CEEC-PCMS2

BOOK OF ABSTRACTS

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2nd Central and Eastern European Conference on Physical Chemistry and Materials Science

16-19 September 2024
Kaunas, Lithuania

The 2nd Central and Eastern European Conference on Physical Chemistry & Material Science

Book of abstracts

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In Central and Eastern Europe, the field of Physical Chemistry has a long history and tradition; on the other side, during the last 3 decades, the field of Materials Science has garnered huge attention from the large public and became a hot topic for the scientific world. Therefore, the scientists belonging to these two communities, which usually are overlapping to a high extent, have now the possibility together to attend symposia "Central and Eastern European Conference on Physical Chemistry and Materials Science (CEEC-PCMS)".

CEEC-PCMS serves as a platform for researchers to convene, showcase their work, elucidate their findings, and engage in discussions about the scientific and technical challenges encountered in both theoretical and applied physical chemistry. The primary objective is to foster a comprehensive understanding of and contribute to the development of innovative and practical materials.

Every two years, CEEC-PCMS aims to gather scientists from Central and Eastern Europe, mainly, but not limited, from the area described by the following countries: Germany and Switzerland to Kazakhstan, and from Scandinavia & the Baltic countries to the Balkans, Turkey & the Caucasians. The previous CEEC-PCMS meeting was held in Split – Croatia (July 2022) and gathered more than 300 participants.

Our conference extends a warm invitation to scientists worldwide engaged in Physical Chemistry and Materials Science, encouraging the sharing of experiences and knowledge with peers in these fields or related disciplines. In addition to the standard symposia, the program includes interactive working sessions (workshops) designed to create a relaxed and informal space for participants to discuss their plans and requirements. This unique format provides a significant opportunity to unite distinguished researchers from Central & Eastern European countries with their global counterparts, fostering the establishment of new connections and encouraging collaborative efforts.

The 2nd Central and Eastern European Conference on Physical Chemistry & Materials Science (CEEC-PCMS2) is based on 30 topics covering the most important areas of Physical Chemistry and Materials Science.

For the venue of the CEEC-PCMS2 Conference (16–19 of September 2024) we have chosen the city of Kaunas, which is the second largest city in Lithuania.

Plenary lectures

THERMODYNAMIC STABILITY OF CRYSTALLINE CALCIUM SILICATE HYDRATES

Barbara Lothenbach[1*], Tamino Hirsch[2]

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The phase diagram of the CaO–SiO$_2$–H$_2$O system between 100 and 250 °C under saturated steam pressure has been revised based on experimentally determined solubility data, observed phase relations considering the kinetic effects resulting in a consistent thermodynamic dataset for crystalline calcium silicate phases as described in detail in [1]. The dataset includes revised thermodynamic properties for 17 calcium silicate phases which form at high temperature, including several phases (Z-phase, kenotobermorite, kilchoanite, pavlovskite and raunhardbraunsite) for which no thermodynamic properties had been available. In addition, thermodynamic properties for hydroxylellestadite, scawtite and spurrite are deduced as those phases occur in autoclaved concrete.

The phase diagrams constructed for the system CaO–SiO$_2$–H$_2$O based on these thermodynamic data indicates that under equilibrium conditions portlandite dominates together with hillebrandite, xonotlite and low-quartz the phase assemblage up to 250 °C. If the formation of the hillebrandite, xonotlite and low-quartz is delayed by kinetic reasons, numerous metastable phases can occur as described in detail in [1]. The presence of minor elements can affect the phases formed, e.g. aluminium promotes the formation of Al-tobermorite destabilizing other calcium silicate phases such as xonotlite [2,3].

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VALORIZATION OF RENEWABLE AND WASTE RESOURCES FOR THE DEVELOPMENT OF HIGHLY FUNCTIONAL SOLUTIONS FOR ADVANCED FIELDS

Paola Fabbri[1,2*], Micaela Degli Esposti[1,2], Davide Morselli[1,2]

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New sustainable solutions for the plastic sector, either bio-based/durable or bio-based/biodegradable, have been intensely investigated in recent years, with the focus almost exclusively kept on the polymer itself. For a long period, new compounds were formulated using standard additives in combination with bio-based polymers, but the new trend is definitely to pursue a strategy fully based on renewable resources, in which bio-based additives are purposely developed to expand the potential and reach of biopolymers. We have demonstrated that novel bioplasticizers synthesised from levulinic acid does not significantly affect the typical biodegradability and biocompatibility of poly(hydroxyalkanoate)s, poly(lactic acid) and others, showing a remarkable plasticization effect, comparable to the best fossil-based commercially available solutions[1]. Glycerol trilevulinate plasticizer produces a significant plasticization effect on both amorphous and semicrystalline polymers and it undergoes enzyme-mediated hydrolysis to its initial constituents, envisioning a promising prospective for environmental safety and upcycling[2]. We also studied wood tannins as fully bio-based biodegradadable multifunctional additives for bioplastics, demonstrating their antioxidant, UV protection, and gas barrier properties on PHB[3]. The presented outcomes show that fully bio-based additives offer new strategies to obtain attractive alternatives to petroleum-based plastic solutions.

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SOL-GEL SYNTHESIS OF GARNET STRUCTURE COMPOUNDS

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Garnet structure compounds are renowned for their exceptional properties, rendering them indispensable in a multitude of applications, including luminescence, magnetism, and laser technology. Despite their established utility, ongoing research endeavors aim to further optimize and enhance these materials for specific applications. This presentation examines the sol-gel synthesis of garnet compounds, with a particular emphasis on recent advancements in structural modification, co-doping strategies, and variations in doping concentrations, with the aim of achieving tailored properties.

The study employs yttrium aluminum garnets (YAGs) as the fundamental matrix and examines the consequences of substituting Y and/or Al with elements such as Lu, La, Gd, Ca, Ga, Ta and/or Sc. The aqueous sol-gel method has been demonstrated to be an effective approach for the synthesis of single-phase garnets. Moreover, the application of annealing at temperatures exceeding 1000 °C has been demonstrated to effectively reduce crystal lattice defects and enhance luminescence properties [1,2].

The presentation will illustrate the luminescence characteristics of with europium, terbium, cerium, and/or cerium doped garnets [1,3–6]. Furthermore, the investigation encompasses cerium-doped yttrium aluminum garnet (YAG) and lutetium aluminum garnet (LuAG) for potential use in scintillator applications. The objective of incorporating boron is to reduce the decay times and enhance the radiation absorption. Samples synthesized via the sol-gel method and doped with 0.05% cerium and varying boron concentrations demonstrate that boron enhances emission intensity and shortens decay times, particularly in samples with 1% and 5% boron [7].

This presentation highlights the effectiveness of the sol-gel synthesis method in producing high-performance garnet materials and explores the potential of diverse doping strategies to further enhance their properties.

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NEW HORIZONS FOR FERROELECTRIC MATERIALS

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Since its discovery more than 100 years ago, ferroelectricity has been a topic a great intellectual interest, and since the middle of the 20th century also of great technological importance. Ferroelectrics have a myriad of applications which exploit their high relative permittivity and also electromechanical properties for a variety of devices including sensors, actuators and memory devices. However, their single biggest volumetric application is in *ca.* 2 trillion multilayer ceramic capacitors produced annually; this makes them are one of the cornerstone materials for consumer electronics. More recently, ferroelectrics research has diversified with the aim of developing both new materials and novel functionality for applications in e.g. nanoelectronics, neuromorphic computing, and piezocatalysis.

In my talk I will touch on a few of these areas to highlight current research and opportunities for the field before discussing some specific examples from our own research activity in the areas of: improper and geometric ferroelectrics for domain wall nanoelectronics; and also 'antiferroelectric' materials for high-field capacitor applications.

SYNERGISTIC DFT AND EXPERIMENTAL STUDIES FOR THE DESIGN OF NANOTUBULAR ALUMINOSILICATES BASED CATALYSTS

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Within the framework of a green transition and a sustainable societal development, the valorization of biomass is one of the most important and challenging topics to handle. Although research has made enormous progress in recent years, today many questions remain open and the reactions yields and efficiencies must be improved through the design and optimization of novel catalysts. Nanotubular aluminosilicates hold a great importance due to their main features (e.g. chemical surfaces and charges, high aspect ratio, low price) which make them promising platforms to be exploited as nanosupports. For this reason, the alkaline activation of Halloysite Nanotubes (HNTs) was studied to enlighten the formation of silanol groups on the external silicic surface. Computational studies, conducted by a cluster approach within density functional theory, provided atomistic details about the formation and the configuration of these active groups which can act as anchoring sites for the attachment of organic molecules.[1] Experimental analysis corroborated the activation of the nanoclay and the efficient functionalization with aminosilanes, such as APTES and AEAPTMS.[2] The presence of nitrogen centers was then used for the decoration of the modified HNTs with gold atoms and clusters, resulting in the formation of an active catalyst for the oxidation of 5-hydroxymethylfurfural (HMF) to furandicarboxylic acid (FDCA), as investigated by ONIOM calculations.[3] Moreover, the modification of the inner aluminum based surface of halloysite by acidic treatment was also focused to have more details about the appearance of acidic sites embedded in the clay nanostructure, which are crucial for the dehydration of fructose to HMF. These results pave the ground for the development of halloysite based materials to be exploited in catalytic processes for the conversion of biomass, through the design of a one-pot multifunctional nanocatalyst with low impact on the environment.

Acknowledgements. This work was supported by “EU - FESR e FSE, PON Ricerca e Innovazione 2014-2020 - DM 1062/2021” and the PNRR SAMOTHRACE.

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NATURE-INSPIRED 3D HYBRID MICRO-NANO-ARCHITECTURES FOR MULTIFUNCTIONAL APPLICATIONS

Ion Tiginyanu[1,2] and Tudor Braniste[2]

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We report on a novel nature-inspired three-dimensional nanoarchitecture of GaN, called aero-GaN or Aerogalnite, which represents the first artificial material with dual hydrophobic-hydrophilic behaviour (see https://en.wikipedia.org/wiki/Aerogalnite) and exhibits properties close to those inherent to a biological cell membrane. The 3D nanoarchitecture is based on GaN hollow micro-tetrapods with nanoscale thin walls, the inner surface being covered by an ultrathin film of zinc oxide. The hollow micro-tetrapods are shown to self-organize when interacting with water, forming self-healing waterproof rafts exhibiting high elasticity and impressive cargo capabilities. Self-propelled liquid marbles based on GaN hollow micro-tetrapods are demonstrated and their rectilinear movement and rotation, including rotation in pulses, are disclosed. Results of characterization of other aero-materials including aero-ZnS, aero-Ga2O3 and aero-TiO2 will be presented and discussed in the context of prospects for their applications in various fields. This work was supported by the Ministry of Education and Research from the Republic of Moldova under the subprogram 020402 ETISEL ”Development of technologies and investigation of the properties of layered semiconductor compounds, hybrid nanostructures and laser sources”.

Invited lectures

EFFECT OF ZINC ON THE PROPERTIES OF POTLAND CEMENT

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In recent years, the amount of zinc in cement and subsequently in concrete has been constantly increasing, mainly due to the use of alternative fuels and the large use of secondary raw materials containing zinc. The presence of zinc in cement significantly prolongs hydration. The aim of this work is primarily to eliminate the negative effect of zinc on cement hydration. Eliminating this effect in the future will allow the use of cement with an increased zinc content. The course of hydration was monitored using isothermal and isoperibolic calorimetry. The formation of new products and phases was monitored using differential thermal analysis and X-ray diffraction. At the same time, the morphology was studied using scanning electron microscopy with EDS microanalysis. Based on the obtained results, the possibilities of elimination of negative zinc influence using appropriately chosen hydration accelerators were found. It is precisely thanks to appropriately chosen accelerators that it is possible to obtain a material with a similar hydration time to ordinary Portland cement, but with better mechanical properties even with a higher zinc content in the mixture.

Acknowledgements. This work was financially supported by the project GA-16646S "The elimination of the negative impact of zinc in Portland cement by accelerating concrete admixtures", with financial support from the Czech Science Foundation.

“ALL-BIO” POLYMERIC MATERIALS REINFORCED BY NATURAL (NANO)FIBRES

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Current research trends in the area of new materials include the design and preparation of polymer composites taking into account the need to reduce the consumption of non-renewable resources and environmental protection requirements, including ease of recycling. The solutions sought include "all-bio" systems that use polymers synthesized from monomers obtained from renewable raw materials, such as biopolyamides, as well as biofillers. Among the latter, an important place is occupied by cellulose, a common natural polymer which, when used as an additive, also with nanometric dimensions, can beneficially improve the properties of the polymer matrix. However, when manufacturing polymer composites modified with natural fillers, their thermal resistance is insufficient and degradation occurs under melt processing conditions. The selection of the matrix-fiber system plays an important role, also to ensure proper adhesion of the components and to prevent (or minimize) the effects of agglomeration during classic processing (extrusion, injection) or using additive technologies. In this context, the appropriate method of obtaining and modifying cellulose becomes crucial. Applied methods of modifying cellulose nanofibers (CNF), including e.g. esterification with acid anhydrides, carboxymethylation or silanization lead to increased compatibility with the polymer matrix and improved thermal stability to enable high-temperature processing. Importantly, for industrial applications, it is required to increase the scale of production of bio(nano)fillers and ensure high quality/repeatability of the characteristics of biomaterials.

The lecture will present the most important aspects of the technology of biopolymer composites modified with natural fibers and the prospects for further development of this promising group of polymeric materials.

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COORDINATION POLYMERS WITH PYRIDINECARBOXYLATE LIGANDS – PREPARATION, CRYSTAL STRUCTURES AND PROPERTIES

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Coordination polymers (CP) are infinite systems constructed by metal ions and organic ligands as main building units. These units are linked together by covalent bonds, resulting in coordination polymers propagation in one, two or three dimensions. Various functional properties of these compounds enable many possible applications e. g. in catalysis, gas and energy storage, gas separation, magnetism, luminescence, molecular sensing and biomedical imaging.

Pyridinecarboxylate ligands, such as pyridine-3-carboxylate (nicotinate) and its derivatives with electron-donating or electron-withdrawing substituents at various spatial positions, are useful candidates for designing coordination polymers of various dimensionalities [1-4], including heterometallic coordination polymers e. g. sodium-palladium(II) coordination networks [2].

The examples of 1D coordination polymers (e. g. cobalt(II) polymer with 2-chloronicotinate and 4,4'-bipyridine as mixed ligands [1]) and 2D coordination networks (e. g. sodium-palladium(II) network with 2-chloronicotinate [2]) (Fig. 1.) will be presented and discussed.

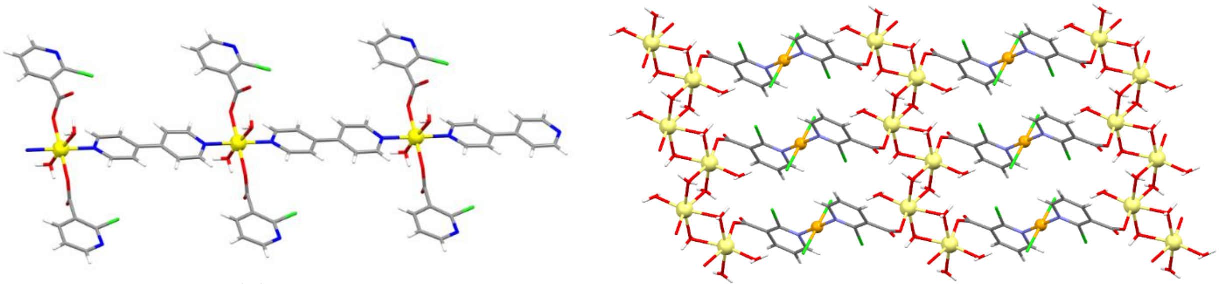

Fig. 1. The 1D cobalt(II) coordination polymer with 2-chloronicotinate and 4,4'-bipyridine ligands [1]; the 2D heterometallic sodium-palladium(II) coordination network with 2-chloronicotinate ligand [2]

The 1D cobalt(II) coordination polymer with 6-aminonicotinate and 1,2-bis(4-pyridyl)ethane may be used as a new electrochemical sensor. Therefore, its electroanalytical application in determination of important analytes (dopamine) will be mentioned [3]. Finally, a different electrochemical behavior of the isostructural 1D cobalt(II) or nickel(II) coordination polymers with 6-oxonicotinate and 4,4'-bipyridine, observed by cyclic voltammetry, will be discussed [4].

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BROAD BAND FREQUENCY MEASUREMENTS OF ADVANCED MATERIALS

Juras Banys*, Saulius Rudys[1], Vidmantas Kalendra[1], Šarūnas Svirskas[1],
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The dielectric response of materials provides information about the orientational adjustment of dipoles and the translational adjustment of mobile charges present in a dielectric medium in response to an applied electric field. Microwave and terahertz dielectric spectroscopy of ferroelectrics and related materials enables the independent determination of the dielectric permittivity and loss in the dispersion region, as well as the parameters of the soft modes related to phase transitions. Besides scientific purposes, microwave dielectric measurements are of increasing importance in telecommunications-related applications and the design of microwave circuit components. These applications include imaging radars, guidance systems, surveillance and secure communications. The magnetic properties are also of crucial importance. Dielectric and magnetic parameters fully characterize the manner in which electromagnetic waves propagate within the medium. The difficulties of making measurements on a wide range of materials over a wide frequency (and temperature) range have led to the development of various direct and indirect methods. At microwave frequencies, the direct single-frequency methods were enriched in the recent years with more convenient broad band frequency domain dielectric spectroscopy (FDDS), time – domain spectroscopy (TDS), Fourier transform spectroscopy (FTS). Computer controlled spectrometers are now the norm in dielectric spectroscopy. Computers allow the computation of electromagnetic fields in entirely new measurement geometries and the use of numerical analysis in the direct measurement process. The use of such spectrometers is now one of the most fruitful factors in new approaches to microwave dielectric spectroscopy. Each investigator employs the method adequate for the size and shape of a sample. The most important problem now is the rigorous mathematical solution of the microwave interaction with the samples in various geometries. Although there is now complete overlap and coverage of the radio frequency to the infrared band, the different experimental methods based on coaxial, waveguide, and resonator and free–space technique are still divided and will be presented. Examples of various materials, such as ferroelectric, relaxor, dipolar glasses and other materials dielectric spectroscopy results will be presented.

ENHANCING THE CATALYTIC ACTIVITY OF COPPER NANOFOAM STRUCTURES FOR ELECTROCHEMICAL CO_2 REDUCTION

Birutė Serapinienė[1], Algirdas Selskis[1], Jurga Juodkazytė[1], Rimantas Ramanauskas[1]*

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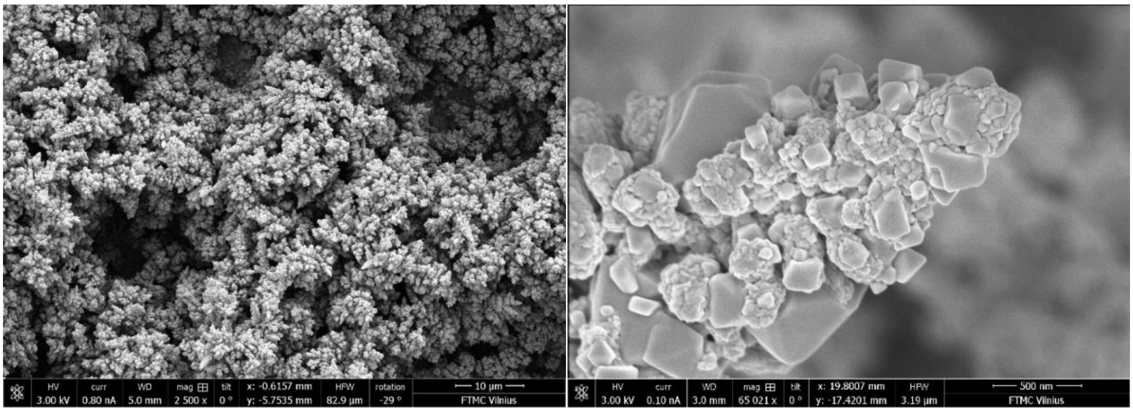
An exclusive feature of the Cu surface is the ability to convert CO_2 into hydrocarbons with significant faradaic efficiency. Simple polycrystalline Cu electrodes have a rather small surface area and therefore their efficiency is low. Porous Cu foam electrodes, suitable for cathodic CO_2 reduction, have been deposited in acidic sulphate solution with different additives to obtain structures with high real surface area and adequate mechanical stability. The SEM image of the Cu foam (Fig. 1a) shows that it is composed of branched dendrites extending in all directions, while the detailed microscopic features (Fig. 1b) indicate that the Cu crystallites in a wall are grape-like deposits.



a

b

Fig. 1. SEM images of electrodeposited Cu 3D structures. Magnification: a - 2500x, b - 65000x.

The influence of electrodeposition time and solution composition on the porosity parameters, microstructure and stiffness of the Cu 3D structures was investigated. Neither ammonium acetate nor polyethylene glycol were found to be effective additives to the Cu sulphate electrolyte to achieve the main objectives. Only the presence of Cl^- ions in the deposition solution resulted in a threefold increase in the real surface area and the achievement of sufficient mechanical stability of the Cu 3D structure. The latter effect is related to the specific influence of Cl^- ions during the electrodeposition process on the microstructural characteristics, such as the size of micropores in the walls of the holes and crystallite aggregates forming dendritic branches. These structural changes, in contrast to the Cu samples deposited in solution without additives, resulted in larger real surface areas, while the denser structures deposited in the presence of Cl^- ions ensured the mechanical stability of the 3D structure.

HOW PRECISE IS PRECISE? PURSUING SPECIFIC EDITING OUTCOMES WITH CRISPR NUCLEASES

Ieva Jaskovikaite[1], Jonas Kubilius[1], Marius Vingradovas[1], Fausta Michnevičiūte[1], Uzair Ahmed[1], Stephen Knox Jones Jr[1]*

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The last decade has produced a massive expansion in the genome editing toolset, paving the way for new therapies, diagnostics and technologies. Cas9, Cas12a, and related CRISPR nucleases form the foundation, offering precise genome editing at target sites that complement their RNA guide (gRNA). Yet, two major phenomena limit their utility: 1) Occasional "off-target" cutting activity at sites with just partial gRNA complementarity, and 2) heterogeneous editing outcomes at the intended target site.

Out team develops high-throughput strategies that benchmark and quantify the off-target propensity of leading CRISPR nucleases, uncovering their precise rates and sites of cutting with nucleotide resolution. Armed with this data, we pursue re-engineering gRNAs to make editing outcomes less variable and more predictable. Our efforts help provide CRISPR users with greater genome editing success without increasing cost or complexity.

ZnO NANOWIRE SYNTHESIS AND APPLICATION

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ZnO nanoparticles, as low cost and low toxic materials, have shown promising performances in sensing, catalysis and optoelectronics. ZnO nanowires (NWs) are the nanoparticles with an elongated, high aspect ratio form. Focus of our work is on the growth mechanisms, developing high yield NW growth methods to the feasible synthesis of ZnO NWs (or ZnO tetrapods), and further exploiting their excellent performance in sensing. We use ZnO NW based sensor platform, which involves chemoresistive sensing mechanism, low temperature preparation methods, therefore virtually any surface could be covered with a controllable thickness and morphology of ZnO NWs implying a wide range of application possibilities. We also established a correlation between the pore size in different ZnO nanostructures because of packing and their electrochemical properties. We expect that the detailed analysis of ZnO NWs will be advantageous for future electrochemical, biotechnological and electronic applications of these materials.

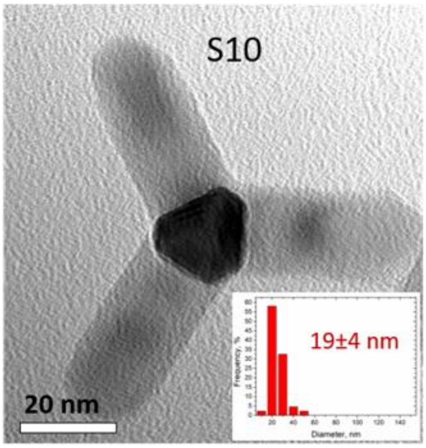

Fig. 1. TEM image of ZnO tetrapod, inset shows leg diameter size distribution

It is recommended that the page of article start and end with text and illustrations (table or a picture) should be placed in page between the text. Citation in the text: [1-5]. Do not use page numbering.

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Oral presentations 1

Material Science and Technology

Conventional Materials: Ceramics, Glasses, Metals and Alloys.

Chemical Thermodynamics

PUTTING THE SQUEEZE ON ENERGETIC MATERIALS - HIGH-PRESSURE STUDIES OF EXPLOSIVES AND PROPELLANTS

Colin R. Pulham[1*], Craig L. Bull[2], Ashfaq Afsar,[1] Aaron Chan,[1] Xiaojiao Liu,[3]
Heather Quayle,[1] and Carole A. Morrison[1]

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Explosives and propellants, known generically as energetic materials, are widely used in applications that include mining, munitions, and automotive safety. Key properties of these materials include: reliable performance under a range of environmental conditions; long-term stability; environmental impact; processability; sensitivity to accidental initiation through stimuli such as impact, shock, friction, and electrostatic discharge. Many of these properties are affected by the crystal structure of the energetic material. Moreover, explosives experience elevated pressures and temperatures under detonation conditions – such conditions often induce phase transitions in the energetic material. Hence detailed studies of pressure-induced structural changes in these materials are essential in order to understand and fully model their behavior.

This presentation will describe some recent high-pressure studies using a combination of X-ray and neutron diffraction techniques on selected explosives and propellants including PETN (pentaerythritol tetranitrate) and 3,4,5-TNP (3,4,5-trinitro-1H-pyrazole). The often highly anisotropic compressibility behavior of these materials can be correlated with their crystal structures and in particular the response of key intermolecular interactions to elevated pressures. The presentation will also describe the interplay between experiment and theory, and in particular how computational studies based on a recently developed vibrational up-pumping model are able to rationalize changes in sensitivity to initiation by mechanical stimulus caused by pressure-induced polymorphism and changes in molecular conformation. The outcomes from such experimental and computational studies have important implications for the safe manufacture, transportation, and use of these technologically important materials.

PORTLAND FLY ASH CEMENT CEM II/A-V 42,5 N MH/LA/NSR USAGE IN MASSIVE CONCRETE CONSTRUCTION

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The usage of fly ash cement CEM II/A-V 42,5 N MH/LA/NSR in massive concrete constructions is the main focus of this study, aiming to evaluate its impact on reducing climate change while enhancing the overall properties of concrete. Concrete incorporating fly ash cement (CEM II/A-V) demonstrates improved resistance properties, including enhanced resistance to chloride penetration, alkali acid reactions, and sulfate attack, critical for ensuring the longevity and durability of concrete structures. An analysis of the heat of cement hydration curves provides (Fig. 1 and Fig. 2) valuable insights into the curing processes, enabling the optimization of construction practices to improve quality.

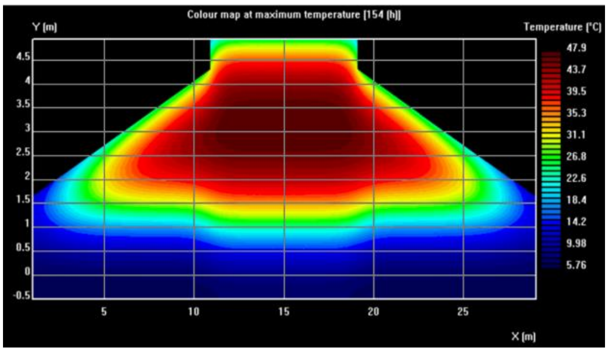

Fig. 1. Temperature modulation on windmill base

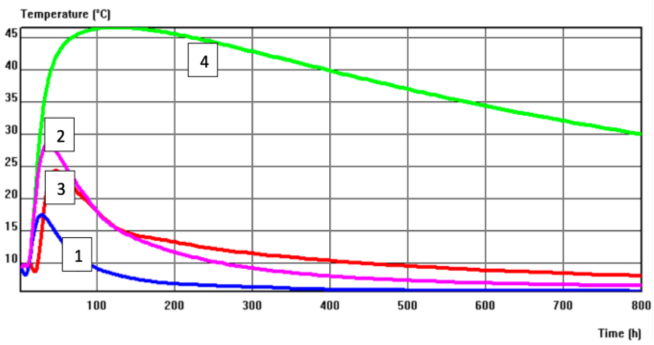

Fig. 2 Graph of temperature change

Portland cement (CEM II/A-V), meeting the additional requirement for moderate heat development (MH) in CEM II/A-V 42,5 H MH/LA/NSR, is well-suited for addressing temperature-related cracks. Studies conducted on actual projects demonstrate the exceptional performance of this cement, with the heat of hydration not exceeding 290 J/g, effectively preventing the formation of micro-cracks in structures. This research contributes valuable insights into the usage of fly ash cement in massive constructions, emphasizing its benefits for sustainable and durable concrete structures.

SOL-GEL SYNTHESIS AND HYDRATION BEHAVIOR OF Cu-DOPED CALCIUM SULFOALUMINATES

Eva Bartonickova[1*], Jiri Svec[1], Radoslav Novotny[1], Martin Sedlacik[1], Jiri Masilko[1], Jakub Palovcik[1], Jan Koplik[1], Lukas Kalina[1], Petr Ptacek[1], Teodor Stanek[2]

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Nowadays, the production of Portland cement is moving towards more sustainable approaches such as recycling or reusing materials as primary sources. The re-use of waste materials mainly has advantages (energy savings and reduced CO_2 emissions), but also disadvantages (heavy metal contamination or impaired hydration behavior) [1]. Calcium sulfoaluminate cements (CSA) belong to the “low-energy cement group” and in comparison, with OPC, their hydration is less influenced by heavy metals presence [2]. Furthermore, when CSA reacts with water, it behaves similarly to OPC, i.e. Aft and Afm are also formed, and metal immobilization can be accommodated by ion incorporation into the ye`elimite or Afm crystal structure. [3].

