UNIVERSITY OF NIŠ
Faculty of Technology, Leskovac

# BOOK OF ABSTRACTS

## 16th INTERNATIONAL SYMPOSIUM „NOVEL TECHNOLOGIES AND SUSTAINABLE DEVELOPMENT“

Leskovac, October, 17-18, 2025.

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# PLENARY LECTURES

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# THE INFLUENCE OF AGEING ON THE RELEASE OF FIBRE FRAGMENTS IN THE STANDARD AND MODIFIED WASHING PROCESS

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The exponential growth of global plastic production has led to widespread pollution of ecosystems worldwide. Microplastics (MPs) have become one of the biggest environmental threats, and their concentration is expected to increase rapidly in the near future. These anthropogenic pollutants result from the fragmentation and degradation of plastics into micro- and nanoparticles through chemical, mechanical, or photochemical processes, creating persistent pollutants that interact extensively with ecosystems. The increasing production and consumption of synthetic textiles have led to significant environmental problems related to microplastic pollution, with polyester materials identified as the main cause of water pollution.

The release of fibre fragments occurs in various ways: during the manufacture of textiles, during industrial washing, household washing, and drying. An estimated 35% of total emissions come from industrial sources, while the remaining releases occur during the consumer use phase, particularly during the first few washes when loose fibres adhering to the surface are removed. Prolonged washing cycles intensify the mechanical degradation processes, leading to increased release, while higher detergent concentrations cause surface changes that accelerate the release of fragments from textile substrates. The importance of this research area is underlined by the recent development of standardised test methods. The first test method (AATCC TM212-2021), developed specifically for measuring the release of fibre fragments in household laundry, published in 2021, describes and defines the terms fibre, fibre fragment, and microfibre. Standardisation in this area was formalised in 2023 with the publication of the first official international standards: EN ISO 4484-1, EN ISO 4484-2, and EN ISO 4484-3, accepted in the Republic of Croatia as HRN standards. It is known that the properties and characteristics of textile materials also change significantly under various environmental conditions. In addition to multiple washing cycles, thermal stress, UV radiation, and mechanical wear also influence these properties. As natural ageing processes take a longer period of time, researchers are increasingly using accelerated artificial ageing techniques to simulate real-life conditions and obtain reliable assessments within practical time frames.

The most important innovation in this research is understanding the relationship between the ageing of textiles and the release of fibres. This is a significant research point in the development of effective strategies to reduce pollution from textiles. While previous studies have investigated the immediate fibre emissions from new textiles, the long-term behaviour of aged materials is still insufficiently studied. This research gap is particularly significant as most textile products are used over an extended period of time during which their fibre-releasing properties can change significantly.

The experimental methodology within this paper includes artificial ageing aimed at accelerating the degradation processes of polyester, including the mechanisms of

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photo-oxidation and hydrolysis. The ageing protocols target the ester bonds (-CO-O-) that are characteristic of poly(ethylene terephthalate) polymer structures to simulate long-term environmental effects. The research focuses on the relationship between artificial ageing treatments and the release of fibre fragments during washing processes.
Within this research, a systematic experimental approach was used to investigate in which way ageing affects the release of fibre fragments from polyester and polyester/cotton fabrics during washing processes under controlled laboratory conditions. The aim is to explore the cause-effect relationship, focusing on how these factors influence the fragmentation of polyester fabrics. A key objective was to develop a modified washing protocol to minimise fragmentation caused by thermal stress during the washing process. The cooling protocol involves a systematic reduction in temperature during the washing process, as a hypothesis that controlled thermal transitions can reduce the mechanical stress on materials exhibiting ageing-related structural changes. This approach targets the potential sensitivity of aged polyester fabrics to rapid temperature fluctuations during conventional washing cycles. The synergy of aged polyester and polyester/cotton fabrics and wastewater after washing with standard and modified protocols presents a scientific challenge and serves as an innovative addition to research in this area.
The scientific contribution of this research can be seen in several aspects: the impact of the individual process parameters of artificial ageing on the properties of polyester and polyester/cotton fabrics; the quantification of the degree of contamination of the wastewater from the washing process; the understanding of environmental issues in the field of textile technology and the verification of the sustainability of the modified process in comparison to the standard washing process. Therefore, in this research, artificial ageing was varied by exposure duration, exposure conditions (direct irradiation versus exposure behind window glass), and combination patterns (continuous irradiation or alternating rain/sun cycles) according to the standards.
The results show that artificially aged polyester fabrics have an increased release rate of fibre fragments compared to non-aged fabrics, with the first wash cycles producing higher particle concentrations than subsequent cycles. Artificially aged polyester fabrics have a higher release rate of fibre fragments than non-aged fabrics. The release of fragments correlates with the extent and duration of previous ageing, and modified washing protocols can effectively minimise fragmentation caused by thermal stress. This research provides important insights into the long-term behaviour of textile materials and their impact on the environment throughout their life cycle. The results contribute to the understanding of the dynamics of microplastic pollution and offer effective mitigation action through optimised washing protocols. The results show the importance of considering the ageing of textiles when assessing environmental impacts and support the development of more sustainable strategies for textile management.

Keywords: polyester fabric, fibre fragment release, artificial ageing, washing protocol, release mitigation

# IMPACT OF Ca-BASED ADDITIVES AND THE TYPE OF PYROLYZER ON THE PYROLYSIS OF PAPER-LAMINATED PHENOLIC RESIN PREINTED CIRCUIT BOARDS (FR2-PCBs)

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One of the most widely used and important components of electrical and electronic equipment is the printed circuit boards (PCBs), accounting for approximately 3–6 wt% of the total waste electrical and electronic equipment (WEEE). PCB consists of an insulating non-metallic polymer substrate (resin), metal foils, laminated layers, and electronic components. Generally, waste printed circuit boards (WPCBs) consist of 30–35 wt% metals, 35–42 wt% refractories, and 24–30 wt% resins. PCBs can be classified based on the substrate type into glass fiber-reinforced epoxy resin (FR4) and paper-laminated phenolic resin (FR2). While FR4 is typically used for small electronic equipment such as mobile phones, FR2 is commonly utilized in household appliances, such as televisions and personal computers.

Halogenated flame retardants (HFRs) are usually added to PCBs to enhance their resistance against heat and prevent ignition. The improper treatment of HFRs in WPCBs can cause serious environmental problems. For example, PCB incineration can generate toxic and carcinogenic components. Moreover, WPCB landfilling causes heavy metals and other additives, used in the structure of PCBs, to leach into groundwater.

Due to the coexistence of metals and non-metallic fractions (NMFs, such as plastic resin, flame retardants, ceramics, etc.) in the structure of WPCBs, their recycling process is complicated and burdensome. In recent years, besides recovering the precious metals of WPCB, thermochemical recycling processes, such as pyrolysis, have been widely deployed to convert the NMF of WPCBs into value-added fuels and chemicals. Pyrolysis is a thermochemical process conducted at moderate temperatures and in the absence of oxygen by which the volatile organic matter of the feedstock is decomposed into solid, liquid, and gas species. The liquid species, known as pyrolysis oil, contain the main and most valuable products of the pyrolysis process. One of the main issues concerning the pyrolysis of PCBs is the production of oil containing high amounts of halogenated compounds, such as HBr, HCl, and organobromine species, which are toxic and corrosive and would contaminate the environment if released into the atmosphere.

In the present study, the impact of the calcium-based additives as well as the pyrolyzer type on the recovery and the halide content of the oil produced from the pyrolysis of FR2- PCB was explored. To this end, the optimal operating conditions (the pyrolysis temperature, the retention time of the feedstock in the reactor, and the PCB-to-additive ratio) maximizing the liquid recovery of the FR2-PCB co-pyrolysis with a 50:50 mixture of CaO and $Ca(OH)_2$ in a fluidized bed pyrolyzer were first investigated through the experiments designed by the response surface methodology. The analysis of the experimental results showed that the highest level of liquid recovery (40.6%) was achieved when $T$ = 620 ◦C, FR2/A ratio = 5.4, and $t$ = 22 min.

In the next step, stydying the impact of the Ca-based additives (CaO, $Ca(OH)_2$, and their mixture) and the pyrolyzer type (fixed and fluidized bed reactors) on the properties of the liquid products under the above optimal operating conditions demonstrated that the highest level of liquid recovery in the fixed and fluidized bed (38.5% and 41.2%) was achieved in the presence of $Ca(OH)_2$ alone. It was also found that CaO alone was more efficacious than $Ca(OH)_2$ in dechlorinating the produced pyrolysis oil. Relative to the oil obtained in the absence of any additive, CaO could reduce the halogen content of the pyrolysis oil up to 80% and 92% for the tests carried out in the fixed and fluidized bed reactors, respectively.
Under similar operating conditions and regardless of the use of the additive, the fluidized bed was always more effective than the fixed bed in enhancing liquid recovery. The fluidized bed pyrolyzer was also more successful than the fixed bed at removing the halide content of the liquid and gaseous products in the presence of all tested additives. However, an opposite trend was observed when no Ca-based additive was used in the pyrolyzers.
In general, it was concluded that when increasing the quantity of the produced pyrolysis oil is more important than improving its quality, using $Ca(OH)_2$ is recommended. Contrarily, when the quality and dehalogenation, in particular dechlorination, of the pyrolysis oil matters, the use of CaO is preferable. The co-pyrolysis of FR2-PCB and CaO + $Ca(OH)_2$ in the fluidized bed reactor was identified as an appropriate approach to enhance both the yield and the concentration of the phenolic compounds of pyrolysis oil while reducing its halide content significantly.

Keywords: Printed circuit board (PCB); Pyrolysis; Dehalogenation; Fluidized bed reactor; Calcium-based additives

This research was funded by the National Fund for Scientific Research, Republic of Bulgaria, grant number KP-06-N71/8 from 8 December 2023.

# LABORATORY MODEL OF MICROBIAL CELLULOSE DEGRADATION - A PROMISING TOOL IN THE CIRCULAR BIOECONOMY

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Various experimental approaches have been developed in search of a way to involve microorganisms in the biodegradation and recycling of cellulose waste, aiming to prevent their accumulation and environmental pollution. Therefore, we focused on the achievement of maximum cellulose biodegradation in laboratory conditions and the development of a useful laboratory model with potential for implementation inwaste management and environmental protection. Bacterial consortia and individual strains isolated from partly destroyed wood (PDW), goat feces (GF), and bioreactor (BR) were evaluated for cellulose degradation capacity by using toilet or filter paper as cellulose substrates. The cellulose biodegradation of mesophilic (37 ºC) bacterial population from methanogenic BR, GF, and PDW varied from 65% to 74% at days 9 and 21 under aerobic, microaerophilic, and anaerobic cultivation.

The metagenomic profiling of the bacterial consortium from BR showed the highest presence of species from the genera Clostridium, Bacteroides, Ruminiclostridium, and, more precisely, the species *Bacteroides oleiciplenus* and *Ruminiclostridium papyrosolvens,* which have high cellulolytic activity. Other genera like Lysinibacillus, Bacillus, Achromobacter, Dendrosporobacter, Ruminiclostridium, Acetanaerobacterium, Alkalitalea, and Treponema were detected in the PDW and GF consortia. Furthermore, six anaerobic strains with proven cellulolytic activity were isolated, and their species identification was done by 16S rDNA sequencing and MALDI-TOF.

By scanning electron microscopy, bacterial diversity, ultrastructural changes, adhesion to the cellulose matrix, and its decomposition are demonstrated. Bacterial consortia and individual fungal strains demonstrate well-preserved viability and cellulose degradation capacity after 12-month storage by using glycerol and skim milk as cryoprotectants.

A reproducible protocol for anaerobic biodegradation of cellulose in a bioreactor (4 l working volume) as a laboratory model was developed. The creation of effective technologies for cellulose biodegradation will contribute not only to the volume reduction of waste but also to its conversion into an additional resource for a circular economy.

Keywords: cellulolytic bacteria, cellulose biodegradation, laboratory model, electron microscopy.

Review paper
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# DESIGN AND PROCESSING OF BIOBASED AND BIODEGRADABLE POLYMERS FOR ADVANCED APPLICATIONS: INTEGRATING EXPERIMENTAL STRATEGIES AND THE POLYMERS-5B VISION

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The urgent need to reduce plastic waste, greenhouse gas emissions, and reliance on fossil-based resources has catalyzed the global transition toward sustainable materials. Conventional plastics, while essential in packaging, automotive, electronics, and textiles, are predominantly derived from fossil raw materials and involve energy-intensive and polluting production methods. Addressing these challenges, the development of biobased and biodegradable polymers—such as poly(lactic acid) (PLA), polyhydroxyalkanoates (PHAs), and poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV)—has gained significant momentum due to their environmental compatibility and versatility in application.

This study investigates the structuring and processing of these biodegradable polymers through techniques like electrospinning, solution casting, extrusion, and thermo-mechanical recycling. Emphasis is placed on enhancing mechanical flexibility, surface properties, and thermal stability, while minimizing energy consumption. The experimental results on PLA/PHB, PHBV/PCL-diol blends, and recycled PLA demonstrate improved film-forming ability, surface wettability, and recyclability over multiple cycles. Such findings are critical for the production of thin films, coatings, and medical scaffolds.

Embedded within this broader research context is the POLYMERS-5B project, a large-scale European initiative that aims to redefine the production of high-performance biobased polymers and copolymers using clean, circular, and sustainable technologies. By applying biocatalysis, green chemistry, and machine learning/AI-based process optimization, the project targets the development of novel bio-polyesters and polyamides that replicate the properties of conventional materials (e.g., PET, ABS, PU) while offering full biodegradability. The project encompasses a comprehensive development pipeline—from basic research (TRL 3) to pilot-scale application (TRL 5)—with applications in the textile, automotive, electronics, and furniture sectors.

In addition, POLYMERS-5B explores the recovery of bioactive compounds (antioxidants, polysaccharides, proteins) during polymer synthesis, supporting cross-sector integration with the food, pharmaceutical, and biotech industries. Life Cycle Assessment (LCA), “Safe and Sustainable by Design” principles, and digital “in-silico” engineering models ensure that environmental, economic, and social impacts are addressed from the earliest stages. By fostering innovation, supporting SME engagement, and enabling the creation of spin-off ventures, the project actively

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contributes to the EU Green Deal, Bioeconomy Strategy, and Paris Climate Agreement objectives.

Keywords: biopolymers, PLA, PHBV, PCL-diol, green chemistry, recycling, electrospinning, circular economy, sustainable materials, bioactive compounds, AI optimization, POLYMERS-5B



Review paper
The research was funded by the Key Research and Development Program of Jiangsu Province (BZ2022030) and Zhenjiang City (GJ2021011), China.

# THE DISTRIBUTION OF MYCOTOXINS IN TRADITIONAL SOLID-FERMENTED FOOD AND DETOXIFICATION STRATEGY

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Solid-state fermentation refers to the conversion of carbohydrates to alcohol and organic acids by the activity of microorganisms on a moist solid substrate without free-flowing water. In East Asian countries, solid-state fermentation has played a critical role in multiple aspects of food and seasoning preparation for thousands of years. In China alone, Chinese soy sauce, Chinese vinegar, and Chinese liquor (Baijiu) are predominantly produced from solid-state fermentation. In addition to significant economic values, solid-state fermented food contains health beneficial components. Recent evidence has also reported the incidence of mycotoxins in solid-state fermented products (e.g., Horse bean-chili-paste, Korean Soybean Paste, and Baijiu). Owing to contamination of raw materials/substrates and the open fermentation environment favorable for toxigenic fungi growth, solid-state fermented food is highly susceptible to mycotoxins. Given the substantial amount of solid-state fermented food consumption, it is essential to assess the occurrence, distribution, and potential health risks from the transfer of mycotoxins during fermentation processing.

Given the significant safety risks posed by mycotoxin contamination in solid-state fermented foods, integrated detoxification strategies are increasingly employed. This lecture examines four key approaches: biological agents, adsorbent amendments, fermentation process optimization, and enzymatic treatments. We further analyze their sustainability, selectivity, and compatibility while addressing inherent limitations. Finally, we present a representative case study on $AFB_1$ detoxification in solid-state fermented vinegar from our prior research.

Keywords: Solid-fermented foods, Mycotoxin, Aflatoxin $B_1$, Deoxynivalenol, Zearalenone, Detoxification

## Section: FOOD ENGINEERING

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# MONITORING THE CHANGE IN THE CONCENTRATION OF GASES IN THE HEADSPACE DURING THE SPOILAGE OF BEEF MEAT

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Meat spoilage is largely driven by microbial metabolism, which produces volatile compounds such as gases that can indirectly indicate microbial activity. Ammonia and carbon dioxide are the most common gases released during protein degradation. This study focused on monitoring the concentrations of these gases in the headspace of minced beef to assess their relationship with microbial growth during storage.
Experiments were conducted using 150 g of minced beef placed in airtight 300 ml jars, stored at 4 °C for 15 days and 7 °C for 10 days. Gas concentrations were measured using a specially designed multi-gas detector capable of detecting carbon dioxide (via non-dispersive infrared spectroscopy), ammonia, and hydrogen sulfide (via catalytic bead sensors), and oxygen (via electrochemical sensing). Microbial growth was monitored by counting total mesophilic bacteria (TMB) on nutrient agar.
During storage, ammonia concentration increased from 0 to 2.5 ppm at 4°C, and to 4.3 ppm at 7°C. The hydrogen sulphide concentration increased from 0 ppm to 39.3 ppm, and from 0 ppm to 159 ppm, at 4 °C and 7 °C, respectively. The initial number of TMB was approximately 4.3 log CFU/g and increased to the value of 7.4 ±0.10 log CFU/g after 15 days at 4°C and 7.6 ±0.16 log CFU/g after 10 days at 7°C.
The results of the experiments showed a correlation between the increase in gas content in minced beef and an increase in the number of TMB. Hence, modern gas content measuring spectroscopic instruments can indirectly determine microbial contamination in closed meat packaging.

Keywords: multi-gas detector device, beef meat, ammonia, hydrogen sulphide, microbial count

Original Scientific Paper
This work was supported by the Ministry of Education, Science and Technological Development of the Republic of Serbia (contract no. 451-03-137/2025-03/200116).

# PHYTOCHEMICAL COMPOSITION AND ANTIOXIDANT PROPERTIES OF ZEIN-BASED EDIBLE FILMS ENRICHED WITH CHICORY BEE-COLLECTED POLLEN

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Edible coatings and films have become highly popular in food preservation due to their ability to prolong the shelf-life of food products, prevent spoilage, and enrich prepared stuff with some bioactive compounds to obtain functional food. Zein, a hydrophobic amino acid-rich protein, is one of the main byproducts obtained during maize processing in the industry. As an excellent barrier against water penetration, it finds a significant application in food preservation with the intention to prevent microbial spoilage as well as to enhance the bioactivity of food. Therefore, the aim of the research was to prepare zein-based edible films (ZEF) enriched with chicory bee-collected pollen (CBCP) as a source of improved bioactivity. Three samples were obtained: control (C) and ZEF-CBCP with 2% and 4% of pollen addition. Phytochemical composition (total phenolic, flavonoid, and dihydroxycinnamic acid derivatives) as well as antioxidant properties (TAC, CUPRAC, FRP, and DPPH$^{\bullet}$) of the prepared films were examined through several standard spectrophotometric assays. For all applied assays, except in the case of total phenolic content (TPC), CBCP addition provoked an increase in observed values, compared to the control sample. However, increased content of CBCP also demonstrated increased TPC value. Among antioxidant assays, prepared films exhibited a great ability to reduce cupric ions measured through standard cupric ion reducing antioxidant capacity (CUPRAC) as well as significantly improved *in vitro* phosphomolybdenum total antioxidant capacity (TAC) with CBCP addition, compared to the control sample. Further examination of the physical and antimicrobial properties of the prepared films will be performed to fully describe the obtained product.

Keywords: pollen, edible zein film, phytochemicals, antioxidant activity

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# STM32F4 MICROCONTROLLER-BASED IOT SYSTEM FOR MONITORING PARAMETERS DURING GOAT MILK FERMENTATION

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This paper presents the design and implementation of a system for the measurement, monitoring, and acquisition of changes in pH and temperature parameters during the fermentation process of fresh goat milk into sour milk using an STM32F4 microcontroller, a pH and temperature sensor. The system can connect to the internet or a local network, which enables remote monitoring and control at a low cost, making it an advantage compared to the other systems. During the fermentation process, there is a change in the pH value (from 6.7 to 3.9) and temperature (from 50°C to 25°C), which results in a change in the voltage at the output of the used pH sensor. The prototype of the device was realized, and the appropriate calibration was performed. All 4 stages of fermentation were monitored, i.e., lag, exponential, stationary, and final stages of ripening, during which there is a change in the activity rate of the bacteria Lactobacillus delbrueckii subsp. Bulgaricus and Streptococcus thermophilus in milk from the family farm "Stanković" from the village of Vrgudinac (municipality of Bela Palanka), with inoculation with starter cultures from the commercial yogurt of the dairy "Milkhouse Niš" containing dominant strains of bacteria. Continuous measurement of pH value and temperature was performed for 12 hours, while periodic measurements of parameters were performed every 30 minutes. The obtained findings on changes in voltage, pH value, and temperature using the proposed system for measuring parameters show the expected profile of acidity and coagulation of milk, and they are presented in the paper.

