Сојуз на хемичарите и технолозите на Македонија

Society of Chemists and Technologists of Macedonia

15th Students' Congress of SCTM

BOOK OF ABSTRACTS

29th September - 1st October 2022
Institute of Chemistry
Skopje, N. Macedonia

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Society of Chemists and Technologists of Macedonia

29th September - 1st October 2022,
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Dear students, colleagues and guests,

It my great honor and privilege to extend to you all a very warm welcome to the Students' Congress of the Society of Chemists and Technologists of Macedonia. This is our 15th students' congress so, for us, it is a cause for a small celebration. The first students' congress was in 1994 (the organizers were Orhideja Grupče, Vladimir Petruševski, Minjas Žugić). The SCTM has a long tradition of organizing chemistry conferences, dating back to 1970; next year we will have the 26th Congress of SCTM, traditionally held in Ohrid.

First, I would like to greet and thank the student participants – I believe for many of you this is the first time to take part in such an event and a good practice for international conferences we believe you will attend in the future. That is why we are having it in English. This year we have about 30 oral presentations, in other words a full two-day schedule.

I would also like to thank the lecturers who have accepted our invitation – this time we have five. We have one from the University of Belgrade and one from the University of Zagreb and I sincerely hope you will enjoy your stay here in Macedonia. The other three are from the University Goce Delčev in Štip, the Faculty of Technology and Metallurgy and the Institute of Chemistry.

I would like to express my special gratitude to our honorable guests:

- Prof. **Elizabeta Gjorgjievska**, our First Lady and good friend of our Society who has found the time in her busy schedule to attend this opening ceremony in support of the young researches presenting their research at this Congress.
- Prof. **Aleksandar Skeparovski**, the Dean of the Faculty of Natural Sciences and Mathematics. I would like to take this opportunity to thank him for all the support he is giving our Society, including the financial support.
- Academician **Gligor Jovanovski** who was a driving force behind the Students' Congresses and we are all indebted to him for this.

Finally yet importantly, I would like to thank the organizers for the time and energy they have dedicated to ensuring that this event is a success. President of the Scientific Committee Prof. **Nataša Ristovska**, the president of the Organizing Committee MSc **Pece Šerovski** as well as the members from the Faculty of Technology, Prof. **Biljana Angjuševa** and Prof. **Dafinka Stoevska Gogovska**.

I do hope you will have a fruitful and enjoyable exchange of ideas these two days at the Institute of Chemistry!

Prof. Zoran Zdravkovski, president
Society of Chemists and Technologists of Macedonia

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PL-1

VOLTAMMETRY-100 YEARS ON

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This year marks the 100th anniversary of the first paper related to development of polarography published by Heyrovsky in 1922 [1]. In 1959, Jaroslav Heyrovsky has been awarded as a first electrochemist with Nobel Prize for the development of polarography. Polarography is an iconic electrochemical technique that is recognized as a predecessor of nowadays modern voltammetric techniques. Since the introduction of polarography in 1922, this technique evolved rapidly in various directions. Initially, the highly toxic mercury working electrode used in polarography was replaced by solid type of working electrodes, thus paving way to the development of voltammetry. Moreover, by using various modifications of the applied potential, the time of analysis has been rapidly shortened, while the sensitivity of novel voltammetric techniques has been increased dramatically, making possible to detect analytes even in sub-nanomolar concentrations [2-4]. Whenever one talks about electrochemistry, one intuitively creates image on processes of corrosion. Now it is well understood that majority of reactions taking place under physiological conditions are also of electrochemical nature. If there is an experimental protocol to measure the energy of electrons/charge exchanged between two conjoined systems in processes taking place in living systems, then it will be possible to get relevant information about enzymes activity, drug-drug interactions, ion transfer across cell membranes, mechanism of action of given biochemical systems and many more. The aim of this talk is to highlight some of the most important achievements of voltammetry and its applications in bioelectrochemistry, in development of biosensors, in kinetic characterizations of various chemical interactions, in ion transfer across liquid-liquid interfaces, in development of bio-fuel cells, in enzymatic redox chemistry and many more. While hints are given to some of the most important theoretical achievements of voltametric techniques, short advice to get better communication between the theoretical and experimental electrochemists is also presented. Since major role of voltammetry is recognized in constructing enzymatic and non-enzymatic biosensors, some of the major achievements and several drawbacks of applying voltametric techniques in designing sensors are discussed. The nanomaterials are almost inevitable part in majority of voltammetric experiments in last 20 years, and short part of the talk is dedicated to the role of nanostructures in voltammetry. Because the scanning electrochemical microscopy (SECM) is recognized as a most promising instrumental electrochemical system that can get voltammetry a step closer to probe even electrochemistry of single cells, some aspects of this technique are also shortly outlined. In the end, a series of directions in which voltammetry will develop in the coming years are also mentioned. The entire talk is designed in a way to motivate younger students to get more intensively involved in doing science.

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Key words: electrochemistry; voltammetry; biosensors; nanomaterials; scanning electrochemical microscopy**.**

PL-2

CHEMICAL AND ATMOSPHERIC PRESSURE PLASMA TREATMENTS, COST-EFFECTIVE WAYS FOR IMPROVING THE JUTE FABRIC PROPERTIES AND EXTENDING ITS LIFECYCLE

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The increased demand for cheap, biodegradable, renewable, and recyclable fibers with good electrical, thermal, sorption, and mechanical properties positioned jute in second place (after cotton) in the natural fiber world market. Multicellular jute fibers are recognized by their heterogeneous chemical composition which includes α-cellulose (58-63%), hemicelluloses (21-24%), lignin (11-12%), and some minor components (like fats, waxes, and pectin). The non-cellulosic components (especially hemicelluloses and lignin) negatively affect fibers' processing leading to limited application. However, cellulose and non-cellulosic components have excellent reactivity due to the presence of a high amount of functional groups (hydroxyl, carboxylic, phenolic, aldehyde), making jute fibers suitable candidates for a variety of chemical (such as alkali and oxidative), and physical modifications and adjustments in their structure and properties.

This paper summarizes the investigation focused on chemical and atmospheric pressure plasma treatments as fast and cost-effective methods for improving the jute sorption and electro-physical properties. Alkali modifications with sodium hydroxide lead to selective hemicellulose removal, increased the content of cellulose exposed on the fiber surface, decreased the crystallinity index, and contributed to elementary fiber liberation. Modifications using > 10% NaOH (so-called mercerization) change the structure of the native cellulose I to cellulose II and increase the number of possible reactive sites. On the other hand, the sodium chlorite modifications are used to selectively remove lignin, which is followed by simultaneous oxidation of fiber carbonyl into the corresponding carboxyl groups. Furthermore, periodate oxidation lead to the conversion of cellulose hydroxyl groups on C2 and C3 atoms to aldehydes, wherein the ring cleavage occurred and 2,3-dialdehyde cellulose was formed.

