodium, phosphorus, magnesium, iron, zinc and iodine (Table 1.2). The studied varieties differ from each other in the ability to accumulate these nutrients in the fruits. The analyzes showed that the fruits collected in Gabala and Gakh regions are the richest in macroelements, and the fruits collected from almost the entire region are rich in microelements, but no notable differences between the regions were revealed in terms of iodine content. Samples of the Gabala region lead in the content of potassium, magnesium and sodium, Balakan, Shaki, Zagatala and Gakh regions - iron and zinc, and the Gakh region - in the content of phosphorus.

The presence of potassium, sodium, calcium, magnesium and phosphorus is an important indicator of the nutritional value of persimmons. Potassium, sodium, magnesium and phosphorus are found in fruits in the form of salts of inorganic acids, calcium in water-soluble, acid-soluble and adsorbed forms. Potassium and sodium actively influence the processes of water-salt metabolism. Calcium is involved in the implementation of the processes of nervous excitability, muscle contraction, blood clotting, and most importantly, the formation of bone tissue. The fruits of the studied samples turned out to be rich in potassium (Gabala and Gakh - 78.7 and 71.8 mg/100 g) and calcium (Gabala and Gakh 10.5 - and 10.7 mg/100 g).

It is known that plant products are rich in magnesium and often provide 2/3 of its intake with food. Magnesium is a cofactor for a number of important enzymes of carbohydrate-phosphorus and energy metabolism. It was determined that in terms of the amount of this macroelement (69.3–74.7 mg/100 g), the studied varieties are second only to watermelon, which contains an average of 200 mg/100 g of magnesium.

Phosphorus plays an essential role in the functioning of the nervous system and in genetic processes, as well as in protein biosynthesis and cell transformation. The recommended intake of phosphorus for an adult in our country is about 1200 mg per day (Guseinova B.M. 2017). The fresh fruits of the studied persimmon contained phosphorus from 23.9 (Zakatala region) to 25.6 mg/100 g (Gakh region).

Microelements iron and zinc present in persimmon fruits in all studied varieties are able to form complexes with the corresponding groups of substances (ligands), which increases their ability to participate as specific catalysts for the most important metabolic processes.

The highest amount of iron was found in fresh fruits of samples from the Balakan and Zagatala regions - 2.7 µg / 100 g. It is known that persimmon fruits, in terms of iodine accumulation, are second only to feijoa fruits.

Table 1. The content of microelements in the composition of persimmon

| Collection point | Microelements, µg/100 g | | |
|---|---|---|---|
| | Fe | Zn | Y |
| Balakan district | 2.7 | 0.6 | 0.4 |
| Gakh district | 2.4 | 0.7 | 0.4 |
| Zagatala district | 2.7 | 0.6 | 0.4 |
| Gabala district | 2.3 | 0.6 | 0.4 |
| Shaki district | 2.3 | 0.7 | 0.4 |

Table 2. The content of macroelements in the composition of persimmon

| Collection point | Macroelements, µg/100 g | | | | |
|---|---|---|---|---|---|
| | K | Na | Ca | Mg | P |
| Balakan district | 47.8 | 49.4 | 8.7 | 69.5 | 24.6 |
| Gakh district | 71.8 | 47.3 | 10.7 | 69.3 | 25.6 |
| Zagatala district | 60.8 | 51.1 | 10.0 | 73.9 | 23.9 |
| Gabala district | 78.7 | 55.7 | 10.5 | 74.7 | 25.3 |
| Shaki district | 61.5 | 53.2 | 9.1 | 70.2 | 24.6 |

Iodine is involved in the formation of thyroxine and the regulation of metabolism. An adult's need for iodine is 0.1–0.2 mg per day. An effective way to optimize the body's supply of iodine is to consume foods rich in this trace element.

Information on the concentration of iodine in persimmon cultivated in Azerbaijan is vital for the population of the Shaki-Zagatala region of our republic, which is a biogeochemical province where the content of this element in water and soil is insufficient.