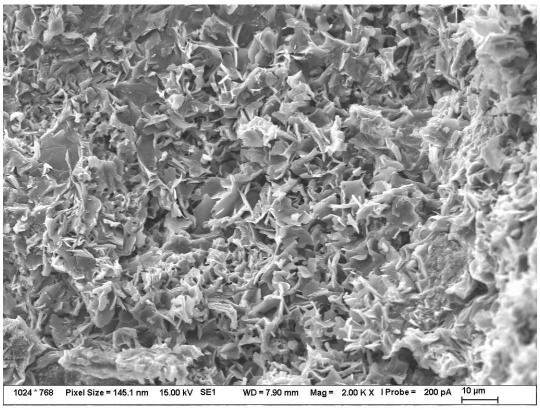

Fig. 1. Morphology of metastable C_3A hydrates and Afm upon hydration of $CuO{\cdot}SO_3{\cdot}CaO{\cdot}Al_2O_3$ system

The modified Pechini synthesis of Cu and S doped calcium aluminates and their hydration behavior are here introduced. Phase composition and crystal structure of the synthetic products was analyzed by means of XRD and Raman spectroscopy, where the ye`elimite presence according to S content was determined. The Cu/S incorporation into the C_3A was successfully observed by XPS analysis. Powders were hydrated and the resulting hydrates were metastable C_4AH_{13} and C_4AH_{19} and Afm with typical lamellar morphology (see Fig. 1 - SEM-EDS).

Acknowledgements. This work was funded by the Czech Science Foundation under the project number GA23-05082S.

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USE OF FLY ASH AND METAKAOLIN IN WOOD BIOMASS ASH-BASED ALKALI-ACTIVATED MORTAR

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Portland cement (PC)-based concrete is widely used as the primary building and construction material. Nonetheless, PC production is responsible for large global anthropogenic emissions of greenhouse gases and industrial energy consumption. Meanwhile, the intensive demand for natural aggregates exerts significant stress on non-renewable resource conservation. Under this background, developing alkali-activated materials based on industrial and agricultural waste as an alternative to traditional PC-based materials is a sustainable move.

As a forestry country, biofuel energy via wood combustion is commonly adopted in Lithuania, contributing to a great deal of wood biomass waste. To explore the application of this waste at a large quantity in producing alkali-activated mortar (AAM) materials, wood biomass fly ash (WBFA) and wood biomass bottom ash (WBBA) were recycled as precursors, and recycled sand (RS) was collected as fine aggregate. Sodium hydroxide (SH) at 7 mol/L, calcium hydroxide (CH) at 10% by the total precursor mass, and sodium silicate (SS) at an SS to SH mass ratio of 1 were ternarily used as alkaline activators. Metakaolin (MK) and coal fly ash (FA) were introduced as a binary precursor to provide extra aluminosilicates at the content of 10, 20, 30 and 40% by precursor weight. Compressive strength, SEM, XRD and TG were tested to analyze the mechanical properties and reaction mechanisms.

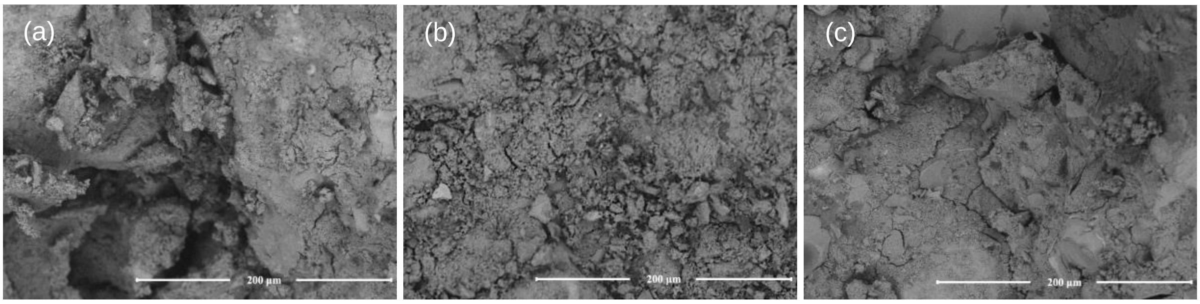

Fig. 1. SEM of AAM at 500 magnification: (a) reference sample; (b) with 30% FA; (c) with 20% MK

According to the results, the addition of FA and MK effectively improves the strength by 47.18 and 33.12%, respectively, with the highest value attaining 22.71 and 20.54 MPa. From SEM images (Fig. 1), the usage of FA and MK densified the microstructure of AAM by introducing more closed pores and decreasing micro-cracks. In the TG-DTG analysis, greater weight loss associated with the activation products was observed for samples with FA and MK, which was further confirmed in the XRD patterns that higher intensive peaks aligned with calcium silicate hydroxide were identified. This indicates that adding FA and MK accelerates the alkaline activation and enhances its degree, favouring the development of mechanical properties.

FIRED CLAY BRICKS WITH ORGANIC LAKE SEDIMENTS (SAPROPEL): STUDY AND CHARACTERIZATION

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Fired clay materials are widely used as building materials around the world because they are ecological, functional, and durable. Furthermore, a new focus on more sustainable development in the building materials manufacturing industry encourages the use of eco-friendly raw materials or waste in the production of fired clay materials [1-6]. In this study, the effects of organic lake sediments (sapropel) on the physical-mechanical, thermal properties, and durability of fired clay bricks were investigated. It was observed that shrinkage after firing and bulk density values decreased (Fig. 1), while apparent porosity and water absorption values increased with the increment in organic lake sediments (sapropel) content.

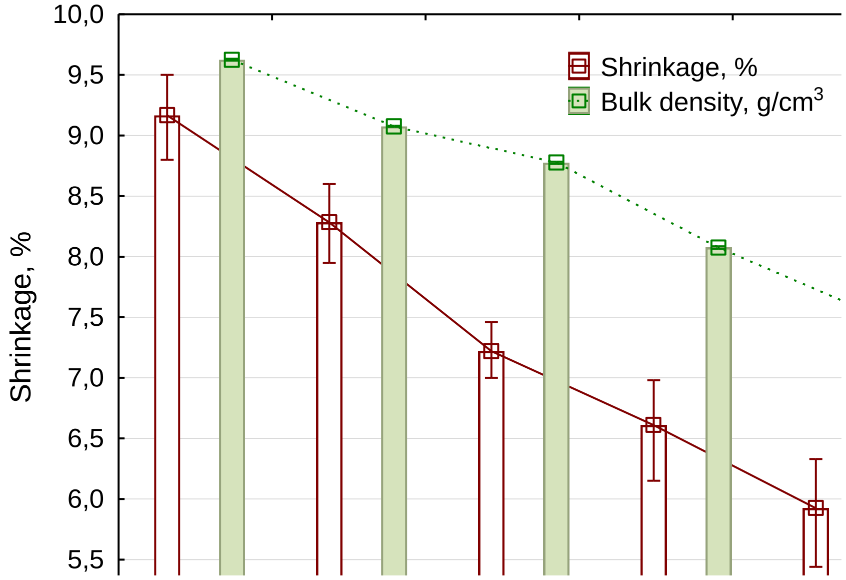

Fig. 1. Effect of organic lake sediments (sapropel) on shrinkage and bulk density of fired clay samples (control: clay samples without sapropel addition, GWS5 - GWS30: clay with 5-30 wt.%, sapropel addition, respectively).

Even fired clay samples containing 30 wt.% organic lake sediments (sapropel) showed good results. The fired clay samples containing up to 30 wt.% organic lake sediments (sapropel) presented a compressive strength above 10 MPa. It can be concluded that organic lake sediments (sapropel) can be effectively used in fired clay brick production.

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THE EFFECT OF NATURAL AND CALCINED SODIUM BENTONITE ON EARLY-AGE HYDRATION AND RHEOLOGY OF METAKAOLIN-SLAG BASED GEOPOLYMER PASTE

Eliška Křivánková[1, 2*], Dana Kubátová[2], Vlastimil Bílek Jr. [1], Martin Boháč[2], Martin Nguyen[2], Michaela Krejčí Kotlánová[2], Theodor Staněk[2]

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In this work the influence of bentonite clay addition on rheological properties of geopolymer paste during early-age hydration was examined. The primary aim was to demonstrate that the bentonite clay can effectively modify rheology unlike traditional chemical admixtures for cement, which typically fail when used in geopolymer systems. The differences between behavior of natural bentonite and its calcined form were also described.

The presented geopolymer pastes combined metakaolin and slag as a precursor, activated with sodium waterglass. The selected type of the bentonite clay was a high-swelling (sodium) type with high content of montmorillonite. Either natural or calcined clay was added in amounts of 8, 12 or 16 wt. % as a replacement from the total mass of the precursor. The main experiments were conducted using rotational rheometer and isothermal calorimeter, supported by Vicat needle tests. The microstructure was studied using scanning electron microscopy.

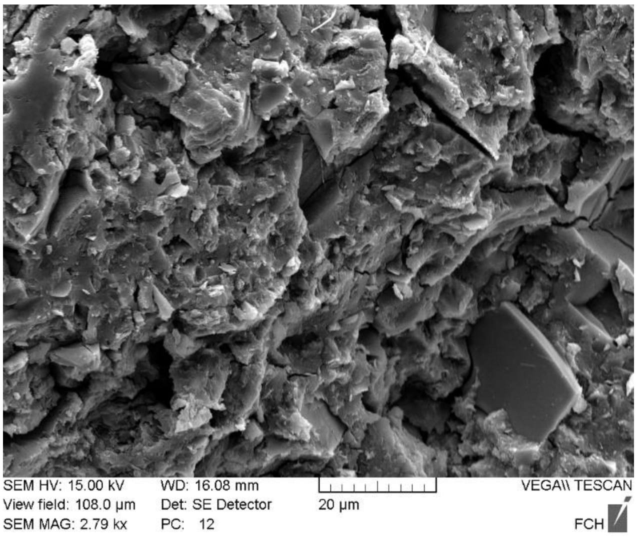

Fig. 1. The fracture surface microstructure of geopolymer containing 16 wt. % of calcined bentonite clay after 24 hours of geopolymerization.

The results revealed that the natural bentonite significantly increased the pastes' yield stress and enhanced plasticity, along with structural regeneration during early age geopolymerization. This confirms that natural bentonite can effectively adjust the rheological properties. In contrast, the addition of the calcined form had a minor influence on the reversible structural recovery, as demonstrated by the evolution of shear moduli.

Acknowledgements. Thanks are due to Czech Science Foundation project no. 23-05122S.

CHITOSAN-BASED SOLUTIONS FOR CONTROLLING SALT DAMAGE IN HISTORIC BUILDING MATERIALS: A NOVEL APPROACH

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Crystallization of soluble salts is a major cause of deterioration for porous building materials like stones, bricks and mortars in historic architecture. Upon repeated dissolution and recrystallization cycles, salts generate stress that can exceed the tensile strength of the substrate, leading to damage such as exfoliation and material loss. The most accepted theory on salt damage states that as salt crystals grow in pores, repulsive forces arise between the crystal and the pore wall, leading to the formation of a thin salt solution layer and generating a disruptive crystallization pressure. Current methods to fight salt damage mainly involve rising damp removal and salts removal by poulticing, however they are not always fully effective. An alternative promising strategy has recently been investigated using salt crystallization modifiers, capable of inhibiting crystallization and promoting salt transport to the walls surface, thus allowing the formation of less harmful efflorescence instead of sub-florescence. Moreover, substances as surfactants and polymeric coatings could eliminate the disjoining pressure between crystal and pore wall, allowing the former to grow without causing damage. In the present research, chitosan, a biopolymer naturally found in the shells of crustaceans and well known for its valuable properties in terms of biocompatibility and sustainability, was selected as crystallization inhibitor. Chitosan aqueous solutions (at different concentrations) were applied to limestone and brick substrates previously subjected to a consolidating treatment based on diammonium phosphate (DAP), as studies have shown that hydroxyapatite (formed thanks to the reaction between DAP and the substrate) ensures a better adhesion for the chitosan coating [1-3]. To assess the effectiveness of the treatments in reducing salt crystallization damage, untreated and treated samples were subjected to a new accelerated weathering test, developed by the RILEM TC 271-ASC [4] to reproduce the salt damage mechanism occurring in the field. Unlike existing standardized tests, which often fail to accurately replicate on-field conditions and may lead to exaggerated degradation, the new procedure involves a first accumulation of salts underneath the surface and then a propagation phase which induces salt crystallization damage through wetting and evaporation cycles. Although one of the developed treatments seems to provide a beneficial effect, the results also highlight the challenges in interpreting the effectiveness of the selected treatments within its parameters and point out also some limitations of the testing method. These complexities highlight the ongoing necessity for advancing methodologies tailored to the unique challenges of cultural heritage conservation, underscoring the continuous quest for scientific innovation to effectively preserve our collective heritage.

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PRODUCTION OF NOVEL PECTINATE GELS USEFUL FOR 3D BIOPRINTING FROM CITRUS PEELS

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Citrus peels (CPs) are the main waste generated by the citrus processing industry, with an annual production of about 10 million tons. Generally, CPs are incinerated or landfilled, but these disposal strategies are inadequate, having negative impact on environmental and economic aspects. To reduce these problems, CPs have been tested for different uses such as additive in animal food or as organic soil conditioner. More recently, its valorization through the extraction of high value products (e.g., essential oils, pectin, and flavonoids) has been exploited. Furthermore, interest in pectin has increased as its properties make it useful not only in food but also in biomedical, pharmaceutical, cosmetic and food packaging applications.

This contribution reports on characterization of pectin obtained from five different CPs (orange, tangerine, lemon, pomelo, and grapefruit) in terms of molecular weight, galacturonic acid content, degree of methylation and rheological properties. In addition, a new ionotropic method employing bivalent ions to cross-link both low and high methoxy pectin to form stable gels will be presented.

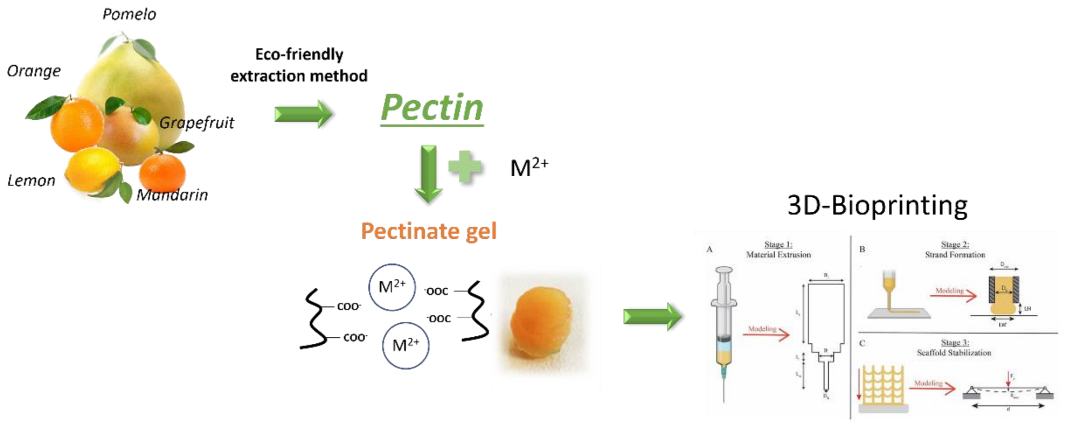

Fig. 1. Representative diagram of the pectin extraction process, pectinate gel synthesis and application in 3D-Bioprinting.

The complete rheological characterization of pectinate gels resulted in the determination of yield stress, zero-shear rate viscosity, flow index and consistency index. These parameters have been analyzed using a previously developed open-source tool to predict printability through extrusion bioprinting. This analysis made it possible to identify the most suitable composition for printing with the ultimate goal of synthesizing new 3D printable pectin-based gel with potential applications in the biomedical field.

SYNTHETIC YE'ELIMITE INFLUENCE ON THE HARDENING PROCESSES OF PORTLAND-FLY ASH BLENDED CEMENT

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As part of the sustainability movement, there is a notable focus on preparing different cement blends by replacing Ordinary Portland cement (OPC) with supplementary cementitious materials (SCMs) like fly ash [1]. Unfortunately, SCMs often delay cement setting time and reduce early compressive strength. Therefore, admixtures (accelerators), which increase the rate of cement hydration, are often applied. However, common accelerators such as sodium chloride (NaCl) and calcium chloride ($CaCl_2$) cause steel corrosion, while sodium thiocyanate (NaSCN) is expensive, toxic, and triggers alkali-aggregate reactions, reducing compressive strength [2]. Therefore, there is a growing demand for new types of accelerators.

Ye'elimite ($C_4A_3\hat{S}$) is the predominant phase in calcium sulfoaluminate cement, also known as green cement. This phase plays a crucial role in controlling the initial hydration process of cement paste (sets and hardens within the first 24 h) and determining the final mechanical properties of concrete [3]. Thus, this work aims to examine the influence of synthetic ye'elimite additive on the hardening processes of Portland-fly ash blended cement and evaluate its potential as an additive to regulate the setting time of mortars.

For the study, synthetic ye'elimite was produced using a mixture of analytical grade materials (CaO, Al_2O_3, $CaSO_4{\cdot}2H_2O$), referring to its ($Ca_4Al_6O_{12}(SO_4)$) stoichiometry. The required materials were weighed and subjected to the following treatments: 1) mechanochemical activation (milling at 900 rpm with three 10-min on-off cycles), 2) hydrothermal treatment (for 8 h at 110 °C), and 3) calcination (for 1 h at 1150 °C). The synthesized ye'elimite (purity of ~97%) was used to prepare ternary mix compositions: OPC ranging from 78 wt.% to 80 wt.%, siliceous fly ash (Class F) from 18 wt.% to 20 wt.%, and synthetic ye'elimite from 0.25 wt.% to 2 wt.%. The prepared cementitious blends were used to examine the standard consistency and setting time of the pastes as well as the compressive strength of mortar samples, which were cured in water (20±1 °C) for 2, 7, and 28 days.

The results demonstrate that the ye'elimite additive effectively acts as a setting accelerator for Portland-fly ash blended cement. It reduces the initial and final setting times of the prepared pastes by up to 1.7 times without compromising the compressive strength of the mortars after 28 days of hydration compared to neat OPC mortars. Meanwhile, the X-ray diffraction and thermogravimetric analysis confirmed that the ye'elimite additive induces the formation of ettringite, hemi- and monocarboaluminates.

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INVESTIGATION OF THE HIGH TEMPERATURE BEHAVIOR OF ALUMINOSILICATE MICROSPHERES

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Aluminosilicate microspheres also known as cenospheres (CS) are a byproduct formed during the coal combustion in thermal power plants. Since CS are mainly consist of SiO_2 and Al_2O_3, they can be considered as a valuable component for high-temperature materials. The exact chemical composition of CS from different plants may visibly differ, influencing their properties at high temperatures. The present work investigates four different CS with SiO_2+Al_2O_3 content from 82.2wt.% to 93.2wt.% (Table 1). X-ray fluorescence (XRF) spectroscopy, X-ray diffraction (XRD) analysis, differential thermal analysis (DTA), thermogravimetry (TG), scanning electron microscopy (SEM) along with other methods were employed to characterize CS and investigate their behavior at temperatures up to 1300 °C.

Table 1. Chemical composition of the studied cenospheres (by XRF, in wt.%)

Mark	SiO_2	Al_2O_3	C	Fe_2O_3	TiO_2	K_2O	CaO	Na_2O	MgO	Other	Total
CS-1	58.3	34.9	0.811	1.64	1.27	0.825	0.844	0.373	0.233	0.804	100.00
CS-2	54.3	36	1.85	2.36	1.43	0.84	1.19	0.555	0.255	1.22	100.00
CS-3	54.6	28.4	1.47	4.59	1.29	3.35	2.44	0.761	1.96	1.139	100.00
CS-4	50.9	31.3	2.62	5.53	1.1	4.93	0.875	0.88	1.19	0.675	100.00

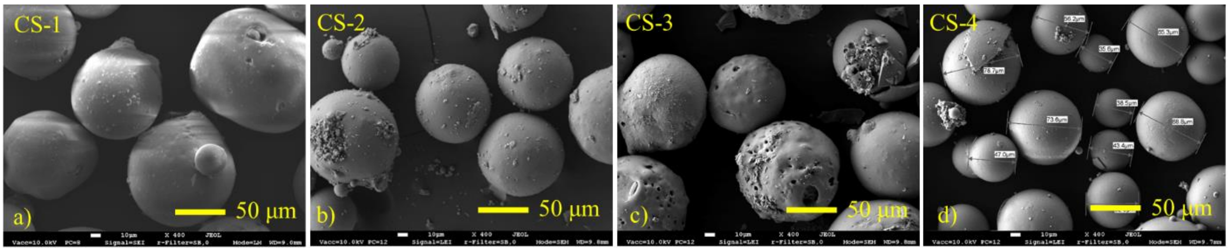

Fig. 1. The outside morphology of non-treated CS from different plants

The research showed that the studied CS differ in external and internal morphology, particle size and its distribution, density, phase composition. Despite the major phase for all cenospheres was mullite, minor components (other than SiO_2 and Al_2O_3 and especially alkalis) significantly influenced the behavior of CS at high temperatures. The results obtained allowed stating that in the CS with high SiO_2+Al_2O_3 content (90.3% and 93.2%) the amorphous phase is SiO_2 glass rather than aluminosilicate. The diffraction patterns of CS-1 and CS-2 heat-treated at 950°C and over showed appearance of reflections attributable to cubic β-cristobalite (high temperature polymorph), indicating that CS with high SiO_2+Al_2O_3 content may be potentially considered as a source for refractory material containing stabilized cristobalite.

Acknowledgements. The equipment and infrastructure of Civil Engineering Scientific Research Center of Vilnius Gediminas Technical University was employed for investigations. This research was supported by the center of excellence project “Civil Engineering Research Centre” (Grant No. S-A-UEI-23-5).

ENHANCED SUSTAINABILITY OF AERATED BUILDING BLOCKS: UTILIZATION OF ACCELERATED CARBONATION AND OIL SHALE ASH IN CEMENT COMPOSITES

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Cement, the second most used material globally, contributes significantly to CO_2 emissions during its production. Autoclaving, or steam curing, is used in the concrete industry to speed up hydration reactions. However, it is energy-intensive and generates additional CO_2 emissions from fuel combustion. This work evaluates using oil shale ash (OSA) and accelerated carbonation curing (ACC) to create more sustainable binders and CO_2 capture methods for lightweight pre-cast building materials. The current research examines the effects of ACC (100%CO_2, 1.5 bar, ~65%RH) on the microstructural changes and phase formations in aerated cement composites. Cement (CEM-1 42,5R), OSA, and fine Al powder (pore making agent) were used for the preparation of aerated blocks. The compressive strength, density, and CO_2 uptake values (Table 1) were obtained as one of the first indicatives for performance evaluation and further analyses were carried out for hardened state microstructural and mineralogical characterization.

Table 1. compressive strength, density, and CO_2 uptake values at 28 days (UC: Uncarbonated, C: Carbonated)

Block Composition	**Water/Solid**	**Density (kg/m^3)**	**Compressive strength (MPa)**	**CO_2 uptake (%)**
100%Cement, UC	0.5	644.2	3.7	14.1
100%Cement, C	0.5	735.1	5.7	
30%Cement/70%OSA, UC	0.6 - 0.625	633 - 650	1.5 - 1.9	13.7
30%Cement/70%OSA, C	0.6 - 0.625	715 - 735	2 - 2.7	

ACC particularly in porous composites, shows potential for CO_2 sequestration as results show improved strength with ACC treatment and enhanced sustainability with CO_2 uptake. According to SEM image analysis densification of the CSH-networks has been achieved with the carbonation induced transformations to a granular-textured calcite. Using ACC to manufacture porous cement composites can be viable strategy for producing lightweight building components and recycling lime, portlandite, $CaSO_4$-rich alkaline waste.

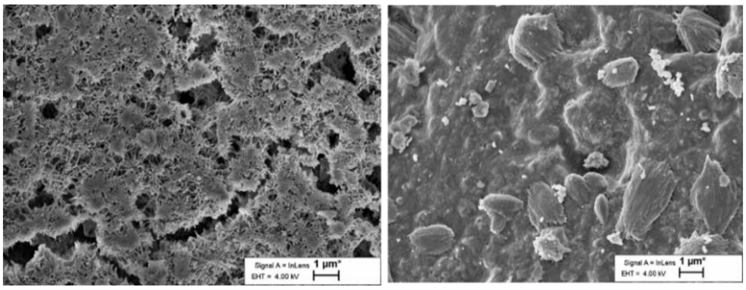

Fig. 1. 30%Cement /70%OSA, UC (left), C (right)

FABRICATION OF CARBON COATED IRON OXIDE FILMS ON COPPER SUBSTRATE FOR ENERGY STORAGE APPLICATIONS

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The global energy demand increases day by day, creating serious challenges to the environment and sustainable development [1]. One of the main energy demands is the fuel for onboard applications, meeting mostly from burning of fossil fuels. Therefore, intensive efforts are underway to transform fossil fuels powered vehicles to electric powered vehicles (EVs) as soon as possible. Despite, the substantial EVs production, high capacity, fast charging, low cost and safe energy storage systems are still needed to be explored to meet the future energy storage for grid and onboard applications. One of the critical components of these systems is the electrodes material [2]. The present day commercial electrode materials are suffered by their low specific capacity, high cost, and instability. Iron oxide possesses a great potential to be used as anode material in lithium ion batteries (LIBs) and post-LIBs energy storage systems [3]. However, the high volume change during (dis)charge cycles limits their practical uses, as they disintegrate over a span of few cycles. This leads to fading of capacity and eventually dying of the energy storage system. To mitigate the volume changes and thus ensure stability of the iron oxide anodes, one of the solutions is encapsulation of the particles of electrode material in highly conductive but flexible shells [3]. We demonstrated a successful preparation of iron oxide films directly on the current collector *i.e.,* copper (Cu) used in practical LIBs via a simple, scalable and cost-effective solvothermal method. The films were then coated with carbon jackets/shells using state-of-the-art chemical vapor deposition (CVD) techniques, which include microwave plasma (MPDV) and thermal CVD. The films were then optimized as anodes for LIBs by electrochemical characterizations.

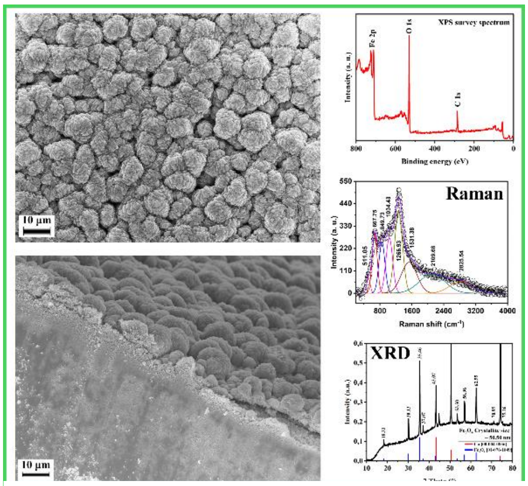

Fig. 1. SEM images (top and side view), powder XRD pattern, and Raman and XPS spectra of a representative sample

Acknowledgements. This project has received funding from the European Union's Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant agreement No 945422

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CHARACTERIZATION OF DEGRADATION PROCESSES IN FRNC COATINGS OF OPTICAL FIBER CABLES

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Fibre optic cables are commonly used in long-distance and high-performance data networks, including telecommunication, military, and medical purposes. Generally, fibre optic cables could be divided into two groups according to their application areas: indoor and outdoor [1]. Cables dedicated to indoor application must meet rigorous fire-safety requirements in the European Union, which guide the selection of Flame Retardant, Non-Corrosive (FRNC)/Low Smoke Zero Halogen (LSZH) for their sheath materials. Commercially available thermoplastic FRNC compounds dedicated to fiber optic cables are based on linear low-density polyethylene /ethylene-vinyl acetate composites highly loaded with aluminum trihydroxide and magnesium dihydroxide fillers, ensuring a higher level of human safety during fires in buildings [2].

Constantly increasing telecommunication market demand requires development of cable manufacturing technology to provide higher production capacity and cost optimization as well. Speed of cable production lines are often limited by extrusion performance of FRNC materials. A further increase in extrusion speed could cause unpredictable changes in the cable FRNC sheaths, which could negatively affect the quality of the cable.

Objective of this work was to characterize an influence of temperature and thermomechanical degradation in extrusion process on properties of commercial FRNC cable compounds. Thermal degradation of cable materials was studied with use of thermogravimetry and rheometry. The series of jacket samples under different processing conditions were produced. Material processing behavior was characterized, and tensile and heat ageing performance of cable jacket were tested. The thermal analysis of materials allows to extend processing temperature to 200°C with satisfactory processing performance. Moreover, rheological analysis shows insignificant rheological changes up to 220$^{\mathrm{o}}$C. Characterization of mechanical properties of the cables shows relationship between production speed, processing temperature and processing profile type. The result and general approach to the FRNC investigation can be successfully used in cable industry or in other industries involving the extrusion of FRNC materials.

Acknowledgements. The work was financially supported by Ministry of Science and Higher Education, Republic of Poland in frame of Fifth Edition of Implementation Doctorate Program for years 2021-2025.

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SUPPLEMENTARY CEMENTITIOUS MATERIAL FROM EXPANDED CLAY DUST AND OPOKA

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This work examines the influence of a two-component supplementary cementitious material consisting of additionally calcined expanded clay kiln dust (KKD) and opoka (O) on Portland cement hydration and hardening. After preparing mixtures of different proportions with Portland cement, differences in hydration, composition, and compressive strength were studied compared to the control sample of pure Portland cement. The composition of the prepared mixes is shown in Table 1.

Table 1. The composition of the prepared mixes

Amount of components, wt.%			Abbreviations
Portland Cement	KKD	Opoka	
100	-	-	CEM
75	7.5	17.5	3K7O
75	12.5	12.5	5K5O
75	17.5	7.5	7K3O

The results of the compressive strength of cement samples with different hydration durations and additives are summarized in Fig. 1.