Nevertheless, the alterations of molecular-, fine, and microstructure, these chemical modifications also homogenize jute fiber structure, and thus, provide unique sorption and electro-physical properties. Some of the biggest benefits of the jute fibers' alkali and oxidative modifications are improved accessibility of the cell wall components to water vapor, and the total water holding capacity, which lead to the increase in the moisture sorption, water retention power, and degree of fiber swelling. The jute electro-physical

properties such as dielectric loss tangent, AC specific electrical conductivity, effective relative dielectric permeability, and volume electrical resistivity are very sensitive to fibers' chemical composition, crystallinity, and their ability for moisture sorption. More precisely, alkali modifications under mild conditions lead to a decrease in the volume electrical resistivity, while the resistivity of mercerized jute is mostly dictated by the presence of cellulose II polymorph as well as pronounced fiber liberation and fabric crimp. The overall improved AC specific electrical conductivity of the jute fabrics with lower hemicellulose content is the sum of three contributors: moisture sorption, crystallinity index, and hemicellulose content. In the case of jute fabrics with lower lignin content, the moisture sorption and crystallinity index significantly influences the AC specific electrical conductivity only at a lower relative humidity (30% RH), while, at a higher relative humidity (80% RH), the moisture sorption and bulk-free water have a higher influence. The obtained increase of the effective relative dielectric permeability after the alkali and oxidative modifications is attributed to the changes in the structural characteristics and decrease in the content of non-cellulosic components. Having in mind that the metals are highly conductive, one of the strategies that can be used to improve the fibers' electro-physical properties is their functionalization by incorporation of ions, nanoparticles, or oxides of various metals. The incorporation of silver ions leads to a decrease in jute fabrics' volume electrical resistivity by 3.0-38.5 times and provided maximum bacterial reduction for *E. coli* and *S. aureus*. Further amelioration of fibers' electro-physical properties could be achieved by the treatment with $CuSO_4$ and *in situ* synthesis of Cu-based nanoparticles on their surfaces by reduction. Exploitation in specific conditions that contribute to copper reduction will make jute fabrics able to store 21-163 times more energy from an external electric field than before the exploitation, which will extend their lifetime. The measurements of jute electro-physical properties as a function of different internal and external factors enable the prediction of its behavior in real application conditions, making it possible to design fabrics with desired properties. The chemically modified jute fabrics are particularly stable to achieve good energy accumulation in the presence of an electric field and they can be successfully used in flexible electronics, as well as, for electrical applications such as electrostatic discharge and fabric-based electromagnetic shielding devices, etc.

Except for the above-mentioned high-performance technologies, jute fabrics with improved dielectric properties could be also used for some ordinary products, such as protective clothing or textile of a specific behavior in environments sensitive to electrical discharges and home textiles (carpet). The increased production of these ordinary, as well as products with high performances, brings a considerable amount of waste in the form of fabric. Recycling in the way of producing filters for wastewaters minimizes the disposal costs of such fabrics thus contributing to "closing the loop" of their lifecycle, which is in agreement with the circular economy concept. To move towards a circular economy and to ensure the recycling and re-use of recycled fabrics, the jute fabrics with improved sorption properties were evaluated as adsorbents for various heavy metal ions, and anthraquinone dye C. I. Acid Blue 111 and Congo Red (C. I. 22120). It is worth mentioning that jute fabrics obtained after the adsorption of Zn^{2+} and Cu^{2+}-ions provided maximum bacterial reduction for *E. coli* and *S. aureus* and can be further utilized as filters for water disinfection. After use, these adsorbents can be burned and the metals recovered (*e.g.*, for catalysis), while the lignocellulosic material as abundant and low-cost waste can be used to prepare activated carbons.

Our latest investigations are focused on the preparation of jute geo-prebiotic support for cyanobacteria growth as a novel solution for damaged land rehabilitation. More precisely, raw jute fabric was subjected to atmospheric pressure dielectric barrier discharge (DBD) under different conditions (power and frequency of discharge, air as working gas, constant time of 120 s) to tailor its wettability properties which were monitored by wetting time and capillary height measurements. Special emphasis was put on the effect of aging on the mentioned properties. The biocrust inoculum survival and efficiency of biocrust restoration could be improved by increasing the availability of water during the initial phase of damaged soil rehabilitation. By tailoring geo-prebiotic polysaccharide supports' sorption properties, the viability of the cyanobacterial inoculum will be improved, the development of the biocrust accelerated, and finally, the efficiency of the biocrust carpet significantly increased.

Keywords: jute, chemical modification, atmospheric pressure plasma treatments, sorption properties, electro-physical properties, adsorbent, wastewater treatment, geo-prebiotic support, damage land rehabilitation

Acknowledgment: This work was supported by the Ministry of Education, Science and Technological Development of the Republic of Serbia (Contract No. 451-03-68/2022-14/200287 and 451-03- 68/2022-14/200135) and the Science Fund of the Republic of Serbia, #7726976, Integrated Strategy for Rehabilitation of Disturbed Land Surfaces and Control of Air Pollution - RECAP.

PL-3

THE ROLE OF DIHYDROCHALCONES AND THEIR DERIVATIVES IN THE TREATMENT OF DIFFERENTIATED THYROID CARCINOMA

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The role of dihydrochalcones and their derivatives in the treatment of hepatocellular carcinoma as well as differentiated thyroid carcinoma. In previous studies (Mayr et al., 2020; Kafka et al., 2020) diverse natural chalcones were identified as potential AKR1C3 inhibitors. Since increased expression of AKR1C3 is associated with castration-resistant prostate cancer as well as with resistance to enzalutamide and abiraterone acetate, different natural chalcones were investigated on prostate cancer cells. The three compounds MF-11, MF-14 (isolated from the plant Melodorum fruticosum) and MF-15 (synthetically derived from MF-14) showed effective repression of AKR1C3 and growth inhibition of prostate cancer cells. The most potent compound was thereby MF-15[1]. The aim of the project was thus to analyze the effects of MF-15 and its derivatives on the proliferation of differentiated thyroid carcinoma cells. Furthermore, the effect of MF-15 combined with already approved multi kinase inhibitors should be tested. In further experiments the mechanism of action should be lighted up. Additionally, a mechanism of action will be proposed based on in "silico" activity profiling.

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Keywords: thyroid carcinoma, MF-15, chalcones, treatment.

PL-4

ANALYSIS OF METALS AND METALLOIDS IN ENVIRONMENTAL SAMPLES

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Quantification of so-called major and trace elements, *i.e.* metals and metalloids, in samples from environment usually requires a use of instrumental analytical techniques that are sensitive enough to determine the analytes in such samples or their digestates. Generally, under the phrase 'samples from environment' water, soil and plant samples are considered. Also, it is well known that analysis of metals and metalloids is of a great interest of researchers of various science branches worldwide, because the roles that such chemical elements (can) have in the context of a food chain and, consequently, human's health [1].

A procedure of analysis of the analytes in question generally consists of the steps as follows: (1) sample collection, (2) storage, (3) sample preparation for measurement, (4) quantification of the selected analytes ('measurement'), (5) calculation and expression of the results, and (6) interpretation of the results (Fig. 1). It is essential to mention that only accurate and appropriate approach to each of these steps leads to accurate and precise results that reflect the analysed properties of a material. Thus, a procedure of collecting of samples should be designed in such a manner to preserve that an aliquot reflects the properties of the population. Then, the samples should be appropriately handled and stored in order to avoid contamination or possible losses of the analytes [2]. Preparation of water samples often implies filtration, sometimes also dilution. On the other hand, solid samples (soils, sediments, biological material) usually should undergo decomposition with aim of the transferring of the analytes into state of water solution; that is because the analytical techniques that are dominantly in use nowadays measure metals and metalloids in solution. So a selection of a solubilization method (reagent and conditions) is an important step that is related to the samples characteristics but also to the research goals [1,2-4]. The last part of a laboratory work in such a research is a quantification of the analytes, and in this context various atomic spectrometric techniques are widespread in use (*e.g.* ICP-AES(OES), ICP-MS) [1,5].

In the presentation a brief overview of the dominant practice as well as a discussion of challenging aspects and requirements of a procedure will be given.