## 4. Conclusions

Studies of the biochemical composition of persimmon fruits collected in the Shcheki-Zakatala economic region showed that the natural conditions of the region contribute to their accumulation of valuable nutrients. According to the content of Ca, K, Mg and Na, samples of the Gabala and Gakh regions

are distinguished. According to the accumulation of Fe – Balakan and Zagatala regions, Zn - Gakh and Sheki regions, more phosphorus is present in the sample fruits of the Gakh region than in samples of other regions. No significant differences were found between the samples in terms of iodine content. The data obtained can be used in the development of recipes for new functional products containing persimmon.

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**PP – 31**

# Separation and Sensitive Detection of Listeria Monocytogenes Using Specific Aptamer Immobilized Magnetic Adsorbent and a Novel QCM Apta-sensor

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In this work, a novel quartz crystal microbalance (QCM) aptasensor is designed for the diagnosis of *Listeria monocytogenes* bacteria. It is an important food-borne pathogen bacteria which cause illness in humans and animals. Because of Listeria monocytogenes's ability to grow and multiply at refrigerated temperature. Listeriosis are frequently occurred by consuming the contaminated food with L. monocytogenes. The method is based on the selection of *L. monocytogenes* bacterium from samples using *L. monocytogenes* specific binding aptamer (Apt) attached magnetic nanoparticles. In the first step, the surface of the polydopamine coated magnetic particles was grafted spontaneously with diaminopolyethylene glycol (DAPEG) as a second polymer layer by conjugation of PDA with DAPEG without using any activating agent ($Fe_3O_4$@PDA@DAPEG). Then, the selected specific binding aptamer for *L. monocytogenes* was immobilized on the as prepared magnetic particles. The saturation magnetization was determined as 36.4 emu $g^{-1}$ for $Fe_3O_4$@PDA@DAPEG. The magnetization curves of the samples exhibited zero refraction tendency exhibiting superparamagnetic properties. This result showed that the prepared $Fe_3O_4$@PDA@DAPEG particles could be easily separated from the solution using an external magnet. The aptamer immobilized magnetic nanoparticles were used for the pre-concentration of the target bacterium, and the same aptamer sequence was also immobilized on the QCM chip using above route and used for the quantitative detection of *L. monocytogenes* using QCM aptasensor. The contact angles and surface energy parameters of bare sensor chip, poly(dopamine), diaminopolyethylene glycol-coating and aptamer ligand attachment were determined using different test liquids (i.e., water, glycerol and diiodomethane). Accordingly, the hydrophilicity of the chip surface increased with the addition of hydrophilic functional groups and prevent non-specific interactions with the sensor surface. The detection limits of the QCM aptasensor were less than 10 CFU/mL. The synthesized magnetic particles exhibited a good permanence and high isolation recoveries for the pull down of the target bacterium from food samples, after recycling ten times. The method was successfully applied to target bacterium examination in food samples.

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**PP – 32**

# Preparation of Molecular Imprinting Polymer for Arsenic Removal from Aqueous Solution

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With the metal, petrochemical, paper, coating and electroplating, mining, and textile industries and increasingly vital activities, aquatic environments contaminated with heavy metals have become the common problem of the world. Due to the negative effects of toxic heavy metals on the ecological system and human health, environmental improvement processes including the development of alternative enhancement techniques or methodologies are still among the research topics that are emphasized. Removal of toxic metal ions from wastewater systems with high selectivity and efficiency is often a challenging process, depending on many parameters, and may require expensive equipment. Recently developed molecular imprinting technique can provide these requirements. Molecular imprinting is a technology for creation of specific binding sites with retention of the shape, size and binding sites of the template molecules. In this technique three-dimensional micro-cavity can be created in a polymer structure via copolymerization of functional monomers and cross-linkers in the presence of target ion that act as template molecules based on coordination or electrostatic interactions. After removal of the template metal ions with an acidic desorption agent, recognition cavities can be realized in the cross-linked polymer structure.

