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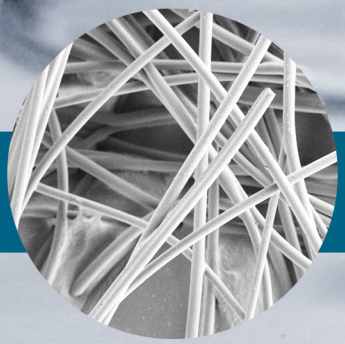

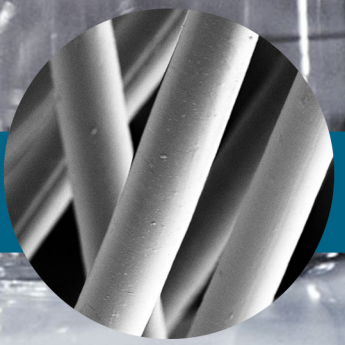


Book of Scientific Research Achievements of Program Group

TEXTILE CHEMISTRY AND ADVANCED TEXTILE MATERIALS

P2-0118 1st Annual Meeting 2024

University of Maribor
Faculty of Mechanical Engineering

Book of Scientific Research Achievements of Program Group Textile Chemistry and Advanced Textile Materials

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Editors
Tatjana Kreže
Lidija Fras Zemljič

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PREFACE

The Programme Group (PG) **Textile Chemistry** has a rich history, and was established in 1999. In 2021, we changed our name to PG **Textile Chemistry and Advanced Textile Materials**, as we focus on developing advanced textile materials and technologies, not only for the garment and textile industry, but also for promising industrial sectors, such as automotive, polymer, electrical engineering, construction, agriculture, medicine, etc. Throughout our history we have been one of the most successful groups at the University of Maribor and in Slovenia, as well as internationally.

Our research guidelines are aligned constantly with the latest global trends and requirements. We focus on developing textiles and textile composites for various applications, with an emphasis on creating multifunctional and smart textiles, which is a major challenge, especially for obtaining simultaneous functionalities while maintaining the basic required material properties. To achieve these objectives, we use advanced technologies such as Nanotechnology and Biotechnology, and concentrate on the principles of a green and sustainable economy.

Fundamental research plays an important role in our research work, analysing the physical, chemical, structural, thermal, mechanical and morphological properties of fibres, focusing on surface chemistry. We also study the interaction phenomena between textiles and functional liquids, and between functional textiles and microorganisms such as bacteria, fungi and viruses. Understanding these surface and interaction phenomena allows us to optimise materials for different functionalities and applications. The main objectives of our research work are defined in two pillars:

- **PILLAR I.** presents the development of **smart and multifunctional textiles and composite materials** for packaging, medical and technical/engineering applications.
- **PILAR II.** is dedicated to **circular and sustainable approaches.**

A specific part of the research found in both pillars focuses on the care of textiles. In both pillars we integrate nanotechnologies and biotechnologies, and develop new methods and analytical tools to understand the interactions between materials better.

In the field of Functional Textiles and Composites, we develop high-performance, multifunctional and smart fibres, based on advanced technologies such as N**anotechnology** (the production and use of functional nanoparticles, nanocapsules, and the production of nanofibres, nanosensors, etc.), B**iotechnology** (enzymatic processing, microbial products, microbiota, cell wall biopolymers, molecular microbiology, microbial engineering, synthetic biology and environmental biotechnology), and advanced **surface functionalisation** and "bulk functionalisation" techniques, where we integrate functional materials into the material (electrospinning, extrusion, 3D/4D printing, etc.). In addition, we perform **surface modifications** such as plasma treatments and coating applications, using state-of-the-art techniques including impregnation, dipping under controlled conditions, various printing techniques (matrix printing, screen printing, inkjet printing and 3D printing), as well as spraying, sputtering and electrospraying, depending on the chemical and process conditions, to achieve an optimal coating on the surface.

The integration of all these processes is key to the development of intelligent, functional and high-performance textiles and other textile composites with antimicrobial, bactericidal, biofilm-inhibiting, self-cleaning, fire retardant, hydrophilic/hydrophobic, antistatic properties, magnetic, electrically conductive and cosmetic properties, depending on the end use in different industries, such as textiles, chemicals, medicine, pharmaceuticals, cosmetics, transport, plastics, agriculture, dyes, cultural heritage, construction and architecture.

Circular economy concepts are the key to sustainable development, and include the use of bio-waste for functionalisation, green production technologies, reuse of materials, development of advanced recycling processes, isolation of secondary raw materials, and the use of natural fibres. At the same time, we are developing purification technologies for textile hygiene, and the reduction of impurities and emissions of micro- and nano-plastics in wastewater. Our research takes into account the entire product life cycle and supports circular business models.

Our programme is highly interdisciplinary, combining the fields of Fibre-forming and other polymeric materials, Chemistry, Chemical Technology, Environmental Science, Textile Chemistry and Technology and Biotechnology. We work with experts in Microbiology, Biology, Physics, Medicine, Pharmacy, Food Biotechnology, and many more, both at the national and European levels.

The proceedings we are presenting provide an overview of our research achievements from 2021 to 2023. Members of the Programme Group present their research stories and innovations, as well as the progress made within the Programme Group's activities. We would like to thank all those whose efforts have contributed to the visibility and excellence of our work. We are particularly proud of the younger researchers, who bring freshness and innovation to our programme.

Thanks also to the Organising, Programme and Editorial Committees for their contribution. We look forward to the challenges ahead, and especially to our first in-house conference, where we will also present our results to industry, promote knowledge transfer to industry, promote our equipment and expand our research network.

Thank you all again! Let's step forward boldly on our research path and spread our creative wings, and let's bring the importance of textile and composite materials, which are versatile, creative and advanced, just like us, closer to the younger generations in particular.

Head of the Programme Group Textile Chemistry and Advanced Textile Materials P2-0118
Prof. Dr. Lidija FRAS ZEMLJIČ

UVODNA BESEDA

Programska skupina (PS) **Tekstilna kemija** ima bogato zgodovino, saj je bila ustanovljena že leta 1999. Leta 2021 smo se preimenovali v PS **Tekstilna kemija in napredni tekstilni materiali**, saj se osredotočamo na razvoj naprednih tekstilnih materialov in tehnologij ne le za oblačilno in tekstilno industrijo, temveč tudi za perspektivne industrijske panoge, kot so avtomobilska industrija, polimerna industrija, elektrotehnika, gradbeništvo, kmetijstvo, medicina itd. Skozi celotno obdobje našega delovanja smo bili med najuspešnejšimi skupinami na Univerzi v Mariboru in v Sloveniji, kot tudi v mednarodnem prostoru.

Naše raziskovalne smernice se nenehno usklajujejo z najnovejšimi svetovnimi trendi in zahtevami. Osredotočamo se na razvoj tekstilij in tekstilnih kompozitov za različne namene, s poudarkom na ustvarjanju multifunkcionalnih in pametnih tekstilij, kar predstavlja velik izziv zlasti za pridobivanje sočasnih funkcionalnosti ob ohranjanju osnovnih zahtevanih lastnosti materialov. Za doseganje teh ciljev uporabljamo napredne tehnologije, kot sta nanotehnologija in biotehnologija, ter se koncentriramo na načela zelenega in trajnostnega gospodarstva.

Pomembno vlogo v našem raziskovalnem delu igrajo temeljne raziskave, ki zajemajo analize fizikalnih, kemijskih, strukturnih, termičnih, mehanskih in morfoloških lastnosti vlaken, pri čemer se osredotočamo na površinsko kemijo. Preučujemo tudi interakcijske pojave med tekstilijo in funkcionalnimi tekočimi sredstvi ter funkcionalno tekstilijo in mikroorganizmi, kot so bakterije, glive in virusi. Razumevanje teh površinskih in interakcijskih pojavov nam omogoča optimizacijo materialov za različne funkcionalnosti ter namembnosti. Glavni cilji našega raziskovalnega dela so opredeljeni v dveh stebrih:

- **I. STEBER** predstavlja razvoj **pametnih in večfunkcionalnih tekstilij ter kompozitnih materialov** za embalažo, medicinsko uporabo in tehnično/inženirsko uporabo.
- **II. STEBER** je namenjen **krožnim in trajnostnim pristopom**.

Poseben del raziskovanja, ki ga najdemo v obeh stebrih, se osredotoča na nego tekstilij. V obeh stebrih integriramo nanotehnologije in biotehnologije ter razvijamo nove metode in analitična orodja za boljše razumevanje interakcij med materiali.

Na področju funkcionalnih tekstilij in kompozitov razvijamo visoko zmogljiva, večnamenska in pametna vlakna, ki temeljijo na naprednih tehnologijah, kot je **nanotehnologija** (proizvodnja in uporaba funkcionalnih nanodelcev, nanokapsul in proizvodnja nanovlaken, nanosenzorjev itd.), **biotehnologija** (encimska obdelava, mikrobni proizvodi, mikrobiota, biopolimeri celičnih sten, molekularna mikrobiologija, mikrobno inženirstvo, sintetična biologija in biotehnologija okolja) ter napredne **tehnike površinske funkcionalizacije** in »bulk funkcionalizacije, kjer integriramo funkcionalne snovi v material (elektropredenje, ekstrudiranje, 3D/4D tiskanje itd.). Poleg tega izvajamo **površinske modifikacije**, kot so plazemske obdelave in nanašanje premazov z najsodobnejšimi tehnikami, vključno z impregnacijo, omakanjem pod nadzorovanimi pogoji, različnimi tiskarskimi tehnikami (matrični tisk, sitotisk, brizgalni tisk in 3D tiskanje), ter brizganje, razprševanje in elektropršenje, odvisno od kemijskih in procesnih pogojev, da dosežemo optimalni premaz na površini. Vključitev vseh teh postopkov je ključna za razvoj inteligentnih, funkcionalnih in visoko zmogljivih tekstilij ter drugih tekstilnih kompozitov s protimikrobnimi, baktericidnimi, biofilm zaviralnimi, samočistilnimi, ognjevarnimi, hidrofilnimi/hidrofobnimi, antistatičnimi, magnetnimi, elektroprevodnimi in kozmetičnimi lastnostmi, odvisno od končne uporabe v različnih industrijah, kot so tekstilna industrija, kemična industrija, medicina, farmacija, kozmetika, promet, plastika, kmetijstvo, barvila, kulturna dediščina, gradbeništvo in arhitektura.

Koncepti **krožnega gospodarstva** so ključni za trajnostni razvoj in vključujejo uporabo bioloških odpadkov za funkcionalizacijo, zelene proizvodne tehnologije, ponovno uporabo materialov, razvoj naprednih postopkov recikliranja, izolacijo sekundarnih surovin in uporabo naravnih vlaken. Hkrati razvijamo čistilne tehnologije za tekstilno higieno in zmanjševanje nečistoč ter emisij mikro in nanoplastike v odpadnih vodah. Pri raziskavah upoštevamo celoten življenjski cikel izdelka in podpiramo krožne poslovne modele.

Naš program je izrazito interdisciplinaren, združuje področja vlakno tvornih ter drugih polimernih materialov, kemije, kemijske tehnologije, okoljevarstva, tekstilne kemije in tehnologije ter biotehnologije. Sodelujemo s strokovnjaki s področij mikrobiologije, biologije, fizike, medicine, farmacije, živilske biotehnologije in še mnogih drugih, tako na nacionalni kot evropski ravni.