Figure 1. Scheme of a metals and metalloids analysis procedure

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Keywords: environmental samples, heavy metals, sample preparation, spectrometric techniques, trace elements

PL-5

MARATHON EXPERIMENTS

ON THE SPONTANEOUS TRANSFER OF GASES THROUGH WATER: A MARATHON EXPERIMENT WITH UNEXPECTED RESULT

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Marathon experiments or marathons are long-lasting experiments (also known as corridor demonstrations). They typically last from a few hours to several months (even years; the term supermarathons seems to be appropriate for the latter). Several marathon experiments devised in our laboratory will be presented during the lecture.

Spontaneous transfer of gases trough water deals, in our case, with common gases that are the main constituents of the air. Two identical cylinders are filled with equal volumes of nitrogen (cylinder 1) and oxygen (cylinder 2) and are placed bottom up each in its own beaker filled with distilled water. The cylinders are left for many months (distilled water is periodically added to the beakers to compensate for the loss due to evaporation).

After a few days, it is clear that the volume of gas in cylinder 2 decreases. After a couple of weeks it is also clear that the volume of gas in cylinder 1 **increases**! These findings are in a way counterintuitive: one could understand the volume decrease, but the increase is *a priori* unexpected.

An explanation is offered in terms of diffusion of gases through water. Due to different solubilities of oxygen and nitrogen in water, oxygen diffuses (transfers) faster, hence the volume decrease in cylinder 2. Due to the same reason (faster influx of oxygen from the air, then the rate at which nitrogen escapes) the volume increase is witnessed in cylinder 1. Both the volume increase (1) and the volume decrease (2) correspond to asymptotic processes. After more than a year, the oxygen content in both cylinders is almost the same, due to equilibrium being approached and (almost) reached.

Students Presentations

SP 1

APPLICATIONS OF ELECTROCHEMISTRY IN CYANOBACTERIAL TAXONOMY: PRELIMINARY FINDINGS

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Extensive research data from the previous decade have unequivocally demonstrated that live cyanobacteria produce photocurrent by using the photosynthetic systems [1], suggesting a prospective utility of electrochemistry in the complex polyphasic approach for cyanobacterial taxonomy and classification. Since cyanobacteria can deposit electrons onto the anode surface when exposed to light [2], both via direct or indirect electrochemical transfer [3], it is hypothesized that electrochemical light/dark signatures at the cyanobacteria/electrode interface might be unique for each cyanobacterial species [2, 4], thus potentially enabling species recognition via photo-responsive current production. In the present study we aimed to evaluate the utility of different electrodes and methodological approaches in electrochemical measurements, for gaining light/dark electrochemical signatures in different homocytic filamentous cyanobacterial strains. The strains in this study were taken from the North Macedonian Culture Collection of Cyanobacteria (NMCCC). All experiments were performed in PBS buffer, pH = 7.4, by using different working electrodes and different modifications (addition of thin layers of polydopamine or, agarose in different concentrations in Z medium). In the experiments with direct transfer (without any modification of the working electrode), the paraffin-impregnated graphite electrode (PIGE) was shown superior, when compared to the basal plane graphite, and glassy carbon electrodes, although the differences in the light/dark electrogenic activities were low. The modification of the PIGE electrode with polydopamine did not give any satisfactory results, while the utility of agarose-modified PIGE showed great improvement in the obtained photocurrents. In conclusion, the utility of modified PIGE working electrode with 1% agarose in Z medium can give satisfactory light/dark differences in the detected photoelectric current, thus resulting in unique electrochemical signatures for different homocytic filamentous cyanobacteria.

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Keywords: electrochemistry, cyanobacteria, paraffin-impregnated graphite electrode.

SP 2

POLYMER MODIFIED ELECTROCHEMICAL BIOSENSORS BASED ONSCREEN-PRINTED ELECTRODES FOR PHARMACEUTICAL CONTAMINATED WASTEWATER DETECTION

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Release of pharmaceutical waste into the environment is becoming a great concern since many pharmaceuticals are resistant to conventional waste treatments. Diclofenac, being an analgesic non-steroidal anti-inflammatory drug with broad usage, often contaminates wastewater through urine excretion, causing aquatic pollution and human health problems. In need of a rapidand sensitive in-situ analysis of biochemical entities such as non-biodegradable pharmaceuticals,a promising application of modified screen-printed electrodes (SPEs) was obtained. Polymer- modified SPEs (graphene and carbon nanotubes) were explored as biochemical sensors, used for ultrasensitive detection of Diclofenac in various pH media. Polyethylene glycol and polyvinylidene fluoride were used for electrode surface modification by drop cast deposition. Electrochemical activity was monitored using cyclic voltammetry (CV). Surface morphology of the obtained sensors was characterized using scanning electron microscopy (SEM). Ultraviolet-visible spectroscopy (UV-Vis) measurements were used for detection of potential electrochemical effect induced by the current in CV in pharmaceutical solutions. The electrochemical characterization shows strong electrochemical response in acidic environment indicating better electrode active area and oxidation-reduction reactions at low pH.

Keywords: wastewater, pollution, biosensors, screen-printed electrodes, carbon nanostructures,polymers, drop casting, cyclic voltammetry

SP 3

INCORPORATION OF THERMOCHROMIC NANOPARTICLES AND PRODUCTION OF SMARTWINDOWS COATINGS VIA MINIEMULSION POLYMERIZATION

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One very frequent problem nowadays is energy consumption, and this issue is expected to spread even wider with the rise of population. It is a very challenging dilemma amongst experts and scientists. Many teams are working hard to find possible solutions and have concluded that the most cost-efficient ones are passive and energy-free technologies, such as smart windows with thermochromic properties.

One key material for the production of smart windows with thermochromic properties is vanadium (IV) oxide (VO_2), because of its ability to undergo a phase transition and with this to modulate from a fully transparent to reflective material on a given temperature [1]. Its unique properties make this material a perfect candidate in the field of green buildings. Nevertheless, one major obstacle that can be observed is the high critical temperature on which the phase transition occurs (≈68 °C) [2]. This low critical temperature limits the use of the film for everyday use, so use of a doping agent is needed in order to adjust this temperature. Transition metals are usually used as dopants for VO_2, such as tungsten [1]. To produce the thermochromic coatings, it is necessary to incorporate the VO_2 nanoparticles in acrylic latexes. This paper follows the synthesis of a W-doped VO_2, its surface modification and subsequent nanocomposite film formation, by using the miniemulsion polymerization method [3]. By choosing this method, we are choosing an eco-friendly technique for the fabrication of the final product – the thermochromic glazing. The obtained film has several potential applications, such as buildings or cars windows coatings.

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Keywords: vanadium (IV) oxide, W-doped, thermochromic, miniemulsion polymerization, phase transition.

SP 4

SYNTHESIS, STRUCTURE AND ELECTRICAL PROPERTIES OF $Gd_{1-x}Er_xFeO_3$ (x = 0, 0.2 and 0.4) PEROVSKITES

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Perovskites are a scientific hotspot, extensively studied because of their versatility and flexible structure. The never-ending possibilities of partial substitutions at cationic positions result with complex perovskites having intriguing properties. Incorporating two rare-earth cations in A-position into the structure of perovskites opens up great possibilities for unique properties, which in the future may lead to brand new applications.

In this research, the results of synthesis and investigation of perovskites that contain Fe at B-position and gadolinium partially substituted with erbium are presented. The studied perovskites may be represented by the following general formula: $Gd_{1-x}Er_xFeO_3$ (x = 0, 0.2 and 0.4). These compounds were synthesized using the combustion sol-gel method with citric acid as a fuel, under controlled temperature and pH value of the initial mixtures. The combustion sol-gel method was shown to be an appropriate method for synthesis of these perovskites.