In the presented study, it has been shown that As(V) heavy metal ions known to have toxic and carcinogenic effects, can be removed from the aqueous medium with high selectivity and efficiency through an adsorption process on the prepared As-ion imprinted polymer. For the preparation of As(V)-MIP, the polymerization mixture was prepared from 4-vinyl pyridine as a functional monomer, ethylene glycol dimethacrylate as the cross-linking monomer and As(V) as a template ion. The polymer was synthesized by using azobis-isobutyronitrile as an initiator and ethanol as a pore forming agent in a die. The adsorption amounts for As(V) was 73.8 mg/g by the prepared MIP. The Langmuir model fitted well with the obtained adsorption curves, with $R^2$ values higher than 0.992. The effect of various cations such as nitrate, sulfate and phosphate ions on the selective adsorption efficiency of As(V) was also tested in aqueous medium and it was found that the presence of other ions did not cause any significant effect on arsenic removal performance.


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# Degradation of Mussels (Mytilus Edulis) Samples for Microplastics Identification

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Microplastic pollution in the aquatic ecosystem has been a worldwide environmental problem. The consumption of commercial fish and seafood is one of the most important routes of human exposure. The degradation of biological tissues is the first step in the pre-processing to identify microplastic in biota. Many types and concentrations of chemicals (NaOH, H2O2, KOH, HNO3, NaClO, HCl, NaI, trypsin etc.) are used for this purpose [9-11].

Microplastic assays are studies with a high risk of contamination. Necessary precaution should be taken to minimize this risk. The study was carried out both in cleanroom and normal laboratory conditions. In this study, microplastic levels were determined in commercial mussels (Rope Mussels; Mytilus Edulis) from the Baltic Sea (Baltimore, Cork, Ireland) by degradation with sodium hydroxide and hydrogen peroxide. The mussel samples were divided into three groups, with 20 mussels in each group, by washing the outer parts with deionized water. After biometric measurements, the inner part of each mussel was washed with deionized water, and the muscle part was separated from the shell and batched in a clean container. Degradation solutions (NaOH,1M; $H_2O_2$, 30% and 12%) were added at approximately twice the amount of the mussel sample. The mixtures were stirred overnight and then left to stand for 5-6 hours. The degraded samples were filtered with 0.45 μm filter paper and placed in petri dishes. It was concluded that the use of sodium hydroxide as a degradation solution is more suitable than the use of hydrogen peroxide in terms of analytical processes.

The MPs were picked using a tweezers and placed gently on a prepared glass slide with double sided tape already in position to determine the chemical characterization by Raman spectroscopy [12].

Numerous contaminants were found in the blank samples made under normal laboratory conditions.

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# Surveillance of Selected Pharmaceuticals in Erzurum Biological Wastewater Treatment Plant

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**ABSTRACT**

Classical wastewater treatment plants do not sufficiently remove the increasing level of micropollutions in the receiving water bodies, especially with the growing use of pharmaceuticals and industrial chemicals. The wastewater treatment plants are considered the main source of micropollutants spreading to the aquatic ecosystem, whose diversity and concentration have increased in surface waters in recent years. Increasing concerns about their potential impacts onto human health and aquatic environment have caused the implementation of analyzing studies to monitor the quality of tap water and surface water bodies. The surveillance of selected micropollutants, were investigated in this study. For this purpose, five pharmaceuticals; diclofenac, amitriptyline, sertraline, paracetamol, metformin were selected as the target compound due to their increasing concentrations in surface waters in recent years. These selected pharmaceuticals were monitored in the inlet and outlet wastewater flow in Erzurum Biological Wastewater Treatment Plant (BWWTP) for a one-year period considered the operating parameters of the Plant. For monitoring studies, diclofenac, amitriptyline, sertraline, paracetamol and metformin were analyzed by LC-MS/MS method in wastewater samples taken monthly for 12 months period.