Keywords**:** microcontroller, pH, temperature, milk, fermentation.

Original Scientific Paper

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# OPTIMIZATION OF MEDIUM COMPOSITION AND CULTIVATION CONDITIONS TO ENHANCE CELL GROWTH OF THE *Pichia kluyveri* K-1 YEAST ISOLATE

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This research aimed to identify the key variables influencing the cell growth of the yeast isolate *Pichia kluyveri* K-1 in order to maximize the number of viable cells and biomass yield. Initially, the one-factor-at-a-time method was used to assess the effect of various carbon sources (glucose, fructose, galactose, maltose, and glycerol) at different concentrations (5-150 g/L) on yeast growth. Glucose proved to be the most effective carbon source, with the highest viable cell count (8.77 log CFU/mL) and biomass yield (68.15 g/L) observed at 150 g/L. However, considering cost efficiency, a glucose concentration of 50 g/L was chosen for further work, as it reduced production costs per unit by 245% with only a 20% decrease in yield. To evaluate the effects of medium composition and process conditions, a Plackett-Burman experimental design (Design-Expert, Stat-Ease Inc., Minneapolis, USA) was applied. Based on the literature, 11 potentially significant variables were tested across 12 runs. The design efficiently identified the significant variables and proposed predictive models. Six variables (agitation speed, yeast extract, $(NH_4)_2SO_4$, $MgSO_4 \cdot 7H_2O$, $KH_2PO_4$, and $CaCl_2$) positively affected viable cells count, while seven (including riboflavin) enhanced biomass yield. However, only temperature and agitation speed were identified as statistically significant parameters ($p<0.05$) for both responses. In contrast, temperature, $ZnSO_4 \cdot 7H_2O$, biotin, and $MnCl_2 \cdot 4H_2O$ negatively impacted both responses. High $R^2$ and adjusted $R^2$ values (>93%) confirmed the strong predictive ability of the proposed models. These findings provide valuable insights into the initial stages of optimization and lay the foundation for subsequent optimization using the Box-Behnken design and response surface methodology.

Keywords: *Pichia kluyveri*, one-factor-at-a-time method, Plackett-Burman design

Original Scientific Paper

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# UHPLC-QTOF-MS CHARACTERISATION OF ANTHOCYANINS IN INDIGENOUS SERBIAN GRAPE SKINS: PROKUPAC AND KADARKA

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In Serbia, wine production is predominantly based on international grape varieties. However, there is a growing interest in the cultivation and commercialization of indigenous cultivars, particularly Prokupac and Kadarka, which are gaining increasing domestic recognition. These red grape varieties are commonly used for the production of red and rosé wines, resulting in substantial quantities of winemaking by-products, including pomace, skins, stems, and seeds. Recent studies have highlighted the potential of these by-products as sources of valuable bioactive compounds, especially phenolics, which can be extracted and repurposed into value-added products [1]. Most existing research has focused on quantifying and characterizing the phenolic profiles of grape pomace or its individual components derived from regionally significant cultivars. However, the detailed anthocyanin profile of the skins of indigenous Serbian varieties, such as Prokupac and Kadarka, remains insufficiently explored. Therefore, the aim of this study was to perform an untargeted analysis of anthocyanins in the grape skins of Prokupac and Kadarka, using the advanced UHPLC-QTOF-MS technique. The anthocyanins were identified based on their monoisotopic mass and characteristic MS fragmentation patterns, and confirmed using available standards or data from the literature. Kadarka grape skins contained a significantly higher amount of anthocyanins compared to Prokupac. In both varieties, malvidin derivatives were dominant, with malvidin-3-*O*-glucoside being the most abundant compound. Derivatives of petunidin, peonidin, delphinidin, and cyanidin were also identified in both grape skins, although at much lower levels than malvidin derivatives. The anthocyanin profiles of Prokupac and Kadarka skins highlight their potential application as functional food ingredients.

Keywords: UHPLC Q-ToF MS, anthocyanins. grape skin, Prokupac, Kadarka

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•The industry partner UNIVEG LLC Žabalj, Industrijska zona BB, Žabalj, Serbia

# RAPESEED OIL POWDER AS A VALUABLE NUTRITIONAL INGREDIENT IN WHOLEMEAL SPELT PASTA

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Pasta is an excellent matrix for incorporating “nutraceutical” active ingredients due to its widespread popularity among consumers, as well as its ease of processing, storage, and preparation. However, the addition of non-traditional ingredients may alter the rheological properties during processing or negatively impact the sensory acceptability of the final product. Therefore, careful formulation and optimization of technological parameters are essential to maintain product quality.

One such nutritionally valuable ingredient is powdered rapeseed oil, which is rich in monounsaturated fatty acids and the essential omega-3 alpha-linolenic acid, both known to support cardiovascular and cognitive health. Its low saturated fat content, combined with natural vitamin E levels, makes it well-suited for daily consumption and the prevention of chronic diseases. Moreover, microencapsulation technology ensures that this oil form is stable, easy to handle, and highly suitable for incorporation into functional and instant food products.

In this study, powdered rapeseed oil was incorporated at 10% (w/w) of the total flour blend (comprising 80% whole spelt flour and 20% durum wheat semolina) and successfully integrated into the formulation of wholemeal spelt pasta, significantly enhancing its nutritional profile. The resulting pasta contains 0.68 g of alpha-linolenic acid per 100 g, satisfying the criteria for the nutrition claim “high in omega-3 fatty acids” according to the Regulation on nutrition and health claims. After cooking, the pasta retains 0.34 g of alpha-linolenic acid per 100 g, meeting the requirement for the claim “source of omega-3 fatty acids.” Additionally, a favorable omega-3/omega-6 fatty acid ratio of 1:3 was achieved. The incorporation of powdered rapeseed oil substantially improves the nutritional value of wholemeal spelt pasta without adversely affecting its sensory attributes or other physical characteristics.

Keywords: wholemeal spelt pasta, powdered rapeseed oil, omega-3 fatty acids, omega-3/omega-6 ratio, sensory quality

# DEVELOPMENT OF NUTRITIONALLY FORTIFIED COOKIES USING WHOLE BUCKWHEAT FLOUR AND ARONIA JUICE

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The aim of this study was to develop nutritionally fortified cookies by incorporating whole buckwheat flour and varying concentrations of aronia (Aronia melanocarpa) juice, focusing on improving their antioxidant properties. The cookies were formulated using 10%, 20%, and 30% of whole buckwheat flour, and 0%, 10%, 20%, and 30% of aronia juice. The antioxidant activity, as a measure of the cookies' functional potential, was evaluated using the DPPH radical scavenging assay. The results indicated an increase in antioxidant activity from 22.7% in the samples containing only buckwheat flour to 25.6% in the samples enriched with both buckwheat flour and aronia juice, demonstrating a synergistic effect of these ingredients. The observed antioxidant enhancement is attributed to the presence of naturally occurring polyphenolic compounds found in buckwheat and aronia. With rising consumer demand for natural alternatives to synthetic additives, these fortified cookies offer a promising fortified food product with improved nutritional quality and extended shelf life.

Keywords: Buckwheat, Aronia, Cookies, Polyphenolic antioxidants, Fortified food

Original Scientific Paper
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# ANTIOXIDANT AND ANTIMICROBIAL PROPERTIES OF CARRAGEENAN-BASED FILMS WITH THE ADDITION OF HAWTHORN AQUEOUS EXTRACT

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This study investigated the antioxidant and antimicrobial properties of carrageenan-based films with the addition of hawthorn aqueous extract at concentrations of 5%, 10%, and 15%. The antioxidant activity was determined using the DPPH method, showing that the radical scavenging capacity increased with higher extract content, with the film containing 15% extract showing the highest activity (the DPPH radicals degree of neutralization: 63.69%). The total phenolic content and migration of bioactive compounds also increased proportionally with extract concentration, with the highest migration observed in the film with 15% extract (8.64 mg GAE/g). The control carrageenan film without hawthorn extract exhibited a certain level of antioxidant activity, with a DPPH radical inhibition of 48.5% and a total phenolic content of 1.12 mg GAE/g of film. Although these results were significantly lower compared to the extract-enriched films, they suggest that the carrageenan matrix itself can contribute to antioxidant potential to some extent, which can be attributed to the presence of sulfate groups in its structure. Antimicrobial activity was tested using a modified disk diffusion method against eight bacterial strains - *E. Coli* (ATCC 25922), *K. Pneumoniae* (ATCC 700603), *P. Vulgaris* (ATCC 8427), *L. Monocytogenes* (ATCC 15313), *P. Aeruginosa* (ATCC 27853), *B. Subtilis* (ATCC 6633), *S. aureus* (ATCC 25923), and *B. Cereus,* and yeast *C. Albicans* (ATCC 2091). All samples showed weak antimicrobial activity, with no significant differences among the various film formulations, and no activity was observed against *Staphylococcus aureus* and *Candida albicans*. The obtained results indicate that the addition of hawthorn extract significantly enhances the antioxidant properties of carrageenan films, while their antimicrobial effect is limited, likely due to the low extract concentration and its interactions with the film matrix.

Keywords: carrageenan, hawthorn extract, antioxidant activity, phenolic content, biodegradable film

Original Scientific Paper

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# SURVIVABILITY OF MICROENCAPSULATED *LACTOBACILLUS PLANTARUM* DURING KOMBUCHA FERMENTATION AND STORAGE

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Kombucha is a traditional beverage which is usually prepared by fermenting sweetened black tea using a symbiotic consortium of acetic acid bacteria (AAB) and yeast. Kombucha exhibits several functional properties, such as antioxidant, antimicrobial, anticancer, etc. Most often, kombucha cannot be labeled as a probiotic product, since the number of probiotic microorganisms usually does not reach the specified criteria. Lactic acid bacteria (LAB) are major microorganisms used for probiotic purposes. For LAB to exhibit health benefits, they should be able to survive sometimes harsh conditions during fermentation, storage, and passing through the gastrointestinal tract. Several methods of microencapsulation of probiotic microorganisms are widely used to improve their viability. In this study, the probiotic strain *Lactobacillus plantarum* ATCC 14917 was encapsulated in pea protein by the lyophilisation method and added at the beginning of kombucha fermentation. Encapsulation efficiency was measured and amounted to 91.17%. The survivability of *L. plantarum*, as well as chemical (pH and titratable acidity) and microbiological (number of yeasts, AAB, and LAB) parameters, were monitored during fermentation. The number of LABs was also determined during the storage of the final product. Pea protein showed good efficiency in protecting LAB cells since their number on the last day of fermentation was 7.22 logCFU/ml and after 21 days of refrigerated storage, 6.55 logCFU/ml, exceeding the recommended minimum level for probiotic products, defined as 6-7 logCFU/ml. The obtained results show that pea protein is a promising agent for microencapsulation of probiotic bacteria to obtain kombucha beverages with probiotic characteristics.

Keywords: kombucha; *Lactobacillus plantarum*; encapsulation; probiotic survivability

Original Scientific Paper

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# VARIATIONS IN RAW MILK QUALITY IN THE MOUNTAINOUS LIVESTOCK REGION OF THE PIROT DISTRICT

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The Pirot District, one of the leading livestock regions in southeastern Serbia, is recognized for its traditional production of high-quality raw milk and distinctive dairy products such as Pirot kačkavalj cheese. The chemical composition and physicochemical properties of milk vary due to seasonal factors, primarily feeding conditions and climatic influences. This study aimed to identify potential qualitative differences in raw milk composition resulting from dietary changes during seasonal transitions, specifically between the winter and summer of 2024. Milk samples were collected from three “Milk House” dairy stations (Pirot, Babušnica, and Bela Palanka), sourced from individual farms. Laboratory analyses included milk fat, protein, total and non-fat dry matter, acidity, and presence of added water. The results showed pronounced seasonal differences, particularly in milk fat and protein content. During the winter period, higher values of milk fat and protein were consistently recorded across all collection stations: in Pirot, milk fat was 4.12% and protein 3.29%; in Babušnica, fat content was 4.00% and protein 3.36%; while in Bela Palanka, fat reached 4.11% and protein 3.40%. In contrast, during the summer, values were slightly lower but more uniform across locations, with fat content at 3.96% and protein ranging from 3.19% to 3.22%. The results highlight the strong influence of seasonal diet on milk composition and underscore the need to adapt processing to ensure consistent quality. Despite growing climate change effects on livestock, the geographical and agroecological features of the Pirot District’s mountainous areas offer an advantage in preserving milk quality, supporting the region’s potential for sustainable dairy production.

Keywords: Dairy; Raw milk; Mountain regions; Variations in chemical composition

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# INFLUENCE OF MICROENCAPSULATED RAPESEED OIL ON THE QUALITY OF WHOLEMEAL TAGLIATELLE MADE WITH WHOLE RYE FLOUR

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Rapeseed oil is distinguished by its high content of monounsaturated fatty acids, particularly oleic acid, and a significant proportion of the essential omega-3 fatty acid alpha-linolenic acid (ALA), which plays a vital role in maintaining cardiovascular and nervous system health. Its low saturated fat content (approximately 7%) makes rapeseed oil suitable for daily consumption and the prevention of chronic non-communicable diseases. Moreover, it is a natural source of vitamin E (tocopherols), a potent antioxidant that protects cells against oxidative stress. Rapeseed oil contains no industrial trans fats, and its innovative powdered microencapsulated form facilitates broader application in the food industry—especially in functional, instant, and dietary products—due to enhanced dosing accuracy, improved stability, and extended shelf life.

In this study, a formulation and production process were developed for tagliatelle made from a flour blend consisting of 70% wheat semolina and 30% wholegrain rye flour, enriched with 10% powdered microencapsulated rapeseed oil (based on total flour weight). The resulting tagliatelle contains 0.62 g of alpha-linolenic acid per 100 g, thereby satisfying the criteria for the nutrition claim “rich in omega-3 fatty acids” according to national regulations on nutrition and health claims. Additionally, a favorable omega-3/omega-6 fatty acid ratio of 1:2.6 was achieved. Incorporation of the microencapsulated rapeseed oil did not significantly affect the technological and sensory quality, color, or texture of the pasta containing whole rye flour.

Keywords: microencapsulated rapeseed oil, whole rye flour, tagliatelle, omega-3 fatty acids, omega-3/omega-6 ratio, sensory quality

# Section: BIOCHEMICAL ENGINEERING

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# LOW-ENERGY NANOEMULSIONS WITH CITRUS ESSENTIAL OILS: FORMULATION DEVELOPMENT AND ANTIOXIDANT ACTIVITY

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Citrus essential oils are popular raw materials for natural cosmetics, known for their characteristic refreshing scent and flavour, but are also being investigated as potential antioxidants, antimicrobial, and anti-inflammatory actives. The aim of this study was to develop nanoemulsions with different citrus essential oils suitable for topical application and to evaluate their stability and antioxidant activity. The following 6 citrus essential oils (EOs) from the *Rutaceae* family were tested: *Citrus aurantium bergamia* – bergamot, *Citrus limon* – lemon, *Citrus aurantifolia* – lime, *Citrus aurantium dulcis* – sweet orange, *Citrus nobilis* – mandarin orange, and *Citrus paradisi* – grapefruit peel oil. The nanoemulsions were prepared using the low-energy phase inversion composition (PIC) method at room temperature and regularly characterised (Z-average droplet size and polydispersity index – PDI, pH value, and electrical conductivity). The nanoemulsions were stabilised with 8 to 10 wt% polyglyceryl-10 laurate (main surfactant) and further optimised by adding 0.5 wt% glyceryl caprylate as co-surfactant and/or 0.5 wt% phenoxyethanol/ethylhexyl glycerine as preservative/co-surfactant. Ethylhexyl stearate was used as the carrier oil, and the optimal EO concentration was 1 wt% for all citrus EO-loaded nanoemulsions. However, after three months of storage, only the nanoemulsions loaded with bergamot, lime, or lemon EO remained stable (Z-ave< 50 nm, PDI< 0.15). This could be related to the medium limonene content and the presence of linalool, linalyl acetate, and citral in these EOs, which was confirmed by Raman spectroscopy. The tested EOs scavenged ABTS free radical in the following order: sweet orange ~ mandarin orange (≥95%), lime (~68%), grapefruit (~64%), lemon (~56%), and bergamot (~47%). The optimised nanoemulsions with bergamot, lime, or lemon EO also scavenged the ABTS radical (~28%, ~42% and ~19%, respectively) and retained their antioxidant activity after three months. Therefore, their potential application as cosmetic nanoemulsions for antioxidant skin protection was confirmed.

Keywords: citrus essential oils, nanoemulsions, low-energy production, Raman spectroscopy, antioxidant activity.

Original Scientific Paper
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# VALORIZATION OF SUNFLOWER HULLS IN FUNGAL CELLULASES PRODUCTION: SELECTION OF CULTIVATION TECHNIQUES AND PRODUCTION TIME

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Sunflower is one of the most widely cultivated crops in Serbia, and its processing generates significant amounts of residues, mainly hulls, a by-product of hulling sunflower seeds. Approximately 20-30% of the weight of sunflower seeds is hulls, which are typically used as solid fuel or for bio-briquette production. Given the high cellulose and hemicellulose content, this research was conducted to explore a more sustainable approach for utilizing sunflower hulls by investigating the potential of fungal cellulase production for application in the detergent industry. Therefore, this study aimed to select the most suitable cultivation technique for the cellulase production from sunflower hull-based media using the wild-type *Aspergillus niger* strain, as well as the time at which maximum production of cellulolytic enzymes is achieved using the selected cultivation technique under given conditions. The bioprocess success was evaluated based on the protein content and cellulolytic activity of the crude enzyme preparations at the temperature conditions of detergent application, i.e., at 30°C and 60°C. Solid-state cultivation techniques (with spontaneous and intensive aeration) were the most suitable in terms of both analyzed bioprocess success indicators. Submerged cultivation techniques (with spontaneous and intensive aeration) followed in terms of suitability, while emulsion cultivation on a liquid medium with spontaneous aeration proved to be the least suitable. Monitoring the cultivation of the applied fungal strain using solid-state techniques for 168 h revealed that the bioprocess was most successful after 72 h, which is significantly shorter compared to the production time in the experiments conducted to select the cultivation technique.

Keywords: biotechnological production, sunflower hulls utilization, cellulases production, *Aspergillus niger*, fungal cultivation optimization

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# ANTIMICROBIAL ACTIVITY OF MICROENCAPSULATED AND UNENCAPSULATED EXTRACTS FROM THE UNDERGROUND PARTS OF *GENTIANA ASCLEPIADEA* L.

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*Gentiana asclepiadea* L. (Gentianaceae), commonly known as willow gentian, is a medicinal plant traditionally used for alleviating gastrointestinal and liver disorders. Owing to its pronounced bitterness, it is also utilized in the food industry, particularly as a flavoring component in liqueurs. This study aimed to evaluate the antimicrobial activity of an extract from the underground parts of *G. asclepiadea* and to assess the effect of microencapsulation on this property. The extract, obtained under previously optimized ultrasound-assisted extraction conditions (50 min, 65°C, 53% v/v ethanol, 40 mL/g liquid-to-solid ratio), was microencapsulated by spray-drying using 20% whey protein as a carrier. Antimicrobial activity was tested against a panel of enteropathogenic microorganisms (three Gram-positive, four Gram-negative bacterial strains, and one fungal strain). Both extract forms exhibited growth-inhibitory effects against all tested strains. Minimum inhibitory concentrations ranged from 3.13 to 25.0 mg/mL for the unencapsulated extract and from 3.13 to 50.0 mg/mL for the microencapsulated extract. *Enterococcus faecalis* was the most sensitive strain. The unencapsulated extract showed slight selectivity toward Gram-positive bacteria, whereas the microencapsulated extract exhibited no such selectivity. Microencapsulation enhanced activity against *Escherichia coli* and *Salmonella enteritidis*, while in most other cases it reduced antimicrobial activity, though inhibition was still present. Mild antifungal activity against *Candida albicans* was observed for both extracts, without a fungicidal effect. The obtained results suggest that *G. asclepiadea* extract possesses antimicrobial potential, and that the microencapsulated form could be a valuable functional ingredient in pharmaceutical and food products.