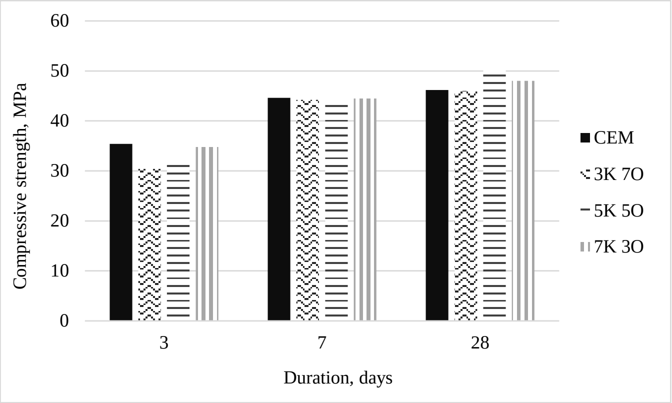

Fig. 1. Compressive strength of cement samples with different hydration durations and additives

It was found that the two-component additive has a complex effect on Portland cement hydration: the carbonate component promotes the formation of monocarboaluminates and the activated dust component promotes the formation of calcium silicate hydrates and hydrogarnets. The pozzolanic reaction and the formation of monocarboaluminates in the samples with the tested additives occur already after 28 days of hydration. A two-component supplementary cementitious material from opoka and expanded clay kiln dust waste is a very effective additive to Portland cement, as this material can replace up to 25 wt.% of Portland cement without reducing the compressive strength of the cement.

THE FUNCTION OF THE GRANULES BINDERS ON THE HYDRATION AND CONVERSION OF CALCIUM ALUMINATE CEMENT MORTARS

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Calcium aluminate cement (CAC) is considered an eco-friendly option due to its lower carbon emissions during production. Additives used in CAC can take various forms including crushed powder, ground powder, or granules, each with different geometric compositions. Granulation creates granules as composite materials, where the primary material is enclosed within a binding matrix that imparts additional properties. The binder in granules can serve merely as a protective layer or play an active role in enhancing the interaction between the granule and cement. This dual functionality can significantly influence the overall performance and durability of the cement mortars.

Materials are used in the work: Calcium aluminate cement Gorkal 70, ("Gorka", Poland) and the fire-clay in ground form for manufacturing the granules with two type binders. The granulation binders were a sodium silicate solution with a density of 1,180 kg/m³ and a ratio of SiO_2/Na_2O of 3.3 and colloidal silicon acid solutions Lithosol 1540 (“Zschimmer&Schwarz”, Germany). The relative viscosity investigation of the mortar's CAC with granules was applied by vibroviscometer SV-10 from A&D. The behaviour of mortar’s hydration was measured using a TONICAL III calorimeter during for 48 hours at an operating temperature of 20°C. The samples hydrated were analysed by X-ray diffraction using a DRON-7 diffractometer. The thermogravimetric (TG) and differential thermal analysis (DTA) curves were obtained using a Linseis STA PT-1600 thermal analyser. The internal and outside morphology and microstructures of mortar were analysed by scanning electron microscopy JSM-7600F SEM (JEOL). The compression strength of mortar samples with granules after 110°C and 1100°C was defined by Tinus Olsen compressive test machine.

The research shows the binder used in fire-clay granules plays a crucial role in influencing the hydration of CAC. Its effects on hydration kinetics, microstructure development, filler effect, compatibility, water retention, and thermal properties are key factors that determine the performance and durability of CAC mortars. The binder can accelerate or retard the hydration reaction depending on its chemical composition and properties. An active binder participates in the hydration process, forming additional hydration products and potentially enhancing the rate of hydration, leads to a denser and more uniform microstructure, which contributes to higher strength and durability, overall stability of the cement matrix. The binder can affect the thermal properties of the granules, which is particularly important in high-temperature applications.

Acknowledgements. The equipment and infrastructure of Civil Engineering Scientific Research Center of Vilnius Gediminas Technical University was employed for investigations. This research was supported by the center of excellence project „Civil Engineering Research Centre“ (Grant No. S-A-UEI-23-5).

RESEARCH ON THE COMBUSTION AND ENERGY RELEASE CHARACTERISTICS OF HIGH-ACTIVITY ALUMINUM ALLOY-POLYVINYLIDENE FLUORIDE COMPOSITE MATERIALS

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The investigation of aluminothermic agents has garnered significant attention due to their ability to generate substantial heat and energy during chemical reactions. These agents are widely utilized in energetic materials, including propellants, explosives, pyrotechnics, and welding agents. Currently, nano-aluminothermic agents are one of the focal points in the research of energetic materials. However, several challenges persist with nano-aluminothermic agents, including the tendency of aluminum powder to agglomerate, the presence of a significant inert oxide layer on its surface leading to delayed ignition, incomplete energy release, and sintering during combustion. Research has demonstrated that the use of high-activity aluminum alloys as fuels in nano-aluminothermic agents can not only enhance their combustion performance but also modulate their energy release characteristics [1, 2]. Additionally, the incorporation of fluorides into nano-aluminothermic agents has been shown to significantly improve their combustion properties [3].

In this study, we aim to investigate the combustion performance and energy release characteristics of composite materials composed of high-activity aluminum alloys as fuels and polyvinylidene fluoride (PVDF) as an oxidant. Initially, polyvinylidene fluoride-coated aluminum-magnesium (Al-Mg) and aluminum-titanium (Al-Ti) samples were prepared using the electrostatic spray deposition method and were compared to pure aluminum as a fuel under identical conditions. The morphological features and compositional information of the prepared samples were characterized using focused ion beam/scanning electron microscopy (FIB/SEM), energy-dispersive spectroscopy (EDS), and X-ray diffraction (XRD). The thermal properties of the samples were analyzed through TG-DSC curves. High-speed and infrared cameras were used to characterize the flame morphology, diffusion area, ignition delay, combustion rate, and combustion temperature of the samples. The combustion pressure of the samples was measured through closed-volume pressure experiments. Finally, by comparing the data obtained from the coated Al, Al-Mg, and Al-Ti samples, we aim to elucidate the combustion mechanism of the composite material composed of high-activity aluminum alloys as fuels and PVDF as an oxidant. This research not only lays a foundation for future studies on nano-aluminothermic agents with high-activity aluminum alloys as fuels but also provides valuable insights for the modulation of energy release in energetic materials.

Acknowledgements. This work is financially supported by the National Natural Science Foundation of China No. 52176114 and No. 52306145.

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SOLUBILITY AND THERMODYNAMIC PROPERTIES OF XONOTLITE

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Xonotlite ($Ca_6Si_6O_{17}(OH)_2$) is a hydrated crystalline calcium silicate with $CaO/SiO_2 = 1$, which forms under hydrothermal conditions at temperatures above ≈100°C and pressures of 9 bar. Xonotlite has a high thermal stability as it dehydrates to wollastonite ($CaSiO_3$) only at 770 to 800°C [1]. Xonotlite is used as heat insulation material, and is furthermore of interest for the gas and oil industry and for long-term nuclear storage facilities [2].

Several studies have been conducted to better understand the formation conditions of xonotlite in terms of optimum temperature and pressure, synthesis duration and starting materials, but very little is known about the solubility of xonotlite under different conditions. The two solubility data sets available in literature [3, 4] differ by as much as 10 log units without any clear reason for this difference.

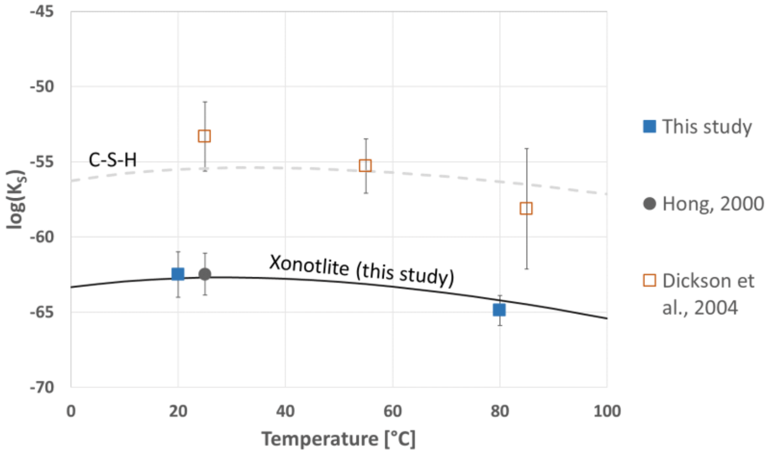

Fig. 1. Xonotlite solubility product ($K_s = 10^{-62.7\pm1.5}$) as a function of the temperature.

The present study determined the solubility product of xonotlite at different temperatures. The derived solubility data are comparable to the data reported by Hong [3] on synthetic xonotlite at 25°C, but are several log units lower than the data reported by Dickson et al. [2] on natural xonotlite, which coincide with the solubility of C-S-H (Figure 1).

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Oral presentations 2

Polymer Science and Technology of Polymeric Systems.

Hybrid Materials, Nanomaterials and Nanotechnologies.

Chemical Engineering and Biotechnologies.

Environmental Chemistry, Geochemistry and Astrochemistry

SYNTHESIS AND CHARACTERIZATION OF A PICKERING EMULSION GEL BASED ON FUNORI AND HNTS

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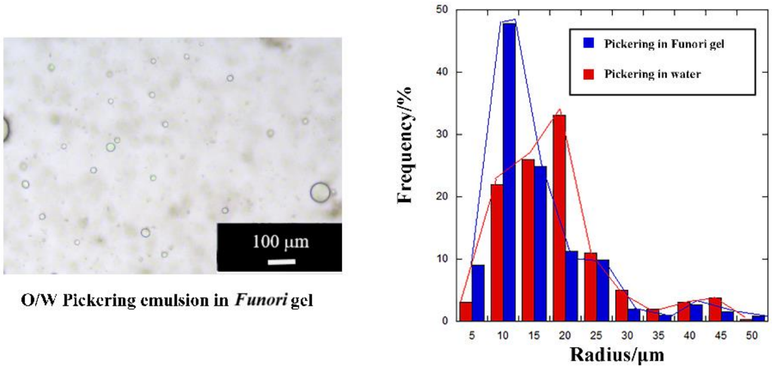
In the art conservation field, the selective removal of varnish layers from paintings without affecting the environment is still an open challenge. Nowadays, the scientific community is emphasizing the use of biopolymers as non-toxic alternatives to the most spread polluting materials [1]. In this regard, we developed a new cleaning system by using a Pickering emulsion gel based on funori and halloysite nanotubes (HNTs). *Funori* gel rheological properties and stability were evaluated by modifying both temperature and crosslinker concentration. Then, the interactions between the polymer and the Pickering emulsion were assessed by studying the interfacial activity of HNTs in aqueous media, both water and funori. The free energy change of halloysite nanotubes, when detached from the oil-water interface, highlighted an enhancement in stability when *Funori* is added. The subsequent optical observation of the emulsion enabled to determine how the presence of the biopolymer affected the stability and the radius size of the oil droplets (Fig. 1)



O/W Pickering emulsion in *Funori* gel

Fig. 1. Optical micrograph of *Funori* gel and radius distributions of isooctane droplets.

Therefore, the developed gel has been applied to remove an hydrocarbon resin from a quartz glass, simulating an hydrophobic layer that could be found on a real artefact surface. The cleaning procedure has been carried out by changing gel application times, monitoring both contact angle and transmittance.

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INFLUENCE OF THE STRUCTURE OF METHACRYLATE POLYMER ADDITIVES ON THE CRYSTALLIZATION BEHAVIOUR OF DIESEL FUEL FORMULATIONS

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During the production of diesel fuel, certain components are removed in order to meet regulatory standards and improve emission control. Despite these efforts to protect the environment, the removal of these components can have a negative impact on the overall performance of diesel fuel. To mitigate this problem, manufacturers are adding additives to diesel fuel and looking for the most cost-effective and energy-efficient solutions. The aim is to minimize the use of additives while improving the various fuel properties. Some of the most important diesel fuel additives are pour point depressants and cold flow improvers. These additives are essential for altering the crystal structure of paraffin wax crystals in diesel fuel, thereby lowering the risk of fuel line and filter blockages. [1,2] In our research, we have synthesized methacrylate polymer additives that have proven to be good pour point depressants and cold flow improvers, improving the pour point of diesel fuel by more than 30 °C and the cold filter plugging point by more than 10 °C. To explain the positive effect of additives on low-temperature properties, in this research we additionally investigated the crystallization behavior of diesel formulations with newly synthesized additives using differential scanning calorimetry and optical microscopy at low temperatures and established correlations of the additive effect at low temperatures with the additive structure.

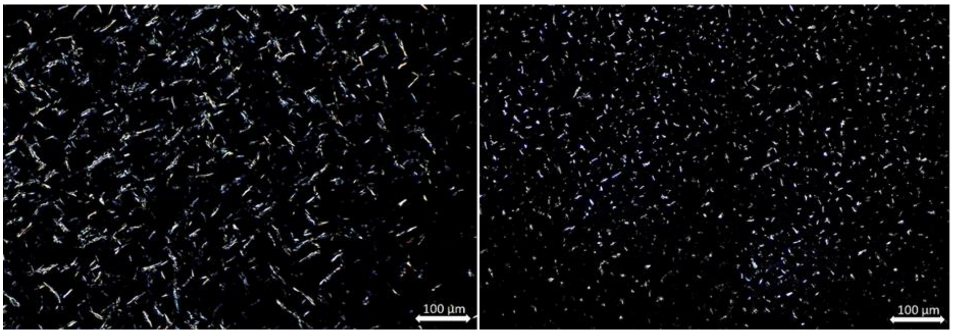

Fig. 1. Morphology of wax crystals in diesel formulations at -20 °C with different additives measured with an optical microscope equipped with a cold stage

Acknowledgements. This research was conducted within the Project of the Croatian Science Foundation Development of functional biofuels and (bio)additives and characterization of blends with mineral fuels (UIP-2019-04-5242).

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RECOVERY OF FIBRES FROM NATURAL FIBRE REINFORCED COMPOSITES BY CHEMOENZYMATIC METHOD TO PACKAGING VALUE CHAIN

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Natural fibre reinforced composites are widely employed in industrial scale especially in the field of wind energy and automotive sector, but managing post-use composites in line with circular economy principles remains a significant concern. This work intends to provide a Demand driven Circular Economy solution that allows for cost-effective reuse of post-use composite materials and components through end of life (EoL) studies into new high-value goods. The shredded fibres from the automotive industry were washed with surfactant and then hydrolyzed using enzyme which decrease the molecular weight of polyester. The reaction is monitored by Fourier Transform Infrared Spectroscopy (FTIR), optical microscopy, Scanning Electron Microscopy (SEM), High Performance Liquid Chromatography (HPLC) and Hyperspectral imaging (HSI) analysis. This characterization techniques confirms the degradation of the polyester into bis(2-hydroxyethyl) terephthalate (BHET), mono(2-hydroxyethyl) terephthalate (MHET) and with high yield of terephthalic acid (TPA). The HPLC examination indicated that the concentration of TPA increased during the reaction period, with the highest level (~9 mM) attained after 5 days of biodeterioration of polymers. As the primary outcome of the study, the recovered fibres are washed and will be transformed into pulp for use in packaging sector through pulp molding. The overview of the work is presented in **Fig. 1**.

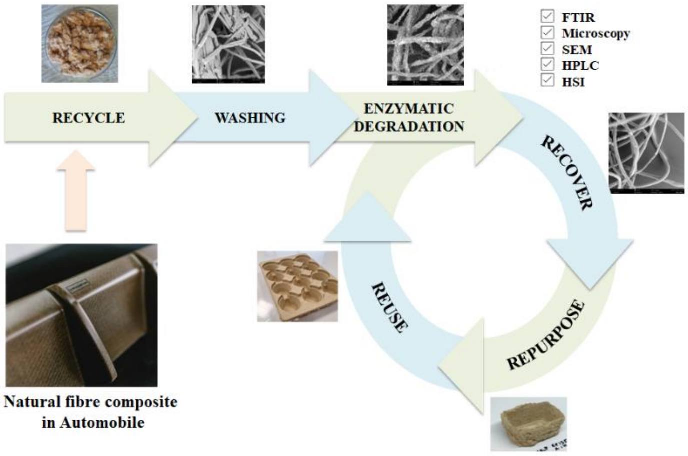

Fig. 1. Overview for the recovery of fibres from the Automotive composites.

Keywords:
Demand driven innovation, Circular economy, Natural Fibre composite, Pulp molding technology, Packaging sector.

Acknowledgement:
This research work was funded by the project "De & Remanufacturing for Circular Economy Investments in the Composite Industry (DEREMCO)"

LIFECYCLE CONSIDERATIONS FOR 1D IMMOBILISED PHOTOCATALYST FILMS

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Previous studies have shown that modifying the anodization process of titania nanotubes can result in films with a reduced level of order, leading to increased microstructural inhomogeneity horizontally and chemical inhomogeneity vertically. Immobilized photocatalyst systems utilizing titania nanotube arrays have demonstrated high efficiency due to the inherent properties of titanium dioxide, which are further enhanced by the advanced morphology provided by the nanotube array configuration which foster high photocatalytic activity.

In this study, we investigate the durability and reusability of the prepared photocatalyst samples. Photodegradation tests were conducted using acetylsalicylic acid and the lifetime of the photocatalyst was discussed. Vibration spectroscopy and depth-sensitive diffraction techniques were employed to monitor the samples before and after extensive use to assess the contribution of different areas formed in the final photocatalytic efficiency. The samples maintained their photocatalytic properties for a significant period.

Furthermore, we propose a novel approach for photocatalyst regeneration, where the used samples are electrochemically resynthesized to enable renewed activation and enhance degradation efficiency. The regenerated films were tested, demonstrating remarkable regeneration of photocatalytic activity. High-resolution electron microscopy of cycled and regenerated samples provided insights into the complex microstructural and chemical changes resulting from photodegradation and anodic regrowth. This innovative concept represents a significant advancement in the simple, cost-effective, and environmentally friendly removal of micro-pollutants through photocatalysis.

Acknowledgements: This work has been funded by UIP-2019-04-2367 SLIPPERY SLOPE by the Croatian Science Foundation.

MICROSTRUCTURE AND MECHANICAL PROPERTIES OF CNTS/CLAY HYBRIDS- HIGH EARLY STRENGTH PORTLAND CEMENT COMPOSITES

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Improving the durability and strength of concrete reduces maintenance. This can lead to sustainable structures and reduced materials consumption. Carbon nanotubes are a material that has outstanding strength properties. Especially, CNTs can play various roles in concrete, such as filling the internal pores and bridging between cracks. This research used Portland cement type III (high early strength) that has high early strength. CNTs/Clay hybrid were added at 0.02 wt.% up to 0.20 wt.%. Compressive strength and density were investigated at an early age until 28 days. Phases characterization and microstructure were also studied by X-ray diffraction and Scanning Electron Microscopy (SEM) with energy dispersive spectroscopy (EDS). CNTs/Clay hybrid enhanced the compressive strength and was highest at 0.10 wt.%. CNTs/Clay hybrid can be seen bridged between the hydration products which help load transfer within the cement matrix.

IMPACT OF PARTICLE SIZE ON THE COMBUSTION PERFORMANCE OF ELECTROSPRAY-COATED ALUMINUM POWDERS IN SOLID PROPELLANTS

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Inorganic metals are often added to solid propellants as burn rate regulators to ensure stable combustion. Aluminum powder, valued for its high energy density, is commonly used for enhancing combustion heat and thermal work capacity. However, micron and nano-sized aluminum powders tend to agglomerate before combustion and sinter during combustion, hindering reactivity. Additionally, an oxide layer on microscale aluminum powder further affects performance. To address these issues, a precursor liquid of microscale aluminum powder, PVDF binder, and nano ZnO can be formulated. An electrospray method achieves uniform coating on aluminum particles, and when subjected to thermal or mechanical shock, these coated particles undergo a vigorous redox reaction, releasing substantial energy and enhancing combustion performance. The coating also mitigates agglomeration and reacts during combustion to produce fluorine gas, removing the oxide layer, inhibiting sintering, and optimizing performance.

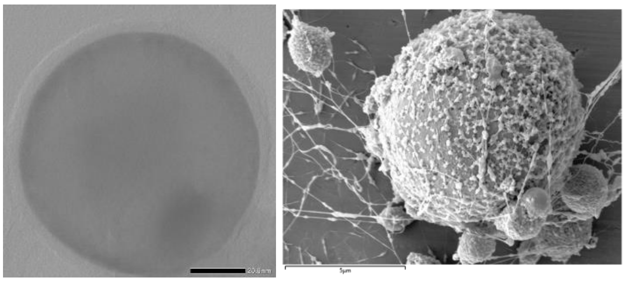

Fig. 1. Examples of electrospray-coated nano and micron-sized aluminum particles

Experiments with 20 nm, 100 nm, 10 μm, and 50 μm aluminum powders investigated the electrostatic spray coating effect. SEM/TEM techniques examined the morphology of sprayed samples, and EDS analysis confirmed the core of aluminum microparticles with a PVDF coating layer and nano ZnO dispersion. TG-DSC and ignition experiments characterized combustion performance. Results showed that the thermite prepared by electrostatic spraying had lower activation energy and higher calorific value compared to physically mixed samples. High-speed photography and infrared technology captured combustion experiments, with SEM analysis of combustion products verifying superior combustion stability and reduced sintering in electrostatic spray samples. A comprehensive evaluation revealed the relationship between coating modification effects and the basic particle size of microscale aluminum powder.

Acknowledgements This work was supported by National Natural Science Foundation of China (NSFC, Grants 52176114 and 52306145).

ADVANCED COMPOSITE GEOPOLYMERS BASED ON HALLOYSITE NANOTUBES WITH VERSATILE APPLICATIONS

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The valorization of natural waste constitutes a significant challenge within the framework of the circular economy. Over the past few years, numerous studies investigated innovative methodologies applicable to new remediation technologies. Halloysite nanotubes (HNTs) prove to be versatile for several technological applications including the production of environmentally friendly building materials. With this in mind, herein we show the development of functional composite materials exploiting HNTs for a variety of applications. Initially, we prepared hybrid films based on halloysite and cellulose recovered from Posidonia oceanica sea balls (egagropili), which are marine wastes accumulating along the Mediterranean beaches [1]. The obtained nanocomposite films possessed promising thermomechanical properties suitable for packaging applications. As a matter of fact, the composite films evidenced macroscopic properties comparable to that of paper based on pristine cellulose despite the large amounts of inorganic nanoclay. Moreover, the specific composition of the films allowed to control the microstructure and the wettability of the nanocomposites. Afterwards, the composite films were converted into geopolymers through the alkaline activation of halloysite. Both geopolymers exhibited a compact structure with no surface fractures. Notably, the geopolymerization process resulted in substantial improvements in tensile properties. On the other side, composite halloysite-based geopolymers with beeswax microparticles have been developed, characterized, and employed as protective layer on steel sheets by brush deposition [2]. In this case, the wettability properties of the steel surfaces changed due to the presence of the microwax particles.

These innovative methods represent a significant advancement in the development of sustainable composite materials, providing promising solutions for both packaging and construction applications.

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THE INFLUENCE OF TREATED COFFEE GROUNDS ON THE PROPERTIES OF GYPSUM BINDER

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The consumption of coffee is increasing every year worldwide, as a result, after the coffee brewing process, there is a large amount of coffee grounds left, which are usually not being used anywhere else. By rotting in landfills, coffee grounds emit CO_2, methane gases and thus contribute to the problems caused by global warming. For that reason, it is important to find alternative ways of reusing coffee grounds [1]. It is known that replacing part of the sand with coffee grounds in concrete gives positive research results [2]. Unfortunately, there are just a few studies analysing the effect of coffee grounds on gypsum binders. Also, the treating of coffee grounds is receiving more and more attention, due to the possibility of extracting various substances from them, for example, oil, which can be used in the production of biodiesel [3]. However, there is a lack of information on how the properties of the treated coffee grounds change, or where this material can be further used to prevent the treated coffee grounds from becoming a waste again. The aim of this research is to determine the influence of solvent-treated coffee grounds on the properties of gypsum binder.

Building gypsum ("Knauf Baugips", Latvia) and Arabica coffee grounds (coffee beans country of origin – Colombia) were used in this study. Several different instrumental analysis methods, such as STA, XRD, XRF and FT-IR, were used to analyse the materials. For the treatment of coffee grounds acetone, isopropanol and bioethanol solvents were selected. Various properties, such as water/gypsum ratio (W/G), setting time, compressive strength (by forming 2×2×2 cm cubes), water absorption, open porosity and density of the building gypsum samples with 5 wt.% coffee grounds additive, were determined.

It was found that the W/G ratio of the building gypsum samples with treated coffee grounds additive decreased from 0.70 to 0.60, the initial setting time was at least 6 minutes, the compressive strength increased by 14-42 %, and the water absorption and open porosity decreased, respectively, by 6-9 % and by 7-8 % compared to the samples in which untreated coffee grounds were used. The density of dry samples with (un)treated coffee grounds additive did not exceed 1100 kg/m^3. The FT-IR and STA results showed that the oil content in the coffee grounds decreased after the treatment, which could have influenced the improvement of the properties of the samples with the treated additive.

In conclusion, by using the coffee grounds additive treated with various solvents, it is possible to produce a gypsum binder with good properties, suitable for use in the further production of building materials, while increasing the possibilities of reusing coffee grounds remaining after treatment, as well as, reducing the amount of coffee grounds in the landfills.

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FERMENTATION EFFECTS ON NUTRIENT PROFILE AND ANTIOXIDANT ACTIVITY OF ARTHROSPIRA PLATENSIS

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Arthrospira platensis, known as spirulina, is a nutrient-rich cyanobacterium with numerous applications in the food and nutraceutical sectors [1]. While fermentation is an ancient preservation technique that enhances food materials' digestibility and nutritional value, its impact on microalgae biomass is poorly understood [2].

This study aimed to evaluate the physicochemical changes occurring during the fermentation of spirulina. Four strains, including two lactic acid bacteria (*Lactobacillus plantarum, Lactobacillus acidophilus*) and two bacilli strains (*Bacillus subtilis, Bacillus coagulans*), were selected for submerged fermentation. Samples were collected and analyzed at six-time points from 0 to 72 hours. Microbial counts indicated significant growth for all strains (~8.5 log CFU) with spirulina as the sole carbon source. All strains exhibited a similar acidification rate, with the pH dropping to an average of 5 within 24 hours of fermentation due to the production of organic acids, as confirmed by liquid chromatography. Spectrophotometric analysis revealed a decrease in phycocyanin and allophycocyanin content by 43% and 55% by the end of fermentation, indicating proteolytic activity. HPLC-MS/MS verified the release of free essential and non-essential amino acids. Notably, it was observed that the ratio of essential to non-essential free amino acids increased with the duration of fermentation, reaching its peak at the end (72 hours). Moreover, the same technique verified the presence of B complex water-soluble vitamins, with pyridoxine (B6) produced by fermenting microorganisms during fermentation. Additionally, the *in vitro* antioxidant capacity was assessed using total phenolic content, CUPRAC, ABTS, and DPPH assays, revealing differences across various fermentation times and strains.

On the other hand, LC-MS/MS analysis showed that biogenic amines (primarily tyramine and histamine) are produced during extensive fermentation, though at relatively safe levels. These results suggest that spirulina may be a suitable fermentation substrate, highlighting the fermentation's impact on its physicochemical properties and the potential health benefits of the resulting extracts. However, further studies are necessary to evaluate the bioactivity and safety of the fermented extracts.

Acknowledgements. The study was financially supported by the Research Council of Lithuania, grant no. S-MIP-23-78.

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PINUS NIGRA CONES-BASED CARBONACEOUS ADSORBENTS FOR REMOVAL OF POLLUTANTS FROM WATER

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The water pollution is one of the most important challenges worldwide. Inorganic and organic contaminants in aqueous environment can seriously affect human health and the environment. Conventional wastewater treatment is not efficient enough to remove all pollutants, therefore many methods are investigated to increase the water quality.

Adsorption on activated carbon belongs to the most popular investigated methods due to its relatively low costs and easy implementation [1]. Activated carbon is prepared from various raw material such as carbon black, wood char, coconut shell, etc. [2]. In recent years, many biomass precursors have been used for the preparation of activated carbons [1]. This approach also contributes to the management of biomass waste.

In this work, five samples of carbonaceous adsorbents based on pinus nigra cones were prepared by microwave. One sample was prepared without an activator, the other four were activated with NaOH, Na_2CO_3, KOH, and K_2CO_3, respectively. The prepared materials were tested for the removal of organic (pharmaceuticals) and inorganic (heavy metals) pollutants from water.

Acknowledgements

This work was financially supported by the European Union under the REFRESH - Research Excellence For REgion Sustainability and High-tech Industries project No. CZ.10.03.01/00/22_003/0000048 via the Operational Programme Just Transition.

Experimental results were accomplished by using Large Research Infrastructure ENREGAT supported by the Ministry of Education, Youth and Sports of the Czech Republic under project No. LM2023056.

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Oral presentations 3

Chemical and Physical Depositions, Coatings and Thin Films.

Surface and Interface Science.

ADVANCING NANOSCALE MATERIAL SYNTHESIS THROUGH NOVEL PLASMA DISCHARGE TECHNIQUES

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Recently, there has been an increased interest in preparing disordered formations. This requires a reduction in the ordering at the atomic scale, which, particularly for soft metals, necessitates very fast cooling rates to prevent thermodynamically favoured crystallization during cooling of molten metals. Meeting these demands has been challenging, but certain physical deposition methods have shown promise. One such method is spark plasma ablation deposition, which offers a powerful, green, effective, and cost-efficient approach. Utilizing only a carrier gas, an electrode, and electricity, this deposition generates a plasma cloud from sparked electrodes that rapidly condenses by cooling, producing atomic clusters. These clusters then coagulate into nanoparticles, which can be streamed for deposition through a nozzle. This deposition even allows for the creation of homogeneous mixtures of precursor metals on a nanoscale when coagulation is induced between multiple materials. The versatility of our deposition configurations allows for the deposition of up to four different elements without lateral manipulation in the point-based configuration, while the printer-based configuration enables micrometric 2D control of deposition.