**Keywords:** Erzurum Biological Wastewater Treatment Plant, Micropollutant, Pharmaceuticals

## 1. Introduction

Pharmaceutically active compounds included in several drug compounds may contain one or more mixtures of active substances produced for the diagnosis, treatment and alleviation of certain diseases, disorders, abnormal physical conditions or their symptoms that can be seen in humans and animals (Bila & Dezotti 2003). Drugs are classified according to their pharmacological action and intended use, their source, chemical structure, place of action and, the way they are prepared. Drugs consist of two parts: the active substance, which is the main chemical that has a physiological effect, and the chemical substances that are used for easier intake of the active substance, which have no physiological effect. The reaction mechanism of drugs in the body occurs in four different ways: absorption, distribution, metabolism and excretion (elimination/excretion), which are the processes after they enter the body (Anonymous 2022). These processes can also be considered as processes that directly control the environmental release of drugs. Drugs, a milestone in the scientific development of human history, have extended life expectancy, cured hundreds of deadly diseases, and thus improved the quality of life. But this success has also led to pharmaceuticals becoming rapidly growing major pollutants in environmental ecosystems. The loading of pharmaceuticals into the environment from livestock activities brings more important problems because, generally, human wastewater can be treated in urban treatment plants, while no treatment can be applied for wastes sourced by livestock activities and they are widely dispersed into the environment from a wide variety of sources. For this reason, the use of antibiotics and hormones promoted growth in livestock

activities, especially in developed countries, has been reduced in recent years (Wise 2002; Anonymous 2020; Sarmah et al. 2006).

The increasing use of pharmaceutically active compounds is increasing their presence in aquatic environments and causing them to become an environmental risk. A wide range of varying levels of pharmaceutical compounds have been reported in different environmental matrices, such as drinking water, surface water, wastewater, sediments and biota, frequently in global studies. However, it varies according to the country, region, area, consumption model and location of the production industry. Lipophilic nature of pharmaceuticals can cause bioaccumulation; metabolism, exposure and other uptake processes increase bioaccumulation. Bioaccumulation studies also reveal potential toxic effects to aquatic organisms. Because many pharmaceuticals exist at low concentrations in the aquatic environment for long periods of time, their toxic effects are more likely to be chronic than acute. The toxicity of pharmaceuticals in aquatic environments varies depending on the type of pollutant, exposure time, concentration, and developmental process to which organisms are exposed (Khan et al. 2020).

In accordance with this explanation, the occurrence of selected pharmaceuticals in wastewater treatment plants where is the main source from which they spread to the aquatic environment, were investigated. diclofenac is an analgesic-antiinflammatory, while amitriptyline is antidepressant-analgesic, sertraline is again antidepressant, paracetamol is antipyrethic and finally metformin is an antidiabetic pharmaceutically active compound. for this purpose, diclofenac, amitriptyline, sertraline, paracetamol and metformin were selected as the target compound and these pharmaceuticals were monitored in the inlet and outlet wastewater flow in Erzurum BWWTP for a one year period using the operating parameters of the Plant.

## 2. Materials and Methods

The target micropollutants were analyzed using the liquid-liquid extraction and LC-MS/MS method in wastewater samples taken from certain points, especially the entrance-exit points of the plant for 12 months between December 2020-November 2021. The samples were taken by expert from BWWTP in accordance with the sampling conditions and quickly delivered to the laboratory. Samples that could not be analyzed immediately were stored at +4 $^{o}$C for a maximum of 48 hours. LC-MS/MS (Agilent Technology 6460 Triple Quad LC/MC) analyzes were made in Atatürk University Central Laboratories (DAYTAM-Eastern Anatolian High Technology Application and Research Center). Liquid-liquid extraction method was used to separate and collect the target compounds in the samples before LC-MS/MS analysis. In this study, considering the methods used in the literature, methylene chloride (DCM) and ethyl acetate (EtOAc) solvents were used for the recovery of target compounds (Duca et al. 2014; Issa et al. 2020).

## 3. Results and Discussions

First of all, the monthly average parameters of the BWWTP where inlet and outlet wastewater samples are obtained have been shown in Table 1 and Table 2, respectively. Table 3 shows the diclofenac, amitriptyline, sertraline, paracetamol and metformin concentrations determined by LC-MS/MS analysis in the inlet and outlet wastewater samples taken from the BWWTP in the period of 12 months. In the first three months of the analysis, in December, January and February, only inlet wastewater samples were taken to detect the presence of target pharmaceuticals in wastewater. As a result of detection and analysis of target compounds

in wastewater, then, both inlet and outlet wastewater samples were started to be taken in the other months and, diclofenac, amitriptyline, sertraline, paracetamol and metformin concentrations were monitored.