Keywords: *Gentiana asclepiadea* L., antimicrobial activity, spray drying, microencapsulation

Original Scientific Paper

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# DESIGN OF LEAF PROTEIN-BASED NANOPARTICLES FOR VITAMIN B12 AND FOLIC ACID DELIVERY

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This research is focused on the design of advanced delivery systems for selected model compounds (folic acid and vitamin B12) based on nanocarrier structures derived from the soluble protein fraction previously isolated from pumpkin leaves. The proteins were isolated from fresh leaves stored at - 80°C after harvesting, using a three-step method: mechanical disruption of plant cells, heat-induced precipitation of the green fraction, and acid precipitation of the soluble white fraction.

Novel delivery systems were produced by combining pH shifting and cold gelation, avoiding the high working temperatures used in other procedures, which could promote the degradation, destabilization, and loss of activity of the active compounds. Nanoparticle size and morphology were characterized by Dynamic Light Scattering and Transmission Electron Microscopy, while encapsulation efficiency was determined by HPLC-UV.

It was determined that an extensive purification was not required to achieve the desired functionality of the protein fraction for high performance in the design of a vitamin delivery system. Since particle size affects the texture and sensory properties of the final food product, the effects of initial pH, calcium and protein concentrations, and the type and concentration of bioactive compounds were investigated with respect to nanoparticle morphology, size, zeta potential, and polydispersity index. Protein nanocarrier appeared to be more effective for vitamin B12 encapsulation, achieving high vitamin loading and enabling controlled release under simulated gastrointestinal conditions. Initial pH had the highest impact, and the optimal formulation was obtained with 5 mM $CaCl_2$, protein concentration of 6 mg/mL, and the final pH of 8.

Keywords: Pumpkin leaves, White proteins, Vitamin B12, Nanoparticle structures, pH Shifting, and cold gelation

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# ANTIMICROBIAL ACTIVITY OF PPIX-MLV LIPOSOMES AGAINST *Staphylococcus aureus* and *Bacillus subtilis*

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Protoporphyrin IX (PPIX) is one of the most naturally occurring porphyrins, employed as a photosensitizer (PS) in medicine and pharmaceutical applications, and demonstrating the considerable potential for use in antimicrobial photodynamic therapy (aPDT). Liposomes are effective drug delivery systems for various bioactive compounds and photosensitizers, improving their clinical efficacy, target cell delivery, and solubility. This study aims to determine the antimicrobial activity of PPIX encapsulated within multilamellar vesicle (MLV) liposomes against two gram-positive bacteria, *Staphylococcus aureus* and *Bacillus subtilis*.
PPIX-MLV liposomes (with a PPIX concentration of $1 \times 10^{-5}$ M) were prepared using the dry PPIX-lipid film method. These liposomes were added to sterile Erlenmeyer flasks containing bacterial cultures and continuously stirred using a magnetic stirrer. Antimicrobial activity was induced by illuminating the samples in a hand-made cylindrical photochemical reactor equipped with “Pure White” LED lamps, delivering a light intensity of approximately 40,000 lux.
The results demonstrated that PPIX-MLV liposomes exert an inhibitory effect on both *S. aureus* and *B. subtilis*. In control samples, bacterial counts were $7.304 \pm 0.047$ and $7.379 \pm 0.090$ log CFU/ml, respectively. Following illumination for 10, 20, and 30 minutes, the colony-forming units for *S. aureus* were reduced to $6.944 \pm 0.005$, $6.673 \pm 0.013$, and $4.633 \pm 0.010$, while for *B. subtilis*, counts decreased to $6.064 \pm 0.011$, $4.853 \pm 0.008$, and $3.559 \pm 0.083$, respectively. These findings suggest that PPIX-MLV liposomes have significant promise for aPDT applications, although their antimicrobial efficacy is influenced by illumination duration and light intensity.

Keywords: Photosensitizer; Porphyrins; Liposomes; Antimicrobial Photodynamic Therapy

Review

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# ADVANCES IN BIOSENSING TECHNOLOGY FOR YEAST CELL DETECTION AND QUANTIFICATION

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The ability to detect and quantify yeast cells, particularly strains of *Saccharomyces cerevisiae*, is crucial in industrial biotechnology, environmental monitoring, and scientific research. Traditional methods such as plate counting and microscopy, while reliable, often lack the speed and sensitivity necessary for real-time monitoring and process optimization. In contrast, modern approaches based on techniques like fluorescence assays, PCR, and flow cytometry allow for faster and more reliable analysis, but require high costs and complex implementation. However, biosensing technologies offer rapid, sensitive, and often label-free detection platforms capable of quantifying yeast cells in complex matrices.

This study presents a theoretical overview of recent advancements in biosensor technologies for yeast cell detection, focusing on electrochemical, optical, and microfluidic platforms. Electrochemical biosensors using impedance spectroscopy have shown high potential in detecting viable yeast cells with minimal sample preparation. Optical biosensors, including surface plasmon resonance (SPR) and fluorescence-based systems, provide label-free and real-time tracking of yeast adhesion and growth. Furthermore, the integration of biosensors into microfluidic devices enables automated, miniaturized, and continuous monitoring, improving the precision of yeast quantification during bioprocesses.

Highlighting the growing importance of tailored biorecognition elements, such as aptamers, antibodies, and engineered peptides, an improvement in the specificity of biosensors for yeast cells has been demonstrated. Theoretical advancements also explore modeling the dynamics of sensor response and the challenges of distinguishing live from dead cells in real-time applications. These technologies pave the way toward fully integrated quality control and automation systems in the bioprocess industry.

Keywords**:** Yeast detection, Saccharomyces cerevisiae, biosensors, electrochemical biosensing, biorecognition elements

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# ENZYMATIC MODIFICATION OF RUTIN WITH L-CYSTEINE

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This study explores the enzymatic modification of rutin with L-cysteine catalyzed by horseradish peroxidase. Rutin, a flavonoid with notable antioxidant properties, suffers from limited bioavailability and solubility. To address these limitations, horseradish peroxidase was employed to catalyze the oxidation of rutin in the presence of hydrogen peroxide, enabling its subsequent conjugation with L-cysteine, a thiol-containing amino acid. The reaction progress and product formation were monitored spectrophotometrically, and the modified rutin derivatives were identified based on characteristic UV-Vis absorption changes. Kinetic analyses, including Michaelis-Menten and Lineweaver-Burk plots, were performed to elucidate the reaction mechanism. The results demonstrated a ping-pong kinetic behavior involving three substrates: rutin, hydrogen peroxide, and L-cysteine. The influence of each substrate concentration on the reaction rate was systematically investigated, and no substrate inhibition was observed. Additionally, non-enzymatic oxidation contributed to the total reaction to a minor extent (~11%). The obtained kinetic parameters indicate that horseradish peroxidase exhibits increased substrate affinity at higher concentrations, supporting the efficiency and potential applicability of this biocatalytic approach for rutin derivatization.

Keywords: rutin; L-cysteine; horseradish peroxidase; enzymatic oxidation; kinetic mechanism

## Section: PHARMACEUTICAL AND COSMETIC ENGINEERING

# INTERACTIONS OF TWO NATURAL AROMATIC $N^6$-SUBSTITUTED ADENOSINES WITH THE HUMAN $A_{2B}R$: *IN SILICO* STUDIES

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Targeting adenosinergic signaling, where $N^6$-substituted adenosines serve as key pharmacophores, is showing promise in the development of new drugs. Adenosine $A_{2B}$ receptors are deeply involved in the mechanisms underlying mediator release by mast cells, the major mechanism by which adenosine induces bronchoconstriction and allergic inflammation occurring in asthma, and thus are potentially important pharmacological targets in treating immune system dysfunction and inflammation.
On these grounds, we performed a preliminary *in silico* screening to identify potential therapy agents by performing molecular docking studies of two natural aromatic cytokinin $N^9$-ribosides as ligands ($N^6$-benzyl and $N^6$-4-hydroxybenzyladenosine) on the human adenosine receptor $hA_{2B}R$ active site. For docking studies, Molegro Virtual Docker (MVD) was used. The crystal structure of the human $A_{2B}$ adenosine receptor, obtained from the Protein Data Bank (PDB ID: 7XY6), was used as the target for docking studies. Three-dimensional structures of the studied ligands were obtained using MarvinSketch 6.1.0 with the application of the force field MMFF94. The performed *in silico* study identified hydrogen bonds, hydrophobic, and hydrophilic interactions between the two studied molecules and key amino acids inside the $hA_{2B}R$ binding pockets, suggesting cytokinin $N^9$-ribosides' potential as starting points for the development of hA2BR-selective modulators and novel anti-inflammatory agents.

Keywords: $N^6$-substituted adenosines; human adenosine receptor $hA_{2B}R$; molecular docking studies

Original Scientific Paper

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# FACIAL EXPRESSION FINE LINES AND WRINKLES: *IN VIVO* EFFECTS OF *ACMELLA OLERACEA* EXTRACT-BASED GLYCOLIPID EMULSION

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Fine expression lines that typically occur on the facial skin at an early adult age due to repetitive facial expressions progressively deepen and become more visible, forming permanent wrinkles with aging. To reduce or improve the appearance of facial expression lines and wrinkles, as the earliest, most prominent and undesirable signs of skin aging, the current study focused on a novel, natural, non-invasive, topical anti-aging emulsion, which is claimed to show a rapid myorelaxant action on facial mimetic muscles. The effects of the developed emulsion on periocular skin wrinkles (crow's feet) were assessed in human volunteers (8 females, aged 31-55 years) by employing silicone skin replica image analysis (Visioline® VL 650–Quantirides®, Courage+Khazaka, Germany). The tested emulsion, stabilized with an innovative natural mixed glycolipid emulsifier (lauryl glucoside/myristyl glucoside/polyglyceryl-6 laurate) and comprising 2% of *Acmella oleracea* (L.) extract (Asteraceae) as a model anti-aging active, was applied to the area around the eye on both face sides, twice daily, for two weeks. For each volunteer, the silicone replicas of assigned periorbital areas, at rest and with expression (at maximal smile), were collected before (baseline) and after 14 days of treatment. The *in vivo* study confirmed beneficial effects of the investigated emulsion on all measured skin wrinkle parameters: a significant ($p<0.05$) reduction in total wrinkle area, mean length, depth, and number of wrinkles, which was more pronounced at rest (40-50%) than with expression (10-20%). The present results highlight the high potential of *Acmella oleracea* extract-emulsion in improving both static and dynamic fine lines and wrinkles caused by facial expressions.

Keywords: expression wrinkles, crow's feet, *in vivo* efficacy, skin replica, wrinkle parameters.

## Section: ORGANIC CHEMICAL TECHNOLOGY

Original Scientific Paper

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# ULTRASOUND-ASSISTED EXTRACTION OF WALL GERMANDER FILTER TEA INDUSTRY WASTE

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The rise of herbal tea manufacturing is a direct result of the expanding global tea market and the increasing demand for healthier dietary choices. Increased tea production results in increased waste formation, which produces waste in the form of powdered plant material. Because of its size (<0.315 mm), this material, also known as herbal dust, is frequently regarded as waste. Its production can range from 10% to 40% of the entire processed plant. In this work, the bioactive compounds from wall germander (*Teucrium chamaedrys* L.) herbal dust, obtained from a filter tea factory, were extracted using ultrasound-assisted extraction (UAE). The extraction was performed by using water as a solvent at 50 ºC, at different extraction times (10, 20, 30 min). The total phenolic content was determined by the Folin-Ciocalteu spectrophotometric method. The highest total phenolic content was obtained at 20 min (475.16 ± 0.74 mg GAE/g DE). UHPLC-DAD-MS/MS analysis of all extracts indicated the presence of citric acids, hydroxycinnamic acids and their derivatives (caffeic acid, dicaffeoylquinic acid), phenylethanoid glycosides (teucreoside, allysonoside, forsythosides B, E, and J), and flavonoid aglycones (cirsiliol, diosmetin, luteolin). The obtained results suggest that waste wall germander herbal dust obtained from filter tea factories possesses significant amounts of bioactive compounds and can be used as a raw material or supplement in the pharmaceutical and cosmetic industries.

Keywords: wall germander, tea waste, ultrasound-assisted extraction, total phenolic content

Original Scientific Paper
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# CHEMICAL COMPOSITION AND ANTIOXIDANT ACTIVITY OF ESSENTIAL OIL FROM WHITE BIRCH LEAVES (*BETULA PENDULA* ROTH)

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*Betula pendula* Roth, commonly known as white birch, has been used since ancient times for the treatment of urinary tract disorders, infections, and inflammations. The aim of this study is the isolation of essential oil from dried white birch leaves, and the determination of its chemical composition and antioxidant activity. The essential oil was isolated from a dried white birch leaf by Clevenger-type hydrodistillation for 2 hours at the boiling point of distilled water, with a hydromodule of 1:10, w/v.
The chemical composition of the isolated essential oil was determined by GC/MS and GC/FID methods on an Agilent Technologies 7890B gas chromatograph coupled with an inert, selective 5977A mass detector of the same company. Components were separated on a non-polar, silica capillary column, HP-5MS (5% diphenyl- and 95% dimethyl-polysiloxane), 30 m×0.25 mm×0.25 μm. Antioxidant activity was determined using the DPPH method. Reaction mixtures were incubated at room temperature, in the dark, for 20-60 min, and the absorbance was measured at 517 nm on a UV/Vis spectrophotometer.
The most abundant groups of compounds were oxygenated sesquiterpenes (30%), oxygenated monoterpenes (22.6%), and sesquiterpene hydrocarbons (14.6%). DPPH radical scavenging capacity increased with incubation time. $EC_{50}$ values of 3.13 mg/cm$^3$, 1.91 mg/cm$^3$, and 1.41 mg/cm$^3$ were determined after 20, 40, and 60 minutes of sample incubation, respectively. Antioxidant activity is related to the presence of monoterpene and sesquiterpene compounds in white birch leaf essential oil. The significant antioxidant potential of white birch leaf essential oil provides the possibility of application as a natural antioxidant.

Keywords: *Betula pendula*, essential oil, chemical composition, antioxidant activity, GC/MS, DPPH.

Original Scientific Paper

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# TOTAL PHENOL AND FLAVONOID CONTENT AND ANTIMICROBIAL ACTIVITY OF ISOLATES FROM WHITE BIRCH (*BETULA PENDULA* ROTH)

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In this study, the total phenolic and flavonoid content was determined, in the collected sap, leaf, and bark extracts of white birch, as well as their antimicrobial activity. The extracts from dried leaves and bark of white birch were obtained using Soxhlet extraction with 96%, v/v ethanol, at the boiling point of the solvent at a solvomodule of 1:15 w/v and 1:10 w/v during 4h and 5h, respectively.

The total phenolic content was determined using the Folin-Ciocalteu method, while total flavonoid content was measured spectrophotometrically using the aluminum(III)-chloride method. The samples were incubated for 90 and 40 min for total phenolics and flavonoids, and absorbances were measured at 765 nm and 415 nm, respectively. The antimicrobial activity of the samples was evaluated using the microdilution method against Gram (-) bacteria*: Escherichia coli*, *Pseudomonas aeruginosa*, *Salmonella enteritidis*, Gram (+) bacteria: *Staphylococcus aureus*, *Enterococcus faecalis*, *Bacillus cereus,* and the fungus *Candida albicans*.

The total phenolic content in leaf and bark extracts of white birch was 112 mg GAE/g d.e. (gallic acid equivalents per gram of dry extract), and 54 mg GAE/g d.e., respectively, while birch sap contained 18 mg GAE/g d.e. The total flavonoid content in the white birch leaf extract was 91 mg RE/g d.e. (rutin equivalents per gram of dry extract), whereas in the bark extract and sap, it was below the detection limit. White birch leaf and bark extracts exhibited antimicrobial activity against all the tested strains of microorganisms. White birch sap showed no antimicrobial activity. The antimicrobial activity correlated with the total phenolic and flavonoid content in the analyzed samples.

Keywords: *Betula pendula*, Soxhlet extraction, antimicrobial activity, phenols, flavonoids

Original Scientific Paper

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# ANTIOXIDANT ACTIVITY OF ROSEMARY LEAF ESSENTIAL OIL (*Rosmarinus officinalis* L.)

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Rosemary (*Rosmarinus officinalis* L., Lamiaceae) is a perennial shrub originating from the Mediterranean, which is grown all over the world as an ornamental plant. It is used both in nutrition and in traditional medicine. Rosemary leaves are rich in essential oil (1-2.5%). According to the recommendations of the European Medicines Agency, rosemary essential oil can be used systemically for the treatment of dyspepsia and spasms of the gastrointestinal tract or locally for the relief of moderate myalgia and joint pain, as well as in the treatment of peripheral circulation disorders. In addition to analgesic and choleretic effects, rosemary essential oil also exhibits hepatoprotective, antimicrobial, and antioxidant effects. This work aimed to determine the antioxidant potential of the isolated rosemary essential oil by applying DPPH, ABTS, and FRAP assays. The essential oil was isolated by using Clevenger-type hydrodistillation (hydromodule 1:15, m/V, distillation time 180 min). The yield of the isolated essential oil was 1.29 $cm^3$/100 g of plant material. The obtained results indicated the ability of rosemary essential oil to neutralize DPPH and ABTS radicals, which increases with a longer incubation period. After 60 min of incubation, the $EC_{50}$ values were 29.7 ± 0.136 $mg/cm^3$ and 38.48 ± 0.47 $mg/cm^3$ in the DPPH and ABTS assays, respectively. The results of the FRAP assay showed that isolated essential oil has an almost negligible reducing capacity (0.062 ± 0.001 mmol $Fe^{2+}$/g of essential oil). Based on the presented results, it can be concluded that isolated rosemary essential oil has a relatively low antioxidant potential.

Keywords: Rosemary, hydrodistillation, essential oil, antioxidant activity.

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# ISOLATION AND CHEMICAL COMPOSITION ANALYSIS OF ROSEMARY LEAF ESSENTIAL OIL (*Rosmarinus officinalis* L.)

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Rosemary (*Rosmarinus officinalis* L.) is an aromatic plant from the Lamiaceae family that originates from the Mediterranean. It is a shrub, up to 2 m tall, with narrow, leathery, perennial green leaves 2-3 cm long. The content of essential oil in rosemary leaves ranges from 1 to 2.5%. Rosemary essential oil is a colorless to light yellow liquid with a bitter taste and a specific aromatic scent. The European Pharmacopoeia (Ph. Eur. 8) prescribes that, based on the content of the most abundant components, rosemary essential oil can be classified into two basic chemotypes: Moroccan-Tunisian type, in which the content of 1,8-cineole is significantly higher than the content of camphor and *α*-pinene, and Spanish type, where the contents of all three compounds are approximately equal. This work aimed to determine the qualitative and quantitative chemical composition of the rosemary leaf essential oil by applying GC/MS and GC/FID methods. The rosemary essential oil was isolated by using Clevenger-type hydrodistillation (hydromodule 1:15, m/V, distillation time 180 min). The yield of the isolated essential oil was 1.29 $cm^3$/100 g of plant material. Thirty-six components were identified in the oil, belonging to the groups of monoterpene hydrocarbons (41%), oxygen-containing monoterpenoids (52.6%), sesquiterpene hydrocarbons (1.4%), oxygen-containing sesquiterpenoids (0.2%), and other compounds (4.8%). The most abundant components in the essential oil were 1,8-cineole (24.2%), camphor (19.5%), and *α*-pinene (17.2%). Based on their content, it can be concluded that the isolated rosemary essential oil belongs to the Spanish chemotype.

Keywords: Essential oil; GC/MS analysis; Hydrodistillation; *Rosmarinus officinalis* L.