In addition to the challenges of preparing soft metals, characterizing them presents its own set of difficulties. Recent advancements in flash scanning calorimetry have proven invaluable in shedding light on fast thermal and energy transitions occurring during high-energy processes such as laser, plasma, and combustion processing. However, conventional methods face limitations due to the inhomogeneity caused by the mixing of micrometre-sized particles. Moreover, our deposition offers the homogenous preparation of soft metals but also facilitates precise and homogeneous positioning of these samples onto flash scanning calorimetry chips.

Investigating systems like tungsten/tungsten oxide, titanium/titanium oxide and copper/copper oxide nanoparticles showcases the ability of our deposition to introduce lattice defects and control organisation at the atomic level, offering insights into surface morphologies and material properties.

In conclusion, spark plasma ablation deposition emerges as a transformative method bridging the gap between material design and deposition, offering a pathway to tailor-made materials with enhanced performance and properties.

Acknowledgements: This work has been funded by the project UIP-2019-04-2367 by the Croatian Science Foundation.

SPECIFIC ION EFFECTS ON FORMATION AND PROPERTIES OF POLYELECTROLYTE MULTILAYERS: THE CASE OF DIVALENT CATIONS

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Polyelectrolyte multilayers are very well known surface coatings which could be prepared by alternating deposition of positively and negatively charged polyelectrolytes (polycations and polyanions) on a solid surface [1-3]. In our study, the multilayer films made of strong polyelectrolytes poly(diallyldimethylammonium chloride), PDADMAC and poly(sodium 4-styrenesulfonate),PSS were built-up on silicon wafer using the layer-by-layer method. The films were built-up in the presence of various divalent cations (Mg^{2+}, Ca^{2+}, Sr^{2+}, Ni^{2+}, Zn^{2+}, and Cu^{2+}) to examine how the nature of cations affects the properties of the film. The results have shown that the thickness, morphology, and roughness of films prepared in the presence of transition metal cations are not significantly different. In contrast, these properties varied for multilayers prepared in the presence of alkaline earth metal cations.

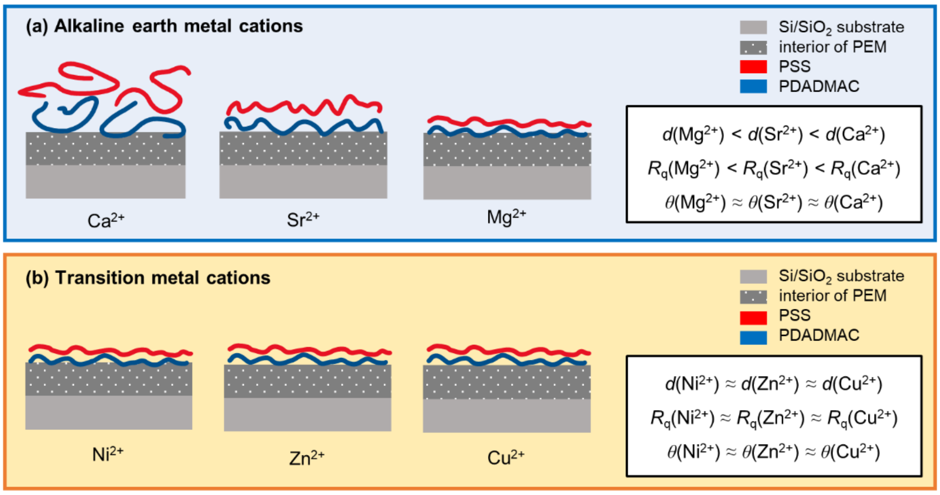

Fig. 1. Schematic representation of PDADMAC/PSS multilayers build-up in the presence of different (a) alkaline earth cations and (b) transition metal cations.

The difference in the ion-specific behavior of these two classes of cations was explained by the difference in the hydration of these ions and by the bridging of polyelectrolyte chains with ions. While transition metal cations have similar hydration parameters, alkaline earth cations have different degrees of hydration and a better ability to form bridging bonds with polyelectrolyte monomers.

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THE MICROSTRUCTURE AND NANOTRIBOLOGICAL PROPERTIES OF THE CHROME AND NICKEL CO-DOPED DIAMOND-LIKE CARBON NANOCOMPOSITE FILMS

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Diamond-like carbon (DLC) films due to the high hardness, low friction coefficient and excellent wear resistance are used as protective coatings in various fields such biological implants, automotive industry, micro/nano-electromechanical systems etc. However, relatively high internal stresses and high brittleness limit the further application of the DLC films. To overcome these issues, doping of DLC films technology should be used [1]. Various metallic elements, such as Cr, Ti, Ag, Cu, Mo and Ni are used to reduce the brittleness and to improve the tribological and mechanical properties of DLC films [2, 3]. The main aim was to deposit Cr/Ni co-doped DLC films and to determine the influence of the metal concentration on the morphology, structure and nanotribological properties of the nanocomposite films.

Ni and Cr with various concentrations were co-doped into DLC films by magnetron sputtering technique. The graphite and the Cr-Ni cathode currents were fixed at 1.5 A and 0.25 A, respectively. The deposition was performed on the Si (100) substrates. The concentration of the Cr and Ni was managed by adjusting a slit wide in a shield mounted above the metallic Cr-Ni target. The structure of the films was analyzed by Raman spectroscopy (RS). The atomic force microscopy (AFM) was used to investigate surface morphology and nano-tribological properties. The elemental composition was determined by an energy dispersive X-ray spectroscopy (EDX) and X-ray photoelectron spectroscopy (XPS). The nano-hardness was determined by nano-hardness intender. The EDX results indicated that the amount of Cr and Ni in DLC films varied from 2 to 5 at.% and from 4.5 to 22 at.%, respectively. The RS and XPS results revealed that the fraction of sp^3 C-C bonds was reduced with the increase in concentration of Cr-Ni metals due to the formation of metal carbides and increase of the oxygen content. The AFM results indicated that the surface roughness was reduced with the addition of low amount of Cr-Ni. The friction coefficient of the film was reduced when the low concentration of the Cr-Ni was used. The DLC film without the Cr-Ni has the lowest nano-hardness (5.5 GPa) and Youngs modulus (40 GPa) values. The increase of the Cr/Ni concentrations enhanced the nano-hardness and Youngs modulus of the Cr/Ni co-doped DLC films.

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CALORIMETRY AS A TECHNIQUE FOR INVESTIGATION OF PROCESSES AT THE MINERAL/AQUEOUS SOLUTION INTERFACE

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Mineral/aqueous electrolyte interfaces play an important role in the environment and technology. The chemical properties of minerals, as well as the composition of aqueous medium determine the processes at the surfaces. In the case of chemically nonreactive materials, surface processes may be simple and involve only the distribution of potential determining ions between the interface and the bulk of the solution. However, at the metal oxide surfaces the surface complexation [1] and adsorption of ions and molecules take place. In some cases, dissolution / crystal growth, surface transformation and degradation/corrosion may occur.

The processes that occur at the mineral/aqueous solution interface are explored using the dissolution calorimetry as well as isothermal reaction calorimetry [2]. Different solid surface are considered: an example of the insoluble oxide (TiO_2) [3], an example of the semi- soluble mineral (SiO_2) [4] and an example of the soluble mineral (CaF_2) [5]. Calorimetric and potentiometric acid base titrations were performed outside of zero charge region, where electrostatic contribution to enthalpy of surface reaction is significant. The results were interpreted by surface complexation model. The interpretation enabled the evaluation of standard protonation and deprotonation enthalpies, and electrostatic contribution to these quantities.

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PICKERING EMULSIONS STABILIZED BY ZnO: IN SITU-FUNCTIONALIZATION AND STIMULI-DRIVEN INVERSION MECHANISM

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Pickering emulsions have attracted increasing attention due to their advantages compared to conventional surfactant-stabilized emulsions and represent promising platforms for applications in numerous fields, such as medicine, pharmaceutics, cosmetics, food industry, and cultural heritage protection [1]. The wetting properties of the stabilizer particles dictate their detachment energy from the oil-water interface [2]. Therefore, oxide-stabilized emulsions generally require a surface functionalization step to tune the oxide wettability, often involving hazardous hydrophobizing agents. We have recently reported the one-step formation of ultra-stable Pickering emulsions stabilized by bare oxides without the addition of any surface modifier [3]. We proved that the formation mechanism is based on the *in situ* functionalization of ZnO by fatty acids naturally present in a broad variety of vegetable oils. The resulting emulsions display excellent stability over time and against temperature variations, mechanical stress and increased ionic strength. Furthermore, these *in situ*-functionalized Pickering emulsions can display phase inversion by multiple stimuli: acidification by mineral and organic acids, UV and sunlight irradiation, addition of multivalent cations and CO_2 bubbling. These versatility can be traced back to the amphoteric and semiconductor properties of ZnO [4]. Depending on the stimulus, the switching kinetics and reversibility can be tailored. Switching by acidification, light irradiation or CO_2 bubbling is fully reversible upon either pH increase, N_2 bubbling or storage in the dark. Even after consecutive cycles, stable oil-in-water Pickering emulsions could be re-obtained. Irreversible destabilization can instead be triggered by excess addition of acids and multivalent cations. The switching kinetics can be modulated achieving either an on-off behavior or a controlled destabilization over several hours. While the inversion of Pickering emulsions is generally related to wetting changes, we here propose an inversion mechanism driven by surface charge-mediated flocculation, which can be reversed by a suitable counter-stimulus able to recover the starting z-potential of ZnO particles.

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Oral presentations 4

Catalysis, Electrocatalysis and Photocatalysis.

Chemical Kinetics.

Porous Media and Sorption

VISIBLE LIGHT PHOTODEGRADATION-ADSORPTION EFFECT OF RECYCLABLE INDUSTRIAL WASTE

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The aim of our research was the development of a procedure to remove organic pollutants from wastewater using recyclable industrial waste. This goal addresses two, nowadays more and more relevant problems, the increasing organic pollution in natural water and the costly recyclying of industrial waste, proposing an environmental friendly solution for both.

The photodegradation and/or adsorption of industrial waste such as red mud was studied using multiple model agents in aqeous and pharmaceutical agents in alcoholic solution. The research work covers the investigation of the reducing effect on model and real organic pollution. The rate of adsorption and photodegradation effect was successfully determined. The effect of the red mud's components were also investigated separately and in their mixture.

These samples were characterized using ultraviolet–visible (UV-VIS), fluorescence and Fourier-transformed infrared spectroscopy (FTIR).

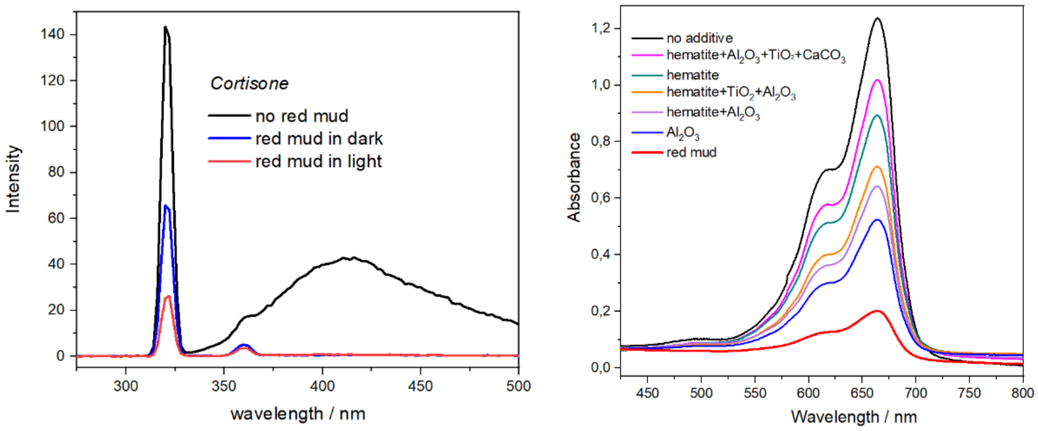

Fig. 1. The fluorescence spectrum of cortisone solutions (left) show the result of the red mud treatment on a prodrug agent, and the UV-VIS spectrum of methylene blue solutions (right) show the degradation of the organic agent in function of the added red mud model mixtures' composition

The results show that the treatment was more effective in the case of red mud, than when the components were applied in any combination, which implies that the unique phase composition of the red mud has a synergetic effect.

PHOTOCATALYSIS OF METHYLENE BLUE DYE BY TIN DOPE HYDROXYAPATITE NANOPARTICLES

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Dyes are essential chemicals needed in the textile, tanning and printing industries. However, the treatment of dye effluents has been problematic, making it a leading contaminant in wastewater. Many solutions are being proposed and studied for the remediation of dyes, including filtration, ion exchange, coagulation, biological treatment, adsorption and photocatalyst. For scientists and researchers alike, the effects of the overabundance of dyes such as methylene blue (MB) in the environment have presented a difficulty. The application of an enhanced oxidation method in wastewater opens the door for the development of a sustainable photocatalyst because of the dye's recalcitrant nature.

Here, hydroxyapatite (HAp) from clamshells (Galatea paradoxa) was used as a photocatalyst to study the photocatalytic degradation of MB. This work used wet chemical synthesis to prepare hydroxyapatite and hydroxyapatite doped with concentrations of 5 wt% and 10 wt% of tin was prepared using the same technique and subjected to calcination at 900°C at 5°C/min for 1, 2 and 3 hours. The powders obtained were characterised using SEM, EDS, FTIR, BET and UV-vis. The photocatalytic study was performed under UV irradiation (90 W, 240 nm) for 180 minutes. The optical band gap observed was approximately ~ 4eV and ~3 eV for hydroxyapatite and tin-doped hydroxyapatite respectively. The findings revealed an increased photocatalytic activity upon the doping hydroxyapatite with tin.

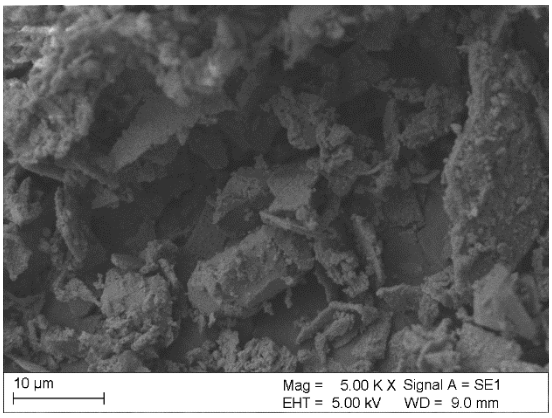

Fig. 1. Micrograph of hydroxyapatite nanoparticles

THE ROLE OF COPPER OXIDE IN TRANSIENT LIQUID ASSISTED GROWTH OF REBCO SUPERCONDUCTING FILMS

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Transient liquid assisted growth (TLAG) is a growth process that allows obtaining high-temperature $REBa_2Cu_3O_7$ (REBCO) superconducing films at growth rates above 100 nm/s and high critical currents (5 MAcm−2 at 77 K) [1,2]. The critical step that controls the growth kinetics is the decomposition of barium carbonate. Thus, understanding the mechanism of barium carbonate decomposition during TLAG is relevant to optimize the processing conditions for REBCO synthesis.

The complete decomposition of $BaCO_3$ powders in an inert argon atmosphere can reach 1300°C [3], which is much higher than the decomposition temperature of YBCO [4]. Thus, it was considered that the presence of $BaCO_3$ hinders the formation of YBCO preventing the synthesis of high performance CC [5]. However, it has been shown that it is possible to grow YBCO films at temperatures as low as 700 °C [1].

Acknowledgements. This work was funded by Spanish Ministry of Science, Innovation and Universities REBCOTLAG project PID2021-127297OBC22.

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KINETIC MODELING OF THE AAEM-CATALYZED PYROLYSIS OF DIFFERENT BIOMASS TYPES – A THERMOGRAVIMETRIC ANALYSIS-BASED STUDY

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In today's carbon-constrained world, renewable energy carrier development is a key component of the strategies adopted to tackle ever-growing energy needs while meeting overarching greenhouse gas emission reduction goals. In this context, the use of low-emission and carbon-neutral renewable energy resources, such as biomass, has attracted interest, with a view to gradually substituting conventional fossil fuels. When being pyrolyzed under an inert atmosphere, the biopolymers composing biomass typically decompose into biochar, bio-oil and incondensable biogas. Pyrolysis products obtained directly from raw biomass, however, usually present certain disadvantages due to their high oxygen content, leading to high corrosiveness and a low heating value. To address this, an interesting option is to implement a catalytic treatment of biomass to optimize selectivity and remove oxygenated groups to produce upgraded pyrolysis products. Among the catalysts currently considered, specific attention is devoted to alkali and alkaline earth metals (AAEMs) due to their low toxicity, affordability and catalytic efficiency. Notwithstanding the findings reported in recent studies aimed at elucidating the impact of AAEMs on the catalytic conversion of biomass, further research, based on a systematic comparison of the impact of AAEMs added to the same feedstocks under the same conditions, are required to determine the respective catalytic efficiency of these additives, notably from a kinetic perspective. The present work thus aims at characterizing the catalytic pyrolysis of 5 different feedstocks (spruce wood, wheat straw, switchgrass, miscanthus and swine manure) impregnated with 4 additives containing AAEMs (NaCl, KCl, $CaCl_2$ and $MgCl_2$). To that end, thermogravimetric analyses (TGA) were carried out with raw and catalyzed samples prepared by wet impregnation. The results obtained were then processed by means of 3 isoconversional models (namely KAS, FWO, and Friedman). Based on the obtained rate constant parameters, the variation of the fuel conversion degree as a function of the temperature was simulated while considering different reaction models commonly employed in the literature. As highlights, this work showed that the computed conversion profiles derived from the implementation of each tested modeling approach allowed to properly reproduce the TGA results, as exemplified in the case of the Friedman model.

Furthermore, the existence of catalytic effects was evidenced by a shift of the decomposition process to lower temperatures when the tested feedstocks were impregnated with the AAEM additives. The analysis of kinetic parameters showed that the rate constants tend to increase when adding catalysts, noting that $CaCl_2$ and $MgCl_2$ exhibit a stronger ability to increase pyrolysis rates as compared to NaCl and KCl.

THERMAL STABILITY AND DEGRADATION OF THREE SIMILAR-STRUCTURED ENDOGENOUS ESTROGENS

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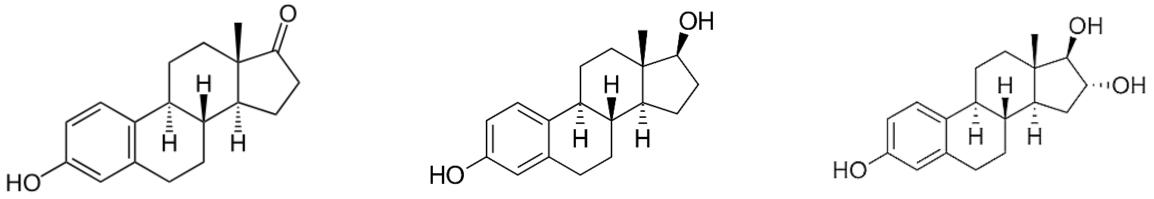
Estrogens are cholesterol-derived hormones, which play essential functions in the human body, beside the role in the reproductive function, such as: their protective effects against neurodegenerative diseases and being a key regulator of bone metabolism [1–3]. There are three types of estrogens produced endogenously, namely estrone, estradiol, and estriol (their chemical structure is presented in Fig. 1). They are used therapeutically in contraceptive therapies for premenopausal women and hormonal replacement therapies for postmenopausal women [3].



Estrone **Estradiol** **Estriol**

Fig. 1. The chemical structure of the three endogenously produced estrogens

Considering the low doses used in the pharmaceutical formulations for these three compounds (≈0.5 mg), it is very important to understand the effect of thermal stress over the kinetics of decomposition of these compounds. As investigational tools, thermoanalytical methods (TG/DTG, DSC) were employed and later the data were processed using several kinetic methods: a preliminary method (ASTM E698), two isoconversional methods (Flynn-Wall-Ozawa and Friedman) and the modified non-parametric kinetic method (NPK).

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IMPACT OF TEMPERATURE ON ZNO SYNTHESIS AND ITS PHOTOELECTROCHEMICAL PROPERTIES

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Temperature significantly influences ZnO synthesis and properties affecting its size, the crystalline structure [1], grain size, and photoluminescence of ZnO thin films, impacting photocatalysis [2]. Calcination temperatures alter the phase composition, crystallinity, and photocatalytic activity of ZnO-SnO_2 nanocomposites, with higher temperatures enhancing photodegradation [3]. Annealing temperatures also affect the photoelectrochemical efficiency of ZnO thin films, with optimal performance at specific conditions [4].

Materials and methods:

Zinc acetate (99.5% purity) was used to synthesize ZnO by heating 1.5 g of $Zn(CH_3COO)_2{\cdot}2H_2O$ in alumina crucibles with perforated foil in a SNOL 8.2/1100 furnace at 400 °C to 600 °C for 1 hour, producing white-grey ZnO powder. For the suspension, 1.5 g of ZnO was dispersed in 150 ml of methanol (10 mg/ml) and sonicated for 30 minutes. ZnO coatings were formed on TEC-15 glass plates using a DC power supply at 15 V to 30 V and deposition times of 5 to 30 minutes, with ZnO deposited at the anode.

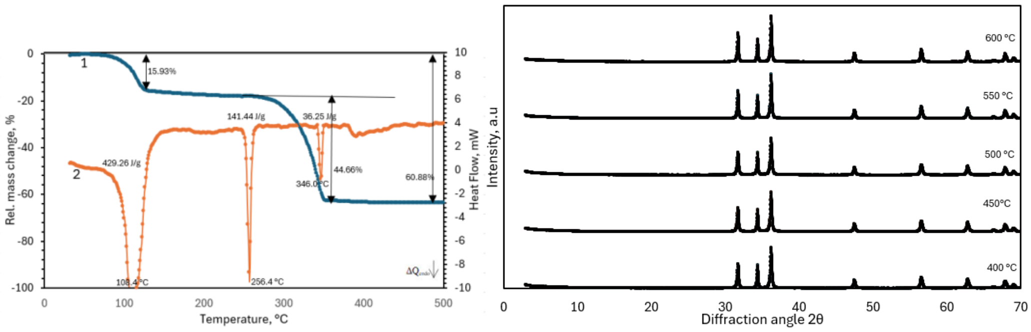

Figure 1: a)-Thermal Transformation Characteristics of Zinc Acetate Dihydrate Precursor: 1-TGA, 2-DSC b) XRD patterns of ZnO synthesized at different temperatures °C: 400, 450, 500, 550, 600

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Oral presentations 5

Advanced Methods for Characterization of Materials.

Photonic Interaction with Matter.

Materials for Nanophotonics and Nanoelectronics.

Thermal Science and Engineering

SPACE TIME OF NON-EQUILIBRIUM SYSTEMS: PHONON IN STATIONARY AND EXCITED STATE

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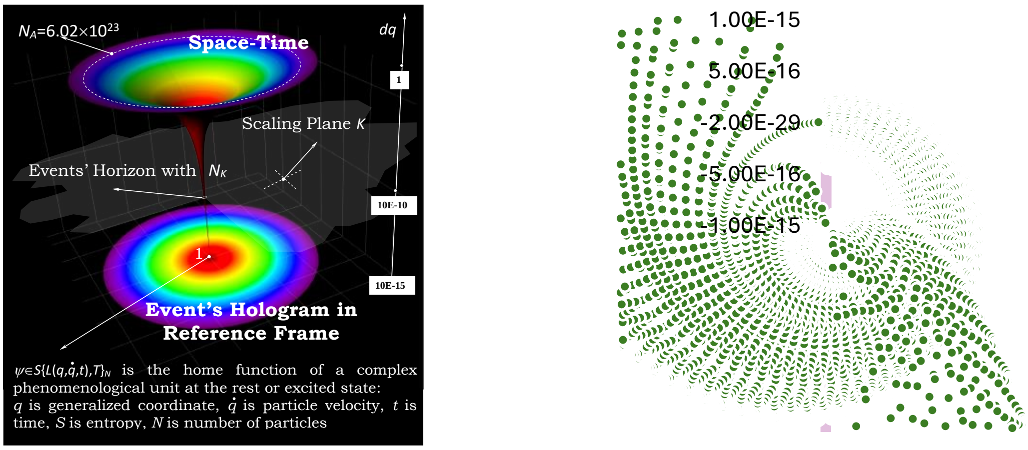
Space Time of Non-Equilibrium Systems is a theory based on the home function of generalized algebraic I-ideal, that commutes the sets of four-dimensional (q_1, q_2, q_3, t) space times with the non-zero curvatures of generalized coordinates q_i of a particle in a period dt modeled in the valence orbital of an element of the ground state. This monomial expression generates transformations that are used to describe the dynamic phases of physical and chemical processes using the experimental time dt measurement capability. The initial structure of the monomial expression contains the singletons $\{q_0\}$ and $\{t_0\}$ related to the electron motion, which are placed in a regular spacetime, what allows us to generalize them on the scale n with their transformations in the so-called commutation ring R:

$$R[It] = \bigoplus_{n=0}^{\infty} I^n t^n \subseteq R[t]. \tag{1}$$

Also, using the operating limits of this generalized function (Eq.1), a physical model with particle and wave properties was developed. This physicochemical model is described as the statistically possible electron states corresponding to a propagating wave of a given density, shown in Fig. 1.



a

b

Fig. 1. The Space time with its hologram (a) and wave function visualization of excited phonon states (b)

The monad has universal properties, regardless of the chosen methods used in an analysis of the physicochemical phenomena. The philosophy of early spacetime led to the emergence of specific paradigms that helped to model the constants and other quantities of pure crystalline substances, experimentally confirmed by many authors.

CLUSTERING-TRIGGERED EMISSION OF CELLULOSE-BASED MATERIALS

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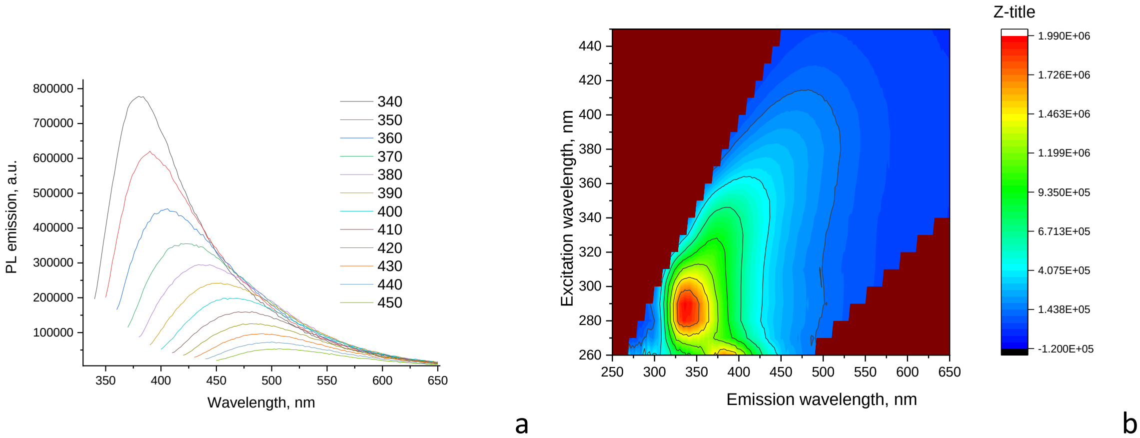
Cellulose-based materials can be classified as non-conventional luminogens that produce photoluminescence in the visible range due to specific intermolecular arrangement. Such an arrangement is usually referred to as clusterization. Here, we demonstrate the importance of intramolecular arrangement of ethyl cellulose macromolecules that yields the features attributed to the clustering-triggered emission, i.e., a tunable photoluminescence (PL), a multiexponent PL decay, and a modest PL quantum yield of about 2% (Fig.1). We show that the observed PL emission is due to n-π* electronic transition of the carboxyl group which probably originates from the chain cleavage in the presence of water, while the emission intensity depends on the form of the sample preparation, either in the powder or spin-coated film, displaying the different density of the emission regions on the microscale as observed by fluorescence microscopy. The emission is also promoted by mechanical stress applied to the sample that probably facilitates formation of the carboxyl groups and/or their specific arrangement. Stress-assisted emission opens up the potential perspective of using this phenomenon in printed photonic devices.



a b

Fig. 1. (a) Fluorescence spectra and (b) excitation-emission map of ethyl cellulose powder.

Acknowledgements. The work was supported by the Knut and Alice Wallenberg Foundation (KAW) through the Wallenberg Wood Science Center.

THERMAL BEHAVIOUR AND THERMOKINETIC STUDY OF SOME CHLORO-AZOMONOETHERS

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From a new class of chloro-azomonoether derivatives of 1-(4'-((4-chlorobenzyl)oxy)-[1,1'-biphenyl]-4-yl)-2-phenyldiazene that were synthesized by using the condensation of different sodium salts of some 4'-phenyldiazenyl-biphenyl-4-ols with 1-chloro-4-(chloromethyl)benzene, the thermal, thermodynamic and kinetic behaviour and properties of five such compounds were investigated.

The thermal analysis (thermogravimetry, derivative thermogravimetry and differential thermal analysis) and calorimetric (differential scanning calorimetry) measurements for these compounds were performed in dynamic air flow (flow rate of 150 $cm^3 \cdot min^{-1}$) in order to determine their thermal behaviour; after melting, two exothermic steps – composed of several chemical reactions for each step – were observed. Also, the decomposition kinetics was carried out for experiments at various heating rates between 2 and 10 $K \cdot min^{-1}$. The kinetic calculations were done with TKS-SP 2.0 software for determining the kinetic parameters, and allowed obtaining the activation energies of the thermoxidative decomposition process (1st step) and burning of the polycondensed residues (2nd step). The activation energy diagrams for these two steps were obtained based on calculations using the modified Ortega isoconversional method, for all five compounds.

The compounds were furthermore studied in terms of their structural and energetic properties using DFT computational methodologies. The equilibrium geometries of the five chloro-azomonoethers derivatives in the gaseous phase were analysed in detail and the structural effects on the aforementioned properties were assessed.