**Table 1.** The inlet wastewater characteristics of Erzurum BWWTP

| | Q | Temp, °C | | pH | Conduc. | TSP | COD | $BOD_5$ | TN |
|---|---|---|---|---|---|---|---|---|---|
| | *$m^3$/day* | *Outside* | *Water* | | *µs/cm* | *mg/L* | *mg/L* | *mg/L* | *mg/L* |
| **January** | 70.848 | -11 | 11 | 8 | 821 | 152 | 366 | | 31 |
| **February** | 75.300 | -8 | 11 | 8 | 826 | 168 | 348 | 177 | 31 |
| **March** | 75.290 | -3 | 11 | 8 | 843 | 175 | 321 | 154 | 27 |
| **April** | 80.893 | 9 | 13 | 8 | 767 | 162 | 368 | 203 | 26 |
| **May** | 65.368 | 33 | 16 | 7 | 814 | 199 | 476 | 286 | 25 |
| **Juna** | 65.787 | 7 | 18 | 7 | 833 | 187 | 485 | 280 | 26 |
| **July** | 71.368 | 11 | 20 | 8 | 826 | 216 | 417 | 252 | 28 |
| **August** | 74.287 | 21 | 20 | 8 | 783 | 190 | 361 | 197 | 27 |
| **Sept.** | 79.343 | 16 | 19 | 8 | 797 | 225 | 398 | 246 | 27 |
| **Oct.** | 92.584 | 6 | 17 | 8 | 809 | 205 | 369 | 210 | 27 |
| **Nov.** | 86.213 | 1 | 13 | 8 | 848 | 208 | 393 | 217 | 28 |
| **Dec.** | 84.997 | -9 | 11 | 8 | 882 | 196 | 392 | 227 | 29 |
| **Mean** | 76.857 | 6 | 15 | 8 | 821 | 190 | 391 | 223 | 28 |

**Table 2.** The outlet wastewater characteristics of Erzurum BWWTP

| | Q | Temp, °C | pH | Conduc. | TSP | COD | $BOD_5$ | TN |
|---|---|---|---|---|---|---|---|---|
| | *$m^3$/day* | *Outside* | *Water* | | *µs/cm* | *mg/L* | *mg/L* | *mg/L* |
| **January** | 66.558 | 11 | 8 | 694 | 16 | 34 | | 7 |
| **February** | 72.000 | 11 | 8 | 703 | 17 | 31 | 18 | 9 |
| **March** | 72.323 | 11 | 8 | 729 | 17 | 32 | 19 | 7 |
| **April** | 75.620 | 13 | 8 | 655 | 14 | 30 | 17 | 5 |
| **May** | 63.813 | 16 | 8 | 690 | 14 | 29 | 17 | 4 |
| **Juna** | 64.750 | 19 | 8 | 723 | 13 | 31 | 18 | 5 |
| **July** | 70.148 | 21 | 8 | 717 | 14 | 30 | 17 | 8 |
| **August** | 72.984 | 21 | 8 | 681 | 15 | 33 | 19 | 8 |
| **Sept.** | 78.000 | 20 | 8 | 664 | 16 | 32 | 18 | 9 |
| **Oct.** | 89.416 | 18 | 8 | 683 | 19 | 37 | 21 | 8 |
| **Nov.** | 83.877 | 15 | 8 | 694 | 15 | 31 | 18 | 6 |
| **Dec.** | 80.906 | 13 | 8 | 742 | 16 | 34 | 20 | 6 |
| **Mean** | 74.200 | 16 | 8 | 698 | 15 | 32 | 18 | 7 |

**Table 3.** The inlet and outlet concentration of diclofenac, amitriptyline, sertraline, paracetamol and metformin in the BWWTP for December 2020-November 2021 period.