The crystal structures of the obtained compounds were refined using the Rietveld method. The crystal structure refinement showed that all synthesized perovskites crystallize in the orthorhombic system (within the space group *Pnma*). The calculations of some crystallographic parameters have shown that the main reason for deviation of the ideal perovskite structure is octahedral tilting. This deformation becomes more pronounced by inserting the smaller erbium in the place of the larger gadolinium. Namely, in all compounds, erbium doping makes the octahedrons more regular but at the same time they become more tilted. This causes greater stress and instability of the structure.

In order to determine how the applied method of synthesis affects the morphology and dimensions of the particles of the obtained powders, SEM images were recorded. These images showed that the morphology of the compounds within the series is similar, i.e. they are composed of particles with nano-dimensions and have porous, spongy appearance.

In order to study some of the electrical properties of the synthesized perovskites, electrical measurements were performed. The results of the analysis showed that the substitution leads to a gradual change in conductivity. Namely, the pure $GdFeO_3$ behaves as a semiconductor, $Gd_{0.8}Er_{0.2}FeO_3$ exhibits semiconductive character at lower temperatures and metallic behavior at higher temperatures. As the substitution increase, $Gd_{0.6}Er_{0.4}FeO_3$ exhibits metallic conductivity.

Keywords: complex perovskites, rare-earths, combustion sol-gel method, Rietveld method, Scanning electron microscopy, electrical measurements

SP 5

INVESTIGATION OF THE SENSING POTENTIAL OF G/MWCNTs HYBRIDS

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Graphene/carbon nanotube (G/CNTs) hybrids are increasingly used for different applications due to their extraordinary properties that are exceeding the properties of their building blocks, the individual graphene and CNTs [1].

In this research work, graphene/multiwalled carbon nanotubes (G/MWCNT) hybrids were prepared using air sonication method. By changing the weight ratios of the G and MWCNTs we were able to obtain several different hybrids. The results from Raman spectroscopy, scanning electron microscopy and transmission electron microscopy suggested that depending on the G/MWCNTs ratio the hybrids had different surface morphology and chemistry.

The sensing potential of the hybrids was investigated using quartz crystal microbalance (QCM). QCM-based sensors are known for their high mass sensitivity and stability, as well as operating simplicity [2,3]. These results showed that by increasing the amount of MWCNTs,the sensitivity of the hybrids also improved, demonstrating the potential of the hybrids to be used as a sensing material for fabrication of mass-based sensors.

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Keywords: G/MWCNTs hybrids, sensors, QCM based sensors

SP 6

STATISTICAL PROCESS CONTROL IN HOSIERY PRODUCTION

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The essential goal of every business is to produce the highest quality products/services in the shortest time with acceptable expenses. In order to be kept and improve its quality, management is needed. So, to help an organization to survive in the world modern market, the Statistical Process Control should be applied, as one of the TQM methods that improves quality and reduces variation. Statistical methods helps in monitoring of the operations and structural changes of business organizations, which is the basis for market analysis and business development. The education and training of the employees in the organizations for the correct use of the statistical methods is a challenge of the modern work that companies meet.

The paper investigates the knitting production of women's tights. The check-lists and Pareto chart are applied for investigating the produced pieces, type of occurred defects and its frequency. Also, Control charts are used for determination whether a knitting process is in a controlled statistical state. This control chart is a diagram which is used to study the changes in the processes over the time, helps to determine whether a particular process is stable, its variability and the influence of external and internal factors. The obtained results show that that the highest percentage of defects occurs from breaking thread 2 (the thread in the panties area). This indicates that efforts should be oriented towards the elimination of this largest percentage of defects. So, the Pareto chart can be the first stage in making improvements. The created control charts signal that the knitting process is not stable. Specifically, the number of defects is above the upper control limit (Kg), as consequence of increased number of defected pieces and reduced production. Step forward should be discovering the causes that led to the unstable production process.

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Keywords: Statistical process control, check-lists, Pareto diagram, Control charts

SP 7

RICE HUSK MODIFIED BY THE CIRCULAR ECONOMY APPROACH FOR PURIFYING COLORED EFFLUENT FROM THE TEXTILE INDUSTRY

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Direct dyes are one of the most widely used dyes for dyeing cellulosic fibers. However, these dyes pose a great danger to the environment because of their highly carcinogenic degradation products. As the effluent from the dyeing process can contain up to 30% dye, a proper effluent purifying treatment must be done in order to protect the ecosystem. Adsorption is one of the most effective and cheapest methods that can be employed for cleaning the colored effluent. The goal of this research is to develop a low-cost and efficient bio-sorbent for direct dye removal. For this purpose rice husk, an agricultural waste that the Republic of North Macedonia has in abundance, was tested as a potential adsorbent for Direct Congo Red (C.I. 20120). Raw rice husk with its lignin-silicon surface shield has low adsorption ability. To improve the ability of the rice husk to purify colored wastewater, effluent from the alkaline scouring of cotton yarn was used immediately after the scouring (without cooling and additionally added chemicals) in order to remove the non-cellulosic silicon-lignin shield from the rice husk's surface. For comparison, the rice husk was modified using an optimized alkaline scouring recipe consisting of 20 g/L NaOH, 2 mL/L Cotoblanc HTD-N, and 1 mL/L Kemonecer NI at 70°C for 30 min. The characterization of modified rice husk was monitored by scanning electron microscopy-energy dispersive X-ray (SEM-EDX), attenuated total reflectance-infrared spectroscopy (ATR-IR), and ζ-potential measurements. The sorption properties of the rice husk were tested by the equilibrium isotherms and sorption kinetics. This rice husk, with 93.8 mg/g adsorption capacity, behaves similarly as the rice husk treated with an optimized alkaline scouring recipe with an adsorption capacity of 88.9 mg/g of direct Congo red dye. This proved that modifying one waste (the rice husk) with another i.e effluent from the alkaline scouring of cellulosic plant fibers, results in obtaining an effective adsorbent in an environment-friendly and economic way. This achievement represents a closed-loop energy-efficient process of the pre-treatment of cotton (alkaline scouring), modification of rice husk using effluent from the alkaline scouring, dyeing cotton fabrics and cleaning its colored effluents with modified rice husk without adding chemicals and energy for heating.

Keywords: low-cost, biosorbent, rice husk, modification, adsorption, direct dyes

SP 8

DESIGN, SYNTHESIS AND QSAR STUDIES OF MONOCARBONYL CURCUMIN ANALOGS AS POTENTIAL ANTI-BREAST CANCER AGENTS

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Breast cancer is the most frequently diagnosed life-threatening cancer in women. Triple- negative breast cancer (TNBC) is a subtype of breast cancer that lacks the expression of the estrogen receptor (ER), progesterone receptor (PR), and human epidermal growth factor receptor2 (HER2). It is considered to be the most aggressive and difficult to treat type of breast cancer. As a result, there is a critical need for the development of a safe and effective drug therapy for its treatment.

Curcumin, a secondary metabolite isolated from *Curcuma longa*, has been shown to exhibit an impressively broad range of pharmacological effects Comprehensive reviews concluded that, among many other naturally occurring compounds, it is an exceptional molecule with anti-cancer activity. Despite its powerful interaction with a diverse set of cellular targets, curcumin has several drawbacks that limit its potential as a therapeutic agent, including poor bioavailability, instability, and rapid degradation under physiological conditions. One of the most common approaches to overcoming these limitations is the design and synthesis of new curcuminanalogs with increased bioavailability and pharmacological activity.