Zbornik, ki ga predstavljamo, prinaša pregled naših raziskovalnih dosežkov od leta 2021 do 2023. Člani programske skupine predstavljajo svoje raziskovalne zgodbe in novosti ter napredek, dosežen v okviru delovanja programske skupine. Zahvaljujemo se vsem, ki so s svojim trudom prispevali k prepoznavnosti in odličnosti našega dela. Posebej smo ponosni na mlajše raziskovalce, ki prinašajo svežino in inovativnost v naš program.

Hvala tudi organizacijskemu, programskemu in uredniškemu odboru za njihov prispevek. Veselimo se prihodnjih izzivov in predvsem naše prve interne konference, kjer bomo rezultate predstavili tudi industriji, spodbujali prenos znanja v gospodarstvo, promocijo naše opreme ter širjenje raziskovalne mreže.

Še enkrat se zahvaljujem vsem! Pogumno stopimo naprej po naši raziskovalni poti in razširimo ustvarjalna krila ter približajmo predvsem mlajšim generacijam pomen tekstilnih in kompozitnih materialov, ki so vsestranski, ustvarjalni in napredni, tako kot mi.

Vodja programske skupine Tekstilna kemija in napredni tekstilni materiali P2-0118
Red. prof dr. Lidija FRAS ZEMLJIČ

Functional Starch Based Films

Lidija Fras Zemljič,[1] Tatjana Kreže,[1] Klementina Pušnik Črešnar [1]

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In the last decade there has been renewed interest in converting starch into a plastic material known as thermoplastic starch, which can replace materials made from fossil sources that currently pose an enormous ecological footprint [1]. In this research work we developed biodegradable starch films, and improved their properties by incorporating various natural plant extracts. Potato starch served as the primary raw material. We combined this with three types of natural substances, i.e. extracts of rosemary, thyme, and biopolymer chitosan-in different ratios, both with and without glycerol (Table 1). Additionally, a control sample was prepared without extracts.

The objective of this study was to determine how different natural extracts (see Table 1) and chitosan, along with varying ratios and the addition of glycerol, influence the final material properties. The methods employed included determining the melting point using a melting table, measuring the Contact Angle using a goniometer, assessing the antioxidation percentage via the 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) ABTS assay, and identifying functional groups through Attenuated Total Reflectance Fourier-Transform Infrared ATR-FTIR analysis. It was demonstrated that the extracts were incorporated successfully into the starch films, resulting in excellent antioxidant activity (Figure 1). Our findings suggest promising initial points for potential practical applications, particularly due to their strong alignment with the principles of the bioeconomy and the circular economy.

Table 1: Description of the samples

Sample	Description of Sample	Sample	Description of Sample
ŠGA	Starch sample with the addition of glycerol	ŠA	Starch sample without glycerol
ŠGAR	Starch sample with the addition of glycerol and rosemary in a 10:1 ratio	ŠAR	Starch sample without glycerol, with the addition of rosemary in a 10:1 ratio
ŠGAT	Starch sample with the addition of glycerol and thyme in a 10:1 ratio	ŠAT	Starch sample without glycerol, with the addition of thyme in a 10:1 ratio
ŠGAH	Starch sample with the addition of glycerol and chitosan in a 10:1 ratio	ŠAH	Starch sample without glycerol, with the addition of chitosan in a 10:1 ratio
ŠGB	Starch sample with the addition of glycerol	ŠB	Starch sample without glycerol
ŠGBR	Starch sample with the addition of glycerol and rosemary in a 1:5 ratio	ŠBR	Starch sample without glycerol, with the addition of rosemary in a 1:5 ratio
ŠGBT	Starch sample with the addition of glycerol and thyme in a 1:5 ratio	ŠBT	Starch sample without glycerol, with the addition of thyme in a 1:5 ratio
ŠGBH	Starch sample with the addition of glycerol and chitosan in a 1:5 ratio	ŠBH	Starch sample without glycerol, with the addition of chitosan in a 1:5 ratio

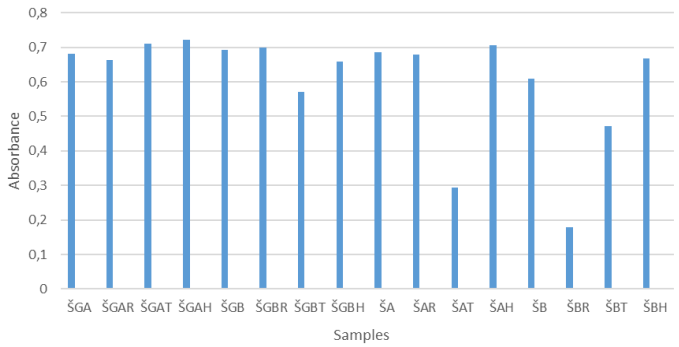

Figure 1: Antioxidant activity of the samples

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Development of Active, Biodegradable PLA Packaging Using Natural Fillers and Coatings

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The demand from consumers and packaging producers for innovative packaging has driven the emergence of solutions like active packaging. This packaging type focuses on enhancing shelf life and safety without compromising quality, achieved by tailoring active packaging systems to specific food needs, thereby notably mitigating food quality degradation [1]. These systems encompass various functionalities, such as oxygen scavengers, carbon dioxide emitters and scavengers, ethylene scavengers, ethanol emitters, moisture absorbers, antimicrobial agents, flavour/odour absorbers and temperature-controlled packaging [1].

Our research approach involves integrating active substances derived from natural sources, such as biopolymers and certain plant extracts rich in polyphenols, into packaging materials. These substances offer antioxidative, antimicrobial, or barrier properties that engage actively with the packaged contents [2]. By utilising natural polymers and polyphenols in coatings, this concept aims to create a protective layer that interacts actively with the packaged product, thereby improving its quality and safety while reducing waste.

Developing multifunctionality (Figure 1) was a key aspect considered and evaluated in this work through several parameters, including elemental composition, wettability, zeta potential measurements, oxygen barrier properties, and bioactivity such as antioxidant and antimicrobial efficiency. Additionally, the influence was examined of the surface properties on the final packaging bioactivity. This exploration aimed to assess if the manipulation of surface properties could serve as a driving force for efficiency and packaging usability.

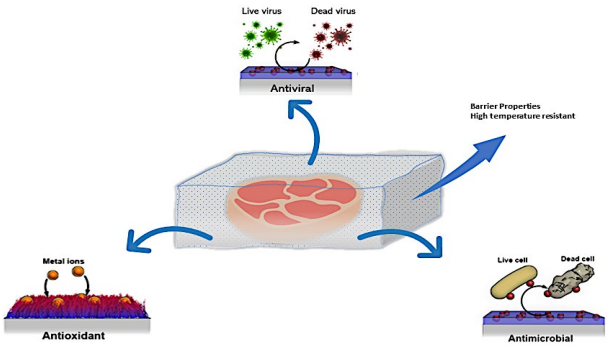

Figure 1: Packaging multifunctionality

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The Effect of Ageing Treatment of PLA-Based Lignocellulosic Composites on the Microstructure and Thermal Behaviour

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Poly(lactic acid) (PLA) is known as the packaging polymer of the 21st century, due to its biodegradability, biocompatibility and bioabsorbability. The limitations of PLA have been studied, attributed to poor hydrophilicity and toughness, inadequate gas barrier properties, slow degradation rate, low thermal conductivity, as well as the weak crystallisation addition of a natural filler such as Kraft lignin (KL) [1,2]. Their unknown long-term properties exposed in the natural environment may cause changes in the chemistry of composites through the degradation process. Therefore, polymer composites (PLA-KL) based on polylactide (PLA, molar mass 75 kg/mol) filled with natural lignocellulosic particles, KL at 0.5, 1.0, 2.5 wt% and prepared by melt extrusion and thermal pressing, were studied here. The main scope is to evaluate the effect of the accelerated weathering of PLA-KL composites on the structural and thermal properties. In addition, accelerated ageing of PLA-KL was carried out according to the ASTM D 4329 Standard. After accelerated ageing, the structural properties were measured by attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR), while differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) were used to evaluate the changes in the thermal properties. After accelerated ageing, the results by ATR-FTIR analysis for neat PLA and all the PLA-KL composites showed an increase of the characteristic vibration peaks of the main characteristic vibration of KL, namely, 3390 cm^{-1}, 1600 cm^{-1}, 1500 cm^{-1} and 1450 cm^{-1}, known as characteristic vibration of the aromatic structure of KL. An increase in the intensity of these peaks may indicate the presence of KL on the surface of the PLA-KL film due to accelerated weathering.

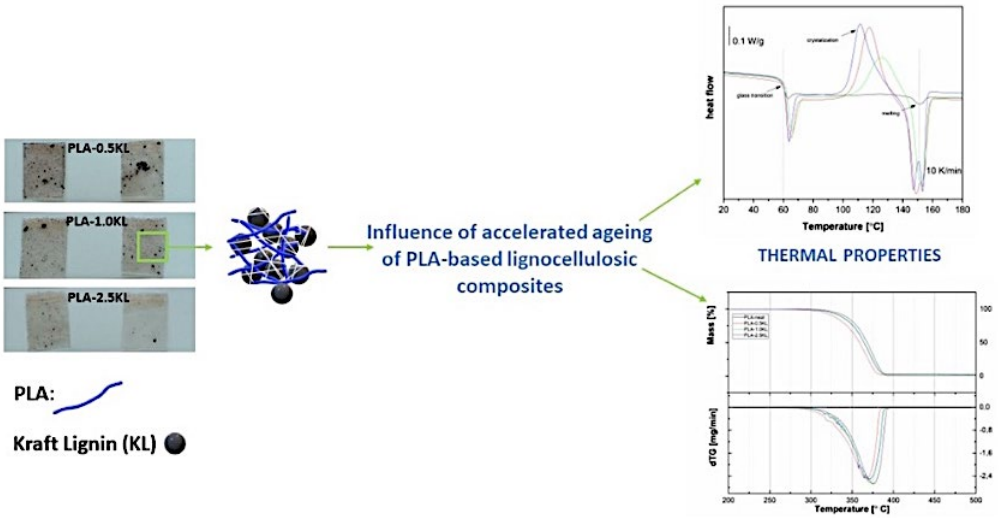

Figure 1: Graphical abstract

The results obtained by DSC showed comparative changes in either glass transition temperature, melting or crystallisation temperature, indicating the degradation process of PLA-KL composites that leads, in general, to rearrangement of the semi-crystalline to the amorphous phase of the composites. Furthermore, the effect of ageing on the thermal degradation of PLA-KL composites filled with the three different contents of polyphenolic fillers was found and studied by TGA analysis. The PLA-KL composites showed worse

thermostability than the composites before accelerated weathering. In addition, the neat PLA, PLA-KL, before weathering, showed no significant mass loss up to 390 °C.

On the other hand, the PLA-KL, after ageing, started to degrade from 350 °C for neat PLA and 325 °C for PLA-KL. Thus, the thermal degradation temperature of PLA-KL composites decreases with the addition of filler, probably due to the catalytic effect of KL, and may reduce the thermal stability of PLA-KL composites.