Original Scientific Paper

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# CHEMICAL COMPOSITION OF INFUSION FROM WILD APPLE WASTE PRETREATED WITH DEEP EUTECTIC SOLVENT

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Wild apple waste generated by tea factories is an insufficiently used by-product primarily composed of peels, pulp, seeds, and stems remaining after extraction processes. Rich in dietary fiber, natural sugars, polyphenols, and organic acids, this biomass presents significant potential for valorization across various industries. Sustainable management of wild apple waste can contribute to environmental conservation and economic efficiency by repurposing it as animal feed, compost, a bioenergy source, or raw material for nutraceutical and cosmetic applications. The chemical composition of wild apple dust infusion, prepared by pretreatment with the deep eutectic solvent betaine/oxalic acid/$H_2O$ was analyzed by using UHPLC-DAD-MS/MS analysis. The herbal dust was submerged with the prepared eutectic solvent, and after 72 h at room temperature, it was strained and filtered, and the infusion was made using a traditional method (a tablespoon of waste was poured over 200 mL of boiling water to steep and covered for 15 min, after which the infusion was poured through a strainer). The analysis indicated the presence of different flavonoids, phenols, phenolic acids, and their derivatives (quinic, rosmarinic, protocatechuic, chlorogenic, dicaffeoyllquinic, 3-*O*-p-cumaroylquinic, 4-*O*-*p*-coumaroyl-quinic acid), flavonoids (apigenin-penthosyl- hexoside, hyperoside, isoquercitrin, rutin, quercetin, quercitrin, methyl-isorhamnetin-*O*-glucoside, kampferide), and phenols (phloretin, phlorizin, and phloridzin). The results show that wild apple waste can be used for the extraction of bioactive components and has a significant role in promoting circular economy practices within the tea processing sector.

Keywords: Wild apple waste, infusion, pretreatment, eutectic solvent, chemical composition

# PHENYLPROPANOIDS AND ANTIOXIDANT ACTIVITY IN AROMATIC PLANT HYDROLATES: *Origanum vulgare* L. AND *Ocimum basilicum* L.

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Hydrolates, as aqueous-based solutions, offer an environmentally friendly strategy for reducing the environmental impact of waste. The recovery of bioactive compounds from these "residual waters" left in the distillation vessel, especially from species of the Lamiaceae family, is receiving increasing attention.
The aim of this study was to evaluate the phenylpropanoid derivatives and antioxidant activity of hydrolates extracted from the aerial parts of oregano (*Origanum vulgare*) and sweet basil (*Ocimum basilicum*). The phenylpropanoid content was quantified spectrophotometrically according to the Arnow method and expressed as chlorogenic acid equivalents (% HA/ml). Anti-lipid peroxidation activity was assessed using the linoleic acid/*β*-carotene bleaching assay. The hypothesis of equality of means was tested using Student's t-test, and linear relationships were assessed using Pearson's correlation coefficient. All analyses were performed using SPSS version 20.0.
The phenylpropanoids content was 0.38 ± 0.01% HA/mL in oregano and 0.43 ± 0.03% HA/mL in sweet basil hydrolate, with no significant difference observed ($p > 0.05$). Antioxidant activity, expressed as $IC_{50}$ values, was significantly different between samples: 11.09 ± 0.02% (v/v) for oregano and 28.97 ± 0.02% for basil hydrolate ($p < 0.05$). A strong positive correlation was found between the phenylpropanoids content and the antioxidant activity of basil hydrolate ($r = 0.982$).
Essential oils from Lamiaceae species are frequently used in industrial and medical research due to their unique physicochemical properties. However, their use is often limited by possible adverse effects. In this context, hydrolates represent a safer alternative for use.

Keywords*:* Anti-lipid peroxidation, CGAeq, Lamiaceae, Natural antioxidants

Original Scientific Paper

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# VALORIZATION OF A NASAL FORMULATION BASED ON SOME HERBAL EXTRACTS INCORPORATED BY ULTRASONIC MACERATION INTO COLD-PRESSED SUNFLOWER OIL

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Due to their valuable phytotherapeutic effects, polyherbal oily formulations are gaining increasing importance in herbal medicine. One of the understudied areas of their application is the treatment of rhinitis, primarily drug-induced rhinitis. The aim of this work is to valorize a potential nasal formulation based on cold-pressed sunflower oil enriched with selected plant extracts. Enrichment of the formulation carrier oil with active principles of some medicinal plants (*Equisetum arvense* L.*, Calendula officinalis* L.*, Taraxacum officinale* L.) was performed by an ultrasound-assisted extraction process (plant-oil ratio of 1:5 w/w, at 23 °C for 2 h, with a frequency of 30 kHz). Qualitative characterization of macerated samples was performed by High-performance thin-layer chromatography (HP-TLC, Silica gel 60, $F_{254}$). The flavonoid content was estimated by UV-VIS spectrophotometry (absorbance at 430 nm), while the alpha-tocopherol content was determined by Gas chromatography/mass spectrometry (GC-MS, capillary column ZB-5MS, helium flow rate of 1.2 mL/min). The antioxidant activity of the oily formulation (a mixture of 3 herbal macerates in equal volume ratio) was evaluated by an indirect DPPH spectrophotometric method (decrease in absorbance at 520 nm). The antioxidant potential of the polyherbal oily formulation ($EC_{50}$ 0.118 mg/mL) was significantly better compared to sunflower oil ($EC_{50}$ 8.532 mg/mL). This confirms the synergism of antioxidant α-tocopherol molecules with the active principles of plant extracts, primarily flavonoid compounds. The conducted research indicates that the obtained and characterized oily macerates are of exceptional importance for the preparation of nasal formulations useful in the treatment of rhinitis.

Keywords: rhinitis, nasal formulation, medicinal plants, ultrasonic maceration.

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# CHEMICAL COMPOSITION OF *HYPERICUM BARBATUM* JACK. ESSENTIAL OIL

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The genus *Hypericum* L. (Hypericaceae), encompassing over 480 species globally (excluding Antarctica), has long been recognized for its medicinal value, exhibiting notable antiviral, antimicrobial, antioxidant, wound-healing, and antidepressant activities.

In this study, the essential oil from the fresh aerial parts of *Hypericum barbatum* Jack., obtained by hydrodistillation using a Clevenger-type apparatus (hydromodule 1:10, w/v), was analyzed by GC and GC/MS. The process yielded 0.03% (w/w) of pale yellow essential oil. A total of 95 compounds were identified, representing 96.2% of the total composition. The major constituents included (*E*)-caryophyllene (10.7%), α-terpineol (9.3%), δ-cadinene (6.6%), β-pinene (4.8%), and α-selinene (4.5%). Other noteworthy abundant components were germacrene D (4.1%) and β-selinene (3.9%). The volatile profile of the oil was dominated by sesquiterpenoids (approximately 60%), particularly sesquiterpene hydrocarbons (44.1%), while oxygenated sesquiterpenoids contributed 15.2%. This fraction was primarily composed of compounds with cadinane and cadinane-related (16.9%), caryophyllane (13.8%), eudesmane (11.9%), and germacrane (5.2%) skeletons. Monoterpenoids constituted 22.6% of the oil, with oxygenated derivatives (13.4%) exceeding the monoterpene hydrocarbons in content. Within this class, compounds bearing *p*-menthane (12.5%) and pinane (7.2%) skeletons were predominant. The *p*-menthane-type compounds were mainly oxygenated (10.6%), whereas the pinane derivatives were largely hydrocarbons (6.6%). Non-terpenoid compounds accounted for 14.0% of the oil, with *n*-alkanes (9.3%) being the principal representatives of this group.

The composition of *H. barbatum* essential oil identified in this study exhibited notable differences from previously published reports.

Keywords: *Hypericum barbatum* Jack., essential oil, chemical composition.

Original Scientific Paper

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# THE POSITIVE EFFECT OF MICROWAVES AND GRINDING OF LAVENDER FLOWERS ON ESSENTIAL OIL COMPOSITION AND ANTIMICROBIAL POTENCY

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This paper investigates the effect of grinding of lavender (*Lavandula angustifolia* Mill.) flowers cultivated in Serbia for obtaining essential oil (LEO) by intensified microwave-assisted hydrodistillation (MWHD) and conventional hydrodistillation (HD). The LEO yield, chemical composition, and antimicrobial activity were greatly affected by the grinding pretreatment. LEO isolated by MWHD from the ground and non-ground plant material was composed of 11 and 17 volatile components, respectively. On the other hand, LEO isolated by HD from ground and non-ground plant material contained 29 and 51 compounds, respectively. Regardless of the extraction technique, the major constituents of LEOs were oxygenated monoterpenes, followed by oxygenated sesquiterpenes and sesquiterpene hydrocarbons. The essential oils obtained by microwave-assisted hydrodistillation were more potent antimicrobial agents compared to the oils obtained by classical hydrodistillation; the most sensitive microorganisms were *B. subtilis* and *C. albicans*. The suggested combination of grinding and intensified MWHD represents a promising approach for producing antimicrobial LEO with the potential to be used in different industries.

Keywords: antimicrobial activity; essential oil; grinding; microwave-assisted hydrodistillation; *Lavandula angustifolia* Mill.

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# ELECTROCHEMICALLY CROSS-LINKED ZN-ALGINATE FILMS: NEW MATERIALS WITH ANTIOXIDANT AND ANTIMICROBIAL ACTIVITY

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Alginate films, widely applied in various industries, are increasingly being explored for pharmaceutical and cosmetic use, particularly in skin regeneration and antibacterial protection. The electrochemical method used for their preparation offers numerous advantages, including compositional flexibility, minimal sample requirements, and the ability to produce biocompatible, renewable, and non-toxic matrices suitable for incorporating natural bioactive compounds. This study focused on the development of electrochemically cross-linked zinc–alginate (Zn–Zn-alginate) films enriched with hyaluronic acid (HA) and/or oregano hydrolate (OH), aiming to evaluate their antioxidant and antimicrobial properties in relation to composition. Film characterization included analysis of swelling behavior, antimicrobial efficacy against both Gram-positive (e.g., *Staphylococcus* spp., *Enterococcus faecalis*) and Gram-negative bacteria (e.g., *Escherichia coli*, *Klebsiella pneumoniae*, *Pseudomonas aeruginosa*), as well as antioxidant activity assessed by DPPH and ABTS radical scavenging assays. The films containing HA exhibited notably enhanced swelling capacity over time, indicating improved hydration potential. The presence of Zn modulated the swelling kinetics, while OH contributed to increased swelling in a time-dependent manner. All tested films demonstrated antibacterial activity against both Gram-positive and Gram-negative strains, which can be attributed to the antibacterial properties of Zn, HA, and OH and/or their synergistic activity. The antimicrobial effect was most pronounced in the samples containing OH, and was comparable to that of standard antibiotics such as amikacin. Antioxidant testing revealed a generally higher scavenging capacity in hydrophilic conditions as measured by the ABTS assay. Freshly prepared films outperformed dried ones in antioxidant assays, and the combination of HA and OH yielded the most favorable results in terms of radical inhibition. These findings suggest that the incorporation of HA and OH into Zn–Zn-alginate matrices enhances their functional properties, supporting their potential application in topical pharmaceutical formulations.

Keywords: alginate films, electrochemical cross-linking, zinc ions, antioxidant activity, antimicrobial activity.

Original Scientific Paper

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# ANTHOCYANINS PROFILE AND ANTIOXIDANT ACTIVITY OF BLACK CURRANT (*Ribes nigrum* L., VARIETY OMETA) EXTRACTS

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Black currant (*Ribes nigrum* L.) is renowned for being a valuable source of various bioactive compounds, including anthocyanins, flavonols, and ascorbic acid. Among these, anthocyanins are the predominant group of polyphenols found in berries, providing the dark purple color of the ripe fruit. Anthocyanins are recognized for their significant antioxidant, antimicrobial, and anti-inflammatory properties, as well as chemoprotective effects. They have been shown to reduce the risk of cognitive decline and improve cognitive behavior. This growing awareness has led to an expanded use of black currants in the food industry, ranging from natural colors and flavors in food and beverages to antioxidants in meat products. The objective of this study is to enhance our understanding of anthocyanin extraction using various solvents, including methanol (MeOH), 80% MeOH, 70% MeOH, ethanol (EtOH), 80% EtOH, 70% EtOH, and water. The analyzed extracts were made from the black currant variety Ometa. To accurately identify and quantify the anthocyanins, high-performance liquid chromatography (HPLC) was employed, while the DPPH method was used to evaluate the antioxidant activity of the extracts. Our findings indicated that delphinidin-3-*O*-rutinoside and cyanidin-3-*O*-rutinoside were the main anthocyanins present in all extracts. The most effective extraction of anthocyanins was achieved using ethanol and 80% MeOH. The highest antioxidant activity was observed in the MeOH and 70% MeOH extracts. This research not only highlights the optimal extraction solvents and the nutritional value of specific black currant extracts but also emphasizes their potential health benefits, paving the way for further exploration of their applications in the food industry.

Keywords: black currants, anthocyanins, antioxidants, extracts.

Original Scientific Paper

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# DETERMINING THE ANTIOXIDANT ACTIVITY OF THE ETHANOLIC EXTRACT OF ELDERBERRY (*Sambus nigra*) BARK

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The elderberry (*Sambucus nigra*) bark has been traditionally used in folk medicine, though it remains less studied compared to the plant's flowers and fruits. Rich in bioactive compounds, it holds potential as a natural source of antioxidants, which play a role in protecting human health by neutralizing free radicals. The aim of this research was to evaluate the total polyphenol content (TPC) and total flavonoid content (TFC), and antioxidant activity of elderberry bark extract using two assays, CUPRAC and DPPH. The results showed that the CUPRAC value was 4.60 mg of Trolox equivalents (TE)/g, indicating a considerable ability of the elderberry bark to reduce copper(II) ions, which reflects its overall antioxidant potential. The DPPH scavenging activity reached 865 mmol TE/g, demonstrating a strong capacity of the extract to neutralize free radicals through hydrogen atom donation. The total flavonoid content was measured at 0.33 mg of catechin equivalents (CE)/g, suggesting the presence of moderate levels of flavonoids, which are known contributors to antioxidant defense mechanisms. Additionally, the total polyphenol content was 0.77 mg of gallic acid equivalents (GAE)/g, confirming that phenolic compounds are present in moderate amounts and likely contribute to the observed antioxidant activity. The results showed that the CUPRAC and DPPH values were relatively high, indicating strong antioxidant activity, while TP and TFC were moderate. Pearson correlation analysis revealed a strong positive correlation between CUPRAC and TP ($r > 0.9$), suggesting that phenolic compounds significantly contribute to the reducing capacity. A moderate correlation was also observed between DPPH and TFC, implying that flavonoids play a role in radical scavenging activity. This suggests that the elderberry bark may contain unique or more concentrated antioxidant compounds contributing to this elevated radical scavenging potential.

Keywords**:** *Sambus nigra,* total polyphenol content, total flavonoid content, DPPH, CUPRAC

Original Scientific Paper

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# THE EFFECT OF MICROWAVE DRYING ON MINERAL CONTENT IN *Capsicum annuum* L. SEEDS

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Pepper seeds are valuable food ingredients, because of their mineral and oil content, which can be influenced by various factors. This work investigated the impact of drying methods - microwave versus traditional - on the macro- and micro-element content in seeds of *Capsicum annuum* L., specifically the homemade sweet variety "*Venčara*". The microwave drying process utilized a Midea, MG7017P-B oven, employing a two-stage drying program: 5 minutes at 55% of the maximum output power of 700 W, followed by 7 minutes at 33%. The traditional drying method was conducted in a drafty area at room temperature over a period of 30 days. The dried samples were ground, and nitric acid was added in a ratio of 1:20 *w/v*. A three-stage microwave digestion in a QWAVE-1000 digestion system (Questron Corporation, USA) was performed: at 160 °C for 30 minutes, at 180 °C for 15 minutes, and at 50 °C for 60 minutes, all at a power setting of 800 W. The minerals were analyzed by using an ICP-OES, ARCOS FHE12, SPECTRO instrument. In both samples, the potassium (7071.7-10320.9 *μ*g/g) was the most abundant element. In seeds dried using the microwave method, the content of calcium, iron, and magnesium was 358.1, 87.7, and 2973.2 *μ*g/g, respectively, and was higher than in seeds dried by the traditional method (213.1, 65.9, and 2684.8 *μ*g/g, respectively). The results suggest that microwave drying is more effective in preserving minerals in pepper seeds compared to traditional drying.

Keywords: drying, microwave, mineral, pepper, seeds

Original Scientific Paper

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# ASSESSMENT OF OXIDATIVE STABILITY OF CHLOROPHYLL-LOADED NANOLIPOSOME SYSTEMS BY USING THE FENTON REACTION

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The Fenton reaction can be used as a tool to monitor the stability of various biomolecules and formulations, enabling the prediction of product shelf life under oxidative stress conditions. It can occur both in biological systems and in systems such as liposomes. Chlorophyll a (chl)-loaded liposomes were prepared using the thin-film hydration method, with a pigment-to-lipid concentration ratio of 1:100 (DPPC or PL-90, C = $5\times10^{-4}$M). To obtain small unilamellar vesicles (SUVs), extrusion was performed through 100 nm filters. In the Fenton reaction, oxidation was initiated by adding 1 cm³ of $FeSO_4\times7H_2O$, 1 cm³ of $H_2O_2$, and 1 cm³ of the liposome-chl dispersion at a defined concentration. The final molar ratio of $Fe^{2+}$ to $H_2O_2$ in the 3 cm³ reaction mixture was adjusted to 1:250. The reaction was monitored over a period of 0–300 minutes using UV-VIS spectrophotometry. The percentage of chl degradation in an aqueous medium, SUV-DPPC, and SUV-PL-90 liposomes was 39.90%, 8.10%, and 17.85%, respectively. In addition to other factors influencing the Fenton reaction, the degradation percentage also depends on lipid composition. The incorporation of chl into PL-90 liposomes, which contain more than 50% unsaturated lipids, resulted in a higher chl degradation percentage compared to SUV-DPPC liposomes. Overall, the results indicate a protective effect of liposomes, suggesting that chl-incorporated nanoliposome systems should be used to preserve chl stability. The Fenton reaction proved to be a valuable tool for monitoring oxidation processes in chlorophyll-loaded liposomes, thereby enabling the assessment of the stability of desired active principles.

Keywords: chlorophyll, liposomes, Fenton reaction, oxidation, stability.

Original Scientific Paper
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# EVALUATION OF POLYPHENOL CONTENT AND ANTIRADICAL POTENTIAL OF ETHANOL AND METHANOL EXTRACTS OF TURMERIC RHIZOME

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The powdered rhizome of turmeric (*Curcuma longa* L., Zingiberaceae) has long been used as a spice and medicinal agent, especially in India and other Asian countries. Traditionally, it has been applied in the treatment of indigestion, fever, skin diseases, liver disorders, rheumatism, etc. Today, turmeric is widely recognized for both its nutritional and medicinal value. Turmeric is rich in phenolic compounds, mainly curcuminoids, with curcumin being the most prominent.
This study aimed to evaluate the antiradical activity of ethanol and methanol extracts of turmeric rhizome, and to determine their total polyphenol and tannin contents. The plant material, originating from India, was kindly provided by the company "Yumis." Extraction was performed using an ultrasound-assisted method with absolute methanol or 96% (v/v) ethanol, at a solvomodule of 1:10 (m/v). Total phenols and tannins were measured using the Folin-Ciocalteu reagent, while antiradical activity was assessed using the DPPH (1,1-diphenyl-2-picrylhydrazyl) assay.
The results showed that the ethanol extract contained significantly higher levels of total phenols and tannins (141.66±3.66 mg GAE per g of the dry extract and 102.49±3.30 mg per g of the dry extract, respectively) compared to the methanol extract. The ethanol extract also demonstrated superior radical scavenging ability ($IC_{50}$=90.09±1.87 µg/ml), in line with its higher phenolic content.
These findings support previous research on turmeric, highlighting its strong antioxidant properties. These effects position turmeric as a promising natural source for health-supporting formulations used in both preventive and therapeutic applications.

Keywords: turmeric rhizome, extracts, phenols, tannins, antiradical effects

Original Scientific Paper

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# DETERMINATION OF ANTIOXIDANT ACTIVITY OF COMMERCIAL AND ISOLATED ESSENTIAL OIL FROM FRANKINCENSE RESIN

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The aim of this work was to analyze the antioxidant potential of commercial and isolated essential oil from the frankincense resin of the species of *Boswellia serrata*. Essential oils, as sources of natural products, represent an alternative to synthetic antioxidants in the pharmaceutical industry. Numerous clinical trials have shown a beneficial effect in the treatment of inflammatory processes such as asthma, rheumatoid arthritis, Crohn's disease, and ulcerative colitis. The essential oil from frankincense resin was isolated by distillation using Clevenger (hydromodule 1:10, m/v, time of distillation 4 hours, volume of isolated oil 1 $cm^3$). Using the ABTS and FRAP tests, the antioxidant activity of the commercial and isolated essential oil of frankincense resin was determined, while both samples showed a relatively low antioxidant potential. The results of the ABTS test indicate a better antioxidant activity of the isolated essential oil ($EC_{50}$ = 131.35 ± 1.10 $mg/cm^3$) in comparison with the commercial sample ($EC_{50}$ = 182.90 ± 4.88 $mg/cm^3$), which confirms lower $EC_{50}$ values during the incubation time of 60 minutes. By increasing the concentration of the essential oil of frankincense resin, the degree of neutralization of ABTS radicals increases, while a longer incubation time leads to a greater degree of neutralization. The isolated essential oil after 60 minutes of incubation shows better antioxidant activity compared to the commercial oil. The results of the FRAP test showed a slightly better reducing ability of the commercial oil, although both oils exhibited a weak ability to reduce $Fe^{3+}$ ions to $Fe^{2+}$, which confirms their limited antioxidant potential.