The good thermal and kinetic stability of these compounds allows for studying their interaction with proteins. For this, the binding with bovine serum albumin (BSA) was investigated by molecular docking, revealing a high affinity of the compounds to fatty acid site 5 (FA5) of subdomain IIIB of BSA by hydrogen bonds and hydrophobic interactions. The type of substituent influences the conformation of the compounds and their binding process with the protein. The effect of binding the dyes to the protein was further investigated by microcalorimetry in order to reveal the thermal stability of these systems.

CO-PYROLYSIS CHARACTERISTIC OF NANO-NITROCELLULOSE WITH STABILIERS: THERMAL STABILITY, THERMAL KINETIC ANALYSIS AND THERMAL DECOMPOSITION MECHANISMS

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Nanoscaling of nitrocellulose reduces mass and heat transfer distances, thereby enhancing combustion performance. However, the thermal stability, kinetic properties, and thermal decomposition mechanisms of nanoscaled NC are not fully understood. This study utilized electrospinning and mechanical mixing to prepare NC of different sizes, incorporating N-methyl-p-nitroaniline (MNA) and 3-methyl-1,1-diphenylurea (AKII) additives to create nano-composite nitrocellulose. Characterization via Focused Ion Beam Scanning Electron Microscopy (FIB-SEM) assessed sample morphology and the influence of stabilizers. Thermal stability and safety were evaluated using Fourier Transform Infrared (FTIR) Spectrometer and Thermogravimetric Analysis-Differential Scanning Calorimetry (TG-DSC) under varying heating rates. The study compared and analyzed the thermal properties of nanoscaled composite nitrocellulose, employing model-free methods to calculate activation energy relationships and kinetic parameters, alongside master-plots for mechanism function determination.

Results highlighted solvent selection's impact on fiber morphology; DMF addition yielded smoother fibers than AC, reducing needle clogging. Additives decreased fiber diameter and increased surface area. Nanoscaled nitrocellulose exhibited size-dependent thermal decomposition characteristics, with stabilizers reducing onset temperatures, broadening reaction ranges, slowing decomposition, decreasing maximum rates, and increasing heat release. Nanoscaling facilitated enhanced heat absorption and nitro group generation during NC thermal decomposition, emphasizing its potential for catalytic effects. This study informs optimization of NC and stabilizer mixtures, advancing applications and providing insights for nanomaterial preparation and thermal stability studies.

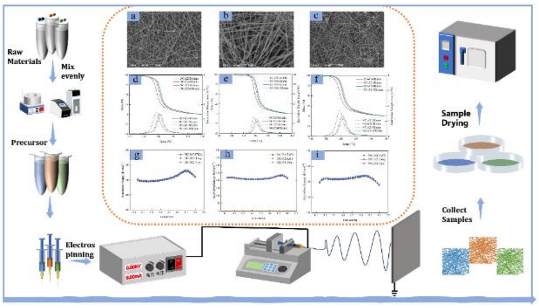

Fig. 1. Graphical abstract

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Poster presentations 1

Biophysical Chemistry, Biomaterials and Biomedical Applications.

Chemical and Physical Depositions, Coatings and Thin Films.

Chemical Thermodynamics.

Materials Science and Technology

COMPATIBILITY STUDIES OF SILDENAFIL-HPBCD INCLUSION COMPLEX WITH PHARMACEUTICAL EXCIPIENTS

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Sildenafil citrate (abbreviated SC, chemical structure presented in Fig. 1) is an inhibitor of phosphodiesterase type 5, which is therapeutically used for pulmonary arterial hypertension, in the form of coated tablets [1]. Due to its low biodisponibility, belonging to the II class of the Biopharmaceutics Classification System [2], and the lack of various pharmaceutical formulations used for this disease, it is necessary to develop new formulations that have an optimised biopharmaceutical profile.

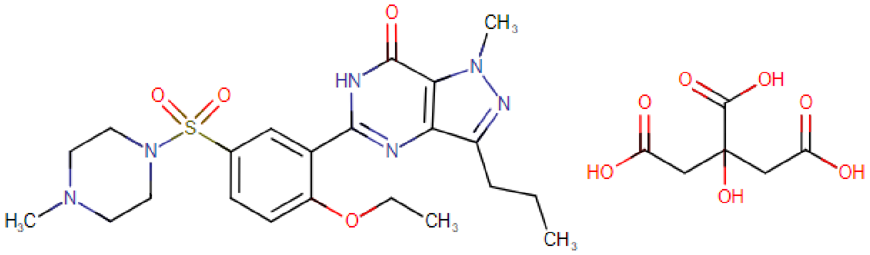

Fig. 1. The chemical structure of sildenafil citrate

The purpose of this study is to obtain and analyse the inclusion complex (IC) between SC and 2-hydroxylpropyl-β-cyclodextrin (HPBCD), a cyclodextrin approved by the FDA for use in pharmaceutical formulations [3]. Furthermore, we set our aim to find out if there are any chemical interactions between the IC and several pharmaceutical excipients, by using the following analytical methods: thermal investigations (TG/DTG, DSC), FTIR, and PXRD. Also, for a better interpretation of the results statistical analysis will be performed.

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BINARY ADDUCTS OF PRASTERONE – PREPARATION AND INSTRUMENTAL INVESTIGATIONS

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Cocrystals are represented by a single crystalline phase formed between the active pharmaceutical ingredient and coformers (such as amino acids, carboxylic acids) linked by non-covalent interactions [1]. Their significant role in the pharmaceutical industry derives from the fact that they increase the dissolution and bioavailability of the active pharmaceutical ingredient. Prasterone or dehydroepiandrosterone (abbreviated PRS, chemical structure presented in Figure 1) is an FDA-approved food supplement used in hormonal therapy, which presents a very low bioavailability, namely 3.1% in the rhesus monkey [2].

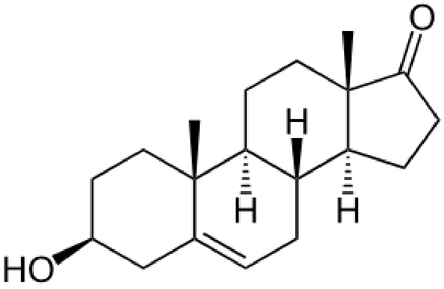

Fig. 1. The chemical structure of prasterone

In order to increase its bioavailability, the purpose of this study is to synthesize cocrystals of PRS with three dicarboxylic acids: succinic acid, malonic acid, glutaric acid, one alpha hydroxy acid: malic acid and one tricarboxylic acid, namely citric acid. The cocrystals obtained were analyzed by instrumental methods, such as: thermal investigations (TG/DTG, DSC), infrared spectroscopy (FTIR) and PXRD.

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IONS RELEASE, MICROHARDNESS AND CYTOTOXICITY OF Mg-DOPED ZrO_2 BIOCERAMICS

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Zirconia-based biomaterials have emerged as promising candidates for various biomedical applications, owing to their exceptional properties such as good fracture toughness, mechanical strength, high ionic conductivity and resistance to corrosion [1-4]. Further, zirconia facilitates osseointegration, presents low ion release in biological environments, such as artificial saliva (AS) and possesses a lower grade of cytotoxicity [5]. In this work, cytotoxicity, dissolution and mechanical properties of $Zr_{1-x}Mg_xO_2$ (x = 0.05, 0.1, 0.15, 0.2, 0.25, and 0.3) bioceramics, prepared by solid-state reactions at high temperature, were evaluated in AS, after immersion for one month. The data delivered by ICP-OES measurements on the elemental concentration after immersion in AS revealed the release of Mg^{2+} ions to be between 34.0 and 60.6 mg L^{-1}, depending on the composition, whereas Zr^{4+} ions concentration was below the limit of detection. Microhardness evaluation of the samples showed that measured Vickers microhardness (VMH) of the bioceramics decreased with increasing the amount of Mg on the samples, with the best value (213.55HV30) achieved for x = 0.05 composition. The biocompatibility of the prepared materials was tested using normal human skin fibroblasts (BJ cell line, ATCC CRL2522). The results confirmed that the ceramic materials are biocompatible, support adhesion and stimulate cell growth.

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SULFOBUTYLETHER β-CYCLODEXTRIN OF QUERCETIN. COMPATIBILITY WITH EXCIPIENTS

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Quercetin (QUE), 2-(3,4-dihydroxy phenyl)-3,5,7-trihydroxychromen-4-one (Figure 1), is a polyphenol possessing a broad spectrum of pharmacological activities, including antioxidant, anti-viral, anti-diabetic, antiplatelet aggregating, anti-cancer, anti-obesity, neuroprotective, hepatoprotective, cardioprotective actions [1–3]. It also shows promising effects on the skin, such as anti-inflammatory, anti-aging, anti-bacterial, anti-fungal, anti-psoriasis, wound healing, anti-itching, skin whitening and photoprotection [3]. QUE low aqueous solubility along with poor percutaneous absorption, chemical instability and rapid enzymatic degradation limit its pharmaceutical and cosmetic potential [2,3]. To overcome these limitations the inclusion complexation with cyclodextrins was performed.

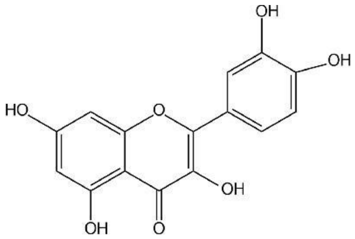

Fig. 1. Structural formula of QUE

The aim of this study was to obtain and evaluate the inclusion complex (IC) of QUE with sulfobuthylether β-cyclodextrin (SBECD). The supramolecular adduct was prepared using the kneading method and was characterized in both solution and solid state in order to evaluate the host-guest interaction. The compatibility of the IC with the most used excipients in oral and topical formulation was also assessed by means of thermal methods (TG/DTG, DSC), and spectroscopic methods such as FTIR spectroscopy and powder X-ray diffractometry (PXRD).

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PREPARATION AND CHARACTERISATION OF INCLUSION COMPLEXES OF CARVEDILOL WITH DIMEB AND RAMEB

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Carvedilol (CARV), an antihypertensive agent prescribed for chronic heart failure, shows poor bioavailability and rapid first pass metabolism when administered orally. In order to develop new oral solid dosage forms with enhanced dissolution profile and bioavailability, cyclodextrin complexation can be employed [1–3]. The structural formula of CARV is presented in Fig. 1.

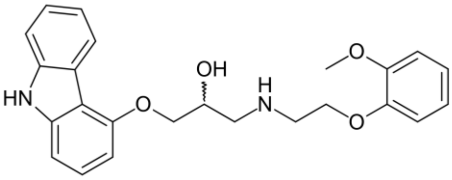

Fig. 1. Structural formula of CARV

The purpose of this study was to prepare and characterize two inclusion complexes (IC) of CARV with two functionalized beta-cyclodextrins, namely randomly methylated β-cyclodextrin (RAMEB) and heptakis(2,6-di-O-methyl)-β-cyclodextrin (DIMEB), respectively. The stoichiometry of the ICs of was determined by applying the Job's method and the values of the stability constants were calculated using the Benesi-Hildebrand method. In solid state, the ICs were characterized by thermal methods (TG/DTG, DSC), FTIR spectroscopy and powder X-ray diffractometry (PXRD). Molecular modeling studies were also carried out for obtaining information over the intermolecular interactions that took place inside the guest-host structure, evidencing the thermodynamic spontaneity of the entrapment process.

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INVESTIGATION OF MULTI-LAYERED ORALLY DISINTEGRATING FILMS COMPOSED OF BIO-BASED POLYMERS AND *SPIRULINA* EXTRACT

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Oral drug administration is the most popular route of pharmaceuticals administration among patients due to its simplicity, non-invasiveness [1]. Orally disintegrating films (ODFs) are thin, flexible and pharmacologically active films that, when applied to the oral mucosa, begin to disintegrate or dissolve and release active ingredients in the target area [2]. Currently, ODFs with prolonged activity are an innovative method for the local treatment of oral cavity diseases. Due to a wide range of inhibitory effects against *Gram-positive* and *Gram-negative* bacteria, as well as strong antioxidant and anti-inflammatory properties, *Spirulina* extract has a great potential to be used as a bioactive material in ODFs, for the treatment of such oral cavity diseases as periodontitis and gingivitis. ODFs composed of bio-based polymers with *Spirulina* extract could be an excellent alternative to surgical intervention and replacement of conventional synthetic antimicrobial agents, which can cause various side effects.

Main goals of this research work were the development of novel orally disintegrating multi-layered films composed of bio-based polymers and *Spirulina* extract and investigation of their properties.

Solvent casting method was employed to attain three-layered films using different bio-based polymers such as methyl cellulose, 2-hydroxyethyl cellulose, chitosan, cellulose diacetate, hydroxypropyl cellulose as film forming materials. Each layer of the multi-layer film contained bio-based polymer, glycerol as plasticizer and citric acid as cross-linker (in some layers). The middle layer also contained *Spirulina extract*. Films without *Spirulina* extract in the middle layer were used as references. Main characteristics of multi-layered films, such as solubility and swelling in a simulated salivary medium, contact angle, mechanical, antioxidant properties were evaluated.

Acknowledgements. This project has received funding from the Research Council of Lithuania (LMTLT), agreement No S-MIP-23-78.

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SYNTHESIS OF MAGNESIUM WHITLOCKITE GRANULES *VIA* A CONTINUOUS DISSOLUTION-PRECIPITATION REACTION

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Synthetic calcium phosphates are extensively utilized in medical and dental applications, including orthopedics, drug delivery systems, dental implants, and alveolar bone augmentation. Their popularity stems from their excellent biocompatibility and strong chemical and biological affinity for bone tissue. The rapidly growing demand for biomaterials in medicine has led to the development of numerous novel synthesis approaches.

This study aimed to create an efficient, cost-effective synthesis route for magnesium whitlockite (Mg-WH; $Ca_{18}Mg_2(HPO_4)_2(PO_4)_{12}$) granules. Our method employs a low temperature dissolution-precipitation reaction to produce granules with the desired Mg-WH phase [1,2]. The study investigated the optimal pH conditions for precipitation of the Mg-WH phase using different combinations of 1 M NaH_2PO_4 and Na_2HPO_4. From an initial 3.00 g of gypsum-based granules, approximately 1.75 g of Mg-WH was synthesized per batch (SEM images of prepared granules are depicted in Fig. 1).

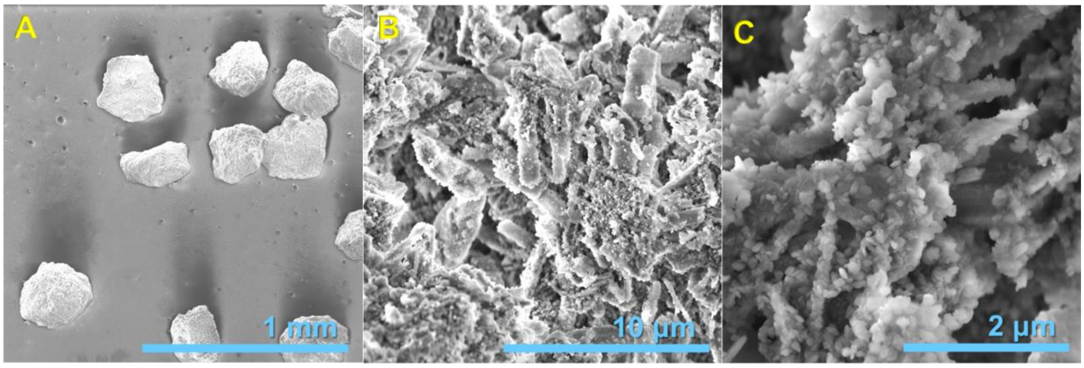

Fig. 1. SEM images of the product of Mg-WH granules at varying magnifications are presented

XRD analysis revealed that the single-phase Mg-WH sample exhibited a crystallinity of 50%, indicating the presence of a mixture of crystalline and amorphous phases. FTIR spectroscopy confirmed the presence of characteristic vibrational modes corresponding to Mg-WH in the synthesized sample. SEM imaging shown irregularly shaped granules with an average size of 329 µm. These granules were composed of interlocked rod-shaped aggregates formed by rhombohedral particles with an average diameter of 70 nm. BET analysis indicated that the synthesized samples exhibited a mesoporous nature.

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PREFORMULATION STUDIES OF PROGESTERONE - AN INSTRUMENTAL APPROACH

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Progesterone (PRG, chemical structure presented in Figure 1) is a human hormone, derived from cholesterol, used in the treatment of endometrial hyperplasia [1–3]. It is available on the pharmaceutical market, in the form of solid (intrauterine devices, tablets, and capsules), semisolid (cream, gel) and liquid formulations. Due to its wide use and numerous pharmaceutical formulations, it is necessary to evaluate the possible chemical interactions between the active pharmaceutical ingredient and the most frequently used excipients, to ensure an optimal dosage of the molecule.

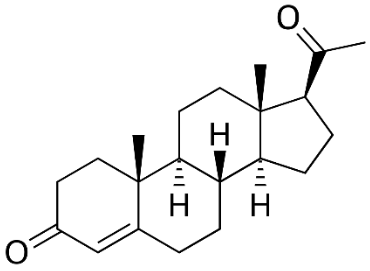

Figure 1. The chemical structure of progesterone

The purpose of this study is to evaluate the compatibility of progesterone with various pharmaceutical excipients used in the development of new solid formulations. All instrumental analyses were performed on binary mixtures of PRG+Excipient, containing a 1:1 mass ratio, and the results were compared to those of PRG and pure excipients. The analytical methods used are thermal analysis (TG/DTG, DSC), FTIR spectroscopy, and PXRD. For a more accurate interpretation of the results obtained, statistical analysis was also applied.

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COMPARISON OF MECHANICAL, PHYSICAL, AND BIOACTIVE PROPERTIES OF SOL-GEL SYNTHESIZED AND MELT-DERIVED LITHIUM DISILICATE-WOLLASTONITE GLASS-CERAMICS

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The aim of the present study is to comparatively evaluate the mechanical, physical, and bioactivity properties of lithium disilicate-wollastonite bioactive glass-ceramics synthesized via sol-gel and melt-derived methods. The phase content and microstructure of the produced samples were analyzed by XRD, SEM-EDS equipments, while the mechanical performance was evaluated by three-point bending and Vickers hardness tests. The physical properties, including bulk density and apparent porosity, were calculated using the Archimedes method. XRD results showed that quartz (SiO_2), lithium metasilicate (Li_2SiO_3), wollastonite ($CaSiO_3$) and lithium disilicate ($Li_2Si_2O_5$) crystalline phases were the main phases precipitated in the glass-ceramic samples produced by both methods at the initial sintering temperature of 750°C. Nevertheless, with the increase in sintering temperature, the intensity of the diffraction peaks corresponding to the lithium disilicate and wollastonite phases increased, while the peak intensities of the quartz and lithium metasilicate phases gradually decreased and became undetectable at the sintering temperature of 850°C. Higher flexural strength and hardness values were obtained in the melt-derived glass-ceramic samples compared to those produced via the sol-gel route. Bioactivity tests were carried out in simulated body fluid according to the protocol described by Kokubo [1] for 3, 7, 14, and 28 days, and the apatite formation on the surface of the glass-ceramic samples after the tests was characterized by TF-XRD and SEM analyses.

Table 1. Chemical composition of synthesized glass-ceramics.

Sample	Lithium disilicate (mol %)	Wollastonite (mol %)
LW-M (Melt-derived)	50	50
LW-S (Sol-gel synthesized)	50	50

Acknowledgements. This work was supported by Sakarya University Scientific Research Projects Units, Project No. 2024-26-62-82.

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PRODUCTION AND CHARACTERIZATION OF ALUMINA-BASED AEROGEL POWDER FROM ALUMINA-CONTAINING RAW MATERIALS AND WASTES

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In this study, it was aimed to produce alumina-based aerogel powder under atmospheric conditions using alumina-based waste materials (secondary aluminum slag, aluminum anodic waste, fireclay mortar and alumina crucible) and a raw material (Seydisehir alumina). Alumina-based materials were dissolved in a base/pure water solution prepared by considering the effects of stirring and temperature. The alumina solutions obtained were filtered. High purity aluminum hydroxide is obtained by precipitating bohamite from aluminum-containing solutions and then washing it from impurity ions. This method is used in the production of alumina-based aerogels because of its relative simplicity [1]. The pH of the solutions was adjusted to neutral by adding an acid/pure water solution and the solutions were then subjected to aging. Aging was continued in solutions containing 20 wt,% pure water/ethanol, ethanol and n-heptane, respectively. Following the aging process, the solutions were filtered and dried under atmospheric pressure at specified temperatures and times to produce alumina-based aerogel powder. The produced alumina-based aerogel powders were investigated by scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), field emission scanning electron microscopy (FESEM), X-ray diffraction (XRD), differential thermal analysis (DTA), Brunauer-Emmett-Teller (BET) analysis, Fourier transform infrared spectroscopy (FTIR). Thermal conductivity coefficients were also measured and density values were calculated. The properties of alumina-based aerogel powder produced using different wastes under different conditions were compared.

The analysis carried out yielded the following results for the alumina-based aerogel powder produced: the particles are nano-sized (10-120 nm) and have a mesoporous structure, thus exhibiting low density values. FTIR analysis revealed the presence of O-H peaks within the porous structure, indicating physical water absorption. The presence of Al-O-H and Al-O peaks indicates that the aerogel powder contains strong alumina bonds. The surface area, particle size and pore volume of alumina-based aerogel powder were determined by BET analysis. The endothermic peaks observed in the thermal analysis results of alumina-based aerogel powder proved the presence of moisture in the structure. The exothermic peaks due to oxidation and new phase formation indicate the response of the powder to temperature change. The thermal conductivity analysis showed a low thermal conductivity coefficient as expected.

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NANOSTRUCTURED SAMARIA AND GADOLINIA DOPED CERIA THIN FILMS FOR LOW-TEMPERATURE SOLID OXIDE FUEL CELLS APPLICATION

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Solid oxide fuel cells (SOFCs) are devices that efficiently convert chemical energy into electrical energy and heat. They consist of a porous anode (660 µm) and cathode (60 µm) separated by a dense electrolyte (30 µm) that conducts oxygen ions [1–3]. The working temperature of SOFC is ~1000 ℃, leading to various problems related to the durability, cost, and application of the device [4]. Therefore, great efforts have been made to decrease the working temperature and increase the power density (*PD*) >1 W/cm^2 by finding new materials exhibiting high ionic and electronic conductivity at low temperatures and creating a new SOFC design. To achieve higher *PD* the thickness of SOFC components should be reduced to <1 µm. the electron beam evaporation technique has advantages against other methods: controlled deposition rate and thickness, material utilization efficiency, purity of thin films, possibility to evaporate even high melting temperature materials, and lower price. In this study, an electron beam evaporation technique was used to produce the samaria and gadolinia doped ceria (SGDC) thin films. The chemical composition of evaporated SGDC thin films with different thicknesses from 200 nm up to 1200 nm (see Fig. 1) were obtained from SEM/EDX analysis that the stoichiometry deviation of the evaporated thin films is, on average, 30 % lower than that of the target materials.

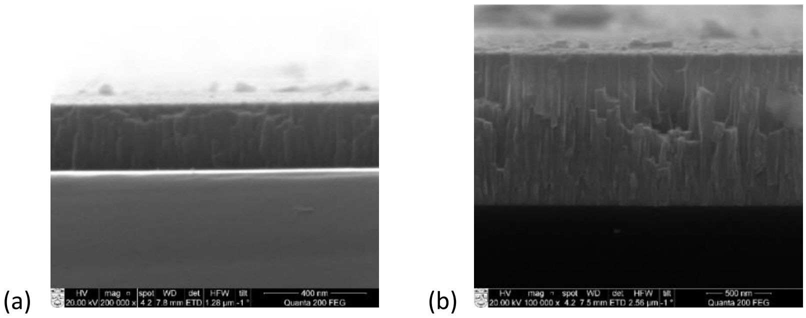

Fig. 1. SEM cross-section views of (a) 260 nm and (b) 1200 nm thick SGDC thin films

To control the chemical composition of SGDC thin films, the target materials should be prepared with a higher stoichiometry using the chemical synthesis routes. The co-precipitation synthesis was used to produce the SGDC as the target material with different stoichiometry.

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CEEC-PCMS 2

STRUCTURAL, MORPHOLOGICAL, COMPOSITIONAL AND OPTICAL PROPERTIES OF Cu_xS FILMS ON FTO GLASS

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The rapidly growing demand for energy is a pressing issue given the growing energy shortages and environmental pollution on a global scale. It is for these reasons that we are looking for ways to produce energy that is durable, inexpensive and low-polluting. The search for alternatives to the burning of fossil fuels has led to interest in copper sulfides due to their favorable optical, electrical and thermoelectric properties. Copper sulfides are non-toxic, inexpensive and have a high energy capacity, which makes them suitable for use in lithium-ion rechargeable batteries, energy storage devices, solar cells and other optoelectronic devices [1–7].

The aim of this work was to synthesize and characterize copper sulfide films on FTO glass by SILAR (sequential ion adsorption and reaction) method which is easy to carry out and is frequently applied to prepare chalcogenide films. The films were obtained by changing the concentrations of cationic ($CuSO_4$ and hydroquinone) and anionic (Na_2S) precursors used. The samples were annealed to improve the crystallinity of copper sulfide films. In this work, the dependence of a composition (XRD, EDS), morphology (SEM) and optical properties (UV-Vis) of unannealed and annealed copper sulfide films on the conditions of film formation was assessed.

In this study by varying the concentrations of the precursors used, copper sulfide films were deposited on FTO glass and the following copper sulfide phases were detected: anilite, digenite, djurleite and chalcocite. It was found that the films of copper sulfides are unevenly distributed, that the crystallinity of the films and particle size increase with increasing annealing temperature from 100 to 400 °C, and that the particles aggregate into agglomerates. The films formed were found to be dominated by copper sulfides and the calculated molar ratio of copper to sulfur varies with the concentrations of the precursors used, ranging from 1.4 to 2.2. The highest bandgap energy value was observed for the unannealed sample when the copper sulfide film was formed using solutions of 0.2 M $CuSO_4$ with 0.2 M hydroquinone and 0.2 M Na_2S. The value of the bandgap energy was not significantly affected by the change of the annealing temperature, varying between 1.2 and 2.6 eV.

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THE METAL-ORGANIC COMPLEX FORMATION AND APPLICATION FOR THE FORMATION OF THIN TIN SULFIDE FILMS

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Metal-organic complexes have gained significant interest in materials science due to their versatile chemical properties and potential applications in various fields, including catalysis, sensors, and thin film deposition. These complexes offer benefits such as tunable composition, controlled morphology, and the ability to form stable precursor solutions for nanoparticle synthesis [1, 2].

This study explores the synthesis and application of a metal-organic complex involving tin(II) chloride, L-ascorbic acid, and ethylenediamine, aimed at developing efficient deposition techniques for tin sulfide thin films. Utilizing the solution synthesis method, we successfully created a stable metal-organic complex, which served as a precursor for tin sulfide nanoparticle deposition via the Successive Ionic Layer Adsorption and Reaction (SILAR) method.

Our findings demonstrate that the synthesized complex is effective in forming porous thin films primarily composed of SnS with traces of SnS_2. Comprehensive structural, optical, and morphological analyses of these films revealed distinct characteristics that highlight their potential for various technological applications. The structural properties indicated a predominantly SnS phase with minor SnS_2 impurities. Optical characterization exhibited favorable band gap values, and morphological studies confirmed the porous nature of the films.

The development of this metal-organic complex and its successful application in thin film deposition present significant advancements in materials science, offering new pathways for the fabrication of tin sulfide-based devices.

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THE STUDY ON CRYSTALLINITY OF IRON OXIDE COATINGS OBTAINED BY MAGNETRON SPUTTERING

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Hematite (α-Fe_2O_3) presenting a corundum structure with Fe^{3+} in octahedral sites, has been identified as a suitable water oxidation catalyst, which conjugates an overall good catalytic performance in both electrolysis and solar-driven water oxidation [1, 2]. Both amorphous and hematite iron oxides have been recognised as promising materials for oxygen evolution as a result of their abundance, nontoxicity, and environmentally friendly nature. The aim of this work was to synthesize iron oxide coatings on stainless steel by magnetron sputtering (MS) technique using pure hematite targets. The MS technique was selected because it achieves high purity, uniformity, and compactness, allowing control of composition, particle size, and crystallinity [3]. Coatings on different types of steels were prepared using three Ar/O_2 ratios applying pulse DC power source. Although the MS technique is more favourable for the formation of crystalline structures, the broadened peaks of the Raman spectrum suggest a semi-crystalline, semi-amorphous structure of the as-prepared coatings (Fig.1A). In order to crystallize the coatings and obtain more ordered structures, the samples were annealed in air at a temperature of 350 °C. Repeated Raman studies demonstrated an increase in crystallinity, as high-intensity peaks characteristic of hematite appeared in the spectra (Fig. 1B). The coatings formed by MS were also electrochemically oxidized to maintain the amorphous structure, but to complete the oxidation of the coatings.