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Enhancing the Properties of PLA Using Coffee Waste Extracts By Surface and Bulk Modifications

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Coffee waste extracts are recognised as a rich source of antioxidants with impressive thermal stability [1]. These extracts also demonstrate antibacterial properties, making them a promising choice for enhancing food packaging materials and extending the shelf life of food products [2]. This study focuses specifically on functionalising poly(lactic acid) (PLA), a versatile biodegradable material used extensively in food packaging, which lacks inherent antioxidative and antibacterial properties. To address these limitations, two approaches were explored: firstly, the in-situ addition of coffee extract powder to L-lactide during polymerisation to create PLA granules, used subsequently for film production; secondly, surface coating of neat PLA films with coffee extract solutions using the spray coating technique [3]. The resulting active PLA materials from both methods underwent thorough analysis through various techniques. These included Contact Angle measurements to assess their wettability, X-ray Photoelectron Spectroscopy (XPS) and Attenuated Total Reflectance Fourier-Transform Infrared (ATR-FTIR) spectroscopy for surface composition analysis, Oxygen Transmission Rate (OTR) measurements to evaluate barrier properties` improvement, and the ABTS assay to determine antioxidant activity. Additionally, antibacterial activity against Gram-positive S. aureus was examined, while Zeta potential measurements were employed to monitor the charge of the PLA films. These findings underscore the potential of coffee waste extracts as active compounds, offering significant promise in enhancing the shelf life and quality of food products packaged using functional PLA films. This eco-friendly approach presents a functional solution for the food packaging industry. Moreover, the comparative analysis between the two production methods highlights the superior efficiency of surface-modified films in enhancing shelf life.

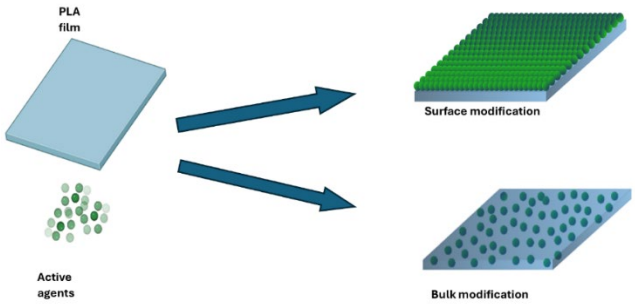

Figure 1: Schematic representation of active agents coated and integrated into bulk of PLA films

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A Battle Against Hospital-Acquired Infections With Antiviral Polysaccharide-Based Coatings as Strategic Protective Textile Development: A Detailed Physicochemical Characterisation of Liquid Media

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The 2019 Coronavirus pandemic outbreak, and many others caused by enveloped viruses, highlighted the critical need for strategic antiviral coatings on textile personal protective equipment (PPE), the first line of defence during global outbreaks. Hospital environments, which, due to their function, have a high level of risk for the cross-spread of pathogenic micro-organisms, commonly experience costly and deadly hospital-acquired infections (HAIs) [1]. Studies report of viral persistence on porous surfaces, such as textiles, from 48 h to 7 days [2]. Textile surfaces are favourable substrates for microbial adherence and colonisation, and PPE lacks inherent antiviral activity, serving solely as a suboptimal physical barrier against viral particles, making it a reservoir for indirect transmission (i.e., a fomite) [3]. Instigated by recent health and environmental concerns with existing antiviral PPE coatings (e.g., based on antiviral metal nanoparticles and synthetic compounds), natural polysaccharides with promising antiviral potency and attributes, such as bioactivity, affordability, water solubility, film-forming capability, nontoxicity, biocompatibility and biodegradability, have become the subject of vigorous research [4]. The strategic design of antiviral biobased PPE coatings is complex, due to the intricate physicochemical and structural properties of liquid and coated media, the surface and specific virus, marking every PPE-virion interaction as individually unique [5]. However, a severe research gap persists in this area, with incomplete understanding of such interactions on the molecular level. Utilising fundamental knowledge in Surface and Colloid Chemistry to comprehend, modify and regulate molecular-level interactions at the coated PPE-virion interface could aid researchers in crafting more efficient antiviral biobased coatings for PPE [5].

Thus, this work is the first phase of this endeavour, wherein we have characterised the key physicochemical properties of differently concentrated aqueous solutions of polysaccharides (1-5 mg/mL) with promising antiviral activity that are either anionic or cationic (poly)electrolytes, i.e., dextran sulfate, λ-carrageenan, chondroitin sulfate A, fucoidan, quaternary chitosan, carboxymethyl chitosan and carboxymethyl dextran. We utilised the techniques of polyelectrolyte and potentiometric titration, molecular composition, zeta potential and hydrodynamic diameter determination, bioactivity assessments (i.e., antioxidant and antimicrobial activity), and determination of the physical properties (i.e., thermal stability, viscosity, surface tension). Attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR), titrations and zeta potential (ZP) determinations confirmed that the polysaccharides contain characteristic active functional groups (e.g., carboxyl, sulfate, sulfonic acid, hydroxyl, primary and quaternary amino, etc. groups), mostly between 1-6 mmol of charge per gram of polysaccharide, and moderate to high values of ZP in the pH range between 3 and 9, respectively. Due to the nature of the PPE application, the ZP value is most important around pH 6.7, i.e., the average pH of saliva. High ZPs (≥ +/-25 mV), suggesting dispersion stability, were expressed by dextran sulfate, quaternary chitosan, fucoidan and λ-carrageenan solutions, as shown in Fig. 1. Particle size distribution, hydrodynamic diameter and turbidimetry assessments revealed that the solutions contained heterogeneous colloidal particles. The polysaccharide solutions did not show significant antioxidant (concentrations between 0.01-1 %) or antimicrobial activity (≤ 0.5 %), thus, we may combine certain plant polyphenols for additional functionality. The further aim of this work includes assessing the polysaccharide solutions for antiviral activity as a key parameter for the antiviral PPE concept.

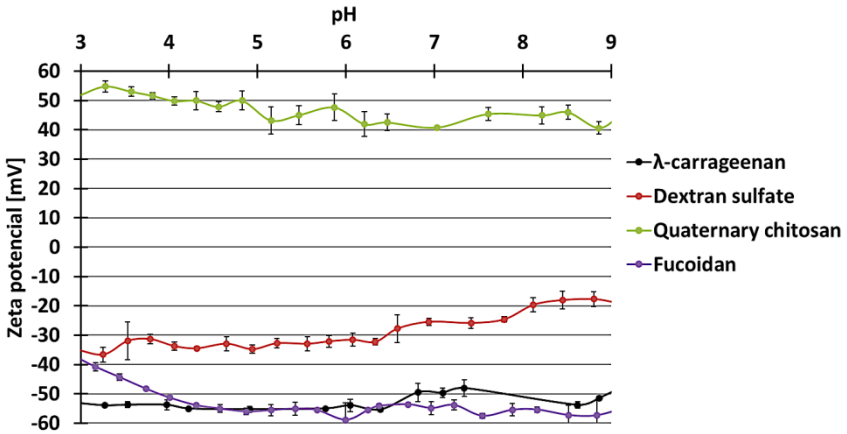

Figure 1: Exemplarily shown zeta potential [in mV] of dextran sulfate, quaternary chitosan, fucoidan and λ-carrageenan solutions in the pH range between 3 and 9.

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New Antimicrobial Cationic Polymers

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New antimicrobial molecules and materials are in high demand, due to the spread of resistant bacterial, fungal and viral strains attributed to many factors.[1] A large group among antimicrobials are cationic polymers, which can be used in the form of functional coatings, or liquid formulations.[2] A cationic charge is very often provided by protonated amino groups or quaternary nitrogen groups, favourably amine or ammonium ions. Many amine or ammonium containing polymers exist, including those with a synthetic or biological origin.[3, 4] Among the known ammonium containing compounds in living cells are choline and betaine, which make up important osmolytes and chemical neurotransmitters. Polymers containing those two monomers could, therefore, be of interest as new antimicrobial agents. Betaine and choline contain, beside amino groups, either a carboxylate or hydroxyl group, which can be bound covalently to various polymer backbones by polymer analogous reactions alternatively to the polymerisation of monomers.[4] In this work, both molecules were attached to the backbone on either polyvinyl alcohol or hydroxyethyl cellulose using carbonyl diimidazole as a coupling agent (Figure 1).[4] The optimum yields and reaction conditions were found and the chemical structure revealed by NMR, IR spectroscopy and charge titrations. Subsequently, the antimicrobial efficacy against Staphylococcus aureus and Pseudomonas aeruginosa was tested, and choline HEC was found to inhibit the former, but not the latter, strain at a polymer concentration of 0.08 mg/ml. While the mechanism of antimicrobial inhibition still needs to be studied and could be attributed to the presence of imidazole traces, betaine containing PVA and choline containing HEC turned out to be relatively toxic for mouse fibroblast cells.[4] Related cationic polymers can be processed into solid materials by electrospinning, probably making them useful as medical wound dressing materials.[3]

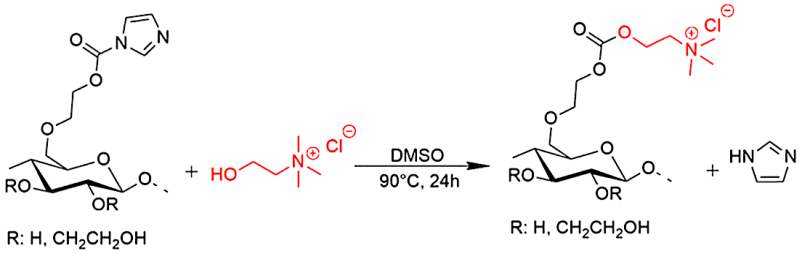

Figure 1: Coupling of choline chloride to previously activated hydroxyethyl cellulose (HEC) leading to a cationic HEC.

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Functional Surface Coatings for Catheters

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In the landscape of healthcare-associated infections (HAIs), Catheter-Associated Urinary Tract Infections (CAUTIs) make up a staggering 80%. Within long-term hospital care, 10-25% of patients utilise catheters, amounting to an annual cost of around 395 million EUR, with each treatment episode averaging approximately 2,550 EUR [1-2]. Among residents in long-term care facilities (LTCFs), 5-10% rely on urinary catheters, mainly men dealing with urinary retention and women managing incontinence. Notably, during hospitalisation, 25% of individuals coping with urinary incontinence receive urinary catheters, impacting nearly half of the elderly population. Given the prevalence of biofilm-related infections, particularly CAUTIs, and the ageing population, numerous preventive strategies have emerged over the last two decades. Future strategies are concentrated on modifying medical device surfaces, especially catheters [3]. This is crucial, because oral antibiotic therapy is becoming less effective due to bacterial resistance, posing a significant global health threat.Surface functionalisation of materials plays a pivotal role in determining material activity and functionality when in contact with biological environments. Meeting various criteria, such as biocompatibility, mechanical properties, hydrophilicity, antimicrobial, antioxidant activity, and antifouling properties, is essential. Introducing properties like antifouling presents a strategic approach to combat CAUTIs and biofilm formation. Therefore, in this study, different colloidal systems based on polysaccharides and natural polyphenols, used individually or in synergy, were developed as surface coatings for catheters.

The primary objective of this research was to explore the anti-infection capabilities (controlled prevention) of novel biomimetic and multi-functional coatings for urethral catheter production. These coatings, based on natural, biocompatible organic biopolymers like chitosan/chitosan derivatives and alginates, serve as carriers for the immobilisation and distribution of antimicrobial/antioxidant-active plant polyphenols. The aim is to ensure hydrophilicity, prevent infections due to friction issues, and deter bacterial adhesion.