This study was conducted in support of the ongoing search for new drug molecules that are effective enough for the treatment of Triple-negative breast cancer while causing only minor side effects. Hence, five symmetric monocarbonyl analogs of curcumin (MACs) with cyclopentanone, cyclohexanone and 4-piperidone as central core, were synthesized by Claisen- Schmidt condensation reaction. Their structures were identified by measuring and comparing the melting points to their literature values, as well as using FTIR and UV/VIS spectroscopic techniques.

To assess the cytotoxic activities of the mono-carbonyl analogs against MDA-MB-231 Triple-negative Breast Cancer Cells and to identify the significant structural features responsible for these molecules' potency, combined 2D- and 3D-Quantitative structure-activity relationship (QSAR) models were also developed. The generated QSAR models demonstrated acceptable internal validation, as well as good external predictive capacity, indicating that they can be used to design similar groups of compounds. These results suggest that the synthesized candidate drugs have promising cytotoxic potential against MDA-MB-231 cancer cells and should be further investigated both *in vitro* and *in vivo*.

Keywords: Curcumin, monocarbonyl analogues, breast cancer, QSAR, MDA-MB 231, FTIR, UV/VIS

SP 9

SYMMETRICAL CYCLIC C5-CURCUMINOIDS WITH CYTOTOXIC ACTIVITY AGAINST HT-29 COLON CANCER CELLS: SYNTHESIS, CHARACTERIZATION AND QSAR SCREENING

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Curcumin or (*1E,6E*)-1,7-bis(4-hydroxy-3-methoxyphenyl)-1,6-heptadiene-3,5-dione, is a natural product originating from the *Curcuma longa* species, which has a potential pharmacological significance. This is due to its extraordinary properties from which the anti-cancer, the anti-inflammatory and the anti-oxidant ones stand out. However, the limiting factors such as low bioavailability, poor absorption, hydrophobicity and fast metabolism make the pharmacological applicative aspect difficult.

In order to overcome these obstacles, chemists have developed methods of structural modification of curcumin by designing synthetic analogues with improved properties, from which MACs (Monocarbonyl Analogues of Curcumin) are of current interest. In this work, five symmetrical cyclic C5-curcumin analogues with cyclopentanone, cyclohexanone and 4-piperidone cores were synthesized via Claisen-Schmidt condensation. Characterization and identification were performed using UV/Vis and FTIR spectroscopic analysis and melting point measurements and comparison with available literature.

In order to assess the bioactivity of the synthetic analogues, hence the cytotoxic effects against CRC (colorectal cancer), as the third most common cancer worldwide, screening was performed using QSAR modeling. Based on previously performed *in vitro* analyzes of a series of MACs on HT-29 cultured cell lines, data were collected on the anti-cancer activity expressed through pIC50 values measured in μmol/L. A multi-linear regression analysis yielded the best model relating the activity to the 2D-structure of the molecules. pIC50 as a dependent variable in the MLR (multiple linear regression) equation was related to topological molecular descriptors calculated from the molecular surface.
The resulting 2D-QSAR model built on the basis of available data was used to screen the synthesized 2,5-diarylidenecyclopentanones, 2,6-diarylidenecyclohexanones and 3,5-diarylidene-4-piperidones for potential therapeutic use and satisfactory predictive values were obtained. However, their clinical evaluation will be critical to assess therapeutic utility.

Keywords: cyclic C5-curcuminoids, Claisen-Schmidt condensation, 2D-QSAR, MLR, HT-29 CRC cells

SP 10

INSTRUMENTAL AND SENSORY CHARACTERISATION OF FUNCTIONAL BISCUITS ENRICHED WITH GRAPE POMACE

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Food industry nowadays generates a significant amount of by–products that are a good source of proteins, minerals, fibers, bioactive compounds and can be used in the functional food production. Consumers have become more aware that proper nutrition is essential to their physical wellbeing and in order to increase their quality of life, they choose functional food that has positive impact on human health besides providing the necessary nutritional requirements. Consequently, functional food market has become very popular and it is rapidly expanding. The aim of this research is to produce functional biscuits using red grape pomace as by–product of wine industry in order to increase the levels of edible fibers and antioxidants in the final product and to examine the products using instrumental and sensory methods.

Red grape pomace, dried and milled, was sieved and used as a substitute for the wheat flour. Functional biscuits were prepared by replacing the wheat flour with grape pomace at different levels. Grape pomace with three different granulations: 1, 0.5 and 0.25 mm was added in four different concentrations: 2.5, 5, 7.5 and 10% substitution in respect of the flour mass, resulting in 12 new formulations plus the control biscuit samples. In this research, instrumental and sensory methods were performed for both biscuits and biscuits' bolus. Color changes and moisture levels were observed in the different formulations of biscuits and biscuits' bolus, as well as rheological properties. Further characterization of biscuits' bolus was followed through determination of dry mass and texture. It was determined that the rheological properties of biscuits differ between the formulations. Formulations in which grape pomace was added needed less saliva during mastication and lower number of bites. Furthermore, the addition of grape pomace increased the brittleness and hardness of the biscuits and there were changes in the bolus as well.

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Keywords: functional products, biscuits, instrumental analysis, food oral processing, sensory analysis

SP 11

QUALITY AND STABILITY OF FOAM IN LAGER BEER

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The stability and durability of the beer foam is crucial for the first impression, the enjoyment, and the whole experience of drinking a glass of cold beer. The foam is a colloidal system, dispersion of gas into liquid, where the volume comes from the gas phase, while the liquid forms a barrier between the gas bubbles.[1] It has been proven that the skeleton of the beer foam is a chemical substance with a protein structure, that forms a flexible and cohesive film and prevents gas migration, coalescence and disproportion of the foam bubbles. The protein substances mainly originate from the barley and are result of enzymatic and chemical modifications that occur in the brewery, especially during the mashing process, when the proteolytic enzymes are activated.[2]

Since the mashing process is one of the key operations for the beer foam quality, two mashing procedures for production of three beer types: light (A), red (B) and black (C) beer are followed. The mashing process involves mixing malted grain and water and heating it with temperature breaks, so the enzymes can break down the starches from the grain into sugars.[3] The two mashing procedures differ in the protein temperature break.

To define the quality of the three beer types, the main parameters such as hight and durability of foam, extract, alcohol content, specific gravity, pH and CO_2 are measured throughout the process. From the analysis of the beer after the first mashing scheme it is evident that although the quality of the three beer types is implacable, there is a problem with the stability of their foam meaning it does not have a height of 3 to 4 cm and is not stable for 3minutes at a temperature of 10°C to 15°C. After the second mashing process, all the parameters are stable and the foam meets the requirements for beer foam quality[4].

All measured parameters were relatively close in the three types, and in this case they cannot be considered as an important influencing factor for the foam. The results show that although the most significant for beer foaming is the time break at a temperature of 45-55°C during which the proteolytic enzymes show maximum activity, if this break is long, it can affect the production of beer with a much higher content of degradation and low-molecular proteins that are necessary nutrients for the yeasts during the fermentation process, but do not contribute to the stability of the foam.[5] Furthermore, the amino acid and protein composition of the wort should be analyzed.