Keywords: *Boswellia serrata*, essential oil, antioxidant activity, ABTS test, FRAP test

## Section: POLYMER ENGINEERING

Original Scientific Paper

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# SYNTHESIS OF GRAPHENE OXIDE – CHITOSAN HYDROGEL COMPOSITE AND ITS APPLICATION FOR THE ADSORPTIVE REMOVAL OF CONGO RED DYE

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The practical use of graphene oxide (GO) for dye removal faces challenges such as aggregation in aqueous solutions, difficulty in post-adsorption separation, and poor mechanical stability. To overcome these limitations, this study integrates GO with chitosan (CS), a natural biopolymer known for its high dye affinity and adsorption capacity. This combination enhances mechanical stability while maintaining superior adsorption performance. GO/CS adsorbent hydrogel beads were synthesized by dispersing 25 mg of GO in 20 mL of 2% acetic acid for 2h in an ultrasonic bath. The suspension was then supplemented with 0.5 g of chitosan and shaken for 24h. The beads were formed by dropping the mixture into 1 M NaOH using a syringe and were left overnight for neutralization. The resulting beads were then immersed in 0.05% glutaraldehyde for 2h to enhance crosslinking. The form of beads was chosen to facilitate future column-based studies for potential industrial applications. Batch adsorption experiments with Congo Red dye were conducted to optimize sorption conditions. GO/CS beads demonstrated effective adsorption across a broad pH range (4–12), with peak capacity observed at pH 6. Their adsorption capacity reached 644.1 mg/g, which is significantly higher than pure CS beads (445.8 mg/g), reported in earlier studies.

Keywords: Graphene oxide, hydrogel, composite, adsorption, Congo red

Original Scientific Paper

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# SYNTHESIS AND CHARACTERIZATION OF GRAPHENE OXIDE-BASED POLY(*N,N*-DIETHYLACRYLAMIDE) HYDROGEL

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In this study, the synthesis procedure and structural characterization of hydrogel composites based on graphene oxide (GO), incorporated into a cross-linked poly(*N,N*-diethylacrylamide) (PDEA) hydrogel network, were presented. The composite hydrogels were synthesized via a free radical polymerization process, thermally initiated using azobis(2-methylpropionamidine) dihydrochloride as the initiator, and 1,2-ethanediol dimethacrylate as the cross-linking agent. Fourier-transform infrared spectroscopy (FTIR) was employed to investigate the chemical structure of the synthesized materials. FTIR spectral analysis confirmed successful polymerization, as evidenced by the disappearance of the characteristic double bonds from DEA and the cross-linker, and the emergence of new bands corresponding to the formation of a cross-linked polymer network. The presence of GO in the final composite was verified by the retention of its characteristic vibrational bands. Notably, a redshift was observed in the bands corresponding to aromatic C–C stretching and C–O–C vibrations, suggesting the existence of π–π interactions between GO sheets and the polymer backbone. The swelling behavior of the synthesized homopolymer hydrogels was evaluated as a function of pH. The synthesized DEA-GO composite hydrogels were utilized to investigate their swelling behavior and responsiveness to pH variations (3.0, 6.0, and 9.0). The highest equilibrium swelling degree was observed in an alkaline medium at pH 9.0 ($\alpha_e$=14.98). In a slightly acidic solution at pH 6.0, the hydrogel reached an equilibrium swelling degree of $\alpha_e$=8.46. The lowest swelling capacity was recorded in a strongly acidic environment at pH 3.0, where the equilibrium swelling degree was $\alpha_e$=6.13. These composites exhibit promising properties for future research and potential applications in various fields.

Keywords: hydrogel, composite, graphene oxide, *N,N*-diethylacrylamide, swelling degree

Original Scientific Paper

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# BIODEGRADABLE POLYURETHANE-BASED SENSORS WITH POLYANILINE FOR REAL-TIME FOOD SPOILAGE DETECTION

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Biodegradable polyurethane (PU) composites with integrated sensing and self-healing potential were developed for intelligent food packaging applications. Polyaniline (PANI) was synthesized and uniformly dispersed within the PU matrix as a sensory component. PANI exhibits pronounced changes in color and electrical conductivity upon exposure to gases released during food spoilage. Specifically, upon contact with sulfide compounds such as $H_2S$, the composite displays an immediate and visible color shift from green to blue, allowing real-time monitoring of food freshness. The materials were characterized using FTIR spectroscopy, DSC, and DMA, confirming structural definition, thermal stability, and viscoelastic properties. Electrical conductivity measurements showed that a minimum of approximately 20 wt% PANI is required to achieve optimal sensing performance. Initial sensitivity tests to $NO_2$ and other spoilage-related gases indicate the practical relevance of these composites. Further modification of the PU matrix is underway to enhance self-healing behavior, aiming to improve packaging integrity and extend its usable lifetime. These results demonstrate the potential of PU/PANI-based biocomposites as smart, eco-friendly materials for active food quality monitoring.

Keywords: biodegradable packaging, food safety, sensors, polyurethane, polyaniline

Original Scientific Paper

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# TESTING THE INFLUENCE OF MONOMER CONTENT, CROSSLINKER CONCENTRATION, AND TEMPERATURE ON THE SWELLING OF THERMO-SENSITIVE HYDROGELS

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Thermo-sensitive hydrogels have been extensively studied in recent years for their applications in biology, medicine, controlled and targeted drug delivery, environmental protection, and many other fields. The aim of this study was to investigate the influence of monomer content, crosslinker concentration, and temperature on the swelling behavior of poly(*N*-isopropylmethacrylamide-*co*-*N*-isopropylacrylamide), p(NiPMAm-*co*-NiPAm), hydrogels in different media. The p(NiPMAm-*co*-NiPAm) hydrogels were synthesized via radical polymerization of N-isopropylmethacrylamide (NiPMAm) and *N*-isopropylacrylamide (NiPAm) monomers at different molar ratios (60/40 and 70/30), with 1.5, 2, and 3 mol% of the crosslinker ethylene glycol dimethacrylate relative to the total molar amount of monomers. The swelling degree of the synthesized hydrogels was examined in solutions with pH values of 4, 7.4, and 8 at temperatures of 25 and 37 ºC. The results showed that the swelling degree of the hydrogels depends on monomer content, crosslinker concentration, and environmental temperature. As the NiPMAm content, crosslinker concentration, and temperature increased, the equilibrium swelling ratio decreased, with crosslinker content and temperature having a more pronounced effect on hydrogel swelling. The highest swelling degree ($\alpha_e$=34.52) was achieved with p(NiPMAm-*co*-NiPAm) 60/40/1.5 hydrogel in a pH 4 solution at 25 ºC, while the lowest swelling ratio ($\alpha_e$=1.6) was recorded for the p(NiPMAm-*co*-NiPAm) 70/30/3 hydrogel in a pH 4 solution at 37 ºC. Changes in the pH value of the medium did not significantly affect the swelling behavior of the synthesized hydrogels. The synthesized hydrogels exhibited a volume phase transition at 37 ºC, indicating their potential application as carriers in so-called "smart" drug delivery systems.

Keywords: Poly(N-isopropylmethacrylamide-co-N-isopropylacrylamide), Thermo-sensitive hydrogels, crosslinker concentration, degree of swelling.

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# MODELING THE SWELLING PROCESS OF POLY(*N*-ISOPROPYLMETHACRYLAMIDE-*CO*-*N*-ISOPROPYLACRYLAMIDE) HYDROGELS USING EXPERIMENTAL DESIGN

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This work aimed to optimize the swelling process of poly(*N*-isopropylmethacrylamide-*co*-*N*-isopropylacrylamide), p(NiPMAm-*co*-NiPAm), hydrogels synthesized by radical polymerization using 2 mol% of the crosslinker ethylene glycol dimethacrylate. To achieve the highest equilibrium swelling degree, the effects of process factors – temperature (25, 30, 35, 40, and 45 ºC) and hydrogel copolymer composition (10, 30, 50, 70, and 90 mol% NiPMAm) – in solutions with pH values of 4, 7.4, and 8 were investigated and optimized using a Central Composite Design. The adequacy of the model, as well as the statistical significance of the independent variables, was evaluated using analysis of variance. A quadratic model best describes the equilibrium swelling behavior of the p(NiPMAm-*co*-NiPAm) hydrogels as a function of copolymer composition and environmental temperature. Both temperature and NiPMAm content had a negative effect on the equilibrium swelling ratio of the hydrogels in all tested solutions. However, the greater absolute value of the regression coefficient indicated that temperature had a more significant effect on the system response compared to the NiPMAm content. Within the investigated range of process variables, the highest equilibrium swelling ratio ($\alpha_e$=31.20) was achieved in the solution with a pH value of 8, while the second-order model describing the equilibrium swelling process of the hydrogels, in terms of actual factor values, is given by the following equation: $\alpha_e = 8.22 - 6.93 \cdot t - 1.13 \cdot \text{quantity}_{\text{NiPMAm}} + 2.11 \cdot t^2$.

Keywords: Poly(N-isopropylmethacrylamide-co-N-isopropylacrylamide), Hydrogels, Central Composite Design, equilibrium degree of swelling.

Original Scientific Paper
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# DESIGN AND CHARACTERIZATION OF POLY(N-ISOPROPYLACRYLAMIDE)-BASED COPOLYMERS WITH TUNABLE LCST FOR SMART WOUND DRESSING APPLICATIONS

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Thermoresponsive polymers are being increasingly investigated for biomedical use, particularly in smart wound dressings that require temperature-sensitive film detachment or drug release. In this work, poly(N-isopropylacrylamide) (pNIPAm)-based copolymers were synthesized with selected comonomers—styrene (St), acrylamide (AAm), acrylic acid (AA), and methyl methacrylate (MMA)—to precisely tailor the lower critical solution temperature (LCST) of the polymer films for wound care applications.
Pure pNIPAm exhibited the LCST near 32 °C. Copolymer compositions were varied with co-monomer contents from 5 to 20 wt%, and the LCST response was strongly dependent on both monomer type and concentration. Styrene incorporation significantly lowered the LCST , reaching ~21 °C at 20 wt%, with a clear decreasing trend as the content increased. Acrylamide showed the opposite effect, with the LCST values rising to ~39 °C at higher concentrations. Acrylic acid induced a modest increase, reaching ~34 °C only at 20 wt%, while methyl methacrylate exhibited minimal influence, maintaining LCST near ~33 °C across all the tested compositions.
FTIR spectroscopy confirmed the copolymer composition and structure, while DSC analysis provided precise determination of the LCST. The synthesized films offer a customizable thermal response, enabling temperature-controlled behavior near the physiological range, which is critical for smart multilayer wound dressings being developed within the SPS project *DRESWOUTRE*.
These findings demonstrate that controlled copolymerization of pNIPAm is an effective strategy for producing responsive biomaterials tailored to specific biomedical applications.

Keywords: poly(N-isopropylacrylamide), microwave-assisted synthesis, LCST, copolymerization

Original Scientific Paper

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# *IN VITRO* TESTING OF HYDROGELS FORMULATED USING BIOSYNTHESIZED SILVER NANOPARTICLES

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Hydrogels are porous three-dimensional structures that absorb a large amount of water, composed of polymer chains. Attention is focused on the treatment of local wounds due to low toxicity, wound hydration maintenance, and prolonged drug release. Hydrogels were prepared using carbomer 940 (polyacrylic acid) and incorporating AgNPs that were previously synthesized at room and boiling temperatures and stabilized using aqueous extracts of blackberry leaves obtained by maceration and reflux extraction. The AgNPs concentration in the hydrogel preparation was 0.1 mg/ml. After preparation, the formulations were stored at room temperature and at +4°C for 12 months. The organoleptic properties and anti-inflammatory activity were tested for the formulations prepared. The examined gels were all odorless, the blank was transparent, and the other gels were green or red-brown, depending on the conditions of the AgNPs synthesis that were incorporated. *In vitro* examination was carried out using the protein denaturation inhibition assay by the hydrogels at a concentration of 0.1 mg/ml. The results showed that the hydrogel formed with the AgNPs synthesized at room temperature using the extract obtained by reflux extraction (BR RT) had a better effect compared to the hydrogel formed with the AgNPs synthesized at boiling temparature using the extract obtained by maceration at room temperature (BM BT), with 84.49±0.00748% and 82.12±0.00986%, respectively. The blank hydrogel showed activity of 78.87±0.00832%. These results showed that storage conditions, synthesis, and extraction methods have an influence on hydrogels' activity.

Keywords: silver nanoparticles; green synthesis; bioactivity; carbomer 940; hydrogels

Original Scientific Paper
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# TEXTURAL PROPERTIES OF CARBOPOL® GEL WITH CURCUMIN AND INCLUSION COMPLEX CURCUMIN:2-HYDROXYPROPYL-β-CYCLODEXTRIN

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Texture Profile Analysis (TPA) is a commonly used technique for evaluating the mechanical characterization of pharmaceutical and cosmetic formulations. The textural properties of a formulation are an important parameter in the optimization of products intended for topical application on the skin. The aim of this work was to investigate the textural properties of Carbopol® gel formulations containing curcumin and the inclusion complex curcumin:2-hydroxypropyl-β-cyclodextrin. For the purpose of textural analysis, three gel samples were prepared: Gel 1-Carbopol® gel prepared according to the instructions given in *Master Formulas* 2008; Gel 2-Carbopol® gel containing dispersed curcumin inclusion complex; and Gel 3-Carbopol® gel containing dispersed curcumin. The textural properties of the samples were assessed using a CT3 Texture Analyzer and a Texture Analyzer-Spread Test Fixture (TA-STF) cone probe, applying the TPA test. During the TPA test, two compression cycles were performed on the samples, and five texture parameters were measured. The obtained values of the texture profile of the tested samples were as follows: hardness 350-500 mN; cohesiveness: 0.61-0.9; adhesiveness: 0.58-0.98 mJ; resilience: 0.12-0.18; elasticity: 3.25-3.95 mm. Based on the results of this study, it can be concluded that the incorporation of curcumin and its inclusion complex into Carbopol® gel did not result in statistically significant changes to the textural characteristics of Carbopol® gel. Furthermore, the absence of statistically significant changes in the textural parameters over the study period may potentially imply the physical stability of formulations. Considering that curcumin exhibits a wide range of pharmacological activities, such formulations may be suitable for topical application.

Keywords: Inclusion complex, Curcumin, 2-Hydroxypropyl-β-cyclodextrin, Carbopol® gel, Texture Profile Analysis.

Original Scientific Paper

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# THERMOANALYTICAL STUDIES ON COMPLEXES OF CLOTRIMAZOLE WITH POLYURETHANE NETWORKS BASED ON CYCLODEXTRINS AND POLYETHYLENE GLYCOLS

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Inclusion complexes between drugs and polymers are a topic of current interest to pharmaceutical research and industry as they may improve the solubility, stability, and bioavailability of the guest molecules. Polyurethanes derived from cyclodextrins show several biomedical applications. In this paper, three cross-linked polyurethane networks based on hydroxypropyl-$\beta$-cyclodextrin (HP$\beta$CD) and polyethylene glycols (PEG2000 or PEG6000) or poly(ethylene glycol)-block-poly(propylene glycol)-block-poly(ethylene glycol) (BLOCK 1100) were synthesized by the usual two-step polymerization method. Both components were linked by reacting polyethylene glycol chains, previously end capped with isocyanate groups and HP$\beta$CD, forming urethane links.

Physical mixtures and inclusion complexes containing clotrimazole were prepared by physical mixing and kneading methods in 1:5 drug:cyclodextrin weight ratios. Drug interactions were studied by thermoanalytical methods such as thermogravimetric analysis (TGA), X-ray diffractometry (XRD), and differential scanning calorimetry (DSC).

The XRD patterns of physical mixtures of the samples with clotrimazole showed the specific peaks at 9.3º, 10.0º, 12.5º, 18.7º, 19.5º, 20.7º, and 23.1º, which correspond to a pure drug. TGA results indicated a slightly increased thermal stability of the drug loaded into inclusion complexes cross-linked with polyurethane networks. The characteristic endothermic peak of tclotrimazole at around 145.90 $^{o}$C did not appear in the DSC curve of the drug-loaded inclusion complexes. The endothermic peak of clotrimazole decreased in the physical mixtures, but the characteristic peak of clotrimazole could be recognized.

Our findings show that these novel cross-linked polyurethane networks based on HP$\beta$CD can be utilized for the *in vitro* slow release of clotrimazole, with potential for *in vivo* application.

Keywords: Cyclodextrin, Inclusion complex, Polyurethane, Thermal analysis

## Section: MATERIALS ENGINEERING

Original Scientific Paper

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# THE INFLUENCE OF SODIUM CHLORIDE AQUEOUS SOLUTION ON AUSTENITE STEEL CORROSION

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Corrosion is an undesirable damage to a material caused by the physicochemical influence of the external environment. Generally, austenitic steels have exceptional physicochemical properties and the highest corrosion resistance. However, their tendency to form corrosion products can reduce their functionality and be a significant problem in various industries (e.g., electronics or medicine).

The objective of this study was to examine the behaviour of austenitic steel SS316 after standing in an aqueous solution of sodium chloride for a defined period. Optical microscopy was used to inspect the changes in the austenitic steel's surfaces. Scanning electron microscopy (SEM) showed a more precise surface topology of steel samples before and after induced corrosion. The microsurface before the experiments was pure without corrosion, due to the formation of a thin passive film on the surface. After exposure to the sodium chloride solution, it was observed that a precipitate had formed on the surface after adsorption of sodium and chloride ions. A semiquantitative method of Energy-dispersive X-ray spectroscopy (EDS) coupled with SEM was used to discover modifications in the ingredients distribution of austenitic steels exposed to the action of sodium chloride aqueous solution. The corrosion changes in the austenitic stainless steel samples were assessed as pitting. EDS spectra showed different elemental contents between the clean surface positions, near the corrosion deposits, in the deep corrosion pit, and the wide pit zone. These findings are important for the safe usage of steel, especially in medicine, to reduce the infection risk from rust and other imperfections triggered by oxidation.

Keywords: corrosion, pitting, SEM analysis, EDS analysis, austenitic steels

Original Scientific Paper
This research was supported by the Ministry of Science, Technological Development and Innovation of the Republic of Serbia, grant numbers 451-03-136/2025-03/200124 and 451-03-137/2025-03/200124.

# COMPOSITES OF GRAPHITE OXIDE AND METAL OXIDES FOR PHOTOCATALYTIC USE

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Semiconductor photocatalysts are a promising solution for pollution mitigation because photocatalysis provides a simple and low-cost technology. However, the use of semiconductors is limited by the fast recombination of electron-hole pairs. Photocatalytic efficiency can be improved by combining semiconductors with carbonaceous materials. The combination of graphene oxide and semiconductors yields photocatalysts with remarkable characteristics. In this study, composites of three metal oxides with graphite oxide as photocatalysts for the degradation of methylene blue (MB) and Reactive blue 19 (RB19) were tested. Graphite oxide was prepared by the modified Hummers method, while metal oxides ($ZrO_2$, $TiO_2$, and $ZnO_2$) were prepared by the sol-gel method. The weight ratio of metal oxides to graphite oxide was 20%. Photocatalytic tests were conducted in batch mode. The influence of time and composite mass was determined.
The composite $ZrO_2$@GO showed the best results for degradation of MB, and the $TiO_2$@GO composite for the RB19 degradation. Generally, better degradation rates were obtained for RB19 dye. The degradation rate increased with the amount of composite in the mixture. Total degradation of MB was obtained for maximum amounts of all three composites. Total degradation of RB19 was obtained only with the $ZrO_2$@GO composite. These are promising results leading to efficient, low-cost, environmentally friendly composites capable of acting as photocatalysts.