| | Diclofenac, µg/L | | Paracetamol, µg/L | | Amitriptyline, µg/L | | Sertraline, µg/L | | Metformine, µg/L | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Inlet | Outlet | Inlet | Outlet | Inlet | Outlet | Inlet | Outlet | Inlet | Outlet |
| **Dec.** | 76.944 | - | 254.111 | - | 2.124 | - | 2.816 | | 156.445 | |
| **January** | 103.020 | - | 350.991 | - | 0.211 | - | 0.282 | | 85.684 | |
| **February** | 139.849 | - | 1342.090 | - | 0.217 | - | 0.275 | | 106.054 | |
| **March** | 2.609 | 3.279 | 81.373 | 9.340 | 0.213 | 0.218 | 0.284 | 0.280 | 78.262 | 7.508 |
| **April** | 0.000 | 0.000 | 61.325 | 43.363 | 0.216 | 0.221 | 0.275 | 0.278 | 205.754 | 0.906 |
| **May** | 0.164 | 0.000 | 177.925 | 7.037 | 0.213 | 0.212 | 0.278 | 0.277 | 79.230 | 1.009 |
| **Juna** | 2.005 | 4.373 | 5.313 | 22.440 | 0.217 | 0.214 | 0.275 | 0.276 | 85.680 | 1.584 |
| **July** | 2.082 | 0.977 | 1.189 | 0.818 | 0.233 | 0.214 | 0.283 | 0.279 | 0.828 | 2.866 |
| **August** | 3.638 | 2.455 | 44.557 | 38.497 | 0.219 | 0.219 | 0.281 | 0.275 | 278.549 | 9.655 |
| **Sept.** | 55.326 | 70.112 | 3568.498 | 15.269 | 0.220 | 0.216 | 0.294 | 0.280 | 238.237 | 32.800 |
| **Oct.** | 22.967 | 26.332 | 997.809 | 8.185 | 0.213 | - | 0.287 | 0.282 | 362.016 | 8.177 |
| **Nov.** | 31.834 | 17.568 | 3935.709 | 5.623 | 0.215 | 0.214 | 0.283 | 0.2795 | 398.400 | 3.467 |
| **Average** | 36.703 | 13.900 | 901.741 | 16.730 | 0.376 | 0.216 | 0.493 | 0.279 | 172.928 | 7.552 |

According to the target pharmaceuticals inlet concentrations, diclofenac concentrations is ranged from 0 to 139,849 µg/L, while paracetamol concentrations is ranged from 1.189 to 3,568.498 µg/L, Amitriptyline is ranged from 0.211 to 2.124 µg/L, Sertraline is ranged from 0.275 to 2.816 µg/L and Metformine is between from 0.828 to 398.4 µg/L. While diclofenac concentrations in the plant inlet wastewater samples were quite low in the period between March and August, it was observed that it was at the highest level in the winter months, which is December-February; It was determined that paracetamol concentrations were at their lowest levels in June and July. According to the average concentrations, the treatment efficiencies were calculated as 62.1% for diclofenac, 98.2% for paracetamol, 42.6% for amitriptyline, 43.4% for sertraline and 95.6% for metformine, respectively. The highest treatment was obtained from paracetamol compounds in BWWT of Erzurum. The advanced treatment methods can be applied to remove residual pharmaceuticals compounds.

## 4. Conclusions

In this study, five important group of pharmaceuticals (diclofenac, amitriptyline, sertraline, paracetamol and metformin) were observed in the inlet and outlet concentration of wastewater from Erzurum BWWTP for 12 months period. The treatability of these selected pharmaceuticals as the indicator of non-treatability capacity of pharmaceuticals in wastewaters were calculated as 62.1% for diclofenac, 98.2% for paracetamol, 42.6% for amitriptyline, 43.4% for sertraline and 95.6% for metformine, respectively. The highest treatment efficiency was obtained from paracetamol compounds in BWWT of Erzurum. It is thought that the change in

treatment efficiencies in a wide range is due to both the seasonal changes affecting the concentrations of micropollutants and the effect of plant operating parameters on the treatability of the target pharmaceuticals. However, it has been observed in some studies in the literature that micropollutants have increased concentrations in the wastewater effluent. It has been suggested from the conversion of metabolites to the parent compound (Tüzün 2017). In order to be a guide for future studies, it is not easy to suggest an advanced treatment processes because the treatability of micropollutants in the wastewater treatment plant varies in a wide range. It is possible to increase the treatment efficiency of these pollutants by changing the plant operating parameters.

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