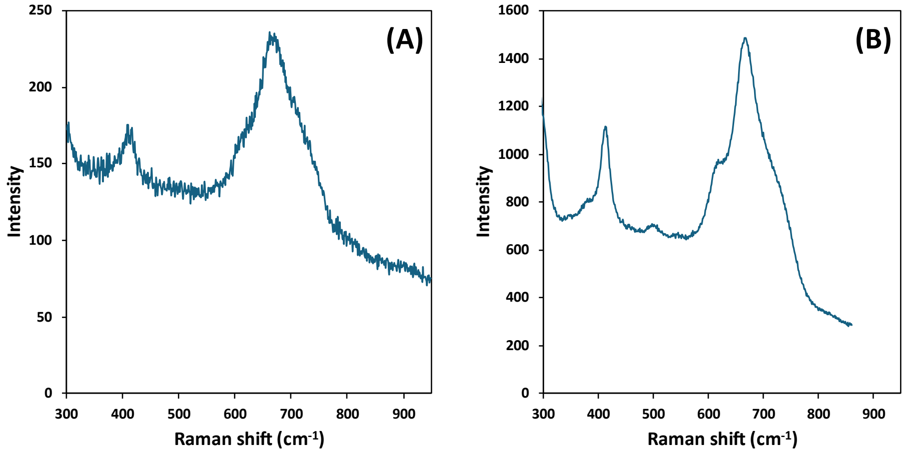

Fig. 1. Raman spectra of as-prepared (A) and annealed at 350 °C (B) iron oxide coatings

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STRUCTURAL AND FERROELECTRIC PROPERTIES OF BISMUTH FERRITE THIN FILMS SYNTHESIZED BY REACTIVE MAGNETRON DEPOSITION

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Bismuth ferrate ($BiFeO_3$) or abbreviation (BFO) is the most promising lead-free ferroelectric materials have lots of attention during the last years. BFO thin films with perovskite structure ABO_3 is assigned to multiferroic material group. Multiferroics have combined two (or more) properties of ferroelectric and ferromagnetic, simultaneously. Multiferroics are characterized as having magneto-electric effect that is to say electric field induces magnetization, and magnetic field induces electric polarization. One multiferroic is not necessarily equal to the other multiferroic, therefore, these materials have attracted interest in material science for potential applications. BFO is ferroelectric material below Curie temperature TC ~ 1100 K temperature it's Neel temperature TN ~ 370°C. BFO films display a greater remnant polarization (Pr) and lower energy band gap than regular ferroelectric materials lead zirconate titanate (PZT). Ferroelectric properties of BFO thin films depend on substrate materials (lattice parameters and thermal expansion coefficient), deposition method, film thickness, measurement frequencies, at al. These properties are possible change with other metals (Zr, Co, Ni, Nb impurities. The influence of the type and concentration of these impurities on the electrical and magnetic properties of BFO is intensively studied. Understanding this influence process is one of the most important tasks in solid state physics.

BFO thin films were deposited on silicon substrate by reactive magnetron layer-by-layer deposition in $Ar+O_2$ gas environment ($p = 1$ Pa) at (500-700°C) substrate temperatures.

The samples were analyzed using scanning electron microscope. The crystallographic structure of thin films was investigated by X-ray diffraction. Sawyer and Tower method for polarization–electric field (P-E) loop measurements were used for hysteresis loops measurements. It was shown that the microstructure and ferroelectric properties depends on deposition temperature. Thin films have dense columnar structure and flat surface. Hysteresis measurements show that all investigated films exhibit ferroelectric properties. The results show that the structure and ferroelectric properties strongly depend on the formation temperature and are very sensitive to stoichiometry of bismuth ferrate films.

FERROELECTRIC AND STRUCTURAL PROPERTIES OF COBALT DOPED LEAD FERRITE THIN FILMS FORMED BY REACTIVE MAGNETRON SPUTTERINGS

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Multiferroic materials are multifunctional materials which demonstrate the interaction of different primary ferroic properties such as ferroelectricity, ferromagnetism, and ferroelasticity. Among the various interactions of ferroic properties the coupling be-tween ferroelectric and magnetic properties attracted the most attention. The possibility to control the ferroelectric and magnetic properties in the same phase provides the ability to develop a wide range of applications on non-volatile memory devices, transducers, magnetic field sensors etc. Special attention is paid to the solid-state memories where multiferroics enable the possibility to write data electrically and read magnetically and this results in high storage density and low-power consumptions. Two mechanisms occur simultaneously – ferroelectric which is result of transition ions having empty d shells and magnetic requires transition metal ions with partially filled d shells. Consequently, these two ordered states mutually exclude each other.

Cobalt doped lead ferrite ($Pb_2Fe_2O_5$) thin films were deposited by reactive magnetron sputtering. The influence of the cobalt concentration and synthesis temperature on the structure, phase composition and ferroelectric properties of $Pb_2Fe_2O_5$ thin films was investigated. It was deter-mined that the increase of the deposition temperature increased the grain size and density of the Co-doped PFO thin films. The XRD data demonstrated that the Co-doped $Pb_2Fe_2O_5$ thin films consisted of $Pb_2Fe_2O_5$, PbO phases with a low amount of CoO and Co_3O_4 phases. The increase of the cobalt concentration in the $Pb_2Fe_2O_5$ films slightly enhanced the cobalt oxide phase content. Po-larization dependence on electric field measurement demonstrated that the highest ferroelectric properties of the Co doped $Pb_2Fe_2O_5$ films were obtained when the synthesis was performed at 550 ^{o}C temperatures. The increase of the cobalt concentration in the films enhanced the remnant po-larization and coercive field values. It was found that the Co-doped $Pb_2Fe_2O_5$ film deposited at 550 ^{o}C temperature and containing a 10 % of cobalt had the highest remnant polarization (72 $\mu C/cm^2$) and coercive electric field (105 kV/cm).

INFLUENCE OF TITANIUM CONCENTRATION ON THE STRUCTURE AND PROPERTIES OF DIAMOND-LIKE CARBON FILMS

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Nowadays various metals (Ti, Cr, Mo, Ni, Ag, Cu, Au) and non-metals (Si, O, N, F) are used to dope the diamond-like carbon (DLC) films to modify the structure and improve the properties [1-3]. The doping of DLC films with titanium enhances the tribological, mechanical, biomedical properties, reduces the compressive stress and increases the adhesive strength [3]. The main aim was to deposit titanium doped DLC films and investigate the influence of the Ti concentration on the structure, surface morphology, nano-hardness, adhesion force and optical properties of Ti-DLC films.

Titanium doped amorphous diamond-like carbon films were deposited on Si (100) substrates by magnetron sputtering. The titanium concentration was controlled by changing the Ti cathode currents (0.25 A, 0.50 A and 1.00 A). Elemental composition was determined by energy dispersive X-ray spectroscopy (EDX). The bonding structure of the films was analyzed by micro-Raman spectroscopy (RS) using 458 nm, 514 nm and 633 nm wavelength lasers. UV–VIS–NIR spectrophotometer was used to measure the transmittance spectra in the range of 400–1200 nm. Nanoindentation tests were carried out by MTS-Agilent G200 nanoindenter. The surface roughness, adhesion forces and friction coefficients of the Ti doped DLC films were determined by atomic force microscopy (AFM). It was observed that the increase of Ti concentration resulted in a higher amount of oxygen in the films. The doping of titanium resulted in the enhancement of surface roughness of the DLC films. The RS results indicated that the addition of titanium increased the formation of more orderly structured C=C sp^2 carbon, C=O and C-O sites. As a result, the nano-hardness of the Ti-DLC films was up to 20 % lower compared to an undoped DLC film. The adhesion force of undoped DLC film was 7.4 nN, while the Ti-DLC films demonstrated the adhesion force values up to 80 % higher. It was observed that the friction coefficient of the Ti-DLC films was up to 3 times lower compared to DLC film.

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EFFECT OF DEPOSITION PARAMETERS ON FILM MORPHOLOGY: A STUDY OF CINCHONINE TRICHLOROCOBALTATE FERROELECTRIC FILMS

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Ferroelectricity remains a fascinating phenomenon in materials science due to the constant uncovering of novel ferroelectric hybrid materials and new features related to its potential applications for developing new technologies [1,2]. Despite their broad utility, metal-organic thin films remain relatively underexplored. Among various fabrication methods, dip coating stands out for its convenience and effectiveness, however achieving high-quality films requires optimization of parameters such as precursor solution properties (concentration, pH, solvent composition) and deposition conditions (temperature, humidity, withdrawal rate, etc.). These parameters strongly affect film properties. Particularly, humidity plays a crucial role, capable of inducing structural changes in compounds leading to a change in material properties [3].

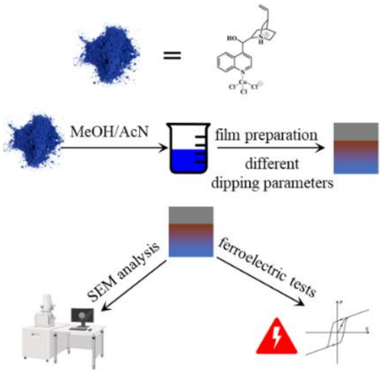

Fig 1. Scheme of research.

This study investigates the effect of deposition parameters on metal-organic thin film morphology in a model system of cinchonine trichlorocobaltate complex. Films were deposited on a Si(100) or ITO:glass substrates using dip coating method under controlled humidity conditions. Results demonstrate that lower humidity levels produce uniform films, whereas increased humidity leads to morphological changes characterized by the formation of nano and mesoscale pinholes. The structural changes which affect morphology were studied by scanning electron microscopy (SEM). Optimized protocols for obtaining high-quality and homogeneous films enabled the ferroelectric characterization of cinchonine trichlorocobaltate thin films.

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DRUG–POLYMER COMPATIBILITY PREDICTION VIA COSMO-RS

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In this work, the solid-liquid equilibrium (SLE) curve for ten active pharmaceutical ingredients (APIs) with the polymer polyvinylpyrrolidone (PVP) K12 was purely predicted using the Conductor-like Screening Model for Real Solvents (COSMO-RS). In particular, two COSMO-RS-based strategies were followed (*i.e.*, a traditional approach and an expedited approach), and their performances were compared. The veracity of the predicted SLE curves was assessed *via* a comparison with their respective SLE dataset that was obtained using the step-wise dissolution (S-WD) method [1]. Overall, the COSMO-RS-based API–PVP K12 SLE curves were in satisfactory agreement with the S-WD-based data points. Of the twenty predicted SLE curves, only two were found to be in strong disagreement with the corresponding experimental values (both modeled using the expedited approach). Hence, it was recommended to use the traditional approach when predicting the API–polymer SLE curve. At the present moment, COSMO-RS may be an effective computational tool for the expeditious screening of API–polymer compatibility, particularly in the case of new APIs (*e.g.*, anticancer drugs), for which experimental datasets are likely limited or non-existent.

Acknowledgments: The authors acknowledge the financial support provided by the Czech Science Foundation (GACR No. 22-07164S).

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STRUCTURAL AND COMPOSITIONAL CHANGES IN LADLE FURNACE SLAG DURING CO_2 CAPTURE

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The mitigation of atmospheric carbon dioxide (CO_2) is a critical challenge, driving the need for innovative CO_2 capture technologies. As the primary greenhouse gas, CO_2 is a major contributor to global warming and climate change, accounting for approximately 76% of all greenhouse gas emissions. Its long atmospheric lifetime and ability to trap heat make it a significant factor in the Earth's radiative balance. Human activities, notably fossil fuel combustion, deforestation, and industrial processes, have elevated CO_2 levels from pre-industrial concentrations of about 280 ppm to over 410 ppm today. Addressing this escalation requires effective carbon capture and storage (CCS) solutions. One promising approach involves utilizing industrial byproducts, such as ladle furnace slag (LFS) from steelmaking, for CO_2 capture. LFS, rich in calcium and magnesium oxides, offers a reactive medium capable of sequestering CO_2 through mineral carbonation processes. This study focuses on evaluating the feasibility and efficiency of LFS as a medium for CO_2 sequestration. By transforming an abundant industrial waste into a valuable resource for carbon management, this research aims to address both environmental and industrial sustainability challenges.

To assess the effectiveness of LFS in capturing CO_2, a series of controlled experiments were conducted. TG-DTA analysis was employed to determine the thermal stability and decomposition behavior of the slag during CO_2 exposure. Changes in porosity were tracked to understand how CO_2 adsorption affects the physical structure of the slag over time. XRD provided detailed information on the crystalline phases formed as a result of carbonation, while SEM was utilized to observe the microstructural changes and surface morphology at various stages of the reaction.

The results indicated that LFS exhibits significant CO_2 capture capacity, with the formation of stable carbonate compounds. The initial stages of the reaction showed rapid CO_2 uptake, followed by a slower phase, suggesting a diffusion-controlled process. The porosity measurements revealed an increase in surface area conducive to further reaction. XRD and SEM analyses confirmed the progressive transformation of slag components into carbonate minerals, highlighting the material's suitability for long-term CO_2 sequestration.

This study underscores the potential of LFS as a sustainable and cost-effective material for CO_2 capture, with a focus on the time-dependent structural and compositional changes. By repurposing industrial waste, this approach not only addresses CO_2 emissions but also contributes to the sustainability of the steel industry. Future research will focus on optimizing the reaction conditions and potentially scaling up the process for industrial applications, aiming to integrate this method into comprehensive carbon management strategies.

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THERMOGRAVIMETRIC ANALYSIS, CALCINATION AND CHARACTERIZATIONS OF LAMPANG CLAY OF DIFFERENT GRADE AND PARTICLE SIZE

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In this work, clay from Lampang province in Northern Thailand passing sieve of mesh number 200 and number 250 was characterized. Fine residual from the clay washing was also characterized and compared. Laser diffraction was used to determine the particle size distribution. Chemical oxide percentages of raw materials were obtained by mean of X-ray fluorescence, and thermal analysis was used to determine the dehydroxilation of kaolinite phase. X-ray diffraction was also used to detect other crystalline phase such as quartz. Scanning electron microscopy was used to investigate the morphology of the clay before and after calcination. The results showed that clay sample passing number 200 mesh has slight more mass loss compared to the clay residual, while the sample passing number 250 mesh has noticeably higher mass loss. At selected calcination temperature, the particle size distribution before and after calcination is very similar to each other. After calcination, metakaolin can be seen in the scanning electron microscopy images.

APPLICATION OF SYNTHETIC MONETITE FOR ZINC ION ADSORPTION

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The global issue of population aging presents significant challenges not only in socioeconomic domains but also within healthcare systems. The inevitable aging processes induces cellular degeneration, which notably impacts the structural integrity of the skeletal system. Contemporary medical interventions utilize implants composed of biocompatible materials to promote bone regeneration and restore locomotor functionality [1]. Among these materials, biodegradable ceramics, particularly those from the class of calcium phosphate, have attracted considerable attention. Monetite ($CaHPO_4$), a calcium phosphate, has been the subject of extensive research due to its superior performance and accelerated resorption. Although pure monetite is rarely found in nature, it can be produced by using microwave-assisted synthesis, which minimizes the risk of secondary by-products [2]. Additionally, the physicochemical properties of calcium phosphate-based materials are often enhanced through the incorporation of various metal ions. In particular, the incorporation of zinc (Zn^{2+}) ions into these compounds has been studied extensively due to their antimicrobial activity, which is especially beneficial of implant manufacturing [3].

Currently, there is limited scientific literature on the adsorption capacity of monetite for Zn^{2+} ions. Thus, the objective of this study is to assess the adsorption capacity of monetite synthesized via microwave-assisted method for Zn^{2+} ions. To evaluate the outcomes, instrumental techniques, including X-ray diffraction (XRD) and simultaneous thermal analysis (STA), were employed.

The adsorption capacity of monetite for Zn^{2+} ions was found to be influenced by the initial zinc ion concentration, the type of anion presents in the solution, and the duration of the adsorption process. In nitrate solutions, monetite initially adsorbed over 80 % of the Zn^{2+} ions, regardless of the initial concentrations (25 mg/g and 100 mg/g); however, a desorption trend was observed as the process duration increased, leading to the release of Zn^{2+} ions back into the solution. In contrast, in acetate solutions, the adsorption process exhibited a different behavior: Zn^{2+} ions were strongly incorporated into the monetite structure at the onset of the process and remained stably retained.

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UTILISATION OF LIGHTWEIGHT WASTE AGGREGATE IN ALKALI-ACTIVATED CONCRETE

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This study reveals that lightweight waste aggregates (LWAs) can be used in alkali-activated concretes (AACs). The alkali-activated binder (AAB) applied in the AAC is composed of wood biomass ash (WBA), phosphogypsum (PG), and an alkaline activator solution (AAS) containing Na_2CO_3 and Na_2SiO_3 (Fig. 1). The AACs were prepared according to the mix design (Table 1) to investigate how the LWA content (0–30 wt.% of the solid) affects the physical-mechanical properties of the WBA-PG-LWA-based AAC. Combined with the test results of the WBA-PG-based AABs, sample WP-0.60 had the optimal performance and was therefore used as the control sample. Thus, the WBA/PG ratio in the binder part of AACs was constant at 4:1. The Na_2CO_3/Na_2SiO_3 solute ratio in the AAS was stable at 0.60.

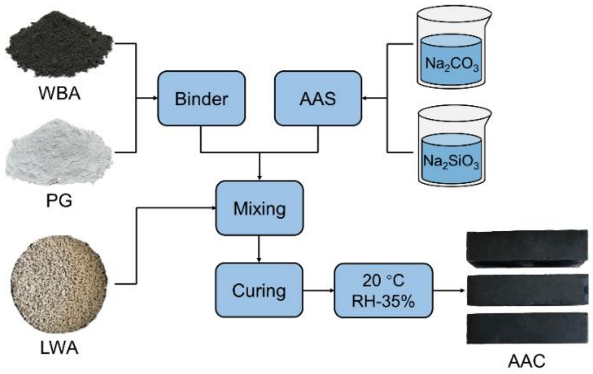

Fig. 1. Schematic of WBA-PG-LWA-based AAC sample preparation

Table 1. Mix proportions of AACs based on WBA, PG and LWA (wt.%)

Sample	WBA	PG	LWA
WP-0.60	80	20	0
WP-0.60-L1	72	18	10
WP-0.60-L2	64	16	20
WP-0.60-L3	56	14	30

Because of the porous structure of the LWA, the density of the AAC samples decreased from 1430 kg/m^3 to 1000 kg/m^3, and the compressive strength dropped from 18.36 to 3.27 MPa as the LWA content increased from 0% to 30% after 28 days of curing. The water absorption and hygroscopicity of the AAC samples increased with increasing RCA content. When the RCA content was increased to 10–30%, the water absorption of the samples increased by 8%, 13% and 17.4%, respectively, compared to the control sample. The thermal conductivity decreased from 0.2116 to 0.1893 W/(m·K) with the rising LWA content, so the AAC containing more LWA has better thermal insulation properties. The products in this research can be applied to interior non-structural components and components that do not require high thermal insulation properties.

INFLUENCE OF DIFFERENT TRIGLYCERIDE FEEDSTOCKS ON THE THERMAL PROPERTIES OF FATTY ACID BUTYL ESTER BASED BIODIESEL

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Biodiesel fuel is a mixture of different components, several of which are usually present in large quantities. For this reason, biodiesel can behave drastically differently during storage, and in severe cases, solidification and phase separation or faster degradation of the fuel can occur. Structurally, it is always an ester, but the “alcohol“ and “acid“ side of the molecule can differ depending on whether the reactant is methanol, ethanol, butanol or another alcohol, or whether the source of the triglyceride is waste oil, sunflower oil, animal fat or other.

In our research, we synthesized biodiesel from butanol and three different triglyceride sources (sunflower, rapeseed and coconut oil) and studied their thermal behaviour as well as the change in viscosity and density with temperature. Gas chromatography was used to determine the type and quantity of the individual esters in various biodiesels. Biodiesel from coconut oil has on average 35 % lower kinematic viscosity compared to other two biodiesels in 0-40 °C temperature range, making it easier to transport via pipelines. It also has inferior low temperatures properties and solidifies at higher temperatures than other two biodiesels, which implies that high amount of unsaturated esters is not desirable in biodiesel.

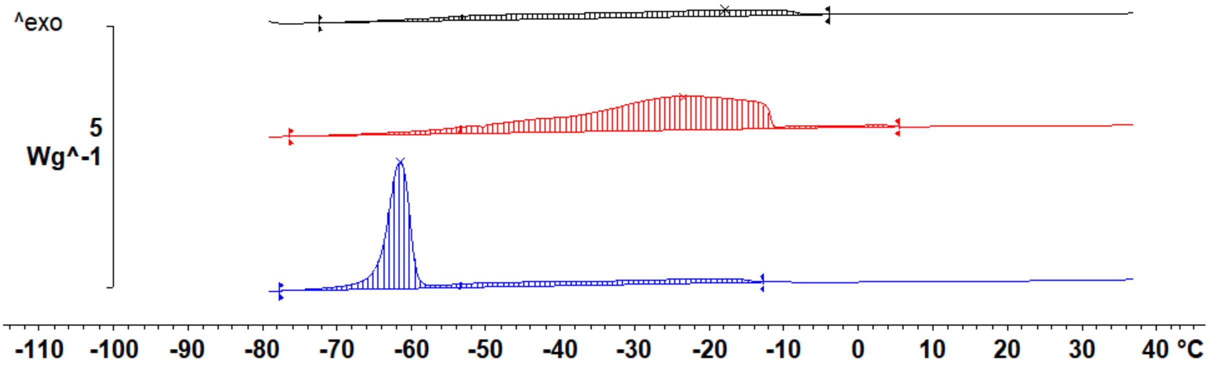

Fig. 1. Crystallization DSC curves of fatty acid butyl ester synthetized from rapeseed, sunflower and coconut oil.

Acknowledgements. This research was conducted within the Project of the Croatian Science Foundation Development of functional biofuels and (bio)additives and characterization of blends with mineral fuels (UIP-2019-04-5242).

INFLUENCE OF AMORPHOUS TiO_2 ON THE GYROLITE FORMATION UNDER HYDROTHERMAL CONDITIONS

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Calcium silicate hydrate gyrolite ($Ca_{16}Si_{24}O_{60}(OH)_8{\cdot}14H_2O$) is utilized as a hydration kinetic modification additive in the cement industry [1]. Lattice spacing between $[SiO^4]^{4-}$ tetrahedrons of ~0,3 nm allows gyrolite to be doped with various metal ions while maintaining neutral charge [2-4]. This property expands gyrolite's properties and broadens its application [5]. Titanium dioxide is prominent in many raw materials utilized in the production of calcium silicate hydrates however there is no data regarding the influence of amorphous TiO_2 on gyrolite during hydrothermal synthesis.

The aim of this research is to investigate the effect of amorphous TiO_2 additive on hydrothermal synthesis of gyrolite and it's thermal stability. Two mixtures of amorphous SiO_2 and CaO equal to (CaO/SiO_2) molar ratio of 0.66 were prepared. Secondary dry mixture was mixed with amorphous TiO_2 additive to match the weight ratio of 80% (CaO/SiO_2=0,66) and 20% (TiO_2). Dry mixtures were diluted to water/solid ratio of 1:10. Hydrothermal synthesis was carried out in unstirred suspensions in a stainless-steel autoclave under saturated steam pressure of 15 bar at a temperature of 200 °C. The duration of isothermal curing was 72 hours. After treatment, the autoclave was cooled to room temperature, the suspensions were filtered, rinsed with acetone to prevent carbonisation of materials. The products were dried at 50 °C ± 5 for 24 hours, and sieved (<80 µm).

It was determined that gyrolite with traces of calcite have formed in both samples. The intensity of diffraction maximums characteristic to gyrolite decreased with increasing amount of TiO_2 additive. During hydrothermal treatment, the additive does not participate in crystallization processes and is intercalated into gyrolite structure instead. TG/DSC results show that at 30-200 °C interval, amorphous TiO_2 additive has no effect on temperature or loss of mass associated with removal of water. At 670-750 °C interval, the sample with the additive has reduced mass losses associated with $CaCO_3$ decomposition. An exothermal effect at 830-860 °C interval shows crystalisation to wollastonite occouring in both samples and an additional formation of titanite $CaSiTiO_5$ occouring in the sample with TiO_2 additive.

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TWISTED STRUCTURE BICARBAZOLE-SUBSTITUTED BENZOPHENONES AS BLUE EMITTERS IN OLEDS

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Organic light-emitting diodes (OLEDs) have ushered in a technological revolution with their remarkable impact on diverse sectors of our daily lives. Currently, there is a strong demand for high-performance blue emitters in full-color displays and solid-state lighting. As the emission peak shifts toward the deep-blue region, the nonradiative transition rate of metal d-orbitals tends to increase, making it challenging to achieve high efficiency with traditional phosphorescent emitters. To address this issue, small-molecule fluorescent materials have regained attention due to their high color purity and low cost [1]. In this context, we introduce a series of donor-acceptor-donor twisted derivatives based on carbazole and benzophenone moieties, as illustrated in Figure 1.

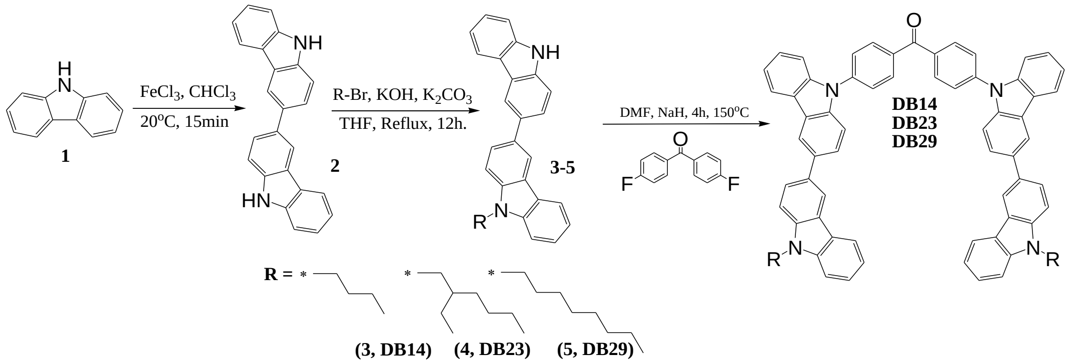

Fig. 1. Synthesis of bicarbazole-benzophenone-based materials

The materials, specifically DB14, DB23, and DB29, were designed with various aliphatic side chains to enhance their solubility and the film-forming properties of layers created via the spin-coating method. These new materials exhibit high thermal stability, with decomposition temperatures exceeding 383 °C, glass transition temperatures ranging from 95 to 145 °C, high blue photoluminescence quantum yields (>52%), and short photoluminescence decay times in the nanosecond range. Due to these characteristics, the derivatives were used as blue emitters in OLED devices. OLEDs incorporating the DB23 emitter demonstrated a high external quantum efficiency of 5.3%, which is very close to the theoretical limit of first-generation devices. This research not only contributes to the understanding of advanced OLED materials but also provides insights into designing efficient blue emitters for future display and lighting applications, thereby advancing the field of organic electronics.

Acknowledgements. This work was supported by the project funded by the Research Council of Lithuania (Grant No. S-MIP-22-84).

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IMPACT OF CALCINATION OF ESTONIAN GRAPTOLITE ARGILLITE (BLACK SHALE) ON THE SOLUBILITY OF METALLIC ELEMENTS IN SULFURIC ACID

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Black shales (BS) are considered as a potential future resource as they are usually enriched with different useful elements such as V, Mo, Ni, Cu, Zn, REEs and others, for which the demand increases year by year with the development of different high-technology sectors.

In this study the effect of calcination, with and without additives (K_2SO_4 and/or Na_2CO_3), on the solubility of metals (V, Mo, Mg, Fe, K, Al) from BS in sulfuric acid was investigated. The experiments were carried out with Estonian BS samples from Pakri peninsula and from Toolse drill core, which mineral part mainly consists of quartz, orthoclase, muscovite and pyrite.

Thermal analysis was performed using Setram Labsys 1600 thermal analyzer coupled with Pfeiffer OmniStar mass spectrometer. The initial BS samples and products of calcination were studied with XRD and SEM, and the specific surface area was determined by nitrogen adsorption method. Leaching experiments were carried out in 10% H_2SO_4 at 90°C for 3h with S/L ratio of 0.75:50 and the solutions were analyzed with Agilent 4210 microwave plasma atomic emission spectrometer.

Thermal analysis indicated emission of hygroscopic and physically bound water between 50-110°C, while thermo-oxidative decomposition of organic matter occurs between 200-550°C, leading to the emissions of CO_2, SO_2, H_2O and different hydrocarbons. Decomposition of pyrite initiates at around 500°C, dehydroxylation of muscovite and chlorite, as well as decomposition of jarosite, dolomite, and unreacted Na_2CO_3 residue, takes place between 400-900°C.

Results of XRD analysis affirm decomposition of pyrite, dolomite, jarosite and, in addition, demonstrate the formation of two different forms of orthoclase – K-sanidine and microcline. Notably, when roasting BS in the presence of Na_2CO_3, changes occur in the lattice parameters of K-sanidine and orthoclase. This may indicate the formation of K-Na-feldspar from V-bearing muscovite which enables the bonds between Al(V)–O to break and liberate V from the lattice.

Analysis of the leaching solutions indicated that calcination of BS samples increased the solubility of metallic elements in sulfuric acid, especially when additives were used. For example, the solubility of V and Mo increased from 33-40% and 60-70%, respectively, up to 70-80% without additives, and up to 90-95% with additives.

Therefore, preliminary calcination of Estonian black shale samples, with or without K_2SO_4 and/or Na_2CO_3 additives, could be a promising method to significantly increase the solubility of different metallic elements in a sulfuric acid environment.

BLUE AGGREGATION-INDUCED EMISSION BIPOLAR MATERIALS CONSISTING OF DIPHENYLSULFONE OR BENZOPHENONE CORE AND TRIPHENYLETHENE-CARBAZOLE FRAGMENTS FOR HIGHLY EFFICIENT OLEDS

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Organic light-emitting diodes are colored light sources, which are widely used in displays of TV screens and smartphones as well as in panels for lighting technologies because of the numerous advantages of the devices, such as high flexibility, low weight, fast response, high brightness, wide viewing angle, high color quality, and cost effectiveness [1,2,3].

Bipolar derivatives having aggregation-induced emission (AIE) properties are shown in Fig.1

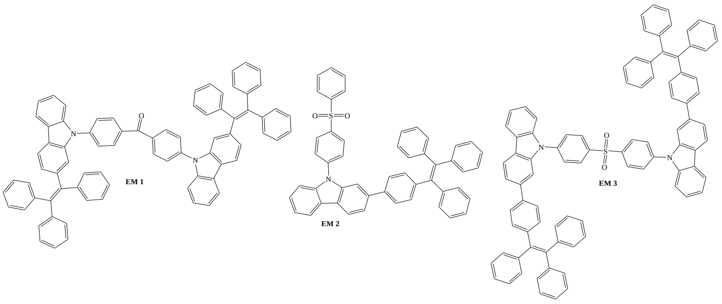

Fig. 1. Objective materials **EM1-EM3**.