The focus was on the functionality of two catheter materials (Figure 1): polyvinylchloride (PVC) and Thermoplastic Polyolefin Elastomer (TPE). The coatings were applied to these surfaces, and their physicochemical properties were examined, including their elemental composition (using ATR-FTIR and XPS).

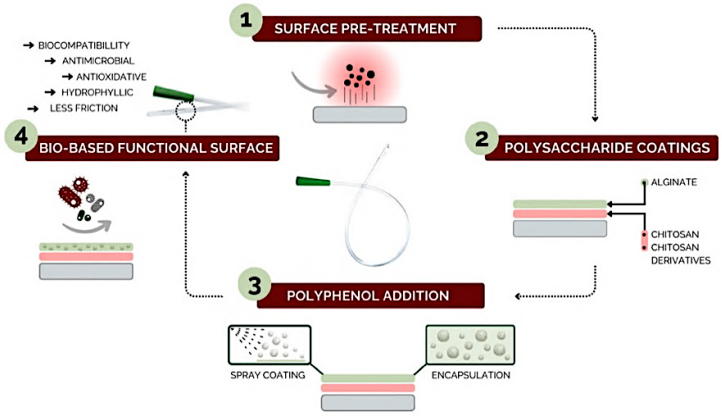

Figure 1: Schematic presentation of our research

The study evaluated hydrophilicity using Contact Angle, antioxidant activity via the ABTS assay, and antimicrobial activity. Additionally, TIK d.o.o., Slovenia's largest company producing medical disposable devices and well-known in the EU, participated in the research to assess catheter usability properties (friction test) and the practical application of this technology in their production processes. The results showed the improved surface hydrophilicity of catheters and introduction of microbial inhibition regarding *S.aureus.*

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The Use of Poultry Feathers and Wool Isolates for the Development of Keratin-Based Nanofibre Structures

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Keratin is one of the most important proteins, useful in various fields, especially in the pharmaceutical industry, medicine, and cosmetics [1]. Rich sources of keratin include poultry feathers, wool, hair, etc. The main challenges in extracting keratin from these sources still lie in the extraction processes and the need for harsh and environmentally harmful chemicals [2]. Therefore, supercritical water is becoming increasingly important as an environmentally friendly medium for the extraction of high-value ingredients in this field [3]. In this study, we investigated the extent to which hydrothermal degradation of poultry feathers and/or wool is a good source of keratin for the production of nanofibres. To this end, the products obtained from hydrothermally degraded poultry feathers and wool waste were analysed in terms of pH, conductivity, turbidity and particle size, and then used in combination with polyethylene oxide (PEO) to produce composite nanofibres using a needleless electrospinning process.

The chemical and physical analyses of the products from the hydrothermal degradation of feathers and wool have shown that the extracted solutions are colloids with a particle size of about 700 nm to 2 µm.

Due to the low average values and the wide range of molecular weights of keratin, the addition of at least 50 % PEO is required for the successful production of nanofibres. It has also been shown that the presence of larger particles in non-dialysed samples hinders the successful formation of nanofibres. However, the ATR-FTIR spectra of the produced nanofibres show all the typical peaks of keratin. In addition, the optical microscopy and preliminary SEM analysis confirmed the formation of nanofibres.

In the further study the purification methods of the isolate will be investigated, to find out which are the most suitable to enable a more successful production of nanofibres. In addition, analyses of different cross-linking processes of PEO will be carried out, in order to reduce the water solubility of the nanofibres produced, and thus improve their usability. This improvement is particularly important for their potential applications as air filters, or medical devices such as wound dressings or fibre systems for controlled drug delivery, etc.

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Predicting the Structural Changes of Textile Materials Appearing in the Household Tumble-Drying Process Using the XRD Analytical Method

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The majority of EU28 households own a tumble dryer, which they use 2.05 times/week [1]. In addition to significant energy consumption and greenhouse gas emissions, the household drying process is also a source of other pollutants, such as wastewaters and microfibres, which are released into the air during and after the drying process and contaminate the household environment. The fact is that existing household textile tumble-drying procedures are based on intensive, and sometimes even aggressive, mechanical actions, such as beating, falling and rubbing of the fabric itself, or with the metal surface of the perforated drying drum. These factors can cause damage down to the molecular scale of fabrics, and, thus, consequently, shorten the lifespan of household textiles, and, at the same time, increase textile waste. One of the research phases of the "Low emission household tumble-drying with an evaluation of damage to textile materials" project was dedicated to investigating and evaluating changes at the fibres' molecular scale after increased dying cycles with the help of the X-ray diffraction (XRD) analytical method. The research was focused on the most used household fabrics, such as cotton (CO), polyester (PES), a PES/CO blend and viscose (CV).

The process, simulating different wetting/drying procedures, was designed as 25 cycles (imitating the household textile care) of repeated sample wetting and drying procedures. The wetting process was conducted by an impregnation padder. After the wetting, the standardised fabrics were exposed to different drying procedures: photothermal, microwave and hot air drying. The XRD method is used for investigations of solid crystalline samples (identification and quantitative analysis of the degree of crystallinity, crystal lattice dimensions and crystal sizes), whereby the study is based on the coherent scattering of X-rays by electrons in atoms. Data from the XRD analysis were used to determine the changes at the macromolecular level of the fibres, mainly their arranged crystalline and disordered amorphous regions. The ratio between these regions can give us information regarding the structural damages in the textile material, occurring during the household tumble drying. In all samples, regardless of their fibre composition, we could observe that the photothermal and microwave drying processes did not influence the ratio between the crystalline and amorphous domains in the fibres significantly. However, the application of hot air drying, performed at 80°C, did show crystalline/amorphous ratio changes above the Standard Deviation, calculated from the raw, photothermal and microwave values. In the case of PES/CO, PAN, WO, SE, and CV changes in the crystallinity were detected, while, in the case of the PA and CO samples, the crystallinity value was in the area of Standard Deviation.

It was also expected that the drying process using hot air would not influence the highly crystalline structure characteristic for PA fibres (51.3%), as well as a cotton sample (32.9%) resistant to heat at 80°C, due to its cellulose crystalline structure The major change in crystallinity was observed in the wool and silk samples, known as materials with low resistance to high heat treatments. In the case of wool, we observed the increase in crystallinity, which can be attributed to the shrinkage and felting of wool fibres during the wetting. In the silk sample, we observed a drastic decrease in crystallinity, from 27.8% in the raw sample, to 8.5% in the hot air-dried sample. Silk and wool fibres are known for their high water and moisture absorbency and shrinking ability, and, therefore, the obtained results were expected.

According to the results obtained during the study, we can conclude that the XRD method shows potential in predicting the structural changes appearing in the tumble drying process of textile materials.

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The Analysis of Epinephrine, Uric Acid, and Methyl Parathion Using Screen-Printed Carbon Electrodes

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This work demonstrates the development of electro-analytical methods for the determination of epinephrine (EP), uric acid (UA), and methyl parathion (MP) using unmodified and modified screen-printed carbon electrodes (SPCE) in real samples, such as drugs, human urine and drinking water.

Quantitative analysis of EP, UA and MP in different samples is crucial, as both high and low mass concentrations (γ) can have an impact on human health [1]. Accurate and precise measurement of these compounds is essential to ensure pharmaceuticals' safety and efficacy, and monitor their impact on the environment [1-3]. Abnormal γ of these compounds can lead to significant health risks, underlining the importance of reliable analytical methods for their detection and quantification [3]. The SPCEs offer fast analysis, low costs and the ability to perform on-site analysis without extensive sample preparation [2].

Individual analysis of EP and UA was performed using an unmodified SPCE (BVT Technologies, Brno, Czech Republic) and a single-drop analysis, where only one drop of 50 µL was required. Simultaneous analysis of EP and UA was performed using a poly-L-cysteine-modified SPCE (pLC-SPCE). An L-cysteic acid-modified SPCE (LCA-SPCE) was used to determine the MP. All three sensors were validated and used for real sample analysis without any sample pretreatment. The γ of EP in an EP-auto-injector and UA in human urine was determined using unmodified SPCE and pLC-SPCE. The γ of MP in drinking water was determined with LCA-SPCE.

The γ of EP in the EP auto-injector and UA in human urine, determined using unmodified SPCE with single-drop analysis, were 478.4 mg/L (the declared γ was 500.0 mg/L) and 316.3 mg/L (within the limits for healthy individuals), respectively. No MP was detected in the drinking water using the LCA-SPCE. Using pLC-SPCE, the γ of EP in the EP-auto-injector was 491.50 mg/L (the declared γ was 500.00 mg/L). The γ of UA in human urine for four different samples, measured with pLC-SPCE, were in the range of 123.4–582.7 mg/L. Three of these samples were within the limits for healthy individuals, while one sample was below. All the results were accurate and precise, as the average recoveries were within 80.0–120.0%, and the relative Standard Deviations were below 20.0 %. The synthesis of LCA-SPCE and pLC-SPCE included activation of SPCE in 0.1 M H_2SO_4 and electro-deposition in 0.1 M phosphate buffer solution, containing 2 mM LCA (for LCA-SPCE) [2] and 2 mM L-cysteine (for pLC-SPCE) [3]. The unmodified SPCE was activated in 0.1 M HCl [1].

The unmodified SPCE is suitable for individual analysis of EP in an EP-auto-injector and UA in human urine. The LCA-SPCE is suitable for the determination of MP in drinking water. The pLC-SPCE is suitable for the simultaneous analysis of EP in the EP-auto-injector and UA in human urine. Future prospects include expanding the range of analytes, including those currently challenging in electrochemistry, and exploring the commercialisation of these methods. Integrating portable potentiostats/galvanostats into smartphones should make electrochemical analysis more accessible and user-friendly..

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Bioactive Bacterial Nanocellulose as a Promising Strategy for Enzymatic Burn Debridement

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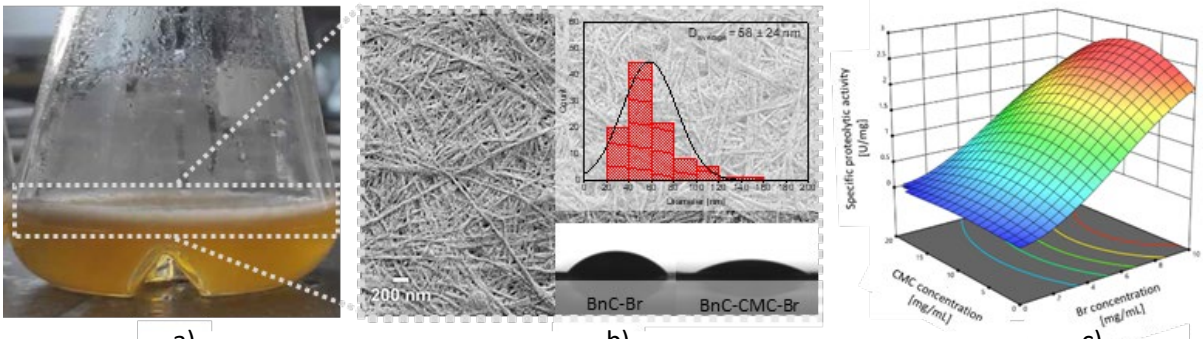
Surgical debridement (tangential excision) is today's most commonly employed method for burn eschar removal. Unfortunately, it leads to the unintended removal of viable tissue, thus prolonging wound healing time and treatment costs. In contrast, enzymatic debridement offers selective removal of non-viable tissue, thus preserving enough viable dermis in the wound bed to support spontaneous re-epithelialization and reduced need for subsequent skin grafting [1-2]. In the field of Burns, bacterial nanocellulose (BnC) stands out as an exceptional material, due to its remarkable properties (high water-holding capacity, biocompatibility, high purity level, non-toxic nature and mechanical stability [3-4]). However, its lack of biological activity, specifically in terms of its ability to break down proteins, can be overcome by immobilising specific bioactive components.