Keywords: composites, graphite oxide, metal oxides, photodegradation, organic dyes

# A NEW CLASS OF HIGH-ENTROPY PYROCHLORE OXIDES: COMPARATIVE INSIGHT INTO STRUCTURAL RESPONSE WITH AND WITHOUT PRESSURE

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High-entropy oxides (HEOs) present a promising platform for engineering novel functionalities through configurational complexity. This study investigates the structural behavior of a new class of high-entropy pyrochlore oxides with the nominal composition $(La_{1/7}Sm_{1/7}Nd_{1/7}Pr_{1/7}Y_{1/7}Gd_{1/7}Yb_{1/7})_2(Sn_{1/3}Hf_{1/3}Zr_{1/3})_2O_7$, synthesized using three different methods: solid-state displacement reaction (SSDR), glycine-nitrate combustion (GNP), and conventional oxide mixing. Initial X-ray diffraction (XRD) results showed amorphous or poorly crystalline phases, which evolved into crystalline pyrochlore upon calcination at 750–900 °C. A mixture of defective fluorite and pyrochlore phases was observed during heating, with phase-pure pyrochlore obtained after pressureless sintering at 1650 °C without additives.

To probe the mechanical resilience of the structure, in situ high-pressure synchrotron XRD was performed up to 25.8 GPa. The results demonstrated excellent structural stability under compression, with no phase transformation. These experimental observations were further validated by theoretical modeling. Rietveld refinement, Raman spectroscopy, and computational analysis confirmed the homogeneity and stability of the final product.

This work demonstrates that both the synthesis route and external pressure significantly influence phase formation and crystallization in high-entropy pyrochlore oxides. The findings underline the structural robustness and compositional flexibility of these materials, offering new possibilities for their application in extreme environments and functional devices.

Keywords: high-entropy oxides, pyrochlore, high pressure, phase stability, synthesis route.

Original Scientific Paper
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# THE CHARACTERIZATION OF PIPERINE AND ITS INCLUSION COMPLEX WITH β-CYCLODEXTRIN USING XRD ANALYSIS

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Piperine, the principal alkaloid of black pepper (*Piper nigrum* L.), limited by poor aqueous solubility and bioavailability, was complexed with β-cyclodextrin to enhance its solubility and stability. The inclusion complex was prepared by the kneading method (1:1 molar ratio, ethanol–water 1:1 v/v) and dried to constant weight at room temperature.
XRD spectra of piperine, its inclusion complex with β-CD, and the corresponding physical mixture were recorded using a Rigaku MiniFlex 600 diffractometer (Rigaku, Tokyo, Japan) with monochromatic CuKα radiation, at 2θ range 5–70°, step size 0.03°, and scan time 5 s; voltage and power were set to 40 kV and 20 mA.
XRD analysis confirmed the crystalline nature of piperine, with sharp diffraction peaks observed at 2θ values (14.66°, 22.22°, and 25.73°) characteristic of polymorphic, the most thermodynamically stable and least soluble form [1,2]. In contrast, the diffractogram of the piperine:β-cyclodextrin inclusion complex exhibited new peaks at 2θ values of 5.87°, 6.65°, 11.75°, and 17.45°, which were absent in the diffractograms of the pure components and their physical mixture. Furthermore, the intensity of the peaks originating from piperine was significantly reduced, indicating the inclusion of piperine into the β-cyclodextrin cavity. The diffractogram of the physical mixture shows the presence of peaks from both piperine and β-cyclodextrin, suggesting that inclusion has not occurred in the physical mixture. These results indicate the need for further investigation of the inclusion complex to optimize its stability, solubility, and potential for pharmaceutical and cosmetic applications.

Keywords: black pepper, piperine, inclusion complex, β-cyclodextrin, XRD analysis

# CARBON CRYOGEL/MULTI-WALLED CARBON NANOTUBE COMPOSITES: PREPARATION AND PHYSICOCHEMICAL CHARACTERIZATION

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This study explores the incorporation of multi-walled carbon nanotubes (MWCNTs) into a carbon cryogel matrix with the aim of improving its structural, textural, and adsorption characteristics. Composites containing varying MWCNT loadings (1%, 3%, and 5% by weight) were synthesized via the sol-gel method, followed by freeze-drying and carbonization. The resulting materials were analyzed using nitrogen adsorption-desorption isotherms and field emission scanning electron microscopy (FE-SEM). Additionally, the point of zero charge (PZC) was determined.

All samples exhibited mesoporous characteristics. Compared to the pure carbon cryogel, a slight increase in the specific surface area value was observed for the samples with 1% and 3% of CNTs, whereas the sample with 5% of CNTs showed a reduction in this parameter. Future work will focus on evaluating the potential of these composites for water purification through batch adsorption experiments.

Keywords: Carbon cryogel, multi-walled carbon nanotubes

# CHARACTERISTICS OF CATALYSTS USED IN PYROLYSIS OF HDPE/PP WASTE MIXTURE

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Conventional plastics are predominantly non-degradable and designed for single-use applications, creating significant environmental concerns regarding their current disposal methods. However, their high energy content presents opportunities for recovery and conversion into value-added products. Pyrolysis has emerged as a promising technological alternative for transforming plastic waste into pyrolytic fuels. The process yields variable distributions of gaseous, liquid, and solid fuel products depending on several factors: (1) catalyst type, (2) process temperature, and (3) feedstock composition.

This study employed four different catalysts, including both natural and synthetic varieties**.** The natural catalysts used for pyrolysis were quartz sand ($SiO_2$) and a natural zeolite (white opalized silicate tuff). Synthetic catalysts consisted of ZSM-5 zeolite and $Al_2O_3$. Catalyst characterization was conducted using FTIR spectroscopy, while their thermal behavior was investigated through thermogravimetric analysis (TGA).

Thermogravimetric (TG) analysis revealed distinct thermal behaviors among the catalysts. The natural catalysts demonstrated exceptional thermal stability at elevated temperatures. In contrast, synthetic catalysts exhibited a moderate mass loss (10-12%), attributable to their hygroscopic nature and large specific surface area. Among the natural catalysts, quartz sand showed minimal hygroscopicity (0.5% mass loss), followed by tuff (1.5% mass loss). FTIR spectroscopic analysis indicated similar functional group profiles across all four catalysts, including characteristic OH, Si-OH, Al-OH, and silanol groups. However, variations in peak intensities were observed, reflecting compositional differences between the materials.

Keywords: catalysts, pyrolysis, FTIR, TGA

Original Scientific Paper

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# INTEGRATED SORPTION CASES OF WASTEWATER PURIFICATION BY BROWN WASTE-DERIVED BIOCHARS

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Heavy metal and pharmaceutical-induced environmental contamination remains a major worldwide problem that requires effective and long-lasting remediation techniques. Sorption has emerged as a key purification method due to its cost-effectiveness and high removal efficiency. The aim of this study was to compare the general sorption capability of biochars derived from two different lignocellulosic precursors by contrasting their methylene blue (MB) numbers along with their structural and textural properties. Biochars derived from the plant dwarf elder (*Sambucus ebulus*) and the sycamore maple seeds were synthesized via pyrolysis and chemical activation to produce mesoporous carbon-based sorbents with high surface areas and diverse functional groups. The preparation procedure involved collecting the precursors, their washing, drying, milling, impregnation with concentrated phosphoric acid, carbonization under a protective nitrogen atmosphere up to a temperature of 700 °C for 60 minutes, cooling down, crushing, rinsing, and final drying of the obtained product. The MB number, which is defined as the quantity of the dye sorbed by 1 g of the sorbent, was estimated in the MB concentration range from 100 to 350 mg/L, contacted with the tested materials at 25 °C for 24 h, and analyzed photometrically at 660 nm. Characterization by FE-SEM, BET, and FTIR confirmed the structural and textural affinity of the obtained biochars for sorption processes in the aqueous phase. MB numbers for biochars derived from dwarf elder and sycamore maple seeds were determined to be 175 and 201 mg/g, respectively, proving the promising sorptive potential of these materials for the treatment of contaminated water.

Keywords: lignocellulosic precursors, dwarf elder, sycamore maple, carbonization.

Review

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# ADVANCED TRANSPARENT INSULATION MATERIALS IN BUILDING DESIGN

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Regarding the building envelope as an essential element in balancing external conditions, especially in regulating temperature, daylight, and airflow, investigation of transparent insulation materials (TIM), which can be used for a building envelope, is of high importance. These materials are constructed with at least one transparent cover and typically occupy the air cavity between the panes of a double-pane unit. Important categories of TIMs are the glazing-perpendicular structure and the homogeneous structure. There are three types of glazing-perpendicular structures: capillary, honeycomb, and parallel slat array. When it comes to homogeneous TIMs, silica aerogel is the most widely used material due to its high porosity resulting in a very low thermal conductivity (e.g. 0.005–0.02 W/mK).

Approximately 96% of the aerogel is air, with the remainder being an open-pored silica structure, most often silicon dioxide ($SiO_2$, amorphous quartz). The pore size and surface area of aerogels can be tailored, and different functional groups can be implemented to provide desired mechanical and physico-chemical properties. Furthermore, improved flexibility along with the pre-shaped forms can be realized through the incorporation of aerogels into textiles and other matrices. In addition, aerogels that are designed for effective transmission of solar energy are produced using supercritical drying and have a density of between 100–150 kg/m$^3$. For these applications, there are two types of silica-based aerogels: monolithic silica aerogel and granular silica aerogel.

The performed and additional investigation of TIMs could contribute to the primary goal of increasing the thermal resistance of the building envelope, while maintaining satisfactory levels of indoor comfort. This is important due to the evolution of building standards and higher demands for occupants' comfort, resulting in a drastic improvement of building envelope performances.

Keywords: Aerogel, building envelope, energy efficiency, transparent insulation materials.

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# USE OF CONTAMINANT-LADEN BENTONITE-BASED GEOPOLYMERS IN CONCRETE PRODUCTION

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The growing global demand for sustainable construction materials and effective wastewater treatment requires innovative solutions. Geopolymers, amorphous aluminosilicate materials formed by alkali activation, offer a promising approach by valorizing industrial by-products and natural minerals while exhibiting excellent mechanical and chemical properties. In this paper, bentonite-based geopolymers for the adsorption and immobilization of pollutants from wastewater, especially phenol and heavy metals, and their integration in the construction industry, particularly in concrete, are investigated.

Bentonite, a montmorillonite-rich clay mineral, has a high cation exchange capacity and a porous structure that makes it an effective adsorbent for various pollutants. When incorporated into geopolymer matrices, it improves the adsorption capacity for both heavy metal ions (e.g., $Pb^{2+}$, $Cu^{2+}$, $Cd^{2+}$, $Hg^{2+}$) and organic pollutants such as phenol. Studies show that geopolymers modified with bentonite remove pollutants with an efficiency of over 90%.

The use of these geopolymers containing pollutants in concrete offers a double benefit: safe disposal of hazardous waste and production of more sustainable building materials. Heavy metals and organic pollutants are immobilized, preventing leaching. Moreover, the addition of treated geopolymers can improve the mechanical properties of concrete (e.g., compressive strength, durability) depending on the type of waste, concentration, and formulation. This approach takes into account environmental concerns and promotes a circular economy by converting pollutants into valuable resources for construction. Future research should optimize the synthesis parameters to improve pollutant uptake and ensure the long-term performance of concrete.

Keywords: bentonite-based geopolymers, concrete production, wastewater treatment

## Section: ENVIRONMENTAL ENGINEERING

Original Scientific Paper

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# CHITOSAN FLOCCULATION OF *BACILLUS* BIOMASS FROM BIOLOGICALLY TREATED DAIRY WASTEWATER: A TAGUCHI OPTIMIZATION APPROACH

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This study investigates the optimization of *Bacillus* biomass recovery from biologically treated dairy industry wastewater using chitosan flocculation, employing Taguchi's experimental design methodology. The primary goal was to determine the optimal process parameters for maximizing biomass recovery efficiency, with a focus on sustainable applications in agriculture. A series of experiments was conducted by systematically varying key factors, including chitosan concentration and mixing rate, to assess their impact on flocculation efficiency. The results revealed that chitosan concentration played a dominant role in determining the biomass recovery, with the optimal recovery achieved at a concentration of 140 mg/L, leading to a 90% biomass recovery. In contrast, the mixing rate showed an inverse relationship, where the lowest mixing speed of 200 rpm produced the highest flocculation efficiency. The study highlights the importance of fine-tuning both the chitosan concentration and mixing speed to maximize biomass recovery in wastewater treatment processes. These findings contribute to the development of more effective and environmentally sustainable methods for wastewater management in the dairy industry, with potential applications in agricultural production. By optimizing the flocculation process, this research paves the way for efficient recycling of valuable microbial biomass, supporting circular economy principles and enhancing the overall sustainability of the dairy production sector.

Keywords: dairy wastewater, flocculation, chitosan, Taguchi optimization

Original Scientific Paper
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# INSIGHTS INTO ELECTROKINETIC TREATMENT OF FINE-GRAINED SOILS

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Extreme rainfalls today represent an increasing threat to the engineering characteristics of fine-grained soils, as they contribute to the deterioration of their physical and mechanical properties, making such soils unsuitable for use in construction. One of the most promising opportunities for preventing such phenomena is an electrokinetic treatment as an innovative and increasingly studied technique. It offers the potential to enhance the soil strength and its overall engineering performance, particularly when integrated with other soil improvement methods. This approach involves the application of a low-intensity direct current (DC) across electrodes inserted into the soil, promoting the movement of pore water through electrokinetic phenomena such as electroosmosis, electromigration, electrophoresis, and electrolysis. As a result of these combined processes, the treatment can significantly improve the physical, chemical, and mechanical properties of the soil.
In contrast to its advantages, there is a major technical challenge associated with electrokinetic soil treatment, such as the corrosion of electrodes. When DC is applied, electrochemical reactions occur at the electrode–soil interface, including oxidation at the anode and reduction at the cathode. These reactions can lead to the dissolution of electrode materials, particularly in the presence of aggressive soil environments. The electrode corrosion not only reduces the service life and efficiency of the electrodes, but also alters the electrochemical conditions within the soil, potentially impacting the uniformity and effectiveness of the treatment. The degradation of electrodes may cause fluctuations in the current density and ultimately diminish the long-term performance of the treated soil.
Given these concerns, this study focuses on evaluating the extent and consequences of electrode corrosion during the electrokinetic soil treatment. It aims to elucidate how variations in operational parameters influence the corrosion behavior and affect the overall efficiency and sustainability of the process.

Keywords: electrokinetic treatment, fine-grained soils, corrosion of electrodes.

Original Scientific Paper

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# KOH-CATALYZED METHANOLYSIS OF WASTE POLYETHYLENE TEREPHTHALATE IN THE PRESENCE OF A COSOLVENT

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Polyethylene terephthalate (PET) is widely used in packaging and textiles due to its excellent chemical and physical properties, such as low gas diffusivity, moisture impermeability, high strength, and chemical resistance. However, the rapid increase in PET consumption has raised environmental concerns regarding PET waste accumulation. Among recycling approaches, chemical recycling via methanolysis offers an effective route for PET depolymerization, enabling closed-loop recycling. This study investigates KOH-catalyzed methanolysis of waste PET strips in the presence of tetrahydrofuran (THF) as a cosolvent to evaluate the effect of catalyst loading on depolymerization efficiency. Experiments were conducted in a stirred batch reactor at 70 °C with a methanol:PET molar ratio of 50:1, a THF:PET molar ratio of 70:1, and KOH:PET molar ratios ranging from 0.5:1 to 1:1. Increasing the KOH:PET molar ratio enhanced PET conversion, achieving 89% conversion at a 1:1 molar ratio within 30 minutes. This improvement is attributed to the higher availability of hydroxide ions at higher KOH loadings, facilitating nucleophilic attack on PET ester bonds and accelerating depolymerization. Additionally, the use of THF as a cosolvent aids in swelling the PET matrix, improving contact with the reagent, and reducing mass transfer limitations. These results demonstrate that KOH-catalyzed methanolysis in the presence of a cosolvent is an efficient method for PET chemical recycling under mild conditions, contributing to sustainable PET waste management.

Keywords: Depolymerization; methanolysis; polyethylene terephthalate, potassium hydroxide, tetrahydrofuran.

Original Scientific Paper

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# THE SORPTION OF METHYL ORANGE DYE BY MgCoAl-LDH: EFFECTS OF CONTACT TIME, pH, AND SORBENT DOSE

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This study investigates the efficiency of layered double hydroxide (MgCoAl-LDH) for the removal of Methyl Orange (MO), a widely used azo dye in the textile industry. MgCoAl-LDH was synthesized by an ultrasound-assisted coprecipitation method at 80 °C, optimizing its structural properties for enhanced sorption performance. The research focused on the investigation of key parameters that affect dye sorption: solution pH (2 to 9), sorbent dose (0.1 to 1 g $dm^{-3}$), and contact time (0 to 180 min), at a temperature of 20 °C. The MO concentration in the samples was determined by a spectrophotometer, Shimadzu UV-vis 1650 PC (Shimadzu, Japan).

The results indicate a strong correlation between pH and sorption capacity. With lower solution pH values, a significant increase in sorption efficiency was achieved. At low pH values, the sorbent surface was protonated and positively charged, so MO molecules could be bound by electrostatic attractions. Since the sorption capacity of the sorbent was the highest at pH 2, all experiments were performed at that initial pH value. The results indicated that the sorption capacity increased with increasing contact time. The optimal sorbent dose was 0.25 g $dm^{-3}$ for sorption of MO with an initial concentration of 100 mg $dm^{-3}$. The equilibrium sorption capacity ($q_e$) of MgCoAl-LDH reached an impressive 210.4 mg $g^{-1}$, demonstrating its potential as an effective sorbent for wastewater treatment. The findings provide valuable insight into the development of advanced materials for dye removal and underscore the importance of controlling pH and sorbent dose in optimizing sorption processes.

Keywords: Layered double hydroxides; Azo dye; Sorption; Sorption capacity

Original Scientific Paper

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# REMOVAL OF METHYLENE BLUE FROM AQUEOUS ENVIRONMENT USING BIOCHAR DERIVED FROM WASTE ROSEHIP SEEDS

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Biochars, obtained by thermal decomposition of carbonaceous biomass, have emerged as promising materials for the removal of pollutants from wastewater. These materials have been found to be effective as adsorbents in removing synthetic dyes such as methylene blue (MB), whose widespread use raises environmental concerns due to its negative effect on the aquatic ecosystem. Therefore, the development of adsorbents derived from waste biomass can contribute to the sustainability of adsorption-based processes. The aim of this study was to evaluate the adsorption performance of biochar derived from waste rosehip seeds produced by slow and incomplete combustion. The batch adsorption experiments were performed at ambient temperature and under atmospheric pressure, with a constant stirring rate of 400 rpm. The initial MB concentration was varied from 1 mg/L to 5 mg/L, while the adsorbent concentration was 1 g/L. The effect of contact time on the removal efficiency of MB was also investigated. The results indicated a significant effect of contact timeon the removal efficiency. As contact time increased, the removal efficiency increased for the initial MB concentration from 1 mg/L to 3 mg/L, while the time required to reach adsorption equilibrium decreased with increasing initial MB concentration. However, at the higher initial MB concentration (5 mg/L), the removal efficiency was lower, and extending the contact time did not enhance removal efficiency. These findings demonstrate the adsorptive potential of biochar derived from waste rosehip seeds, suggesting its application in wastewater treatment. Nevertheless, further optimization of adsorption conditions is necessary to maximize its adsorption capacity.

Keywords: adsorption, biochar, methylene blue, rosehip seeds

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Original Scientific Paper

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# THE ROLE OF ELECTRODEPOSITION CURRENT DENSITY IN THE SYNTHESIS OF SORBENTS BASED ON THE BISMUTH COMPOUNDS

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Textile dye pollution is one of the major environmental problems due to the widespread use of synthetic dyes in industry. A significant amount of the total world dye production is lost in effluent during the dyeing process. Therefore, it is necessary to provide suitable technology for the wastewater treatment. Among the various wastewater treatment methods, the sorption process is one of the effective techniques successfully used to remove textile dyes from water. In this study, the anthraquinone dye Reactive Blue 19 (RB19) was selected as a representative pollutant. The aim of this research was to study the effect of electrodeposition current density in the synthesis of bismuth compounds on the sorption performance of the obtained materials. The materials were synthesized by galvanostatic electrodeposition on a titanium cathode from a solution of bismuth nitrate in nitric acid and further thermal treatment. The effect of electrodeposition current density during material synthesis was determined by varying the current density in the range from 30.0 to 200.0 mA $cm^{-2}$. The electrodeposition time was proportionally reduced (from 25.0 to 3.75 min) with increasing current density, in order to obtain the same amount of deposit in all cases, according to Faraday's law. The results showed that the sorption activity of the material increased with increasing electrodeposition current density. The maximum sorption capacity for the material obtained at 200.0 mA $cm^{-2}$ was very high (1047 mg $g^{-1}$), which means that it could potentially be practically applied in the treatment of wastewater from the textile industry.

Keywords: Sorption; Bismuth compounds; Reactive Blue 19; Sorption capacity.