The objective materials have shown high thermal stabilities. The photoluminescence spectra of the prepared derivatives registered using tetrahydrofuran-water solutions demonstrated strong blue emissions, confirming their AIE property. Thin films of the compounds were tested in multilayer organic light-emitting diodes. The device using emitter **EM3** outperformed the other devices, achieving a peak efficiency of 3.4% (5.6 cd/A and 4.7 lm/W) with maximum luminance of about 11000 cd/m^2. However, OLED using **EM2** as emitter demonstrated higher efficiency at higher luminance values. The maximum efficiencies of the device were obtained at a luminance value of about 100 cd/m^2 and were recorded as 3.2% (5.2 cd/A and 4.1 lm/W) with luminance of 17515 cd/m^2.

Acknowledgements. We gratefully acknowledge the funding support from the Research Council of Lithuania (grant No. S-MIP-22-84 and grant No. S-LU-24-7).

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MORPHOLOGICAL AND ELECTRICAL PROPERTIES OF CARBAZOLE DERIVATIVES/SILICON HYBRIDS

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Carbazoles and their derivatives are widely used as luminescent and phosphorescent materials in various fields of organic electronics, including the fabrication of organic light-emitting diodes [1-3].

The objective compounds **RB-75** and **RB-70** are presented in Figure 1. The newly synthesized derivatives were identified by mass spectrometry and NMR spectroscopy. The data were found to be in good agreement with the proposed structures.

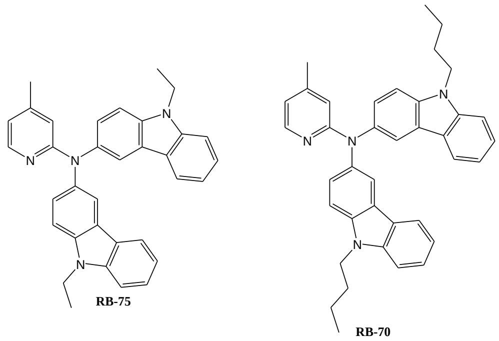

Figure 1. Compounds **RB-75** and **RB-70**

Carbazole derivatives were used for creation of hybrids on silicon patterned substrates using self-organization and spin-coating. The morphology of self-organised and spin-coated films showed two different forms of surface morphology: square-like and network-like. Carbazole derivative hybrids used in this study have three main behaviors: Ohm's law, rectifying and bimolecular recombination. This makes these hybrids promising for use in solar cells.

Acknowledgements. This work was supported by the Research Council of Lithuania (Grant No. S-LU-24-7) and by National Academy of Sciences of Ukraine (project III-4-11).

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TEXTILE ASH WASTE UTILIZATION IN CEMENT STONE

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Textile consumption in the EU ranks fourth in environmental and climate change impact, behind food, housing, and mobility. It is also the third largest consumer of water and land and the fifth largest user of primary raw materials and emitter of greenhouse gases [1]. Sustainable technologies and the utilization of potential waste materials gain more attention as they take an approach to green and circular economy [2]. Considering the given aspects, there is a need to reduce the textile's impact on the environment and search treatment methods for textile reuse [2], [3]. To meet this demand, textile waste is commonly burned or recycled into practical goods, they do not stay usable long enough before they return to being wastes once again [3]. Burned textile ash wastes could be used in making cement, because the large increase in population has increased the need for new and efficient building materials [3], [4].

This study aims to investigate the utilization of synthetic textile ash waste (TAW) and their feasibility in cement stone under hydrothermal hardening in a CO_2 environment. The initial materials used in the experiment were Ordinary Portland cement CEM I 42.5 R (OPC) and synthetic textile ash waste. TAW was used to replace Portland cement at a rate of 0, 2.5, 5, and 7.5 wt.%. Prepared cube samples (3x3x3cm) (w/s ratio corresponds to EN 196 – 3) were kept in moulds for 24 hours (100% humidity) at 23±2℃ temperature, then were transferred to an autoclave and hydrothermally hardened in a CO_2 environment (99.9% concentration) at 15 bar pressure for 4, 24 hours at 25, 45 ℃ temperatures. Subsequently, the absorbed CO_2 content and compressive strength of the hydrated samples were determined. The structural and morphological composition of the raw materials and hydrated cement were characterized by X-ray diffraction, X-ray fluorescence spectroscopy, and thermal stability of the hydrated samples was examined by simultaneous thermal analysis (DSC/TG).

The results show that Portland cement replacement by the 2.5–7.5 wt.% of TAW does not have a significant impact on the earlier cement hydration mechanism but has a positive effect on the compressive strength of thermally hydrated OPC samples under CO_2 atmosphere depending on additive content and hardening conditions. It is found that absorbed CO_2 content and the compressive strength of samples increase with prolonged hydrothermal hardening duration from 4 h to 24 h.

Acknowledgements. This research was funded by grant TEXTIFUEL from funds of Kaunas University of Technology and Lithuanian Energy Institute.

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CALORIMETRY STUDY OF GAMMA DICALCIUM SILICATE DURING THE CARBONATION PROCESS

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Globally, solving the problems regarding the Green Deal, especially the reduction of CO_2 emissions, is a big challenge. One of the many options for this problem could be mineral carbonation, which can use secondary materials and convert them into slightly soluble carbonate compounds. Suitable materials for this process are, for example, various calcium silicate compounds, such as dicalcium silicate, wollastonite, or rankinite. Considering the exothermic nature of the carbonation process, calorimetric methods appear to be advantageous for studying events during the carbonation reaction.

This work is focused on the investigation of the chemical processes during the carbonation of a gamma dicalcium silicate (γ-C_2S). The carbonation reactivity of the γ-C_2S phase was examined by perfusion and isothermal calorimetry under different conditions (temperature, relative humidity, CO_2 pressure). It has been shown that the change in carbonation conditions significantly affects the ratio of formed calcium carbonates (calcite x vaterite, aragonite). The carbonation products were characterized using X-ray diffraction analysis (XRD), thermogravimetric, and differential thermal analysis (TG/DTA), and scanning electron microscopy (SEM).

Acknowledgements. This paper was prepared within the framework of the work on the project no. GA24-12423S: “Carbonation curing as a new approach for the development of alternative binders”, financed by the Grant Agency of the Czech Republic.

POTASSIUM TETRAFLUOROBORATE – A SOLID-SOLID PHASE-CHANGE MATERIAL FOR THERMAL ENERGY STORAGE APPLICATIONS

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Increasing use of renewable energy sources requires energy storage solutions to address the discrepancy between energy supply and demand. Phase-change materials (PCMs) can reversibly store and release large amounts of latent heat during phase transition. Solid-solid PCMs, such as potassium tetrafluoroborate, KBF_4, are of increasing interest for thermal energy storage due to their high energy-storage density, retention of bulk solid properties and removing the requirement to contain hot liquids. KBF_4 is known to undergo a solid-solid phase transition between an ordered orthorhombic phase and a disordered cubic phase (Table 1).

Table 1. Experimentally measured and refined structural properties of solid phases of KBF_4.

Crystal phase	Orthorhombic	Cubic
Temperature	25°C	350°C
Space group	*Pnma* (62)	*Fm-3m* (225)
Refined unit cell parameters	a = 8.66299(1) Å b = 5.484621(8) Å c = 7.033275(8) Å V = 334.173(1) Å^3	a = 7.35718(5) Å V = 398.230(8) Å^3

Suitable PCMs for thermal energy storage need to have reproducible thermal cycling. This requires the phase-transition temperatures and the enthalpy values to remain constant. An experiment was carried out where KBF_4 was cycled 10 times between 25 and 350°C. The results show reproducible thermocycling behaviour for the investigated 10 cycles (Fig. 1).

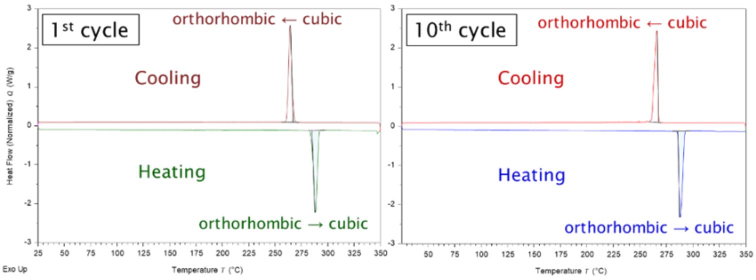

Fig. 1. Comparison of the 1st and 10th cycles of KBF_4 between 25 – 350°C.

Potassium tetrafluoroborate is a promising candidate as a solid-solid phase change material for thermal energy storage applications. Thermal cycling shows reliable reproducibility over 10 cycles and relatively high volumetric energy density. Further work is underway to explore the long-term stability of KBF_4 under a range of environmental and thermal cycling conditions.

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APPARATUS FOR MEASURING THE HYGROTHERMAL PROPERTIES OF POROUS BUILDING MATERIALS

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In recent years, high demands have been placed on the ecological and economic aspects of buildings, whether new or reconstructed. Because of this, a deeper investigation of hygrothermal properties is necessary. Hygrothermal properties include hydraulic conductivity, longitudinal and transverse dispersion coefficients, heat transfer coefficient, and thermal conductivity.

In materials science, experiments are a frequent tool for the investigation of material properties. The theoretical models are also becoming very popular. Modeling of phenomena occurring in materials appears to be a reliable method for predicting untested situations. The models supplement the experiments with important materials parameters. The transport of water and heat in a porous medium is a complex problem. The reason is primarily in the complicated structure of porous materials. Gas, liquid, and solid phases coexist here together. In the scientific literature, many papers can be found in which the flow of heat [1] and water [2, 3] are described separately. In 2019, the authors Kačur et al. [4] published paper that connects the flow of heat and water in a porous medium. This mathematical model was the theoretical basic for constructing the apparatus for measuring hygrothermal properties of porous building materials. The designed apparatus provides an innovative way to measure hygrothermal properties.

A cylindric sample is measured in accordance with the designed model. The upper and lower sides of the sample are protected by a water-resistant layer. This layer ensures that the direct of water flow into the sample is perpendicular to the gravitational driving force. The whole sample is placed in a water bath. The temperature of water in the box is controlled by a temperature regulator and is recorded during the measurement at three different locations. Using a thermocouple introduced into the sample, also the temperature of sample is recorded. Then, using inverse analysis, the hygrothermal properties of the investigated sample are determined.

Acknowledgements. This work was supported by NAWA STER Programme Internationalization of the Doctoral School of Lublin University of Technology - IDeaS of LUT and by Constantine the Philosopher University in Nitra, Grant No. VII/10/2024.

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WORMWOOD AS A POTENTIAL MATERIAL FOR CATALYST SYNTHESIS

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Wormwood (lat. Artemisia) was studied as a potential material for catalyst synthesis due to its unique chemical properties and porous structure. Studies indicate that wormwood can create various catalysts, particularly for environmental and industrial applications. Wormwood-derived support for catalyst has shown promising results. This potential is attributed to bioactive compounds in wormwood, which can be chemically modified to enhance catalytic performance and stability [1]. Wormwood's high surface area and functional groups can enhance the dispersion and stability of active catalytic sites, making it a promising material for sustainable and eco-friendly catalyst development (Fig. 1).

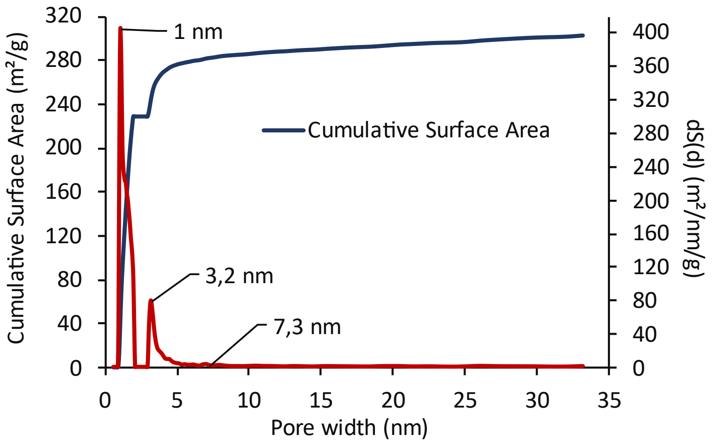

Fig. 1. Pore size distribution analysis result

Modifying wormwood was carried out by tetraethyl orthosilicate (TEOS), which could create a material with enhanced properties suitable for catalytic applications. The process involved impregnating wormwood with TEOS, which acts as a silica precursor [2]. This modification increased the surface area and introduced Si-based functional groups, improving the metal nanoparticle dispersion and stability on the catalyst.

Based on the results of physical adsorption, the following data were obtained: BET = 495 m^2/g; DFT method summary: pore volume = 0.312 cc/g; surface area = 303 m^2/g; pore width = 1.096 nm; average pore size = 3.44 nm; total pore volume = 0.43 cc/g (pore smaller than 183.2 nm); shape of pore – slit and cylindrical. The resulting hybrid material can be used in various catalytic processes, such as pollutant degradation or organic synthesis, offering an eco-friendly and sustainable catalyst option.

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SOLUBILITY OF AL-SUBSTITUTED TOBERMORITE

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1.13 nm tobermorite forms in autoclaved aerated concrete, silicate bricks, thermal insulation materials, etc. Due to its crystal morphology, this mineral is used for the sorption of heavy and radioactive metal ions. The formation mechanism of 1.13 nm tobermorite is a complex process and is highly dependent on the reactivity of the raw materials. It is well known that its formation occurs much more intensively if Al^{3+} ions are presented in the reacting medium. However, there are not many works examining how intercalated Al^{3+} ions affect the stability of 1.13 nm tobermorite. Recently, a fundamental work on this topic was published by Lothenbach *et al* [1], but they examined samples with very high inserted Al^{3+} ions.

CaO and Al_2O_3 were obtained by burning reagents $CaCO_3$ and $Al(OH)_3$ at 950 °C for 1 h or at 475 °C for 4 h, respectively. Source of silica – $SiO_2{\cdot}nH_2O$. Hydrothermal synthesis of 1.13 nm tobermorite was carried out in a stirred suspension (w/s = 20.0, 100 rpm) for 24 h at 180 °C from mixtures with molar ratios $CaO/(Al_2O_3 + SiO_2) = 0.83$ and $Al_2O_3/(Al_2O_3 + SiO_2) = 0$; 0.025; 0.05. Products were characterized by XRD, STA, FT-IR and SEM methods. Solubility tests were performed at 25, 50 and 80 °C in suspensions with w/s = 200, duration – up to 120 h. After the set time, 25 ml of the suspension was taken and was filtered through a 200 nm filter and the concentrations of Ca^{2+}, OH^-, SiO_2 and $Al(OH)_4^-$ were determined.

The results showed that Al-substituted tobermorite is a water-stable compound. In the sample with $Al_2O_3/(Al_2O_3 + SiO_2) = 0.025$, a concentration of all studied ions increases at 25 °C sharply only in the first 20 minutes of interaction with water. It still grows slightly during the first 3 h of dissolution (e.g., from 0.88 to 0.94 mmol/l for Ca^{2+}) and after that it practically does not change. It should also be noted that as the amount of Al^{3+} intercalated in the 1.13 nm tobermorite crystal lattice increases [$Al_2O_3/(Al_2O_3 + SiO_2) = 0.05$], the amount of SiO_2 in the solution decreases almost twice. The concentration of Ca^{2+} ions decreases slightly.

After increasing the exposure temperature to 50 °C, the amount of Ca^{2+} in the solution increases slightly. This indicates that the stability of Al-substituted tobermorite is slightly reduced under these conditions. However, when the temperature is raised to 80 °C, the concentration of Ca^{2+} ions decreases. Maybe it is related to lower solubility of CaO at higher temperature. The concentration of SiO_2 increases intensively during the first 20 min of dissolution, and then fluctuates within the error limits.

Comparing the solubility results of Al-substituted and pure 1.13 nm tobermorite, it was observed that when aluminum is inserted into the crystal lattice of this compound, its stability in water increases. As the amount of intercalated Al increases to $Al_2O_3/(Al_2O_3 + SiO_2) = 0.05$, this difference only increases.

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SURFACE ALIGNMENT,THERMAL BEHVIOUR AND EXCESS-CONDUCTIVITY OF $Bi_{1.8}Pb_{0.3}Sr_2Ca_2Cu_{3.3}O_y$ CERAMIC SUPERCONDUCTOR

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Single 2223 phase was obtained for the off-stoichiometric $Bi_{1.8}Pb_{0.3}Sr_2Ca_2Cu_{3.3}O_y$ superconductor system that was sintered at 847 °C for 600 h, in nitrogen atmosphere. Additional sintering of 20 h in air atmosphere at 845 °C**,** by inserting an isothermal treatment of 5 h at 826 °C (sample A) or 727 °C (sample B) in the furnace cooling process, lead to some important changes in superconducting properties (resistive transition, excess conductivity).

Some aspects of thermal analysis of the resulting samples (after the last sintering) was performed with a horizontal DIAMOND Differential/Thermogravimetric Analyzer from PerkinElmer Instruments, by heating each sample from room temperature (RT) to 1000 °C at a rate of 10 K/min, in dynamic air atmosphere. We observed a relative thermal stability in the RT÷869 °C range. A strong endothermic effect, indicative of melting accompanied by minor decomposition processes, begins at around 869 °C for both samples.

Plots of electrical resistance vs. temperature (four points method) by cooling in helium (30 K), evidenced that the critical temperature (T_c) for the transition to the superconductor phase decreased from 108.47 (sample A) to 107.53 K (sample B). From log-log plots of the excess-conductivity, the cross-over temperatures between 3D and 2D dimensionality were calculated; also, from 2D to SWF (short wavelength fluctuation) behaviour**,** the coupling factor and the coherence length for both samples were obtained. Partial alignment of the crystallites with the c-axes parallel to the pressure axes was evidenced both by X-ray diffraction (XRD) and atomic force microscopy (AFM) techniques.

THERMOKINETIC STUDY OF Ag_2O DOPED YBCO-123 COMPOSITE FROM THERMOGRAVIMETRIC ANALYSIS

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Isothermal and non-isothermal methods have been applied in a thermogravimetric experiment in order to evidence D2 oxygen diffusion taking place in the framework of normal crystal lattice of Ag_2O doped $YBa_2Cu_3O_{7-x}$-(YBCO-123) composite.

In the framework of JMAK (Johnson-Mehl_Avrami-Kolmogorov) model for oxygen diffusion, we used equation, $\ln[-\ln(1-\alpha)]=n\ln t+\ln k$, to calculate the parameter n (the order of diffusion) for some of 12 isotherms inserted in the cooling process in flowing air atmosphere. These isothermal steps were inserted at temperatures ranging from 1373.15 K down to room temperature (specifically, 1233.15 K, 1073.15 K, 1023.15 K, 973.15 K, 923.15 K, 873.15 K, 823.15 K, 773.15 K, 723.15 K, 673.15 K, 623.15 K, and 573.15 K).

The thermokinetic (TK) study of the oxygen uptake in the YBCO-123 sample (A-case) and in the YBCO-123 doped with Ag_2O (5 % Ag) sample (B-case), has demonstrated that the oxygen accumulations due to D2 oxygen diffusion occurred during the following isotherms: at 723.15 K, 773.15 K, and at 823.15 K, respectively). At 673.15 K, the D2 oxygen diffusion is confirmed only for B sample (n=2), while for sample A, n=3.9 was obtained due to the other possibilities of oxygen accumulation (channels or fissures of imperfect product crystals). In order to calculate the activation energy, the Freedman and the Modified Ortega methods equations were employed, giving for the activation energy (A-case) the value of 1.068 eV in a non-isothermal oxygen diffusion (in 1034.92 – 776.15 K range).

UTILIZATION OF ALKALI NITRATES IN TAILORING LITHIUM SILICATE GELS FOR SURFACE TREATMENT OF CEMENTITIOUS MATERIALS

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The durability of concrete-based structures is intrinsically linked to their corrosion resistance and water permeability. In order to extend the service life of concrete, surface treatment with wide variety of sealers and densifiers is becoming increasingly more prominent in the concrete industry [1]. Alkali silicate densifiers work by penetrating the porous structure and forming a secondary silicate gel, which blocks the surface pores of the concrete structure [2]. Compared to sodium and potassium silicate solutions, lithium silicate provides higher water resistance, lower tendency for alkali-silica reaction, and a lower tendency to effloresce on the concrete surface [3]. In cementitious systems, the gelation of silicate solutions is typically caused by the calcium ions present in the pore solution. However, the gelation process can also be controlled by changing the pH or the amount of alkali ions in the solution [4].

In this study, lithium silicate solution was used as the basis for the surface treatment of cement-based materials. Aqueous solutions of $NaNO_3$, KNO_3 and $LiNO_3$ were chosen as inorganic gelling agents and their influence on the gelation process and the properties of the formed silicate gel was studied. Rheological investigation of the gelation process and physical and structural characterization of the prepared gels were carried out as well as chemical composition study of the supernatant formed during gel syneresis. The performance in reducing the water permeability of cementitious structures was also assessed.

Alkali nitrates can be effectively used as gelling agents with the possibility of tailoring the gelling properties. The shrinkage associated with the syneresis process, appeared to be influenced by both the type and concentration of the gelling cation. The concentration ratio of Si to alkali ions in the supernatant remained relatively constant throughout the whole monitoring period and no significant chemical changes occurred after the formation of the initial gel structure. Water permeability test showed, that treatment of cementitious surfaces with such densifier can reduce the water permeability especially in the initial stages of immersion. However, the effectiveness decreases with the increasing amount of gelling agent.

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CEEC-PCMS2

XONOTLITE HEAT-RESISTANT INSULATION MATERIALS FROM MICROSILICA

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The key to reducing energy losses lies in the use of efficient insulating materials. One of the effective varieties of heat-resistant insulating products with an operating temperature of 1050 °C is made from calcium silicate hydrates [1].

Raw materials: CaO, obtained by burning the reagent $CaCO_3$ for 1 h at 950 °C (CaO_{free} = 99.78 %) and mikrosilika (SiO_2 = 99.5 %). The influence of mixture composition (CaO/SiO_2 = 0.7–1.1), suspension (S_1) w/s ratio (10–20), mixing intensity (50–150 rpm), temperature (180, 200 °C) and duration of hydrothermal treatment (2–24 h) on the mineralogical composition of the products (XRD, STA) and the properties of the obtained suspension was investigated. Optimal technological parameters are presented in Table 1. The synthesis duration of 2 h was chosen due to the fact that a sufficiently large amount of xonotlite is already formed, and the remaining 1.13 nm tobermorite is expected to recrystallize in it during the 2nd autoclave treatment. It has been observed that the thicker suspension obtained during hydrothermal synthesis, the lower the density of the samples. This is estimated by determining the relative volume V_r: products were poured into a measuring cylinder, and kept for 24 h. V_r is the ratio of the volume of the sediments in a cylinder and their mass. The samples (20 × 20 × 60 mm) were formed in a stainless steel mold. 1 mm diameter holes were drilled at the bottom of the mold at a distance of 5 mm. A water collection tank with a nozzle for connecting a vacuum pump was welded under it. After the excess water was sucked out and the mass began to dry, it was slowly pressed (1.6 MPa) by the upper punch (also with holes 1 mm in diameter) without interrupting the vacuuming. 3 samples were formed simultaneously. The hydrothermal curing of samples (A_2) was carried out in a Parr Instruments 4555 at 200 °C for 2–8 h.

Table 1. Optimal technological parameters of laboratory tests and main properties of the products

Parameters: S_1 – synthesis; A_2 – autoclaving	Burning temperature, °C	Average density, kg/m^3	Strength, MPa	
			Compressive	Bending
CaO/SiO_2 = 1.0; S_1: w/s = 20; 100 rpm, 200 °C; 2 h, Vr = 19 A_2: 200 °C; 4 h	20	163	0.97	0.59
	650	172	0.98	0.70
	800	168	0.72	0.62
	1000	166	0.64	0.45

Microsilica can be used to produce high-quality heat-resistant insulating products. Their thermal conductivity at 20 °C is ~0.05 W/m·K, working temperature – 1000 °C, the linear shrinkage determined by dilatometer in the range of 20–1000 °C does not exceed 0.5%, after burning for 2 h at a temperature of 1000 °C, compressive strength reaches 65%, and bending – 75% of the initial, the predominant compound is xonotlite.

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IMMERSION STUDIES AND ADHESION BEHAVIORS OF THERMALLY SPRAYED NICKEL ALLOY COATINGS FOR USE AS BOND AND TOP COATINGS

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Nickle alloys have excellent properties for corrosion and oxidation resistance alloyed with high concentration of Cr and Mo especially, Hastellloy C276 and Inconel 625 [1-2]. For the corrosion protection, thermal spraying with bond coatings were used to remunerate the thermal expansion mismatch between coatings and substrates [1]. However, limited studies on immersion test of the double-layer coatings especially, the effect of corrosion on adhesion behaviors of thermally sprayed nickel alloy coatings for use as bond and top coatings. The bond coatings, Ni-5wt.% Al, Ni-20wt.% Cr, or PMET and the top coatings, Hastelloy C276 or Inconel 625 were sprayed by arc spraying (AS) and high velocity oxygen fuel (HVOF) techniques. It was used to produce the coating onto SS304 substrate. The immersion test was followed by ASTM G31-72 in 20 vol% sulfuric acid solution at room temperature. The microstructure of the coatings was studied using Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS). The preliminary study shows that the corroded surface of top coating produced by arc spraying has more severe corrosion than the HVOF spraying due to the electrolyte can occur along the open porosity, interconnected porosity, intersplat and oxide phases led to a rapid corrosion attack of the bond coating. Thermal spraying using the electric arc technique exhibited lower corrosion resistance compared to coatings sprayed using HVOF. Therefore, the bond strength of double-layer coating produced by arc spraying is inferior when compared with those produced by HVOF. The bond strength of the coatings was examined by adhesion test, following ASTM C633 standard. The detailed results will be thoroughly discussed.

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SYNTHESIS AND OPTICAL PROPERTIES OF STRONTIUM BROMAPATITE DOPED WITH PENTAVALENT MANGANESE

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Luminescence thermometry allows monitoring the temperature of materials through the change in the optical properties. Temperature determines the change of different luminescence characteristics: emission spectrum shape, intensity, luminescence lifetime, etc. Optical thermometers are often developed by doping optically active ions into the matrices of inorganic compounds. Currently, one of the most promising ions for optical thermometry is Mn^{5+}.

Mn^{5+} ions are unique due to several reasons. First of all, this oxidation state is not common for Mn ions, which prefer to disproportionate to more stable states. Secondly, optically active Mn^{5+} ions can be excited in the first biological window (NIR-I) and emit a narrow emission line in the second biological window (NIR-II). This property makes these activators particularly suitable for temperature sensing in biological systems or bioimaging. Another feature of the emission of Mn^{5+} ions is a long lifetime values (ca. 10^2 µs at room temperature). Such values are highly desirable for temperature measurements in biological tissues, because the interfering emission of the biomolecules is significantly shorter. Unfortunately, a very limited number of matrices can stabilize Mn^{5+} ions. These matrices include compounds containing phosphorus or vanadium ions in a tetrahedral coordination (PO_4^{3-}, VO_4^{3-}), where Mn^{5+} ions can partially replace P^{5+} or V^{5+} ions.

In the present work, Mn^{5+}-doped strontium bromapatite ($Sr_5(PO_4)_3Br$) powders were successfully synthesized and comprehensively characterized. Phase purity and crystal structure of synthesized samples were studied by X-ray diffraction (XRD), infrared (FTIR) and Raman spectroscopy. Quantification of Mn ions was performed by inductively coupled plasma optical emission spectrometry (ICP-OES). Morphological features of synthesized powders were investigated by scanning electron microscopy (SEM). Optical properties were investigated by means of photoluminescence measurements. Excitation spectra, emission spectra and decay curves of the samples were studied. Temperature-dependent photoluminescence measurements were performed as well.

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INFLUENCE OF HYDROTHERMAL REGIME ON THE SYNTHESIS OF TOBERMORITE AND XONOTLITE

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The key for reducing energy losses is the use of efficient insulating materials. When temperature rise >500 °C, silicate and ceramic materials should be used. One of the effective varieties of thermal insulating products with an operating temperature of 1050 °C is made from calcium silicate hydrates – 1.13 nm tobermorite or xonotlite [1, 2]. The formation mechanism of these compounds is a complex process and highly dependent on the reactivity of the raw materials and hydrothermal regime [2]. It is essential to analyze these factors in detail, as changes in one of the conditions can have an unpredictable effect on the entire compounds formation process and their properties.

The following raw materials were used in the work: CaO, obtained by burning the reagent $CaCO_3$ for 1 h at 950 °C (CaO_{free} = 99.78 %) and mikrosilika (SiO_2 = 99.5 %). Hydrothermal syntheses were carried out in stirred at 100 rpm and unstirred suspensions (W/S = 20) at 180 and 200°C with the duration of isothermal curing of 2, 4, 8, 16, 24, and 72 h. The composition of the mixtures corresponded molar ratios CaO/SiO_2 = 0.7, 0.83, 0.9, 1.0, and 1.1. The products were examined by XRD, STA, FT-IR and SEM methods.

It was found that when synthesizing the compounds at a temperature of 180 °C and without mixing the suspensions, the reactions of formation of crystalline calcium silicate hydrates take place slowly enough and traces of 1.13 nm tobermorite were identified only after 16 h. In stirred suspensions, hydrothermal reactions are significantly accelerated: after 8–16 h of synthesis, not only 1.13 nm tobermorite with a high degree of crystallinity is formed, but also the most thermodynamically stable calcium silicate hydrate – xonotlite. C-S-H type semi-amorphous calcium silicate hydrates without a clear crystal structure were also identified in all products. This means that the synthesis products have not yet reached stoichiometric equilibrium, as compounds of variable basicity are formed.

After increasing the synthesis temperature to 200 °C, the hydrothermal reactions are significantly accelerated: in stirred suspensions 1.13 nm tobermorite with high crystallinity is formed after 4 h (CaO/SiO_2 = 0.83), and xonotlite – after 8 h (CaO/SiO_2 = 1.0) of isothermal curing. The amount of semi-crystalline C-S-H type compounds is also significantly reduced. Products with such a mineral composition should be well suited for the production of insulating heat-resistant materials.