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Keywords: beer foam, mashing process

SP 12

IMPLEMENTATION OF PASSIVE SAMPLING AND GC/MS ANALYSIS FOR MONITORING OF POLYCYCLIC AROMATIC HYDROCARBONS IN AMBIENT AIR

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Polycyclic aromatic hydrocarbons (PAHs) are a large group of organic compounds widely distributed in the atmosphere formed as products of incomplete combustion or pyrolysis of organic matter. PAHs are emitted in ambient air predominantly by anthropogenic activities (incomplete combustion of fossil fuels, motor vehicle emissions, domestic heating, cooking, waste incineration, oil refining etc.) or from natural sources (forest fires, natural losses or seepage of petroleum or coal deposits). These compounds consist of two or more fused aromatic rings and can be present in ambient air in gaseous phase or bound to particulate matter depending on their molecular weight [1]. PAHs are nonpolar lipophilic compounds that can be rapidly and widely distributed in living organisms. They are generally unreactive but because of their high toxic, immunosuppressive, mutagenic, and carcinogenic potential, the U.S. Environmental Protection Agency (U.S.EPA) has listed 16 PAHs as high priority pollutants [2]. Thus, exposure assessment for PAHs is important and reliable sampling and analytical methods are necessary.

In this research, methods and tools have been optimized and applied for sampling and analysis of polycyclic aromatic compounds in gaseous and particle phases. Gaseous PAHs were collected at a municipal and industrial landfill using polyurethane foam disk passive air samplers (PUF-PASs) that were exposed to ambient air for one month from december 2021 to august 2022. Gaseous and PAHs associated with particulate matter collected on filters were desorbed by Soxhlet extraction with 10% diethyl ether in hexane. The final extracts were concentrated and analyzed by gas chromatography-mass spectrometry with electron ionization. Twelve PAHs were identified in the gaseous phase by passive air sampling (naphthalene, acenaphthene, acenaphthylene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, chrysene, benzo[k]fluoranthene, benzopyrene, benz[a]anthracene) and ten PAHs were found in the particulate matter (fluoranthene, pyrene, benz[a]anthracene, chrysene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzopyrene, indeno[1,2,3-c,d-pyrene], dibenz[a,h]anthracene, benzo[g,h,i]perylene) and their average monthly concentrations in ambient air were estimated. Low molecular weight PAHs with two or three aromatic rings were predominantly found in the gaseous phase while high molecular weight PAHs with five or more rings were found in the particulate phase. Naphthalene, phenanthrene, fluoranthene and pyrene were most abundant in the air around both landfills during the sampling period (estimated concentrations ranging from 0,2-14 $ng \cdot m^{-3}$ air), while chrysene and benzopyrene had high average concentrations in the air around the municipal landfill (15-21 $ng \cdot m^{-3}$ air).

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Keywords: Polycyclic aromatic hydrocarbons, air pollution, passive air sampling, gas chromatography-mass spectrometry

SP 13

DEVELOPMENT AND VALIDATION OF METHOD FOR DETERMINATION OF CHLOROBENZENES IN SOILS

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Chlorobenzenes (CBs) are an important class of organic compounds that are used for a wide variety of purposes, such as pesticides, industrial solvents, and intermediate in the synthesis of many organic compounds. Different toxic effects have been attributed to CBs since they are persistent, migratory and with tendency for bioaccumulation [1]. It has been established that excessive exposure to these chlorinated compounds causes brain, spinal cord and skin problems, irritation of the eyes and hematological disturbances including anemia. A wide range of methods is described in literature for the extraction and quantification of CBs in solid matrices mainly using GC [2], but no standard method is yet available.

This study presents a novel, accurate and sensitive method for determination of chlorobenzenes (CBs) in soil samples, including extraction, concentration, clean up and injection in GC-ECD for selective and sensitive determination, supported by GC/MS for identification. The extraction technique and solvent composition was found to have a considerable effect on the yield of CBs. A Box–Behnken response surface methodology was employed for optimization of the extraction efficiency. Three parameters: composition of extraction solvent (mixtures of petroleum ether–acetone), extraction time and soil/solvent ratio were optimized by this experimental design. This approach provided sufficient information to allow selection of extraction conditions that will result in maximum target recovery.

Linearity was established in the range 21.7-151.7 μg/L. Accuracy was tested and satisfactory recovery was obtained that ranged from 98.7-105.2% in the low concentration region, 86.1-93.3% in the medium and 64.3-79.0% in the high concentration region, as well as precision in the range 3.45-23.09% RSD. Sensitivity of electron capture detector and mass spectrometer in the total and selected ion monitoring were compared. LOD were in the range of 2.08-3.84 μg/L using ECD and 10.4-19.2 μg/L using MS in the SIM mode.

The analytical potential of this developed method was demonstrated by determining the target analytes in real soil samples. It was successfully applied for identification and quantification of chlorobenzenes in soil samples from the region of a former chemical factory. The concentration of the detected chlorobenzenes (1,4-DCB, 1,2-DCB, 1,3,5- TCB, 1,2,4 –TCB, 1,2,3-TCB, 1,2,3,5- TeCB, 1,2,3,4- TeCB, PeCB, HCB) ranged between 1.11 μg/kg and 1156 μg/kg.

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Keywords: chlorobenzenes, soil, extraction, GC-ECD.

SP 14

DEVELOPMENT AND VALIDATION OF CHROMATOGRAPHIC METHODS FOR ANALYSIS OF ORGANOCHLORINE PESTICIDES AND POLYCHLORINATED BIPHENYLS IN HEMP (*CANNABIS SATIVA* L.)

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A high percentage of the world population relies on traditional medicines, largely based on plants as alternative medicine for therapeutic purposes, mostly as a plant extracts or as active components of plants. As a medicine *Cannabis sativa L.* is widely used for the treatment of numerous pathological and physiological conditions, but also for the treatment of a high of autoimmune and cancerous diseases. Cannabis is a plant consisting of at least 554 different compounds, among them Δ9-tetrahydrocannabinol (THC) and cannabidiol (CBD) as the most commonly recognized for their therapeutic properties. For the successful cultivation of cannabis, pesticides are often used to control pests as well as to improve yield and quality, but pesticides have a harmful effect on the environment and human health, so, equal importance should be given to the safety of these herbs as it is given to the other foods.

The aim of the research is development and validation of chromatographic methods for the analysis of organochlorine pesticides and polychlorinated biphenyls in samples of *Cannabis sativa* L. For chromatographic separation, detection and quantification of pesticide residues gas chromatographs with MS and ECD detectors have been used and methods were applied to solvent mixture of 36 pesticides. Several solvents and their mixtures including acetone, ethyl acetate, hexane, acetonitrile, and methanol were tested for extraction efficiency. The process of purification was also optimized using SPE C18 and SPE SCX column and also QuEChERS method was applied which was found to be more efficient. The GC-ECD method was validated by evaluating linearity, accuracy, precision, matrix effect, method repeatability, limit of detection (LOD) and limit of quantification (LOQ) and measurement uncertainty.

Keywords: organochlorine pesticides, polychlorinated biphenyls, cannabis, QuEChERS, GC-ECD;

SP 15

SCREENING FOR PYRROLIZIDINE ALKALOIDS IN HONEY SAMPLES FROM NORTH MACEDONIA

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Honey is the most important product from apiculture and it is considered a high quality food. It is a healthy blend of sugars, minerals and antioxidants, which makes it valuable for nutritional and therapeutic use. On the other hand, honey is a food that is directly affected by plant diversity and origin of pollen. This means that honey can be contaminated by various natural toxins, such as pyrrolizidine alkaloids (PA). Many PAs producing plants are widely distributed, and therefore, these compounds can be found in honey and other beehive products. Safety concerns regarding the presence of PA in honey arise from the proved hepatotoxic effect on human health. Moreover, because of the complexity of honey as a matrix, analyzing trace amounts of PAs represents a major analytical challenge. The aim of the study was to investigate a general situation of PA contamination in honey samples easily accessible to citizens of North Macedonia.