In this work, the proteolytic enzyme bromelain (Br) was immobilised within BnC membranes produced under static fermentation conditions by the addition of various concentrations of carboxymethyl cellulose (CMC) to the culture RAE media, to develop a proteolytic active wound dressing that can be used for eschar removal in burn injuries. The obtained BnC-CMC membranes were *ex situ* modified through bromelain entrapment and covalent binding, using 1-ethyl-3-(3-(dimethylamino)propyl) carbodiimide (EDC) crosslinker in the presence of N-hydroxysuccinimide (NHS). Response surface methodology (RSM) with a central composite design was employed, to achieve the highest specific proteolytic activity by investigating Br, CMC and EDC concentrations. The physicochemical, morphological, mechanical, bioactive and cytotoxic properties of the membranes were evaluated. Hyperactivation of the immobilised Br was observed under optimal immobilisation conditions (c_{CMC}=8.8 mg/mL, c_{EDC}=0 mg/mL, and c_{Br}=10 mg/mL, Fig. 1), resulting in a specific proteolytic activity of 2.3 U/mg, a more compact arrangement of fibrils with stronger affinity for water, as well as an improved water rehydration ratio and elasticity compared to an unmodified BnC membrane. The in vitro cytocompatibility was observed for the BnC-Br and non-bioactive BnC membranes after 24h exposure to normal human dermal fibroblasts. Overall, the results suggested that a combination of BnC membrane with bromelain has the potential to be used as an effective non-surgical debridement agent.



a) b) c)

Figure 1: a) BnC membrane produced over 4 days of static fermentation, **b)** SEM image of BnC-CMC membrane with the hydrophilic nature of the BnC/BnC-CMC membrane, **c)** 3D RSM plots for specific proteolytic activity of bromelain as a function of Br and CMC concentration

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Acknowledgements: *Financial support was received in the framework of the Young Researcher Programme P2-0118/0795 and Project J2-2487, both financed by the Slovenian Research and Innovation Agency (ARIS).*

PILLAR I: FUNCTIONAL TEXTILES AND COMPOSITES – ENIGNEERING APPLICATIONS

ICP-MS Elemental Analysis of Food Samples via Homogenisation and Acid Digestion using HNO_3 and H_2O_2

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This study demonstrates a protocol explaining the step-by-step process and theory for the preparation of food samples (homogenisation and acid digestion) and subsequent elemental analysis with inductively coupled plasma mass spectrometry (ICP-MS) [1]. Currently, elemental analysis is an important technique for the quality control of food samples. ICP-MS is employed frequently for this purpose, due to its advantages, such as low limit of detection (LOD) and a wide linear concentration range for various elements [2]. To analyse samples with ICP-MS, they need to be converted to a liquid state, converting metal analytes into water-soluble salts simultaneously. Thus, microwave-assisted wet acid digestion (MAWD) is used widely for the digestion of food samples, as it enables rapid digestion at high temperatures and pressure and low reagent consumption [3]. The samples need to be homogenised properly before digestion. Food samples (i.e. broccoli, mushrooms, sausages, noodles) were cut manually with a ceramic knife, and then dried in a laboratory dryer at 105 °C until a constant weight was obtained. The samples were then homogenised using a laboratory mixer (BÜCHI Mixer B-400) with ceramic blades. A mass of 250 mg of the homogenised samples was transferred to polytetrafluoroethylene (PTFE) reaction vessels, followed by the addition of 5.0 mL of 68 wt.% HNO_3 and 1.0 mL of 30 wt.% H_2O_2. The saamples were digested by MAWD using an ETHOS LEAN microwave system. The digestion protocol was: 10 min increase to 160 °C, 10 min increase to 200 °C, and 15 min at 200 °C. The digested samples were diluted with ultrapure water and filtered through polyamide syringe filters (25 mm diameter, 0.20 µm pore size). ICP-MS analysis of the samples was performed using an ICPMS-2030 (Shimadzu, Kyoto, Japan). Quantification of 4 elements (Cu, Fe, Mn, and Zn) was performed with the ICP-MS. The linear concentration range for all the elements was 1.0 µg/L to 50.0 µg/L. The determined LODs were 0.5 ng/L, 2.8 ng/L, 2.8 ng/L, and 3.2 ng/L for Mn, Cu, Fe, and Zn, respectively, while the limits of quantification were 1.6 ng/L, 9.2 ng/L, 9.5 ng/L, and 10.8 ng/L for Mn, Cu, Fe, and Zn, respectively. Broccoli contained an average concentration of 5.9 ± 0.5 µg/g (Standard Deviation), 32.5 ± 2.7 µg/g, 42.8 ± 0.2 µg/g, and 63.0 ± 1.9 µg/g of Cu, Mn, Zn, and Fe, respectively. For mushrooms, the content of Zn, Fe, Cu, and Mn was 35.6 ± 1.4 µg/g, 30.4 ± 1.3 µg/g, 18.5 ± 1.0 µg/g, and 5.4 ± 0.3 µg/g, respectively. The sausages contained 0.9 ± 0.3 µg/g of Mn, 42.2 ± 0.9 µg/g of Fe, 25.1 ± 2.6 µg/g of Zn, and 1.0 ± 0.1 µg/g of Cu. The contents of Zn, Fe, Cu, and Mn in the noodles were 5.4 ± 2.8 µg/g, 10.3 ± 1.2 µg/g, 1.6 ± 0.3 µg/g, and 7.5 ± 0.2 µg/g, respectively.

Several critical steps influenced the preparation of food samples and subsequent ICP-MS analysis. The samples need to be completely dry before weighing. Homogenisation was performed with ceramic blades to eliminate possible contamination caused by the abrasion of metal blades. High-purity chemicals were used for the MAWD. H_2O_2 was added to the HNO_3, to improve the oxidation conditions while allowing the regeneration of acid [4]. The solutions of samples were diluted with ultrapure water, to decrease the concentration of residual acid and the concentration of the component of the sample matrix before the ICP-MS analysis. Filtration was done, to eliminate possible particulates from the solution of the sample. The ICP-MS analysis was performed using a collision cell, which minimised the spectral interferences (polyatomic ions). Homogenisation with a laboratory mixer and MAWD with 5.0 mL of 68 wt.% HNO_3 and 1.0 mL of 30 wt.% H_2O_2 is an effective method for the preparation of food samples for ICP-MS analysis. By following the instructions given in the protocol, one should be able to prepare food samples for elemental analysis with ICP-MS.

With slight modifications (different homogenisation processes, the use of different acids in different ratios for acid digestion, modified temperature step), the protocol can be used for the preparation of other samples, such as inorganic samples (soil, sediments, and electronic waste).

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High-Performance Chitosan/Nanocellulose-Based Composite Membrane For Alkaline Direct Ethanol Fuel Cells

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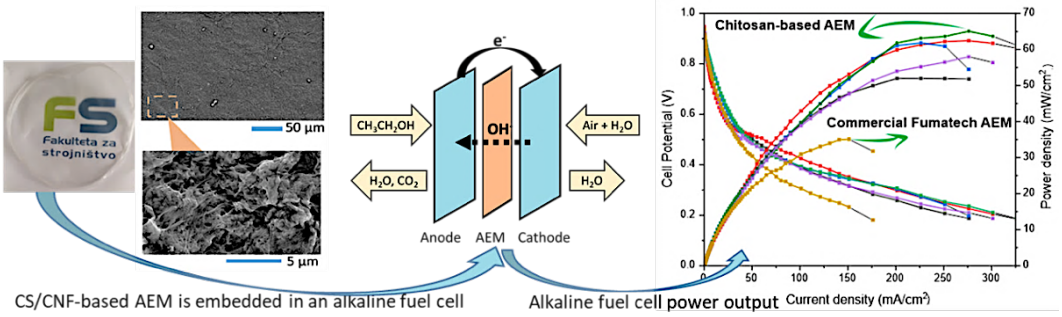
Fuel cells, such as alkaline direct ethanol fuel cells (ADEFCs), are effective energy conversion devices that have attracted increasing research interest in recent years, due to the growing demand for renewable resource technologies and the depletion of fossil fuels. [1] This work focused on the fabrication of high-performance biopolymer-based anion exchange membranes (AEMs) for ADEFCs, produced by a simple and low-cost solution casting process using green chemistry principles. The current, commercially available AEMs, the heart of a fuel cell, are inadequate, pointing to the need to develop new highly efficient, easy-to-manufacture, environmentally friendly and economically viable AEMs. Naturally derived materials such as chitosan (CS) and cellulose nanofibrils (CNF) are of great potential, as CS is insoluble at alkaline conditions, resulting in high alkali resistance. [2]–[5] The composite membranes prepared by the solvent casting method were analysed extensively for morphology, alkaline uptake, swelling ratio, ethanol permeability, mechanical properties and ionic conductivity, and tested in a single lab-scale ADEFC system. The prepared CS-based composite AEMs with CNF-based fillers were superior to the commercial Fumatech membrane in terms of Young's modulus and tensile strength (69 % and 85 % higher, respectively), ion exchange capacity (169 % higher) and ionic conductivity (228 % higher). Single fuel cell tests have shown excellent performance of the CS-based AEMs with CNF and modified CNF fillers, as they exhibited up to 86 % higher power density at 80 °C compared to the commercial membrane (65.1 mW/cm^2 vs. 35.1 mW/cm^2), and higher maximum power density at all test conditions. The data obtained indicate the applicability of the newly fabricated CS-based composite membranes as AEMs in ADEFCs. In the future durability tests would provide valuable information on the lifetime of such an ADEFC.



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Development of a Novel, Grape Waste Culture Medium and Its Influence on Bacterial Nanocellulose Properties

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Bacterial nanocellulose (BnC) represents an exopolysaccharide synthesised extracellularly by various bacterial strains, undergoing static cultivation to generate a 3D gelatinous pellicle comprising entangled cellulose nanofibre[1,2]. The cost-effectiveness of this biotechnological process is tied intricately to the culture medium selection, with the carbon source constituting approximately 30% of the overall expenditure, where the sugar-rich waste and by-products present viable alternatives to the defined media in terms of cost and availability, where diverse BnC applications [3–5] necessitate specific quality attributes influenced mainly by the biotechnological process itself.