Original Scientific Paper

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# CONGO RED DYE REMOVAL USING MAGNETIC BIOMASS OF *Ambrosia artemisiifolia*

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Congo Red (CR) is a synthetic anionic dye widely used in the textile and paper industries, as well as a pH indicator. Due to its toxicity, potential carcinogenicity, and resistance to degradation, it represents a serious environmental pollutant when released into aquatic ecosystems. Efficient removal of such dyes from wastewater is crucial to minimizing ecological and health risks. Biosorption offers a simple, cost-effective, and environmentally sustainable method for treating dye-contaminated water. This study investigates the use of magnetically modified biomass derived from the invasive plant *Ambrosia artemisiifolia* as a green and economical material for CR removal from aqueous solutions. Experiments were performed at unadjusted pH, evaluating the effects of sorbent dosage (1 – 5 g/L), ionic strength (0 – 1 M), contact time, and initial dye concentration (5 – 250 ppm). CR removal was monitored using UV-Vis spectroscopy, while FTIR analysis confirmed dye binding to the magnetic biosorbent. The results showed that the biosorption efficiency was 68.4 % after 40 minutes, while the maximum biosorption capacity was 33 mg/g. It was observed that the process followed the pseudo-second-order kinetic model, indicating chemisorption. The Langmuir isotherm model best described this interaction, implying monolayer biosorption. These findings suggest that magnetically modified *Ambrosia artemisiifolia* biomass offers promising potential as an efficient biosorbent for the treatment of dye-contaminated water, providing a green solution for industrial wastewater management.

Keywords: Congo Red, magnetic biosorbent, wastewater treatment, FTIR.

Review

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# PHENOLIC POLLUTION IN WASTEWATER: THE ROLE OF MAGNETITE-BASED CATALYSTS IN ADVANCED OXIDATION PROCESSES

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According to the World Health Organization, phenol is the third most serious water pollutant after petroleum products and heavy metals. Phenol's resistance to biodegradation and its harmful effects on aquatic life, soil ecosystems, and human health underscore the urgent need for effective treatment technologies that can neutralize phenol-containing wastewater and mitigate its environmental impact. Magnetite-based catalysts have emerged as promising materials for advanced oxidation processes (AOPs) for the removal of phenol from wastewater, in order to meet the urgent need for efficient, sustainable, and cost-effective treatment technologies. Due to their inherent magnetic properties, chemical stability, low toxicity, and economics, these catalysts enable easy separation and recycling after treatment, minimizing secondary pollution and operational costs. Magnetite-based catalysts can effectively activate various oxidizing agents, such as persulfates or hydrogen peroxide, facilitating the heterogeneous catalytic oxidation of phenol to harmless end-products such as carbon dioxide and water. In addition, the incorporation of magnetite in composite materials or on suitable supports (such as clays, zeolites, or aluminosilicates) improves catalytic performance by increasing surface area, active sites, and multifunctional properties.
Despite significant progress, the complete removal of phenolic compounds remains a challenge due to the complexity of industrial effluents and the economic considerations associated with catalyst regeneration and waste disposal. Therefore, the development of magnetite-based catalysts for AOPs remains an important area of research. By providing an overview of the different types of magnetite-based catalysts used in AOPs to remove phenol from wastewater, researchers are better able to select and develop more effective catalysts that are tailored to specific AOP systems and have great potential for large-scale practical applications in environmental remediation.

Keywords: water pollution, phenol-containing wastewater, advanced oxidation processes, magnetite-based catalysts

Original Scientific Paper

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# GREEN EVALUATION OF DISPERSIVE SOLID-PHASE MICROEXTRACTION USING AMINO-FUNCTIONALIZED POLYMER

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As environmental concerns continue to shape scientific research, aligning analytical techniques with Green Analytical Chemistry (GAC) principles is fundamental to the development of sustainable and eco-friendly analytical practices. The twelve GAC principles serve as key guidelines for evaluating the greenness of analytical methods. Recent trends in method development emphasize the creation of extraction techniques that require minimal solvent use, while also aiming to reduce the number of steps, energy consumption etc. In this study, the greenness of dispersive solid-phase microextraction (DSPME) utilizing amino-functionalized polymer as a sorbent was assessed using various green analytical metrics, including Analytical GREEnness (AGREE), the Green Analytical Procedure Index (GAPI), and the Analytical Eco-Scale (AES). The investigated methodology was employed for the preconcentration of the organic dye Congo red. The results demonstrated that the proposed method is consistent with the GAC principles. The overall score on AGREE pictogram, with a value of 0.72 and a light green color, indicates that the proposed procedure is environmentally sustainable. Furthermore, according to the result of 94 points on the AES, the proposed DSPME method is classified as an excellent green method.

Keywords: GAC principles, greenness, DSPME, polymer, organic dye

Original Scientific Paper
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# OXYGEN CONSUMPTION IN THE SOUTH MORAVA RIVER AS AN INDICATOR OF ENVIRONMENTAL PROTECTION

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The protection of surface waters is crucial for preserving ecological balance and ecosystem safety. The oxygen consumption indicator, according to the applicable Regulation on the National List of Environmental Protection Indicators (Official Gazette of the Republic of Serbia, No. 37/2011), includes monitoring of biochemical oxygen demand over five days (BOD-5) and ammonium ion ($NH_4$-N) parameters. The data for this analysis were obtained from the Surface Water and Watercourse Monitoring Program in the city of Leskovac, which is regularly updated and publicly available. Precise monthly measurement data for $BOD_5$ and $NH_4$-N were used at the monitoring site on the South Morava River, 100-200 meters after the confluence with the Veternica River, in 2024. Based on the exact monthly parameter measurement values, the arithmetic mean annual values for 2024 were calculated for each of the parameters, as follows: $BOD_5$ = 2.723 mg $O_2$/l and $NH_4$-N = 0.21 mg/l. The obtained values indicate that the water quality of the South Morava River at the observed location falls within the moderate ecological status category, according to the applicable criteria. Although these values do not indicate a serious level of pollution, there is still a need to implement preventive measures to preserve water quality and reduce potential further contamination. It is recommended to continue regular monitoring and to implement appropriate protective measures, particularly in terms of reducing nitrates and phosphates through the regulation of agricultural activities and the improvement of wastewater treatment systems in urban areas.

Keywords: oxygen consumption, environmental protection, ecological status.

## Section: CHEMICAL ENGINEERING

# INVESTIGATING THE KINETICS OF ESSENTIAL OIL EXTRACTION FROM *JUNIPERUS COMMUNIS* THROUGH HYDRO-DISTILLATION

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*Juniperus communis* grows in mountainous regions at higher altitudes where the climate is moderately continental. Juniper berries are rich in essential oils, terpenes, flavonoids, and organic acids, which contribute to their wide application in the pharmaceutical, cosmetic, and food industries.
In this work, the kinetics of essential oil separation from juniper berries by hydro-distillation in an Unger-type apparatus was studied. The hydromodule (the ratio of material to water) showed minimal influence on the kinetics, yield, and composition of the extracted oil. The yields for hydromodules of 1:5 and 1:3 were nearly the same (2.78% compared to 2.98%), although the smaller hydromodule (1:3) facilitated a quicker initial extraction due to a higher concentration of plant material. Kinetic analysis indicated that fast distillation occurred during the first 120 minutes, but this rate decreased significantly by 180 minutes, at which point equilibrium was achieved. After 180 minutes, the efficiency of extraction suggested that complete oil recovery had been achieved. In this work, four empirical models with one to four constants were used to treat the hydrodistillation process as hydrodistillation with two mass transfer mechanisms, the first where the hydrodistillation is kinetically controlled, and the second where the mass transfer is controlled by the diffusion of the essential oil through the pores of the plant particles. The estimation of the empirical parameters of the selected models was made by applying a method of fitting the experimental data to the model data. The models exhibited high correlation coefficients ($R^2$=0.85-0.99) and low mean relative deviation (MRD=2.13-8.72%).

Keywords: *Juniperus communis*, essential oil, hydro-distillation, kinetics, modelling

# FRACTIONATION OF ESSENTIAL OIL FROM *JUNIPERUS COMMUNIS* BY HYDRO-DISTILLATION

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This work focuses on the fractionation of essential oil extracted from *Juniperus communis* berries collected from mountainous areas of North Macedonia, using hydro-distillation enhanced with a Vigreux column. The main objective was to selectively isolate terpene groups and better understand how they distribute across distillation fractions. Gas chromatography–mass spectrometry (GC-MS) analysis revealed three major fractions with distinct compositions. The first fraction was dominated by monoterpene hydrocarbons (84.6%), particularly α-pinene (51.7%). In contrast, the third fraction showed a strong presence of sesquiterpenes (78.4%), with germacrene D dominance (10.0%). The intermediate second fraction exhibited a more mixed profile. Overall, the essential oil consisted primarily of monoterpenes (64.5–66.4%) and sesquiterpenes (32.9–34.6%), with α-pinene, sabinene, myrcene, and germacrene D identified as key constituents. Fractional hydro-distillation successfully separated these chemical groups, supporting its use as a reliable method for isolating specific terpene classes. Interestingly, variations in hydromodule ratios (1:3 vs. 1:5) had little effect on either the chemical composition of the fractions or the overall yield, which ranged narrowly from 2.78% to 2.98%. The kinetic behaviour observed during distillation revealed that most monoterpenes were extracted quickly in the early stages, the first 15 minutes (56% of extracted essential oil), and the following 120 minutes (37% of extracted essential oil). Sesquiterpenes appeared more abundantly in the third and less in the second fraction. These findings contribute valuable knowledge for the design and optimisation of essential oil processing, especially where precise terpene separation is important. The results are particularly relevant for the pharmaceuticals, perfumery, and cosmetics industries.

Keywords: *Juniperus communis*, essential oil, hydro-distillation, composition, fractionation

Original Scientific Paper

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# EVALUATION OF *PRUNUS* KERNEL OILS AS UNCONVENTIONAL FEEDSTOCKS FOR SUSTAINABLE BIODIESEL PRODUCTION

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The physicochemical properties and performance of biodiesel are closely linked to the fatty acid composition of the oils used in its production. This study evaluates the potential of oils extracted from kernels of five *Prunus* species (cherry, plum, apricot, nectarine, and peach) as feedstocks for biodiesel production, focusing on rapid assessment based on fatty acid composition and calculated fuel properties. The fatty acid composition of the oils was determined using gas chromatography with mass spectrometry (GC-MS). Biodiesel properties, including kinematic viscosity, density, higher heating value, cetane number, iodine value, cold filter plugging point, and oxidation stability, were estimated using established empirical equations[1]. Oleic acid was the predominant fatty acid in all oils, ranging from 41.3% in cherry kernel oil to 77% in plum kernel oil, followed by linoleic acid, which ranged from 17% in plum kernel oil to 48.9% in cherry kernel oil. The calculated biodiesel properties were: kinematic viscosity of 4.1–4.4 $mm^2/s$, density of 864–880 $kg/m^3$, cetane number of 49.6–57.9, oxidation stability of 5.0–9.5 h, and iodine value of 99.2–118 g $I_2$/100 g, complying with EN 14214 specifications except for the slightly lower cetane number of peach kernel oil-derived biodiesel. The higher heating value ranged from 39.3 to 39.8 MJ/kg. The cold filter plugging point, ranging from –14.1 °C to –7.6 °C, indicated biodiesel suitability for colder climates. The results demonstrate the potential of *Prunus* kernel oils as unconventional and sustainable feedstocks for biodiesel production, contributing to the valorization of agro-industrial by-products within the renewable energy sector.

Keywords: biodiesel; fatty acids; kernel oil; physicochemical properties; *Prunus*.

1. Górnaś P., Rudzińska M., Soliven A., Ind. Crops Prod. 100 (2017) 77–84.

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# FATTY ACID COMPOSITION OF FODDER RADISH SEED OILS OBTAINED BY DIFFERENT EXTRACTION TECHNIQUES

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Fodder radish (*Raphanus sativus* L. var. *oleiformis* Pers.) is gaining recognition as a valuable oilseed crop due to the high content of essential fatty acids, antioxidants, and bioactive compounds in its seeds. This study aimed to determine the fatty acid composition of crude fodder radish seed oil (FRSO) obtained using different extraction techniques: screw pressing under optimal conditions (a nozzle diameter of 8 mm, a press head temperature of 80 °C, and a screw frequency of 50 Hz), hydraulic pressing, and Soxhlet extraction. The oils were analyzed by gas chromatography with flame ionization detection (GC-FID) using an Omegawax® 250 column. Regardless of the method used, the fatty acid profile of FRSOs revealed a predominance of monounsaturated (~62%) and polyunsaturated (~27%) fatty acids (MUFAs and PUFAs, respectively), with saturated fatty acids (SFAs) accounting for less than 10%. The major components were oleic (30.9%), erucic (20.8%), linoleic (14.9%), and linolenic (11.9%) acid, with palmitic acid (5.3%) as the most abundant SFA. The high erucic acid content classifies FRSOs as high-erucic acid rapeseed (HEAR)-type oils. The average chain length remained stable at 18.7, while the total unsaturation degree ranged from 127.4 (screw press) to 133.6 (Soxhlet). The oleic-to-linoleic (≈ 2.1) and linoleic-to-linolenic (≈ 1.25) ratios indicate favorable nutritional properties. Tukey's test showed no significant differences in major fatty acids across extraction methods, except for higher palmitate, behenate, and linoleate in Soxhlet-extracted oils. The extraction method moderately influenced the FRSO composition, with a consistent fatty acid distribution of MUFAs > PUFAs > SFAs observed across all samples.

Keywords: fatty acids, fodder radish, extraction, hydraulic pressing, seed oil

This work was supported by the Republic of Serbia - the Ministry of Science, Technological Development and Innovation (Programs for financing scientific research: 451-03-137/2025-03/200133 assigned to the University of Niš, Faculty of Technology, Leskovac, 451-03-18/2025-03/200155 assigned to the Faculty of Technical Sciences, Kosovska Mitrovica), and the Serbian Academy of Sciences and Arts (SASA) (Project F-78).

# OPTIMIZING SCREW PRESSING OF FODDER RADISH SEEDS FOR MINIMAL RESIDUAL OIL IN PRESS CAKE

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Fodder radish (*Raphanus sativus* L. var. *oleiformis* Pers.), a member of the *Brassicaceae* family, is recognized for its high productivity and short growth cycle, contributing to its increasing global agricultural relevance. In addition to its value as a forage crop that supports sustainable livestock production and reduces feeding costs, fodder radish also represents a promising raw material for biofuel production. In this paper, oil extraction was performed using a screw press (Model SPU 20, Elektromotor-Šimon, Senta, Serbia), producing both oil and press cake from seeds of the NS Bora variety, developed at the Institute of Field and Vegetable Crops (Novi Sad, Serbia). To optimize the screw pressing process, Response Surface Methodology (RSM) was employed. The investigated process parameters included nozzle diameters (8 mm, 10 mm, and 12 mm), press head temperatures (40 °C, 60 °C, and 80 °C), and screw rotation frequencies (40 Hz, 50 Hz, and 60 Hz). A quadratic regression model was developed to predict the residual oil content in the press cake. The minimum predicted value (20.7%) closely matched the experimentally obtained value (22.1 ± 0.7%), under conditions of an 8 mm nozzle diameter, a press head temperature of 80 °C, and a screw frequency of 40 Hz. All three process parameters significantly influenced the oil yield, with the nozzle diameter having the most pronounced effect. The developed model provides a robust framework for future research, underscoring the importance of multi-factor optimization in achieving efficient and sustainable oil extraction.

Keywords: fodder radish, mathematical modeling, oil extraction, optimization, screw pressing

Original Scientific Paper
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# THERMODYNAMIC PROPERTIES OF Γ-VALEROLACTONE + 2-METHYLTETRAHYDROFURAN MIXTURE AT HIGH PRESSURES RELEVANT FOR THE PRODUCTION OF BIOFUEL

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The necessity of the transition from a fossil-based economy to a circular bioeconomy has intensified research related to the optimisation of the production of energy and chemicals from biomass. Lignocellulosic biomass has proven to be an excellent carbon-neutral feedstock whose complex structure significantly complicates its conversion into biofuels and biochemicals. Biomass valorisation is a challenging process, due to numerous chemical reactions that overlap, the design of which requires knowledge of the thermodynamic properties of all components present in the process.[1]
Some of the platform chemicals obtained from biomass are γ-valerolactone (GVL) and 2-methyltetrahydrofuran (2-MTHF). GVL has a wide application as a green solvent or the basis for the production of chemicals such as 2-MTHF. Both mentioned components represent promising biofuels or fuel additives and are often found together in the reaction mixture during biomass valorisation. Therefore, the thermodynamic properties of the GVL + 2-MTHF mixture were investigated in the temperature range of (293.15 - 413.15) K and at pressures up to 60 MPa. The measured density values were correlated using the modified Tammann-Tait equation, which enabled the calculation of the valuable derived volumetric properties, such as the isothermal compressibility and the isobaric thermal expansivity.[2] The observed positive deviation of the measured density from the behaviour expected for an ideal mixture indicated the attractive interactions between the mixture components.

Keywords: density, modelling, derived volumetric properties, biomass valorisation, platform chemicals, biofuels.

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# KINETIC STUDY OF PHOTOCATALYTIC DEGRADATION OF METHYLENE BLUE AND BRILLIANT CRESYL BLUE USING ZnCrAl-LDH BASED PHOTOCATALYSTS

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Water pollution resulting from various organic dye contaminants poses a significant environmental and public health concern. Azo dyes, including Methylene Blue (MB) and Brilliant Cresyl Blue (BCB), are known for their stability, toxicity, and general harmful effect on aquatic ecosystems. Among wastewater treatment methods, photocatalysis has gained attention as an efficient and eco-friendly approach. Mixed metal oxides derived from layered double hydroxides (LDHs), especially those containing Zn, have shown strong photocatalytic activity, making them promising materials for dye degradation in water. The aim of this study was to investigate the kinetics of MB and Brilliant BCB photodegradation with ZnCrAl-LDH-based mixed oxides. ZnCrAl LDH was synthesized by the low supersaturation coprecipitation method, and dried precipitates were calcined at 500, 700, and 900 °C for 5 h (ZnCrAl 500, ZnCrAl 700, and ZnCrAl 900). XRD analysis showed that, in addition to the dominant ZnO phase, a mixed oxide phase was present only in ZnCrAl 500, while ZnCrAl 700 and ZnCrAl 900 exhibited spinel phase formation alongside ZnO. Photocatalytic tests under solar irradiation showed that ZnCrAl 500 achieved the highest degradation efficiency (~60% for MB and 78% for BCB). The increase in calcination temperature led to higher crystallinity and the formation of a spinel phase, which possibly caused a decrease in photocatalytic efficiency. Kinetic studies indicated that MB degradation followed pseudo-first-order kinetics (correlation coefficient $R^2$=0.99), while BCB fitted a pseudo-second-order model ($R^2$=0.96). These differences probably arise from variations in dye adsorption mechanisms, molecular size, and orientation on the photocatalyst surface.
The results demonstrate that ZnCrAl-LDH-derived photocatalysts hold strong potential for application in water purification.

Keywords: layered double hydroxides, photocatalysis, mixed metal oxides, wastewater treatment

Original Scientific Paper
This research was supported by the Ministry of Science, Technological Development, and Innovation of the Republic of Serbia (Grant No. 451-03-136/2025-03/ 200134).

# ZnCrAl LDH-BASED ADSORBENTS FOR EFFICIENT CONGO RED DYE REMOVAL

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Water pollution caused by various organic dye contaminants represents a great concern due to harmful effects on the environment and public health. Dyes are chemically stable and resistant to degradation, even under light exposure. Congo Red (CR) is a widely used anionic dye with notable mutagenic and toxic properties. Zn-based layered double hydroxides (LDHs) and derived metal oxides have gained attention as effective adsorbents due to their low cost, ease of use, and satisfactory adsorption capacity. The aim of this study was to investigate the efficiency and kinetics of Congo Red adsorption with ZnCrAl-LDH-based adsorbents. ZnCrAl LDHs were synthesized by the low supersaturation coprecipitation method (Zn(II) 70 mol%; Cr(III) 5 mol%; Al(III) 65 mol%) and dried (100 °C, 12 h, ZnCrAl 100). Mixed oxides were obtained after calcination (500 °C, 5 h, ZnCrAl 500). Reflections characteristic of a layered structure detected by XRD analysis confirmed successful synthesis of LDH. XRD analysis also revealed the layered structure collapse and mixed oxide phase formation after calcination. Textural analysis showed an increase in adsorbed gas, due to the formation of large mesopores after thermal treatment. Furthermore, an increase in BET surface area (86 $m^2 g^{-1}$) of ZnCrAl 500 when compared to ZnCrAl 100 (69.7 $m^2 g^{-1}$) was noted. Adsorption tests showed high removal efficiency for both adsorbents: ZnCrAl 500 achieved maximum efficiency (100%) after 10 min, while ZnCrAl 100 adsorbed 72% of dye after ~20 min. Kinetic studies suggest that the adsorption rate fits the pseudo-second-order model. Both materials show great potential for future application in wastewater purification.