A study of commercially available honey samples, from large-scale mass and small scale producers, on the territory of North Macedonia was conducted. Several types were investigated, including: flower blossom honey, forest honey, acacia honey and orange blossom honey. The samples' colour was determined by comparison with Pfund grading system classification. A mixture of formic acid/methanol was used as an extraction solvent. Following extraction, a clean-up step was carried out, using the QuEChERS EN 15662 method. Each sample was analyzed by liquid chromatography for simultaneous detection of PAs and their *N*-oxides.

All of the analyzed samples were positive for PAs. Results show presence of several types of PAs, including monoesters, open chain-diesters and macrocyclic diesters. Among the detected PAs, lasiocarpine was the most common, followed by jacobine and lycopsamine. This data imply the possibility of intoxication with PAs originating from honey. Future research should be conducted to evaluate the risk of exposure to these natural toxins on daily basis.

Keywords: pyrrolizidine alkaloids, natural toxins, honey

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SP 16

PHENOLIC PROFILE OF MERLOT WINES DETRMINED BY UPLC-ESI-IT-MS

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In this study, the phenolic profile of Merlot wines (*Vitis Vinifera* L.) produced with classical fermentation, roto process and punchdown method (harvest 2021) has been determined using an UPLC technique coupled with DAD and ESI-IT-MS. Identification was performed by ESI-IT-MS method with alternating ionization polarity [1]. Analyses were performed on a CORTEX UPLC C18 column (1.6 μm x 2.1 x 150 mm) with gradient elution using a binary mobile phase consisting of 1% (*v/v*) acetic acid in water as solvent A and 1% (*v/v*) acetic acid in methanol as solvent B. For the elution programme, the following proportion of solvent B was used: 0-10 min 5-20% B, 10-45 min 20-50% B, 45-50 min 50-80% B, 50-60 min 80-90% B. The injection volume was 2μL. Identification of the compounds was based on retention time, UV-Vis and mass spectra compared with the available standards and data from the literature. In total, 50 phenolic components were identified, divided into the following groups: phenolic acids and derivates, stilbens, flavonols, dihydroflavonols, flavan-3-ols and antocyanins. UPLC-MS extracted ion chromatograms were calculated by summing up the intensities of the specified masses in the mass spectra. Ion intensities were extracted at the *m/z* values of the molecular (M^+) or the quasi-molecular ($[M+H]^+$, $[M-H]^-$) ions of the detected compounds. From the group of hydroxybenzoic acids, gallic acid was detected producing the deprotonated ion in negative ion mode at 169 and 153, respectively, forming fragments at *m/z* 125 and 109 as a result of loss of CO_2 from the carboxylate group. From the group of anthocyanins, the presence of glucoside, acetylglucoside and *p*-coumaroylglucoside derivatives of delphinidin, cyanidin, petunidin, peonidin and malvidin were confirmed in the Merlot wines. All of them had similar fragmentation pattern containing two signals, the original M^+ molecular ion, and the fragments $[M-162]^+$, $[M-204]^+$ and $[M-308]^+$ which are result of elimination of glucose, acetylglucose and *p*-coumaroylglucose residues, respectively. Considering the influence of winemaking method, it was observed that wine produced with roto process presented highest content of phenolic compounds. Compared to the classical fermentation, the content of phenolic compounds was about 30% higher in the wine obtained by the roto method.

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Keywords: Wine, polyphenols, UPLC- ESI-IT-MS, vinification, Merlot.

SP 17

IDENTIFICATION AND QUANTIFICATION OF BIOACTIVE COMPOUNDS IN ESSENTIAL OILS AND PLANT MATERIAL FROM *ORIGANUM VULGARE* AND *ORIGANUM MINUTIFLORUM* USING GAS CHROMATOGRAPHY

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Origanum vulgare and *Origanum minutiflorum* are important herbaceous aromatic plants belong to *Lamiaceae* family and they are wide spread in theMediterranean region. These aromatic herbs are widely used in agriculture, pharmaceutical and cosmetic industry and also as a traditional medicine through the years. It is known that *Origanum* species are rich natural source of bioactive components including phenolic glucosides, flavonoids, tannins, sterols, triterpenes, resins and essential oil and possess antimicrobial, cytotoxic and antifungal activities.

In the present study, the variation of the essential oil compositions and volatile compounds of dried herbamong *Origanum vulgare* (culinary herb) and *Origanum minutiflorum* (wild oregano) was investigated using gas chromatography mass spectrometry (GC-MS). GC-MS analysis of essential oil and dried herb resulted in the identification of 9 and 14 compounds in *Origanum vulgare* and *Origanum minutiflorum*, respectively, representing more than 98% of the total composition.

The main compounds in the wild oregano (*O.minutiflorum*) essential oils were: carvacrol (63.92 %), *p*-cymene (13.56 %), *γ*-terpinene (4.32 %) and caryophyllene (3.69 %). The main compounds in culinary herb (*O. vulgare*) oil were: carvacrol (62.90 %), *p*-cymene (21.32 %), thymol (6.96 %), and *γ*-terpinene (2.38 %).

The results supported the assumption that the studied overground parts of the wild growing oregano are rich in essential oil containing the valuable aromatic compounds carvacrol and thymol, which are the reason for its properties.

Keywords: Essential oil, herb, GC-MS, terpenoids, carvacrol.

SP 18

IDENTIFICATION AND QUANTIFICATION OF POLYPHENOLIC COMPOUNDS IN PLANTS OF *BORAGINACEAE* FAMILY USING LIQUID CHROMATOGRAPHY

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The *Boraginaceae* family includes approximately 2700 species worldwide, mainly in Europe and Asia. Some of the species from Boraginaceae family have pharmacological and cosmetological importance as a source of many useful natural constituents including terpenoids, polyphenols, flavonoids, and naphthoquinones. The high diversity in secondary metabolites isolated from these plants possess anti-inflammatory, antimicrobial, antiviral, antitumor, cardiotonic, contraceptive and antiplatelet activity.

In this work systematic study of extraction efficiency polyphenolic compounds from plant material for subsequent LC-MS analysis was carried out. The most efficient extracting solvent "Methanol + H_2O (50:50)" was applied to all analysed plant species.

LC-DAD-ESI-MS/MS method was used for qualitative and quantitative analysis of *Echium vulgare*, *Echium italicum*, *Onosmaheterophylum* and *Cynoglossumcreticum*, collected from different locations in Macedonia.

These widespread *Boraginaceae* species contain various classes of polyphenols such as hydroxycinnamic acids and flavonoids. In total, 16 compounds were identified and most abundant compounds were 3- and 5- caffeoylquinic acid, kaempferol 3-*O*-rutinoside, salvianolic acid, dihydrokaempferol-*O*-hexoside, quercetin 3-*O*-ruthinoside (rutin), diosmetin 3-*O*-rutinoside, rosmarinic acid andluteolin 3-*O*-glucoside.

The total polyphenolic content was the highest in *Cynoglossumcreticum*, followed by *Onosmaheterophylum*, *Echium vulgare* and *Echium italicum.*

Keywords: *Boraginaceae*, *Echium vulgare*, *Onosmaheterophylum*, *Cynoglossumcreticum*, *Echium italicum*, polyphenols, HPLC, mass spectrometry

SP 19

OPTIMIZATION AND VALIDATION OF ETAAS METHOD FOR DETERMINATION OF SELENIUM IN SELECTED FOODS FROM NORTH MACEDONIA

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Not presented.