Our work describes BnC produced by the bacterial strain *K. melomenusus* AV436^T from the microbial culture collection at the Faculty of Natural Sciences and Mathematics, University of Maribor, using grape extract (GP) as the culture medium. The GP was derived from Slovenian grapes (2021 season) through a controlled process of grape waste hydrolysis (Patent pending). The morphological examination of BnC membranes through Scanning Electron Microscopy (SEM) (Fig. 1) unveiled ribbon-like microfibrils interwoven randomly into a dense network-like structure. In the GP medium the nanofibrils were twice as wide, attributed to a "glueing" effect induced by specific components in the culturing medium. The X-ray diffraction data displayed peaks at 2θ = 14.4°, 16.8°, and 22.6°, corresponding to the (101), (10$\bar{1}$), and (002) planes of cellulose I, where the amorphous segment at 2θ ~18° was notably higher in the samples produced in the GP extract, and the crystallinity index (CI %), calculated through deconvolution, was significantly lower (< 50%) compared to 72% as in the case of the reference culture medium. It is postulated that different types of reducing sugars in the culture medium can impact the arrangement of cellulose chains. The organisation of BnC fibrils within the network facilitates stress transfer and cohesion, whereas non-cellulosic components may disrupt compactness, leading to the loosening of the BnC network. In comparison to the reinforced acetic acid-ethanol (RAE) medium, lower tensile strength and elongation were measured in the GP extract BnC.

This study highlights substantial differences in the properties of BnC membranes produced by the same bacterial strain when using different culture media, particularly in terms of morphology and crystallinity, which can be utilised in the preparation of new hybrid materials for diverse applications, uncluding, but not limited to, medicine, food, pharmacy, cosmetics, etc..

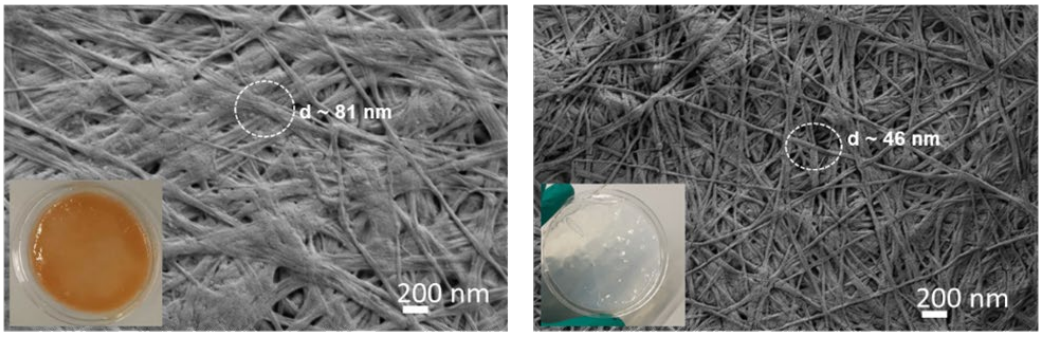

Figure 1: SEM micrographs from the BnC membrane produced by K. melomenusus AV436T, using RAE and GP extract as culture medium.

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The Role of Cellulose as an Additive in Deacidification of Books

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Degradation of paper artifacts is a cause for great concern, as the information about our cultural heritage needs to be preserved in its original form for a core of valid reasons, like national, political, legal, historical, economic, scientific and emotional.[1] The degradation of paper is caused by physical, chemical and biological factors. Endogenous factors, e.g., the pH value of the paper, are crucial determinants for paper degradation and its longevity.[1] Paper acidity arising from the addition of aluminium sulphate $Al_2(SO_4)_3 \cdot 18H_2O$ (alum), as a sizing agent in the final stages of paper making from the mid-19th century until the final decades of the 20th century, is generally accepted as a main cause of paper degradation. Consequentially, enormous effort and financial resources are put into the development of special deacidification treatments to preserve paper artefacts. The potential for such treatments is huge in Slovenia alone, as 230 km of bookshelves full of archival material exist and ≈ 80% of it was produced after 1830. The role of cellulose in the preparation of non-aqueous colloidal dispersions of alkaline particles and their role in preserving the mechanical integrity of compromised paper artefacts is presented in this work. The stability and particle size in the colloidal dispersions of alkaline particles are evaluated by dynamic light scattering (DLS) and turbidimetry. The successful application of the dispersions on model paper artefact was evaluated by chemical analysis (Attenuated total reflectance – Fourier transform infrared spectroscopy; ATR-FTIR) and penetration depth using scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDX) of paper cross-sections. Neutralisation of the paper's acidity and the added alkaline reserve were determined by standard acid-base titration methods and new protocols for pH measurement and alkaline reserve determination using an automated titrator and a phototrode for the colourimetric determination of the titration end-point. Finally, the visual appearance of the paper artefacts was evaluated by UV-VIS spectroscopy.

New perspectives of using functional polysaccharides to achieve the multifunctionality of speciality treatments for acidic paper artefacts will be presented briefly as well.

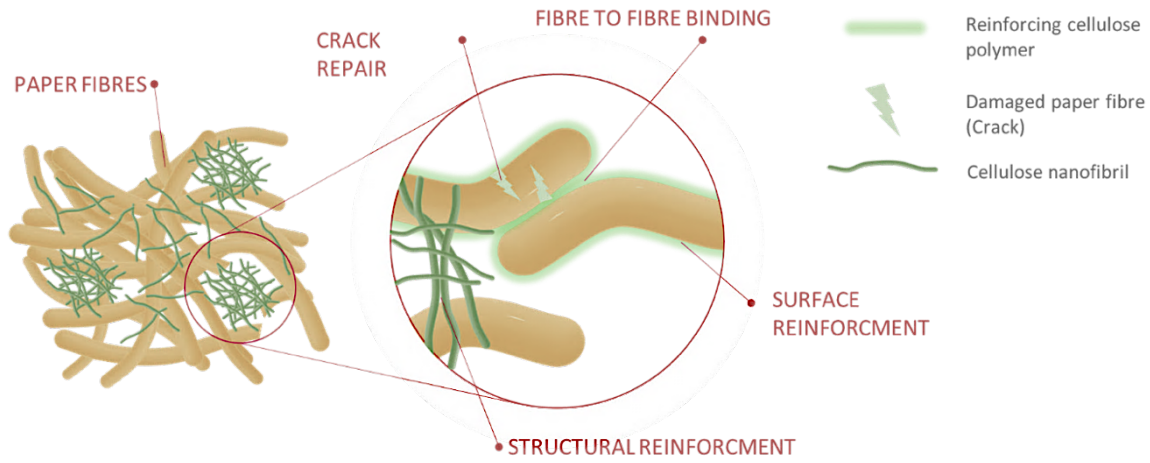

Figure 1: The many roles of cellulose in preserving the mechanical integrity of damaged paper fibres

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Zeta Potential Analysis of Functionalised Monolithic Columns for Chromatography of Biomolecules

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Chromatography is a well-known method for the separation and purification of biomolecules such as proteins, antibodies, DNA, or viruses. Unlike chromatography columns that contain particulate gels for separation, porous monolithic columns represent a promising alternative approach with superior properties and ease of handling for a scalable purification process. The homogeneity and integrity of the surface-active groups determine the applicability of such monolithic columns. Zeta potential analysis provides insight into the surface charge under various conditions and is a versatile technique for studying the surface and interfacial properties of materials. Surface charge is an essential property of chromatographic materials for optimizing their performance in various separation and purification applications [1]. Using the streaming potential method to determine the zeta potential has been employed for the characterization of functional groups of monolithic chromatographic columns used in the purification of biomolecules.

In our study, zeta potential analysis aimed at selecting appropriate chromatographic monoliths and investigating their stability in contact with an aqueous solution. With the streaming potential method, it was possible to estimate the performance of cationic monolithic columns based on the location of their isoelectric points in the alkaline pH range. The monolithic materials differ in the type of surface-conjugated ligands [2, 3] and the difference between them is observable. The strong anion exchanger shows a constant positive zeta potential of about 11 mV, while the weak anion exchanger shows a decrease in surface charge with IEP at pH about 9.3 (Figure 1). The applicability of the streaming potential method as a sensitive and robust analytical tool for selecting the appropriate chromatographic monolith for a specific purification application could be demonstrated.

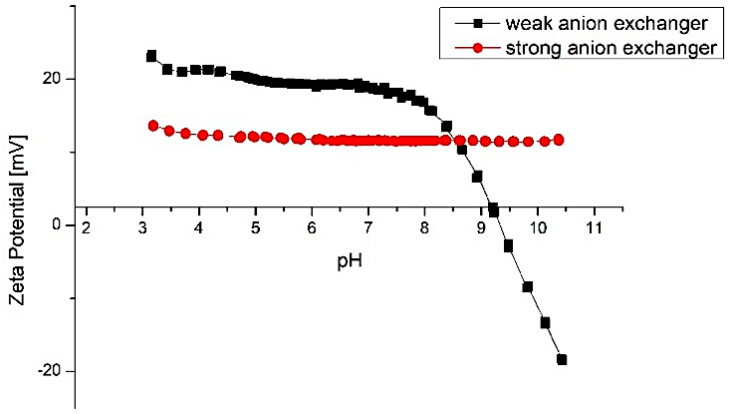

Figure 1: Surface characterisation of two different chromatographic monoliths by the zeta potential as a function of pH.

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2D Transitional Metal Carbides for Advanced Functionalisation of Fibrous Polymers - Optimisation of Synthesis Conditions

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Two-dimensional (2D) transitional metal carbides and nitrides, also known as MXenes, have gained tremendous research interest recently, due to their unique properties such as high electrical conductivity (up to 20,000 S/cm), pseudocapacitance, excellent dispersion in aqueous solutions, excellent ion intercalation behaviour, and mechanical strength and stiffness, making them ideal candidates for diverse applications, including wearable electronic textile devices.

Therefore, the main goal of the presented study was to synthesise stable $Ti_3C_2T_x$ MXenes with high capacitance by selective etching of A-element layers (the top-down approach) from the small-size (40 µm) and large-size (100 µm) MAX phase precursor for further advanced functionalisation of fibrous polymers. Herein, different process parameters were varied, i.e., time, temperature, the ratio between solvents and their concentrations. The as-prepared MXene nanosheets were characterised according to their morphology, structure, composition, flake size/distribution, chemical bond action, etc., using various analytical skills, such as Scanning Electron Microscopy (SEM), X-Ray powder Diffraction (XRD), Dynamic Light Scattering (DLS), Fourier Transform InfraRed spectroscopy (FTIR) and zeta potential measurements. Selected results are presented in Figure 1.