Keywords: adsorption, layered double hydroxides, mixed metal oxides, wastewater treatment

Original Scientific Paper

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# OPTIMIZATION OF BIODIESEL PRODUCTION FROM FODDER RADISH OIL IN THE PRESENCE OF TRIETHANOLAMINE:PROPYLENE GLYCOL DEEP EUTECTIC SOLVENT

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Biodiesel has emerged as a viable alternative to fossil diesel due to its favorable combustion characteristics, lower environmental impact, and biodegradability. The selection of raw materials and process conditions is crucial for achieving high efficiency and quality in biodiesel production. Vegetable oils, in particular, serve as primary feedstocks, offering a sustainable pathway for biodiesel production while effectively utilizing agricultural resources. This study investigates the methanolysis of fodder radish (*Raphanus sativus* L.) seed oil using uncalcined CaO as a catalyst and triethanolamine:propylene glycol (TEOA:PEG) deep eutectic solvent as a cosolvent, aiming to maximize the content of fatty acid methyl esters (FAME). Experiments were conducted at 60 °C with varying catalyst loadings (2–10% by oil mass), methanol-to-oil molar ratios (6:1–12:1), and TEOA:PEG amounts (2–10% by oil mass), following a Box-Behnken design. A second-order polynomial regression model was developed to predict FAME content as a function of process parameters. All three factors significantly influenced the FAME content, with catalyst loading having the most pronounced effect. The maximum predicted FAME content of 99%, obtained at a catalyst loading of 10%, a TEOA:PEG amount of 4%, and a methanol-to-oil molar ratio of 9:1, closely matched the experimentally achieved content of 97.9%. This study highlights the effectiveness of using uncalcined CaO with TEOA:PEG as a cosolvent in biodiesel production and demonstrates the importance of statistical optimization in enhancing yield and process sustainability.

Keywords: biodiesel; fodder radish; methanolysis; optimization; triethanolamine:propylene glycol.

# ELECTROCHEMICAL OXIDATION OF DIBUTYL PHTHALATE: EFFECT OF ELECTROLYTE ON KINETICS AND EFFICIENCY

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This study investigates the electrochemical degradation of dibutyl phthalate (DPA) using boron-doped diamond (BDD) anodes in three different supporting electrolytes: sodium sulfate ($Na_2SO_4$), sodium chloride (NaCl), and sulfuric acid ($H_2SO_4$), each at a concentration of 2 g/L. Experiments were conducted under galvanostatic conditions at a current density of 10 mA/cm$^2$, with an initial DBP concentration of approximately 5 mg/L and a treatment duration of 120 minutes. The removal efficiency varied depending on the electrolyte: 93.4% for $H_2SO_4$, 90.7% for $Na_2SO_4$, and 75.5% for NaCl. Kinetic analysis applying both first- and second-order models demonstrated that DBP degradation follows second-order kinetics in all cases, with apparent rate constants of 0.02170 mg$^{-1}$·min$^{-1}$ in $H_2SO_4$, 0.01650 mg$^{-1}$·min$^{-1}$ in $Na_2SO_4$, and 0.00527 mg$^{-1}$·min$^{-1}$ in NaCl. The good performance observed with sulfuric acid can be attributed to the *in situ* formation of highly reactive persulphate radicals ($S_2O_8^{2-\bullet}$). In sodium sulfate, sulfate radicals also contribute to DBP degradation, albeit to a slightly lower extent. The lower efficiency observed with NaCl is likely due to the formation of chlorine-based oxidants ($Cl_2$, HOCl), which exhibit lower oxidative capacity under the applied conditions. These findings highlight the critical role of electrolyte composition in determining both the degradation efficiency and kinetic behavior of DBP electro-oxidation, providing valuable insights for optimizing electrochemical treatment processes of phthalate-contaminated waters.

Keywords: dibutyl phthalate, electrochemical oxidation, supporting electrolytes, degradation kinetics.

Original Scientific Paper

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# PRELIMINARY COST ESTIMATION FOR BIODIESEL PRODUCTION FROM PLUM KERNEL OIL

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Cost estimation of biodiesel production is crucial for ensuring its sustainable production. This study examines the production cost of biodiesel obtained by methanolysis of plum kernel oil in the presence of an eco-friendly cosolvent, the triethanolamine:menthol deep eutectic solvent (TEA:M DES), and a waste-derived catalyst (35CaO/CFA-Z). The cost analysis included the expenses related to plum kernel oil, methanol, catalyst, and DES preparation, as well as energy consumption per equipment unit. Catalyst preparation costs encompass the materials and chemicals, as well as the energy required for calcination, activation, drying, and hydration. Although the initial cost of preparing 35CaO/CFA-Z was relatively high (€12/kg), it can be reduced through catalyst reusability. The catalyst was successfully reused under the optimized reaction conditions in 5 cycles with insignificant loss in catalytic activity. The reusability reduced the catalyst's cost to €2.42/kg, contributing approximately 13% to the total production cost. In contrast, the TEA:M DES was estimated to be the largest cost contributor, accounting for 30% of the total production cost. The plum kernel oil represented 21% of the total production cost, deviating from the commonly reported trend where oily feedstock typically accounts for 70-80% of the overall cost. This highlights the potential of utilizing waste-derived feedstocks to reduce the overall biodiesel production costs significantly. To enhance cost-efficiency and process sustainability, the recycling and reuse of DES should be investigated.

Keywords: biodiesel, costs, deep eutectic solvent, methanolysis, plum kernel oil

# COMPARATIVE STUDY FOR THE KINETICS OF PYROLYTIC REACTION USING COATS-REDFERN AND THE DYNAMIC METHOD

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The hydrogen- and carbon-rich composition of household waste plastics makes them particularly suitable for producing liquid fuels. Researchers typically employ thermogravimetry to analyze thermal degradation kinetics, as this information is vital for pyrolysis optimization. Determining the apparent kinetic parameters through comprehensive kinetic studies represents a critical step in developing efficient plastic waste conversion processes.

This study investigates the kinetic parameters of catalytic degradation for a waste polyolefin mixture (76.2% HDPE, 23.8% PP) via thermogravimetric analysis (TGA). The waste samples were re-granulated and palletized by manufacturers. Non-isothermal conditions were applied at five different heating rates (3, 5, 7, 10, and 20 °C/min) under a nitrogen atmosphere, covering the temperature range from 30 °C to 700 °C. The apparent kinetic parameters of thermal and catalytic degradation were determined using the integral Coats-Redfern method and the dynamic model-free method. A comparison of the obtained kinetic parameters, including activation energy ($E_a$) and the pre-exponential factor, was conducted.

The results obtained using the integral Coats-Redfern method indicate that a reaction order of “n”= 1 provides the best fit to the experimental data. The average activation energy ($E_a$) for thermal degradation with “n” = 1 was 322 kJ/mol, while for catalytic degradation over 2%, 5%, and 8% ZSM-5 catalysts, the values were 272, 260, and 254 kJ/mol, respectively. Using the dynamic model-free method, the activation energy ($E_a$) was determined to be 309 kJ/mol for thermal degradation, compared to 302, 277, and 271 kJ/mol for catalytic degradation with 2%, 5%, and 8% ZSM-5 catalysts.

Keywords: pyrolysis, TGA, ZSM-5, HDPE / PP, kinetic parameters

Original Scientific Paper
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# ISOCONVERSIONAL KINETIC ANALYSIS OF THE THERMOOXIDATION OF NETTLE (*URTICA DIOICA* L.) SEED OIL

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Lipid oxidation is a chain reaction of free radicals that leads to the development of unpleasant aromas and flavors, the loss of nutrients, and the formation of harmful compounds. In this work, kinetic parameters dependent on the degree of conversion ($0 \leq \alpha \leq 1$) were determined, in order to better understand their variation during the oxidation process. This provides an insight into the complexity of the oxidative mechanism and enables a better assessment of oil stability and behavior during heat treatment. Kinetic parameters such as the activation energy (*Ea*), the pre-exponential factor (*A*), and the reaction rate constant (*k*) were characterized using the iso-conversion Ozawa-Flynn-Wall method, and the oil oxidation was carried out by the DSC method. The results show the dependence of the activation energy on the conversion can be explained through three domains: from a conversion 0.1-0.6 it increases from 63.11 kJ/mol to 78.25 kJ/mol; from 0.6 to 0.75 it remains constant at about 78.9 kJ/mol; from 0.75 to 1 it increases again to 80.40 kJ/mol. The *A* values increased with increasing conversion and were in the range of $4.47\times10^{7}$ - $1.47\times10^{9}$ $min^{-1}$. The *k* values increased with heating rate for each degree of conversion (in the range of 0.239 - 0.251 $min^{-1}$ for $\beta = 5$ ℃/min, 0.464 - 0.492 $min^{-1}$ for $\beta = 10$ ℃/min, and 0.834 - 0.90 $min^{-1}$ for $\beta = 20$ ℃/min). The results indicate the complexity of the reaction mechanism, which is probably a consequence of the non-ideal behavior of the system, but also confirm the significant influence of the heating rate on the kinetic response of the system.

Keywords: nettle seed oil, oxidation, DSC, kinetic parameters, Ozawa-Flynn-Wall method

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# OPTIMIZATION OF LABORATORY-SCALE LAVENDER OIL EXTRACTION: A RESPONSE SURFACE METHODOLOGY APPROACH

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This study presents a laboratory-scale optimization of lavender *(Lavandula angustifolia* Mill.) essential oil extraction by hydrodistillation, aiming to maximize oil yield through the selection of optimal process conditions. A combined historical design of experiments and response surface methodology (RSM) was employed to investigate the effects of three key variables: distillation flask volume (250, 500, 1000, and 2000 mL), distillation rate (three distinct heating rates, ranging from 0.6 to 8 mL/min, adjusted to flask volume and specific water-to-flower ratios), and water-to-flower ratio (10:1, 15:1, and 20:1 mL/g).

A linear regression model describing the relationship between these variables and essential oil yield was developed, with analysis of variance (ANOVA) confirming the statistical significance of the model and all three factors. Optimization was performed using the desirability function method, which identified the most favorable conditions for achieving a high essential oil yield. The maximum predicted yield of lavender essential oil was 2.28%, obtained under the following optimized conditions: a water-to-flower ratio of 10:1 mL/g, a distillation flask volume of 2000 mL, and a distillation rate of 7.5 mL/min.

These findings demonstrate the importance of carefully selecting distillation parameters to maximize oil recovery, especially in small-scale or pilot setups where yield is critical. The study provides practical guidelines for optimizing lavender essential oil production and represents the foundation for further research focused on process scale-up and energy efficiency improvements.

Keywords: essential oil, hydrodistillation, *Lavandula angustifolia* Mill., mathematical modeling, optimization

Original Scientific Paper
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# ULTRASOUND-ASSISTED EXTRACTION OF TOTAL EXTRACTIVE MATTER FROM WILD CYCLAMEN TUBERS (*CYCLAMEN PURPURASCENS* MILL.)

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Wild cyclamen (*Cyclamen purpurascens* Mill.) tuber has been used for intestinal cleansing, treatment of migraines, nasal polyps, wounds and ulcers, menstrual pain relief, and alleviation of swelling and rheumatism. It is rich in various bioactive compounds, including saponins, tannins, flavonoids, polyphenols, alkaloids, and essential oils. The aim of this study was to investigate the effects of different parameters on the ultrasound-assisted extraction of total extractive matter. Four parameters, including extraction time (10–50 min), extraction temperature (15–75 °C), ethanol concentration (0–96%, v/v), and the liquid-to-solid ratio (5–25 mL/g), were varied. Optimization was performed using the one-variable-at-a-time approach. The total extractive content was determined by drying an appropriate amount of the obtained extract at 105 °C to a constant mass. The extraction time, extraction temperature, and liquid-to-solid ratio had a positive effect on the total extractive matter. Ethanol concentration also had a positive effect, with the total extractive matters reaching a maximum at 25% (v/v) ethanol, followed by a sharp decline with further increases in concentration. The highest yield of extractive matter (36.46 g/100 g dry matter) was obtained at the temperature of 50 °C and liquid-to-solid ratio of 10 mL/g, using 25% (v/v) ethanol solution for an extraction time of 20 min. The proposed operating conditions align with those used for the extraction of valuable compounds, thereby minimizing the risk of their degradation. Further studies will focus on a detailed analysis of the chemical composition and biological activity of the obtained extract to evaluate its potential for pharmaceutical applications.

Keywords: ultrasound-assisted extraction, wild cyclamen tubers, total extractive matter, bioactive compounds.

# FLOW REGIMES IN MODIFIED SPOUT-FLUID BED WITH DRAFT TUBE AND HYDRAULIC BARRIER

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Spouted beds are widely recognized for their efficiency in gas-solid contact and flexibility for various industrial applications. The Draft Tube Spout-Fluid Bed (DTSFB) with a hydraulic barrier represents an advanced modification designed to minimize gas bypass and prevent the mixing of spouting and aeration gas streams, making it suitable for carrying out a chemical reaction or adsorption process in the annular part of the bed. This system ensures stable operation, optimizes particle circulation, reduces bed pressure drop, and the gas flow rate required for spouting. In this study, a comprehensive experimental investigation of the flow regime transitions in a DTSFB with a hydraulic barrier is presented. Understanding the flow regime in such systems is essential for determining optimal process parameters for stable system operation and for determining the minimum spouting velocity. The experimental system consisted of a cylindrical column with a diameter of 115 mm and a hydraulic barrier with a diameter of 60 mm, and a draft tube with a diameter of 15 mm and 20 mm at a variable distance from the bottom. Spherical glass particles with diameters of 1, 2, and 3 mm were used in the experiments, and air was used as a spouting agent. In this work, different flow regimes were visually identified by setting different values for the flow rates of the spouting and aeration fluid. The influence of spouting and aeration fluid flow rate, draft tube diameter, particle diameter, and the distance of the draft tube from the bottom on the flow regimes was investigated.

Keywords: spout-fluid bed; flow regimes; stable spouting; hydraulic barrier

## Section: TEXTILE ENGINEERING

Original Scientific Paper

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# APPLICATION OF NATURAL DEEP EUTECTIC SOLVENTS IN THE ECO-DYEING OF POLYESTER FABRIC WITH DISPERSE DYE

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Polyester dyeing usually requires high pressure, high temperature, a large amount of water, and auxiliary agents to achieve better solubility of disperse dyes and uniform dyeing. This paper describes the use of a natural deep eutectic solvent (NADES) as a green alternative dyeing medium for polyester. The fabric samples of polyester fabric were washed in a non-ionic detergent and dried for 24 h before dyeing. To compare the dyeing efficiency, standard dyeing was performed at 120 °C for 110 min in an Ahiba „Nuance“ Top Speed dyeing machine by using distilled water, acetic acid, dispersant, and a leveling agent. The concentrations of Disperse Red 60 were 1, 2, 3, 4, and 5% for both methods. Dyeing in NADES (prepared with equimolar amounts of urea, glycerol, and betaine hydrochloride) was performed at 100 °C for 60 min in laboratory conditions on a hot plate with a closed glass Erlenmeyer under pressure, imitating the process of industrial, standard dyeing in an AHIBA dyeing machine. After dyeing, the samples were dried in a dryer. The efficiency of the dyeing process was analyzed using CIELab parameters and K/S values. The results showed that dyeing in a NADES improved dye adsorption and color strength without using auxiliaries. This study presents a sustainable method for polyester dyeing at lower temperatures and shorter dyeing times.

Keywords: Sustainability, natural deep eutectic solvent, dyeing, polyester, disperse dye

# APPLICATION OF 3D PRINTING TECHNOLOGY IN THE FASHION INDUSTRY

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The textile industry is a branch of industry that deals with the processing of fiber raw materials and the production of fabrics, knitwear, and clothing. A turn in the fashion industry in the direction of sustainable fashion is necessary, which implies a reduced consumption of natural resources and the application of a circular business model. This paper discusses the possibility of using 3D printing technology in the clothing industry as a technological innovation for the purpose of developing a new concept of the fashion industry. It describes the techniques used in 3D printing, the materials applicable for 3D textile printing, as well as the advantages and limitations of this technology. Three-dimensional (3D) printing creates 3D objects quickly and efficiently - layer by layer, based on computer-aided design (CAD) and computer-aided manufacturing (CAM). Technology 3D printing allows designers to experiment with complicated patterns and structures that are difficult to achieve with traditional textile production methods. To achieve the desired result, several 3D printing techniques are used, including material extrusion, powder layer fusion, and bath photopolymerization. Thermoplastic polymer filaments with a low melting point, such as polylactic acid (PLA), acrylonitrile butadiene styrene (ABS), and thermoplastic polyurethane (TPU) are commonly used for 3D printing. 3D printing offers fashion designers greater creative freedom, enabling them to push the boundaries of fashion product design. It can be concluded that instead of the traditional mass production of clothing, which results in waste material and unused stock, 3D printing enables production "on demand", reducing waste and contributing to environmental sustainability.

Keywords: 3D printing, CAD, CAM, thermoplastic filaments

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# MORPHOLOGY ANALYSIS OF TEXTURED MULTIFILAMENT POLYESTER YARN AT DIFFERENT LOADS

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The paper analyzed the surface appearance changes of textured multifilament PES yarns loaded up to the elastic limit and the yielding limit.
Changes in surface appearance of textured PES multifilament yarns were recorded using a (stereo) Leica microscope, supported by Video Analyzer 2000 software. Previously, samples of textured PES multifilament yarns were subjected to appropriate tensile forces (ISO 2062) on a Tinius Olsen dynamometer, supported by the “Horizon” software.
Several samples of textured multifilament PES yarns with a linear mass of 167f36x1 dtex were prepared. All analyzed samples were recorded before loading on a dynamometer, and then loading was performed to the specified force values. The samples were relaxed after loading and recorded again.
The results show minor changes in the appearance of the surface of the yarn that was loaded with a force up to the elastic limit. Insignificant changes are evidence that in multifilament PES yarns, even small loads cause deformations, which should be taken into account when predicting the characteristics of textile materials.
Analysis of the surface appearance of the relaxed textured multifilament polyester yarns, which were previously loaded with force to the yield point, showed significant changes in the surface appearance after relaxation compared to the surface appearance of the relaxed yarn before the loading process on the dynamometer. However, monofilament breaks were not observed on the recordings, which can be very significant when predicting the limit loads of more complex textile structures intended for application under conditions of higher loads.

Keywords: textured PES multifilament yarns, mechanical characteristics, deformation characteristics, morphological characteristics.

## Section: TECHNOLOGY AND SUSTAINABLE DEVELOPMENT

# POTENTIALS FOR APPLICATION OF INNOVATIVE AI-BASED SOLUTIONS IN CONSTRUCTION WASTE MANAGEMENT IN THE CITY OF LESKOVAC

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The management of construction and demolition waste (CDW) is one of the most complex aspects of local environmental policy, particularly in urban areas such as the city of Leskovac. Despite the existence of a legal framework, several practical challenges persist: the lack of data on waste flows, the absence of an efficient monitoring and recycling system, and weak coordination among relevant institutions. This presentation emphasizes the critical role of information systems and software solutions in addressing these challenges. The implementation of digital tools for tracking the generation, transportation, and disposal of CDW, based on AI, can establish a transparent and efficient waste management system. Information systems enable improved planning and control, as well as automated reporting to competent authorities. At the core of the system lies a centralized software platform designed to coordinate all stakeholders — municipal departments, contractors, waste operators, and citizens — through automated workflows. The proposed platform employs AI-driven waste categorization algorithms based on project documentation and geolocation data, enabling proactive identification of hazardous and recyclable materials. Upon project registration (e.g., demolition, construction), the system generates automated notifications, tracks declared vs. delivered waste volumes, and assigns optimal collection, storage, and treatment points, while generating detailed reports for inspection bodies and tracking fulfillment of recycling obligations. This model can be replicable across Serbia's future network of regional landfills, aligning with the National Waste Management Program. Ultimately, it transforms CDW from an environmental burden into a valuable resource, operationalizing circular economy principles through intelligent coordination, local empowerment, and digital innovation.

Keywords: construction waste, information systems, AI, Leskovac, circular economy

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