SP 20

SOLID STATE INTERACTIONS OF L-ASCORBIC ACID WITH MAGNESIUM STEARATE

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Since its discovery 80 years ago, vitamin C (ascorbic acid) has proven to be necessary for numerous metabolic processes, with some applications in treatment of different types of cancer [1, 2]. In the form of L-ascorbic acid, vitamin C is a potent water-soluble antioxidant, proficient in neutralizing oxidative stress through electron transfer processes [3]. L-ascorbic acid is rarely administered as a pure chemical substance alone, but rather combined with a variety of excipients, which can be a cause for various physical or chemical interactions [4]. One of the known incompatibility factors, in high dosage ascorbic acid tablets, is the commonly used lubricant magnesium stearate [5]. Incompatibilities between magnesium stearate and acidic APIs are well known in literature [6], and even though the ascorbic acid and magnesium stearate interaction is reported, it has not been systematically studied yet.
This work aims to examine the nature of the solid-state interaction between vitamin C and magnesium stearate by interpreting the results of analyzed initial and stressed binary mixtures between L-ascorbic acid and magnesium stearate. FT-IR analysis showed a shift in vibrational bands from around 1577 cm^{-1} to 1538 cm^{-1} under stress conditions at 40 °C and 75 % RH. DSC analysis of the abovementioned stressed binary mixture shows a new endothermic peak at around 57 °C, which could be a sign of an enantiotropic phase transition or a byproduct of the interaction between ascorbic acid and magnesium stearate, most likely (+)-magnesium L-ascorbate. Hygroscopicity test and temperature-controlled ATR analysis suggests that the interaction originates not only from a change in one parameter, but rather from a combination of both humidity and temperature.

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Keywords: vitamin C, ascorbic acid, magnesium stearate, compatibility study, solid-state interactions

SP 21

MECHANICAL CHARACTERISTICS OF THE STEEL SHEETS IN DEPENEFENCE OF THE CHEMICAL COMPOSITION OF STEEL

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Not presented.

SP 22

EFFECT OF $BaTiO_3$ AND MWCNT ON DIELECTRIC PROPERTIES OF FLEXIBLE AND LOW COST TPU COMPOSITE FILMS

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Dielectric materials have received great scientific attention in different electrical applications as a vital segment in high-frequency electronic circuits and capacitors [1]. In order to achieve high energy storage density, several key parameters such as high dielectric permittivity, high electrical breakdown strength and low dielectric losses are crucial [2]. Ferroelectric ceramics possess excellent dielectric properties and ability to store a large amount of electrical energy.

$BaTiO_3$ as a typical ferroelectric and enviormentally friendly material, possessing high dielectric constant and breakdown strength, is considered as a common filler in composites designed for supercapacitors [3]. However, in order to increase its flexibility and the overall mechanical properties, $BaTiO_3$ is usually mixed with inexpensive and flexible polymer matrices.

The aim of this study was to prepare lead-free composites based on thermoplastic polyurethane (TPU) polymer matrix, $BaTiO_3$ and MWCNTs as a conductive filler, via solvent casting method. $BaTiO_3$ and MWCNTs particles were surface modified in order to introduce proper functional groups. The results showed specific interactions between TPU and the fillers, confirmed by FTIR analysis. Consequently, the dielectric constants of the samples increased and dielectric losses decreased with addition of $BaTiO_3$ and MWCNTs fillers, while improved dielectric breakdown strengths were achieved. Overall, the TPU composite films showed improved dielectric and mechanical properties suitable for their energy storage application.

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Keywords: Dielectric materials, $BaTiO_3$, TPU, capacitors.

SP 23

ELECTROSPUN PVDF/$BaTiO_3$ NANOCOMPOSITES

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Electrospinning is one of the most popular techniques for fabricating polymer nanofibers. Accordingly, piezoelectric polymer nanofibers, which are widely employed in microelectronic applications, are frequently produced by electrospinning [1]. The produced nanofibers are characterized by a higher piezoelectric response, excellent flexibility, and high surface-to-volume ratio. The most common piezoelectric polymer is poly (vinylidene fluoride) (PVDF), which can be easily electrospun into nanofiber mats. In general, PVDF crystallizes in five different phases (α, β, γ, δ and ε), but only the beta phase exhibits the best piezo-, ferro- and pyroelectric properties [2]. Therefore, it is necessary to achieve a high beta phase content in PVDF. This can be achieved by employing specific processing methods, such as spin-coating and electrospinning, or post-treatments such as annealing or stretching. The incorporation of different types of fillers can also lead to crystallization into the desired beta phase [3].

In this study, ceramic nanoparticles of $BaTiO_3$ were added to PVDF solution in amounts of 5,10,15 and 20 wt.%, and the electrospinning method was employed to prepare nanocomposite fibers. The obtained nanofibers were further morphologically characterized and their physical, thermal and mechanical properties were investigated. The density and porosity of the nanofibers were calculated by the liquid displacement method.

The obtained results confirmed that electrospinning is an effective method for inducing a high beta phase content in PVDF, while the addition of ceramic nanoparticles enhanced the tensile strength at break and further stabilized the high beta content. The addition of nanoparticles also had an influence on the processing parameters and the morphology of the nanofibers.

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Keywords: electrospinning, fibers, piezoelectric composites

SP 24

MICROSTRUCTURAL AND MECHANICAL CHARACTERIZATION OF ASTM A242 STEEL SHEETS

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Not presented.

SP 25

THE APPLICATION OF ESCAPE ROOM ACTIVITIES IN 9th GRADE CHEMISTRY TEACHING

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Looking at the changes happening in the modern society, changes in the field of education are also inevitable. In contemporary teaching it is necessary to introduce new student-centered activities that will try to keep their attention and increase their motivation to learn.In the last few years, the Escape Room [1] activity has been applied in different teaching contents. The purposes of this study were to apply an innovative approach in chemistry teaching aimed at increasing the engagement and motivation of 9th grade students in reviewing the Exothermic and endothermic reactions topic, improving their communication skills, encouraging creative, critical and logical thinking, and improving the ability to solve problems. The primary idea of the research was to encourage students to participate actively in the classes and to increase their interest and motivation to learn chemistry. The application of this activity, besides helping the students to master the teaching content in a more interesting way, can also be used to encourage discussion and collaboration within the group as well as competition between the groups.

The research included 244 9th grade students who attend classes in different languages of instruction (195 in Macedonian and 49 in Turkish), from five primary schools in Krivogashtani, Prilep, Skopje, Kicevo and Radovish. In order to examine how these activities affect the students' engagement and motivation, two different questionnaires were used: Students' Motivation towards Science Learning (SMTSL) [2] and Activity Perception Questionnaire (APQ) [3]. The APQ questionnaire was used to assess students' subjective opinions and experiences related to certain activities conducted in the classroom. The modified SMTSL questionnaire examined the level of students' motivation to study chemistry, while the IMI questionnaire was focused on students' opinions and experiences regarding the activities conducted in the class. The obtained results from both questionnaires were analyzed using the SPSS Statistic 26 software package. The analysis of the collected data showed that the application of the Escape Room activities in a great extent affects the improvement of teaching. Students were more engaged during the lessons and actively participated in all activities. In this way they were more motivated and more satisfied with the achieved results.

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Keywords: Ativity Perception Questionnaire (APQ), chemistry teaching, Escape Room, Exothermic and endothermic reactions, Students' Motivation towards Science Learning (SMTSL).

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