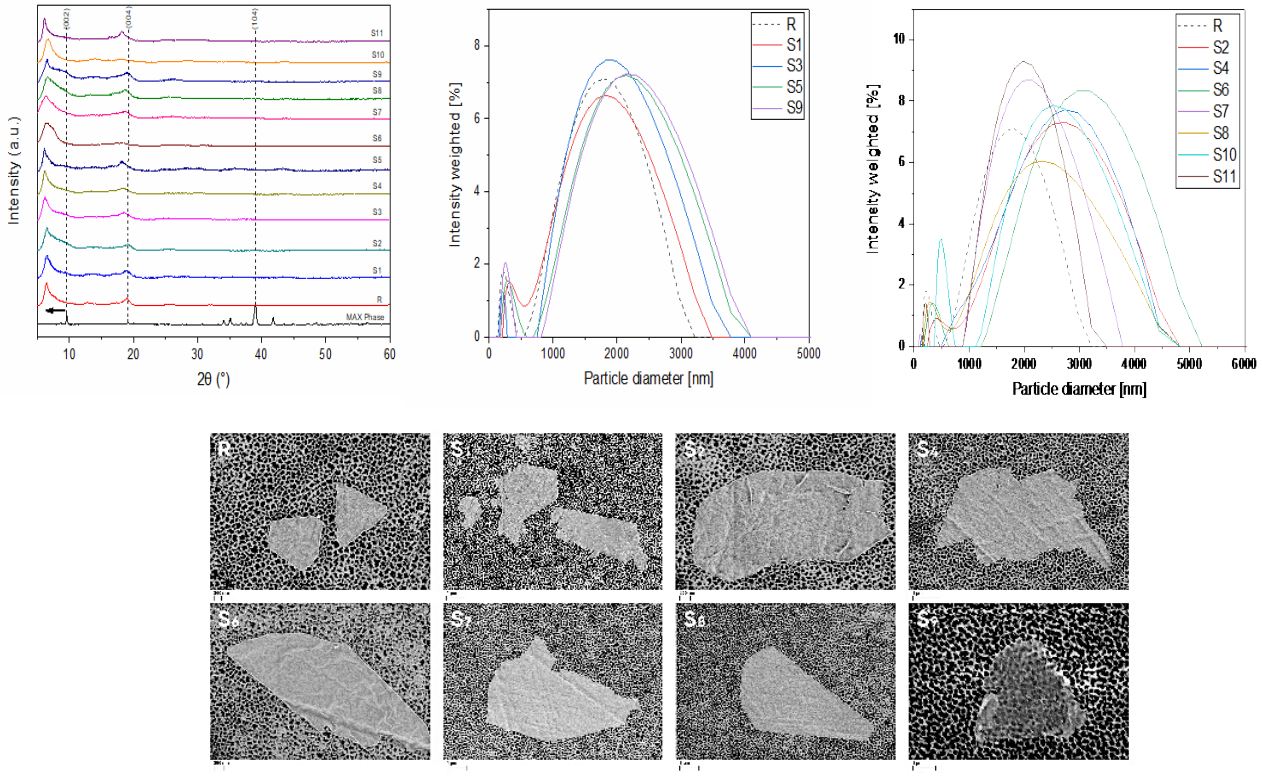

Figure 1: XRD (above-left), DLS (above-right) and SEM (below) of differently prepared MXenes.

MXenes were prepared successfully using the proposed procedure, as can be perceived from the XDR and SEM results. The process parameters influenced the size and morphology of the synthesised particles.

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Spray-Dried Lignin and Tannic Acid Particles: The Influence of Size and Composition on the Processing and Functional Properties of Biomass-Based Resins

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Urea formaldehyde (UF) and melamine urea formaldehyde (mUF) are the most used adhesive systems in European medium density fibreboard (MDF) and low-density fibreboard (LDF) production, especially in interior applications. Because of the nature of these adhesives, particularly the formaldehyde component, the re-use, incineration, recycling and land-filling of fibreboards is problematic. To tackle issues related to adhesives, there has been interest in using bio-based sources to replace fossil-based adhesives. Research into lignin, starch, tannins and plant proteins have been the main areas of interest for adhesive applications. Lignocellulosic biomass is the world's most abundant renewable material that has been recognised as a potent feedstock to produce chemicals; as one of the three main components of lignocellulosic material, wood-based polyphenolics are among the most abundant biopolymers and the largest source of aromatic building blocks on the planet, with a great potential to serve as starting material to produce biobased products. Two types of wood-derived polyphenolic bio-macromolecules were employed - tannic acid and lignin - whose capacity to form different macromolecular architectures was employed in particle design. The spray drying technique was used for particle fabrication; here, a polymer solution is atomised into fine droplets and dried in a flow of drying gas to produce dry particles, ranging from 200 nm to 1 µm. The spray dried particles were used for the preparation of epoxy thermosetting resins. Epoxy thermosets are characterised by excellent mechanical properties, high chemical and thermal resistance and low water and moisture uptake. Conventionally produced from diglycidyl ether of bisphenol A, which is a result of a reaction between epichlorohydrin with bisphenol A, and, in most cases, combined with hardeners to achieve the required properties, epoxy resins are burdened with fossil-fuel based constituents. In our work, we have used different types of lignin and tannic acid as molecules with an abundance of aromatic rings and glycerol diglycidyl ether as the carrier of epoxide moieties. Different ratios of both components were tested, as well as the addition of water to help facilitate the inclusion of water-soluble lignin (e.g. alkali lignin) and tannic acid. Fourier Transform InfraRed analysis was employed to confirm the ring-opening of the epoxide group of the glycerol diglycidyl ether, followed by the formation of covalent ether bonds with the admixed polyphenolic polymers. To quantify the rheological properties of bio-based epoxy resins prior to curing, which is important from the point of view of board manufacturing and the introduction of fibres into the adhesive resin formulations, we performed frequency sweep measurements. These are employed to describe the time-dependent behaviour of a sample, with high frequencies used to simulate fast motion on short time-scales (e.g. mixing of the fibre-adhesive formulation), while low frequencies replicate slow motion or rest (setting of the samples). All bio-based epoxy samples possess a non-crosslinked structure before curing: G'' (loss modulus) was, in all cases, higher than G' (storage modulus), but there were significant differences in the values of the moduli between samples; the tannic acid-based epoxy exceeded both lignin epoxies by exhibiting very high values of both moduli, which we ascribed to a multitude of pyrogallol catechol groups offering multiple bonding sites. The alkali lignin-based epoxy exhibited the second highest values for moduli, but, unlike the tannic acid and kraft lignin samples, these values (G' and G'') were much closer, indicating that the alkali lignin reaction with glycerol diglycidyl results in epoxy resin that, even prior to curing, possesses less of a viscous (liquid) component and a more pronounced elastic (solid) part. While not cross-

linked in the wet state, since the lines for G' and G'' never intersected, it does showcase a structure akin to a more fixed state, which is also evident from the complex viscosity results.

Temperature-dependent oscillatory rheological analysis gave further insight into the influence of particle size on the process of epoxidation; smaller particles cross-linked at a lower temperature and exhibited a higher storage modulus, showing clearly the importance of particle morphology. In the present study we have shown that wood-derived polyphenolic polymers (lignin and tannic acid) can be employed in the formation of materials with different morphologies, which can serve as adhesives in fibre composites, as well as in other technical applications. Harnessing the chemical and structural features of these polyphenolic macromolecules in material design has proved to be a viable platform for the development of bio-based wood-derived products.

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PILLAR II: SUSTAINABLE ECONOMY AND CONCEPTS

3D Printed Porous Nanocellulose-Protein Scaffolds for Tissue Engineering Applications

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Tissue Engineering has developed into a promising field of regenerative medicine that aims to develop functional replacements for damaged or diseased tissue [1]. In this context, the integration of nanomaterials in the design of scaffolds has gained considerable attention. This study investigated the application of scaffolds made of nanofibrillated cellulose (NFC), carboxymethylcellulose (CMC) and collagen (Coll), which combine the biocompatibility of collagen with the exceptional mechanical properties of NFC/CMC. The manufacturing process involves a novel approach that utilises chemical cross-linking and 3D printing technology to create biomimetic structures with enhanced structural integrity and tailored porosity. The NFC/CMC/Coll composite was produced through a careful mixing process, where the ratio was optimised to achieve an ideal balance between mechanical strength and biocompatibility [2]. Chemical cross-linking agents (CA: citric acid) were used to increase the stability and ensure the durability of the scaffold in a physiological environment. The scaffolds were produced by a combination of direct 3D printing, freeze-drying and dehydrothermal treatment methods [3,4]. The characterisation studies demonstrated the excellent mechanical properties, long-term dimensional stability and biocompatibility of the nanocellulose-collagen scaffolds. In vitro studies demonstrated the scaffold's ability to support cell viability and grow cells derived from bone tissue. The presented NFC-CMC-Coll scaffolds are promising for applications in various fields of Tissue Engineering, including the regeneration of bone or cartilage. This innovative approach, which combines polysaccharides and proteins, chemical cross-linking and 3D printing, represents a major step forward in the development of biomimetic scaffolds for advanced Tissue Engineering applications.

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PILLAR II: SUSTAINABLE ECONOMY AND CONCEPTS

Significant Fragmentation of Disposable Surgical Masks - An Enormous Source for Problematic Micro/Nanoplastics` Pollution in the Environment

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The pandemic of the COVID-19 disease has brought many challenges in the field of Personal Protective Equipment. The amount of disposable surgical masks (DSMs) consumed increased dramatically, and much of it was disposed of improperly, i.e., it entered the environment [1-4]. For this reason, it is crucial to analyse the waste and identify all the hazards it poses accurately. Therefore, in the present work, a DSM was disassembled, and gravimetric analysis of the representative DSM waste was performed, along with detailed Fourier-transformed infrared spectroscopy of the individual parts and an in-depth analysis of the waste. Due to the potential water contamination by micro/nanoplastics, and also by other harmful components of DSMs generated during the leaching and photodegradation process, the artificial weathering by Xenontest alpha and toxicity characteristic leaching procedure (TCLP) were used to analyse and evaluate the leaching of the micro/nanoplastics. The micro/nanoplastic particles were leached from all five components of the mask in an aqueous medium, as shown in Figure 1. When exposed to artificial weathering conditions, a DSM loses up to 30% of its mass in just 1 month, while micro/nanoplastic particles are formed by the process of photodegradation. Improperly treated DSMs pose a significant hazardous risk to the environment, due to the release of micro/nanoparticles and chloride ion content.

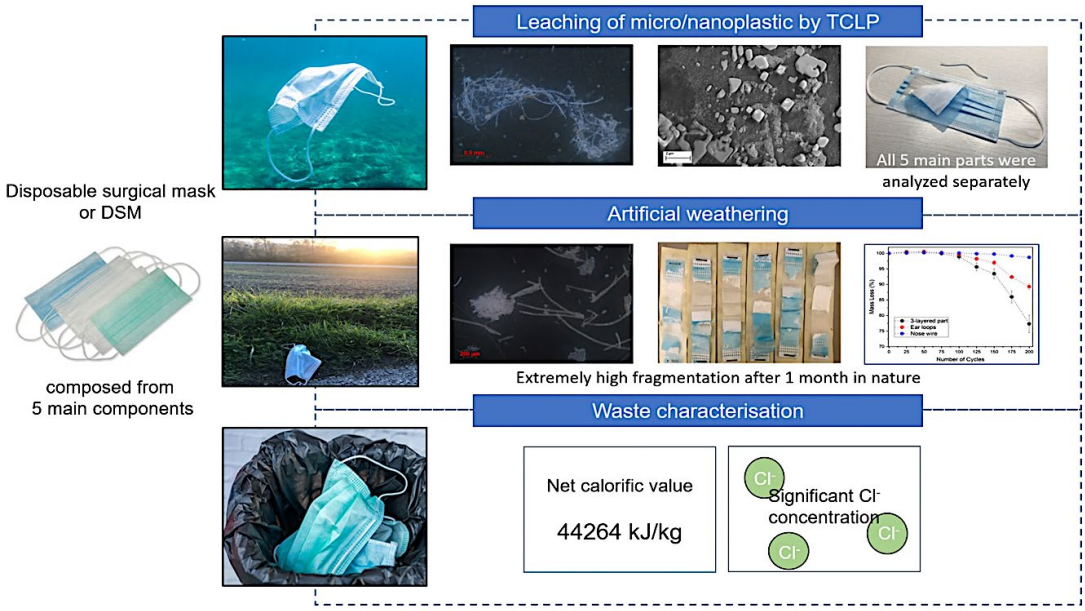

Figure 2: Analysis of the negative environmental impact of waste DSMs

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PILLAR II: SUSTAINABLE ECONOMY AND CONCEPTS

The Morphological, Surface and Thermal Properties of Polylactic Acid Foils, Melamine-Etherified Resin, and Polyethylene Terephthalate Fabric During (Bio)Degradation in Soil

Olivija Plohl,[1] Alen Erjavec,[1] Lidija Fras Zemljič,[1] Alenka Vesel,[3] Maja Čolnik,[2] Mojca Škerget,[2] Yee Van Fan,[5] Lidija Čuček,[2] Gregor Trimmel,[4] Julija Volmajer Valh [1]

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The (bio)degradation of plastics in soil is a complex process that depends on several factors, such as the type of plastic, environmental conditions, microbial activity, and the presence of other organic matter in the soil. Ageing in soil thus also influences the physicochemical properties of plastic materials. To understand the mechanisms and investigate the changes in plastic properties in soil due to decomposition followed by fragmentation, the gravimetric, morphological, surface and thermal properties of plastics were studied, some of them for the first time [1]. The study was performed for three different plastic materials, polyethylene terephthalate (PET-fib) and melamine etherified resin (MER-fib) in the form of nonwoven fabrics and polylactic acid (PLA) in the form of foils. The materials were exposed to soil for one, three and six months, and, only in the case of PLA foil, for a final four months. The overall concept of this study is presented schematically in Figure 1. The results show that remarkable changes were observed, especially for MER-fib and PLA after exposure to soil, which is related to the bio and chemical degradation processes. The biodegradation process was indicated with the soil microorganisms used in the study (lactic acid bacteria, photosynthetic organisms, yeasts, actinomycetes, and enzymatically active fungi), while the chemical degradation showed that it may occur at the surface of the material, with changes in the elemental composition and chemical functionality. The microbial end products of the biodegradation of MER-fib are presumably NH_3 and CO_2, while, for PET-fib it is CO_2 and for PLA it is CO_2 and H_2O, including several proposed conversion products in partial pathways. The study represents an important contribution to understanding the behaviour of the analysed (bio)plastics and the changes in their properties after exposure to natural systems for pollution countermeasures and cleaner production.

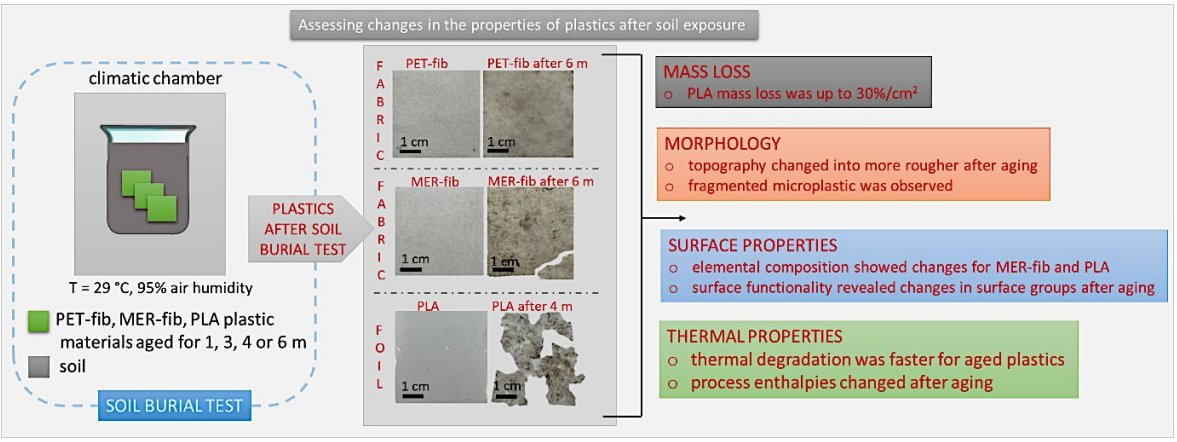

Figure 1: The overall concept of the study

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PILLAR II: SUSTAINABLE ECONOMY AND CONCEPTS

Residues of Pharmaceuticals in Drinking and Wastewater

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Pharmaceuticals are synthetic or natural chemicals in prescription, over-the-counter and veterinary drugs. In recent decades, traces of pharmaceutical products have appeared in the water cycle, including surface water, wastewater, groundwater, and, to a lesser extent, in drinking water. The advances in analytical technology have been a key factor driving their increased detection. Even at low concentrations, their presence in water has raised concerns among the professional public regarding possible risks to human health. Our research aimed to analyse data on the consumption of prescribed medicines in hospitals and outpatient clinics in Slovenia. We also took samples of drinking and wastewater, to determine the presence of selected pharmaceuticals.

We used descriptive statistics to analyse the data on the consumption of medicines. The drinking and wastewater samples were taken at the Central Water Treatment Plant Ptuj following the ISO 5667 1 and ISO 5667 3 Standards. The samples were analysed according to the EPA Method 1694 Pharmaceuticals and Personal Care Products in Water, Soil, Sediment, and Biosolids by HPLC/MS/MS, EPA 821 R 08 002 (December 2007), using the method of LC/MS/MS and LC/HRMS (orbitrap). We determined the presence of 68 pharmaceuticals in the drinking water and 59 pharmaceuticals in the wastewater.

The analysis of the drug consumption data showed that rosuvastatin, which belongs to drugs for changing serum lipid levels, was the most frequently prescribed outpatient drug. In contrast, in hospitals, vitamin D and its analogues were prescribed most frequently. In the wastewater the following pharmaceuticals were detected: Paracetamol (0.035 mg/L), Caffeine (0.043 mg/L), Naproxen (0.018 mg/L), 1-H benzotriazole (0.020 mg/L). In the drinking water samples, all 68 investigated pharmaceuticals were below the limit of quantification (< LOQ). Although the current published risk assessments indicate that trace concentrations of pharmaceuticals in drinking water pose a low risk to humans, there are gaps in knowledge regarding the evaluation of risks associated with long-term exposure to low concentrations of pharmaceuticals and combinations of different drug mixtures.

Future research in these areas could be helpful to characterise the potential health risks of long-term, low-level exposure to pharmaceutical products better, especially for sensitive sub populations. One of the key challenges in assessing exposure to pharmaceutical products through drinking and wastewater is assessing possible risks to human health. It is also crucial to study new possibilities for wastewater treatment procedures, which we want to investigate in the continuation of the research.

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The Influence of Magnetic-Based Nanoparticles on the Efficacy of a Carboxymethyl Dextran Coating as a Prospective Modifier for Electrochemical Sensors

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The escalation of antibiotic discharge into wastewater has emerged as a pressing contemporary public concern. Consequently, there is a critical need for electrochemical sensors, incorporating precisely and controllably designed nanomaterials to enhance the detection of antibiotics in contaminated environments. While numerous nanomaterials have been explored for their heightened sensitivity and unique functionalities, conventional modifications of the working electrode surface involve primarily materials like graphene, carbon nanotubes, metals and conductive polymers. Challenges such as expensive synthesis routes, challenging removal of metal catalysts, hydrophobicity, agglomeration issues and induced defects have limited the promise of these nanomaterials in designing highly efficient modified electrochemical sensors [1,2]. In contrast, the utilisation of magnetic nanoparticles (MNPs) presents a promising avenue, due to their controlled synthesis conditions and subsequent functionalisation with biopolymers. This approach offers advantages, such as a large surface area, good electron conductivity, and increased electroactive interaction sites. However, there are still considerable gaps in research in this area, particularly in the preparation of MNPs functionalised with polysaccharides with specific properties that are intended to serve as modifiers for electrochemical sensors. This study aimed to fabricate magnetic iron oxide nanoparticles (MNPs) functionalised with carboxymethyl dextran (CMD), a polysaccharide with specific functional target groups. The objective was to use this modified material as a potential electrochemical sensor modifier, ultimately enhancing the electrochemical sensing capabilities. Two distinct modification procedures were employed to evaluate the impact of magnetic-based nanoparticles on CMD coating efficiency. Firstly, MNPs were synthesised through hydrothermal synthesis using iron sulphate salts, followed by stabilisation with citric acid, and, further, coated with a roughly 2 nm thick porous silica (SiO_2) layer, to augment the effective specific surface area through porosity. Subsequently, the MNPs were functionalised with aminopropyl-triethoxysilane (APS) to introduce -NH_2 groups, ensuring a positive charge under moderately acidic conditions. The biopolymer CMD was then attached electrostatically to the MNPs@SiO_2@APS (termed MNPs@SiO_2@APS-CMD). In the second procedure, the MNPs were silanised directly with APS, followed by CMD functionalisation (termed MNPs@APS-CMD). The resulting nanocomposites underwent characterisation through transmission electron microscopy (TEM) to assess their morphology, size and the success of the silica coating. Infrared spectroscopy, zeta potential electro-kinetic measurements, and X-ray photoelectron spectroscopy (XPS) were employed for further characterisation, along with thermogravimetric and magnetic property assessments. The results of two different MNPs` functionalisation approaches demonstrated the successful formation of all layers on the MNPs, with the final CMD layer exhibiting functional groups (i.e., hydroxyl and carboxyl groups) that can improve the adsorption of antibiotics, and, consequently, a better electrochemical response. This outcome holds promise for the potential application of CMD-functionalised MNPs as effective modifiers for electrochemical sensors targeting antibiotics.

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Book of Scientific Research Achievements of Program Group Textile Chemistry and Advanced Textile Materials: P2-0118
1ST Annual Meeting 2024

Tatjana Kreže, Lidija Fras Zemljič (eds.)
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The publication presents an overview of the research achievements of Program Group P2-0118 Textile chemistry and advanced textile materials from 2021 to 2023 in the field of development of smart and multifunctional textiles and composite materials as well as circular economy concepts. Members of the program group present their research innovations and progress achieved within the framework of the program group's activities.

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ZBORNIK ZNANSTVENORAZISKOVALNIH DOSEŽKOV PROGRAMSKE SKUPINE TEKSTILNA KEMIJA IN NAPREDNI TEKSTILNI MATERIALI: P2-0118

1. LETNO SREČANJE 2024

TATJANA KREŽE, LIDIJA FRAS ZEMLJIČ (ED.)
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Ključne besede:
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nanotehnologija,
biotehnologija,
površinska funkcionalizacija in modifikacija,
krožno gospodarstvo

Publikacija predstavlja pregled raziskovalnih dosežkov Programske skupine P2-0118 Tekstilna kemija in napredni tekstilni materiali od leta 2021 do 2023 s področja razvoja pametnih in večfunkcionalnih tekstilij ter kompozitnih materialov kot tudi konceptov krožnega gospodarstva. Člani programske skupine predstavljajo svoje raziskovalne novosti in napredek, dosežen v okviru delovanja programske skupine.

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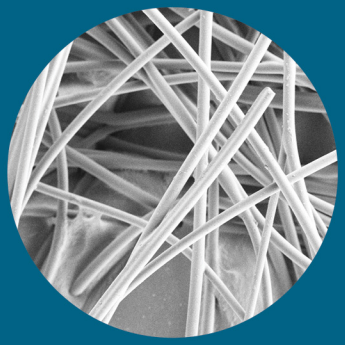

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