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HYDRATION BEHAVIOUR OF CALCIUM ALUMINATE CEMENT BINDER WITH ALUMINATE RICH SLAG

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Taking into account the new Circular Economy Principles, specifically the 2030 targets for materials use and consumption footprint, it should be noted that the current linear "take-make-dispose" economy must be transformed into a truly circular economy. This transformation is based on key principles such as preventing waste and reducing energy and resource use.

The primary objective of this investigation is to carry out investigations, which are related to determination of the possibility of using the waste material by the ferroalloy industry as a binder material. In this purpose was used high aluminate slag (ARS) after aluminothermic smelting, with content: Al_2O_3 = 74.9%, TiO_2 = 19.5%, MgO = 2.2%, CaO = 2.0%, Fe_2O_3 = 1.1 %, SiO_2 = 0.17% and other components ≤ 0,1%. The X-ray qualitative and quantitative diffraction (XRD) analysis revealed the presence of following phases: corundum = 58.3%, hibonite = 22.1%, Spinel = 9.7%, Titanium monoxide = 4.8%, β-Alumina = 2.6% and Ti_2O_3 = 2.4%.

In this work, the hydration of calcium aluminate cement (CAC, Al_2O_3 > 70%) binder mixtures with ARS (0%, 5%, 10%, 15%, 20%, 25%, and 50%) was investigated at W/B ratios of 1 and 0.35. The methods used included calorimetry, thermal analysis, XRF, XRD, and scanning electron microscopy (SEM). ARS (5–25%) was found to shorten the induction period of CAC hydration and accelerate the time of secondary heat release. Significant changes in the CAC paste were caused by the addition of 50% ARS—the induction period was very similar to the results of the reference sample. However, SEM results showed differences in the effect of ARS on the formation of the binder structure during hardening. Notable changes in the CAC binder microstructure were caused by the addition of 10% and 25% ARS (Fig. 1 a-c).

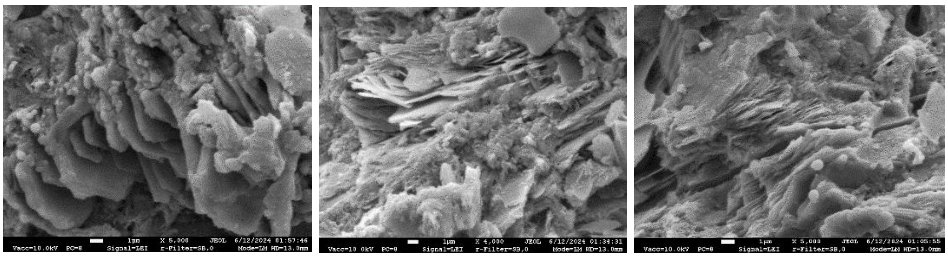
a) b) c)

Fig. 1. The microstructure of samples after 3 days hardening: a – reference; b and c ARS modified

Additionally, it was found that after 3 and 7 days of hydration, crystalline hydration products such as CAH_{10}, C_2AH_8, C_3AH_6, and AH_3 were formed in the CAC binder with ARS. Compared to the control group, the peak heights of hydrates and cement minerals differed.

Acknowledgements. The equipment and infrastructure of Civil Engineering Scientific Research Center of Vilnius Gediminas Technical University was employed for investigations. This research was supported by the center of excellence project „Civil Engineering Research Centre" (Grant No. S-A-UEI-23-5).

COMPREHENSIVE STUDY OF CALCIUM ALUMINATE HYDRATATES USING THERMAL ANALYSIS AND VIBRATIONAL SPECTROSCOPY

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Calcium aluminate phase ($Ca_3Al_2O_6$) strongly affects the early-stage hydration of OPC and influences further workability (i.e. flash set) [1]. The C_3A rapidly reacts with the added water and form solid hydration products. The nature of the precipitates depends on the water and sulfur content. In absence of Sulphur the stable (C_3AH_6) and metastable C_4AH_{13}, C_4AH_{19} and C_2AH_8 are formed [2].

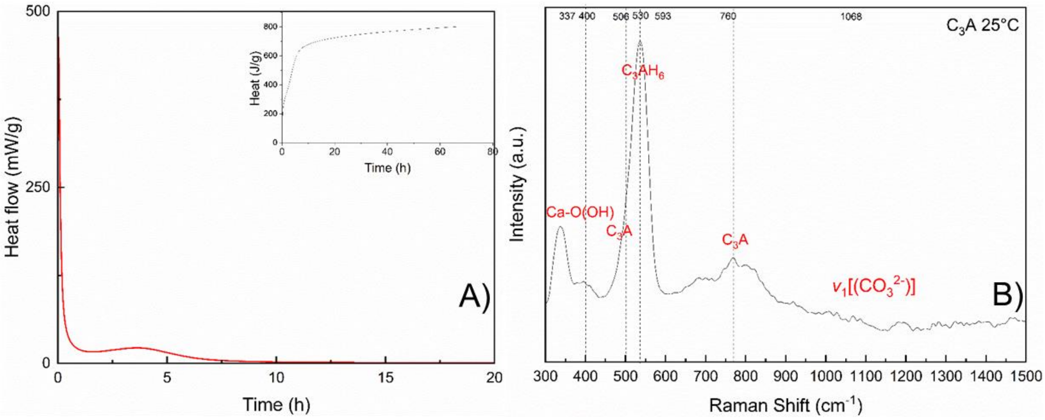

Fig. 1. Calorimetric measurements of calcium aluminate and TG-DTA analysis of reaction products

Here we introduced the comprehensive study of hydration product formed upon hydration at different temperature (15, 25 and 35 °C) by using combined vibrational spectroscopy analysis with TG-DTA observation. Controlled hydration was performed in TAMAir instrument (Fig. 1a). Phase composition of reaction products was strongly influenced by the temperature. With the increasing temperature the content of stable C_3AH_6 hydrates increased and subsequently metastable C_4AH_{13}, C_4AH_{19} were diminished. TG-DTA (Fig.1b), Raman and Infrared spectroscopy was used to order the crystalline and semicrystalline species. Conversion of metastable hydrate lamellar structure to C_3AH_6 cubic morphology was clearly observed by SEM-EDS analysis.

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MICROWAVE ASSISTED AQUEOUS-BASED SYNTHESIS OF COPPER SULFIDE

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Copper sulfide (Cu_xS_y) is a promising transition metal chalcogenide since it has desirable properties for p-type semiconductor, such as high theoretical specific charge capacity (337–560 mAh/g), high electronic conductivity (~10-3 S/m), outstanding discharge characteristics together with potential high carrier concentrations, low thermal conductivity, low solubility in most electrolytes and appropriate direct band gaps for solar light absorption [1]. Due to these properties, Cu_xS_y shows great potential to be used as a photocatalytic, photothermal or energy-storing material [2].

Currently employed methods to produce Cu_xS_y, such as colloidal synthesis, hot-injection method, mechanochemical or solvothermal treatment, typically use fossil-based reagents, which are unsustainable and human hazardous [3]. Moreover, the production usually takes a long duration, high temperatures and results in low yield consequently increasing the processing cost [3].Hence it is crucial to develop a more sustainable approach to fabricate high value compounds.

Microwave irradiation-assisted hydrothermal treatment enables a rapid formation of nanoparticles by employing irradiation of microwaves as a heating source and consequently achieving high temperature and pressure in a significantly shortened duration [4]. Nevertheless, the latter method has not been applied to synthesize copper sulfide. In addition, the aim of this work was to fabricate Cu_xS_y in an aqueous solution by hydrothermal treatment assisted with microwave irradiation.

Copper sulfide (CuS) was synthesized from a mixture of copper oxide and elemental sulfur. Aqueous-based synthesis was carried out at a temperatures of 120–180 °C for 10–120 min in a hydrothermal environment assisted by microwaves. Phase composition, crystal lattice parameters and morphological properties of synthesized products were characterized with analysis methods of X-ray diffraction, X-ray fluorescence and Scanning Electron Microscopy.

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NAPHTALIMIDE-BASED BIPOLAR DERIVATIVES FOR HIGH-EFFICIENCY OLEDs

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Organic light-emitting diodes (OLEDs) transcend the capabilities of conventional diodes, excelling in performance, durability, and manufacturing processes [1]. In addition to these achievements, OLEDs possess a plethora of remarkable features that set them apart from other diode types [2]. Bipolar organic derivatives can be used as materials of emitting layer of OLEDs. Some naphtalimide based derivatives as electroactive materials are already described in scientific literature [3]. Here, we present new potential emitters containing naphtalimide core as electron acceptor and carbazole or arylcarbazole fragments as electron donors.

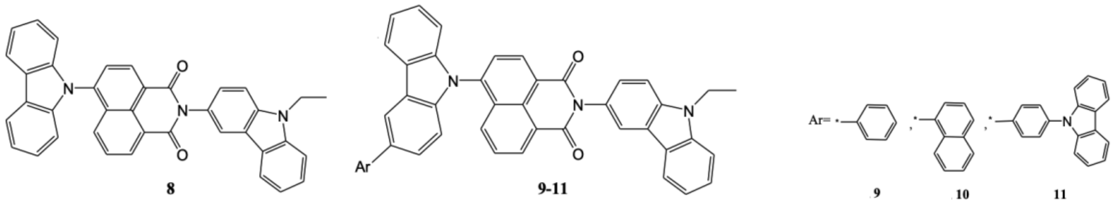

Fig. 1. New synthesized compounds **8**, **9**, **10** and **11**.

A series of naphtalimide-based compounds 8, 9, 10 and 11 were successfully synthesized and characterized. Their photophysical, electrochemical, and thermal properties were examined in regard to their prospective application in green OLED devices. Cyclic voltammetry measurements provided insights into the electrochemical properties of the compounds, and the calculated HOMO and LUMO energy levels confirmed their suitability as emitters with 4,4'-bis(carbazol-9-yl)biphenyl (CBP) host. Furthermore, the materials were thermally and morphologically stable, with high breakdown temperatures and very high glass-transition temperatures. Furthermore, OLED devices were fabricated using the synthesized compounds as dopants in the CBP host matrix. Electroluminescence spectra of the devices displayed green emission, and the devices exhibited favourable performance characteristics. Among the devices, the one incorporating 5 wt% 11 emitter demonstrated superior performance, achieving maximum power efficacy of 7.7 lm/W, maximum current efficacy of 7.9 cd/A and maximum external quantum efficiency of 3.3%. The CIE coordinates of (0.29, 0.52) of 11 emitter based device indicated an efficient and stable green OLED with peak emission at 520 nm. Finally, the synthesized naphtalimide-based compounds show promising potential as efficient green emitters for OLED applications.

Acknowledgements. This work was supported by Research Council of Lithuania (grant No. S-MIP-22-84).

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SOLUTION PROCESSABLE CARBAZOLE-BENZOPHENONE DERIVATIVES AS BIPOLAR HOSTS FOR HIGH-EFFICIENCY STABLE GREEN TADF OLEDs

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Solution processable bipolar materials have been reported as emitters and host materials in organic light emitting diodes (OLEDs) [1]. These materials exhibited a thermally activated delayed fluorescence (TADF) nature and have the capability of harvesting triplet excitons from excited triplet to singlet states by reverse intersystem crossing [2]. Currently, solution processable TADF OLEDs have been attracting attention for being cost-effective, possessing a simpler design structure, and capability of being scaled up for large area displays [3]. Here, we developed two carbazole–benzophenone derivatives (BPBCzO and BCzBCzO).

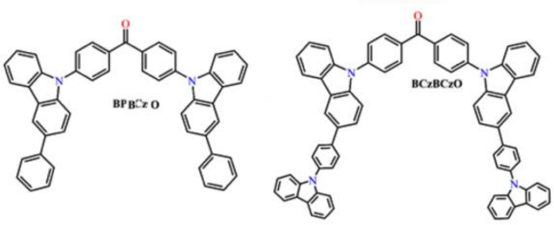

Fig. 1. New synthesized compounds BPBCzO and BCzBCzO.

These resulting materials displayed a bipolar transport characteristic, high triplet-energy, suitable frontier molecular orbital energy levels and solubility in common organic solvents. Consequently, BPBCzO and BCzBCzO were successfully incorporated with 4CzIPN for the fabrication of solution-processed green TADF OLEDs. Remarkably, green TADF device employing BPBCzO host material and 4CzIPN guest material displays a maximum power efficacy (PE_{max}), current efficacy (CE_{max}), external quantum efficiency (EQE_{max}), and luminance (L_{max}) of 55.6 lm W^{-1}, 70.7 cd A^{-1}, 23.2%, and 16500 cd m^{-2}, respectively. Additionally, cross-linkable hole transport material VPEC was successfully incorporated into green TADF OLEDs to achieve stable solution processed OLED with high-efficiency. Corresponding device displayed a PE_{max} of 63.6 l m W^{-1}, CE_{max} of 81.0 cd A^{-1}, EQE_{max} of 25.3%, and L_{max} of 18900 cd m^{-2}. These devices are among highly efficient solution-processable TADF OLEDs.

Acknowledgements. This work was supported by the Research Council of Lithuania (grant No. S-LU-24-7).

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CONCRETE PAVERS SAMPLES HARDENING IN A CO_2 ENVIRONMENT

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One of the ways to reduce the negative impact of Ordinary Portland cement (OPC) production on the climate change, is to use cements in which part of the clinker is replaced by supplementary cementitious materials [1], and to harden the products in a CO_2 environment. Blended cement CEM II/A-LL 42,5R, in which 15% of the clinker is replaced by carbonate opoka and CEN standard sand EN 196-1 were used in this work. After studying the influence of various parameters: composition of the mixture (20–35% PC and 80–65% sand), amount of water (w/c = 0.2–0.35), compacting pressure (7.5–15 MPa), and also, effect of CO_2 pressure (10–15 bar), temperature (25–55 °C) and duration of curing (8–24 h) on the properties of the ø36 × 36 mm samples (Fig. 1), the optimal technological indicators were determined.

According to them (PC : sand ratio – 1: 3, w/c = 0.3), concrete pavers samples (100 × 100 × 50 mm) were compressed with a force of 12.5 MPa at a speed of 1 kN/s with a 30 s hold. Carbonation curing was carried out in a Parr Instruments (USA) pressure reactor. It was first twice purged with CO_2 gas up to ~2 bar, immediately depressurized and afterwards the pressure of gaseous CO_2 (99.9 % concentration) was increased (and decreased afterwards) by 2.5 bar/min to 12.5 bar. Duration of curing at 25 °C – 24 h. The main characteristics and photo of the obtained products are presented in Fig. 1.

	Compressive strength, MPa	Splitting tensile strength, MPa	Amount of CO_2 absorbed, %
1 : 3, w/c = 0.3, MPa, 12.5 bar, 25 °C, 24 h			
1	40.53	4.55	6.94
2	44.71	5.05	7.58

[1] Laboratory samples, ø36 × 36 mm
[2] Concrete pavers samples, 100 × 100 × 50 mm

Fig. 1. The main characteristics and photo of concrete pawer samples

The higher strength of the concrete pavers samples may be due to different dimensions than the laboratory specimens. But there may be another reason – composition of the initial mixtures. In the work, OPC was not used, but with 15% opoka additive. It is known that finely dispersed $CaCO_3$ and amorphous SiO_2 are formed during carbonization, which compact the structure of the product. However, in this case, additional amounts of calcite and SiO_2 were introduced by grinding the clinker with opoka, which contains up to 70% SiO_2 and 20–40% calcite. The amorphous SiO_2 in it is finely dispersed, so it can increae the density.

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THE EFFECT OF BASE RAW MATERIAL AND ALKALI ACTIVATION SOLUTION ON ALKALI ACTIVATED BINDERS SUBSTITUTION BY SYNTHETIC ZEOLITE-LIKE POWDER

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Due to the growing demand for sustainable and high-performance building materials need for new alternatives and innovations in production of building materials is obvious. Alkali-activated binders offer a promising alternative to traditional Portland cement by utilizing industrial by-products like fly ash etc., thereby reducing environmental impact. Incorporating synthetic zeolites into alkali activated binders can enhance their mechanical properties and durability, making them more suitable for a wide range of building applications.

This research contributes to the development of innovative, eco-friendly alternative building materials with improved performance characteristics, addressing both sustainability and technical challenges in the building industry. The aim of this study is to investigate the effect of substituting alkali activated binders with synthetic zeolite-like powder. Two types of synthetic zeolite-like powders were synthesized using low-temperature hydrothermal methods using type F fly ash and metakaolin as main precursor and sodium hydroxide solution. The zeolite-like powders were then used to replace 10% of the solid raw materials in the composition of prepared alkali activated binders. The impact of these substitutions was evaluated by testing the compressive and three-point bending strengths, material density, and mineralogical composition as well as structural changes of the resulting binders.

The results indicate that the incorporation of synthetic zeolite-like powders affects the mechanical properties and microstructure of alkali activated binders. Specifically, a 10% substitution of the solid raw material with synthetic zeolite-like powder resulted in varied changes in strength and density, depending on the type of zeolite and the activation solution used. This research provides insights into optimizing the properties of alkali-activated materials through the strategic incorporation of synthetic zeolites, potentially enhancing their performance for various construction applications.

THE INFLUENCE OF CALCINED AND SONICATED KAOLINITE CLAY ON THE EARLY CEMENT HYDRATION

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Usually, mineral additives for cement are very small, they are less than 1 µm and are smaller than cement particles. The smaller the particle size of the mineral additives, the better it works as a microfiller, and the higher the specific surface area, the better the reactivity. However, the smaller the particles, the more they tend to stick to each other and large agglomerates are formed. Ultrasonic dispersion is a great way to break down the resulting agglomerates. During the research, calcinated kaolinite clay (CKC) was intensively milled and sonicated in the suspension. The influence of the additive amount of calcinated and milled kaolinite clay suspension on the early hydration of Portland cement was investigated.

The composition of the samples is shown in Table 1.

Table 1. Composition of the samples

Sample no.	Amount of components, wt.%	
	Portland Cement	CKC
1	100	0
2	95	5
3	90	10
4	85	15

The results of the compressive strength of the samples after 3 days of hydration are summarised in Fig. 1.

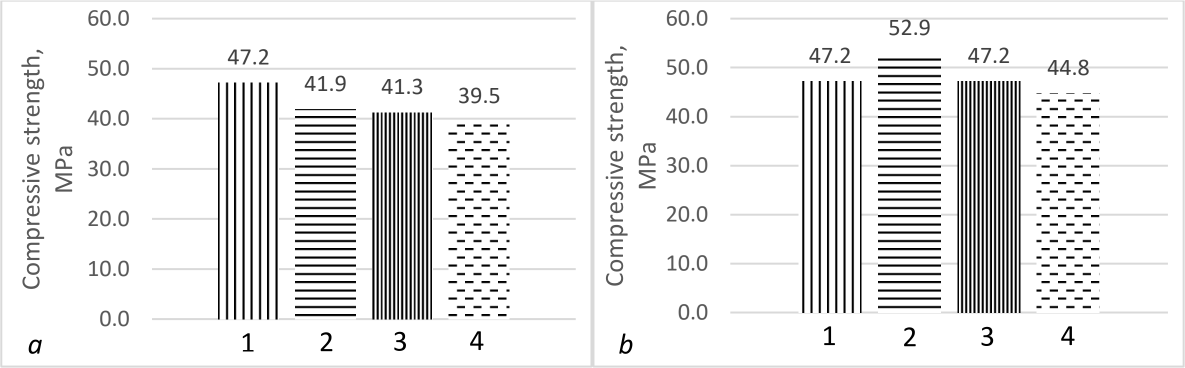

Fig. 1. Compressive strength of the samples after 3 days of hydration

Ultrasonic treatment of the clay additive suspension of ground kaolinite has been shown to increase the strength of Portland cement samples after 3 days of hydration. The maximum sonication effect after 3 days of hydration (a 26% increase in strength from 41.9 to 52.9 MPa) was found in the sample with Portland cement replaced by a clay additive of 5% calcinated sonicated kaolinite due to the acceleration of hydration of C_3S.

UTILIZATION OF BYPASS DUST IN CO_2-HARDENING CONCRETE

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Ordinary Portland cement (OPC) has a huge carbon footprint and produces no less than 2.1 billion tons of CO_2 annually [1]. Global cement production raised by from 1.6 billion tons in 2000 to 4.4 billion tons in 2021 [2] due to the growing population, ongoing urbanization and infrastructure development. So, cement industry is one of the largest contributors to climate change accounting for about 7% of global CO_2 emission. The search for new alternative cementitious materials with a lower CO_2 footprint than OPC is one of the major challenges facing the scientific community and cement industry.

The aim of this work is to investigate the possibility of replacing a part of OPC in CO_2-hardening concretes with bypass dust. Raw materials used: CEM I 42.5 R, CEN standard sand EN 196-1 and Akmenes Cementas AB bypass dust. The samples (ø36 × 36 mm) were compressed with a force of 12.5 kN from OPC : sand (1:3 by mass) mixtures, in which 0; 10; 20; 30 and 40% OPC replaced with bypass dust. They were cured in a reactor filled with 99.9% pure CO_2 gas at temperature of 25 and 45 °C, under a pressure of 15 and 12,5 bar, and the holding time was 24 h. The compressive strength of the samples, the amount of bound CO_2, and the mineralogical composition using XRD and STA methods were determined. The data are presented in table 1.

Table 1. Dependence of sample properties on the amount of bypass dust

Amount of bypass dust, %	Compressive strength, MPa	Amount of bound CO_2, %	Compressive strength, MPa	Amount of bound CO_2, %
	Curing temperature – 25 °C		Curing temperature – 45 °C	
0	28.93	6.03	35.92	5.86
10	23.64	6.02	33.77	5.78
20	21.20	5.86	31.12	5.20
30	16.71	5.54	23.84	5.41
40	12.77	5.20	24.97	4.90

XRD and STA results showed that portlandite, potassium chloride and spurite, which are present in the bypass dust, react within 24 h in hardened OPC-standard sand-dust samples in an environment of 25 °C and 15 bar CO_2 pressure. They are dominated by quartz, also calcite is formed and alite remains. In the sample with 40% dust additive, spurite reacted incompletely. Up to 20% of OPC can be replaced by bypass dust in the composition of CO_2 curing products and such mixtures may be suitable for producing silicate masonry products of strength class 20 (25 °C) or 30 (45 °C), the properties of which are regulated by the standard EN LST 771-2. Bypass dust is not an inert additive because some of the compounds in it react with moist CO_2 gas during carbonization.

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Poster presentations 2

Advanced Methods for Characterization of Materials

Catalysis, Electrocatalysis and Photocatalysis.

Chemical Engineering and Biotechnologies.

Chemical Kinetics.

Conventional Materials: Ceramics, Glasses, Metals and Alloys.

Electrochemistry and Corrosion. Energy and the Environment. Energy Storage and Conversion. Renewable Energy Systems.

Environmental Chemistry, Geochemistry and Astrochemistry.

Hybrid Materials, Nanomaterials and Nanotechnologies.

Life and Natural Sciences. Bio-inspired Materials. Lignocellulosic-based Materials.

Novel Devices, Sensors, and Biosensors.

Physical Chemistry of Colloids and Food Technology.

Polymer Science and Technology of Polymeric Systems.

Porous Media and Sorption.

Quantum Chemistry, Spectroscopy and Molecular Structure.

Surface and Interface Science

PHOTOCATALYTIC ACTIVITY OF A MOLECULARLY IMPRINTED CORE-SHELL PHOTOCATALYST

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Pharmaceutically active compounds have been a growing concern for decades due to their resilience and ongoing release in the aquatic environment. The removal strategies comprise chemical, physical, and biological processes, including filtration, adsorption, electrocoagulation, flocculation, etc. Heterogeneous photocatalysis is a promising advanced oxidation method for removal of pharmaceuticals from wastewater [1, 2]. In most cases, in heterogeneous photocatalysis, photocatalyst is used in the form of suspension. The main drawback of suspended photocatalysts is their separation from the solution after the photocatalytic process [3, 4]. For this purpose, a magnetic nanostructured core-shell photocatalyst with a molecular imprint of a chosen antibiotic was prepared via microwave-assisted synthesis. The magnetite core was coated with a protective layer of SiO_2 and a photocatalytic layer of molecularly imprinted TiO_2. Microwave radiation enables fast and uniform heating rate, rapid nucleation and growth of particles, shortens reaction time, and enables energy savings, while the magnetite core enables easy separation by an external magnet as well as the possibility for reuse [5]. The molecularly imprinted TiO_2 layer possesses specific cavities designed for the target molecule (imprint) that will improve the efficiency of the extraction process, thus creating a synergistic effect of photocatalysis and extraction.

The phase composition of the prepared photocatalyst was determined using X-ray diffraction analysis (XRD) and Fourier transform infrared spectroscopy (FTIR). The BET surface area, pore volume, and pore size distribution were calculated from nitrogen adsorption-desorption isotherms. The bandgap energy value was determined using diffuse reflectance spectroscopy (DRS). The surface morphology was examined using a scanning electron microscope (SEM). The degradation efficiency, kinetics, and mechanism of selected pharmaceuticals by prepared materials were determined and analyzed.

Acknowledgements. This work was funded by the Croatian Science Foundation under the project [IP-2022-10-4400]: Development of molecularly imprinted polymers for use in analysis of pharmaceuticals and during advanced water treatment processes (MIPdePharma).

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PHOTOCATALYTIC DEGRADATION OF PROCAINE VIA G-C_3N_4 SYNTHESIZED BY CONVENTIONAL AND UNCONVENTIONAL METHOD

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A metal-free polymeric semiconductor graphitic carbon nitride (g-C_3N_4) has recently attracted considerable interest in various fields of science, especially in various types of advanced oxidation processes as visible-light-driven photocatalyst [1]. It is a two-dimensional material whose layer-like structure resembles graphene. Typically, g-C_3N_4 is synthesized from precursors that are rich in carbon and nitrogen, such as urea, melamine, thiourea, cyanamide, or dicyandiamide [2]. In this work, g-C_3N_4 samples were prepared using urea as a precursor in two ways: using the conventional method (in the electrical furnace) and the unconventional microwave method (MW). One g-C_3N_4 sample was prepared at 550 °C for 2 h, with a heat rate of 3 °C/min in the electrical furnace (conventional method). Six g-C_3N_4 samples were prepared using the MW method at different microwave power (600 and 800W) and periods of time (10, 12, and 15 min) in order to determine the optimal synthesis parameters. MW synthesis is a fast, simple, and environmentally friendly method [3, 4]. Prepared g-C_3N_4 samples were used for photocatalytic degradation of procaine by irradiation with UV-A and visible light. The degradation rate and mechanisms were determined and compared. The phase composition of the prepared g-C_3N_4 samples was determined by X-ray diffraction analysis (XRD) and Fourier transformation infrared spectroscopy (FTIR). Nitrogen adsorption/desorption isotherms were used to determine Brunauer, Emmett, and Teller (BET) specific surface area and pore size distribution. The band gap energy of the samples was determined by diffuse reflection spectroscopy (DRS).

Acknowledgements. This work was funded by the Croatian Science Foundation under the project [IP-2022-10-4400]: Development of molecularly imprinted polymers for use in the analysis of pharmaceuticals and during advanced water treatment processes (MIPdePharma).

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SYNTHESIS AND CATALYTIC PERFORMANCES OF MANGANESE AND IRON LOADED CARBON COMPOSITES FOR THE SELECTIVE CATALYTIC OXIDATION OF DYES

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Elimination of organic pollutants in water potabilization processes through environmentally friendly methods is a rapidly developing field. Adsorption and oxidation with chemical reagents are the basic methods currently allowing for the elimination of organic pollutants. Catalytic oxidation/reduction methods of pollutants are beginning to be applied on an industrial scale with the synthesis of efficient catalytic materials. This potabilization process has several advantages over classical methods: it does not require the use of chemical reagents, the working time of catalytic materials is longer than that of adsorbents, and the regeneration of catalysts is done directly in the filtration column.

In this study, a series of carbon composites impregnated with Fe (III) and Mn (IV) ions were synthesized and studied. Fractionated walnut shells (0.8-2.00 mm) were impregnated with the respective salt solutions at room temperature for 24 hours. After contacting with metal ions, the walnut shells were subjected to hydrothermal carbonization at 320°C for 3 hours. The use of the hydrothermal method allows for the introduction of metal ions at the carbonization stage, preventing the washing out of ions from the composite structure. After carbonization, the samples were subjected to activation in a fluidized bed with water vapor at 1000 °C for 20-30 minutes. X-ray diffraction analysis on the powders of the obtained samples showed that during the carbonization process, metal ions are reduced to Fe(II) and Mn(II). To oxidize the metal ions, the composites were treated with a KMnO4 solution, which allowed the incorporation of Fe (III) and Mn (IV) ions into the carbon skeleton structure. The obtained samples were washed with distilled water and dried at 110°C.

The obtained composites were tested using gas adsorption methods, X-ray diffraction, FTIR spectroscopy, thermal analysis, scanning microscopy, potentiometric titrations, etc. Tests were also conducted to establish the degree of metal ion elimination under different conditions in aqueous solutions at various pH levels and temperatures. The samples were initially tested in model catalytic processes for the decomposition of hydrogen peroxide and compared in activity with commercial catalytic materials (Centaur activated carbon) and with MnO2. Subsequently, the samples that exhibited the most pronounced catalytic activity were selected and used for the oxidation of organic dyes.

CHARACTERIZATION OF TiO_2 NANOPARTICLES ON AISI 316 STEEL PLATE

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Spraying TiO_2 nanoparticles onto AISI 316 stainless steel surfaces has proven effective for various applications. Research shows that TiO_2 nanoparticle spray coatings can enhance the degradation of chemical warfare agents like chlorpyrifos [1] and facilitates the photocatalytic degradation of antibiotics such as ciprofloxacin. [2]. Additionally Al_2O_3-TiO_2 hybrid coatings on AISI 316 steel improve corrosion resistance while maintaining wear resistance [3]. These studies suggest that TiO_2 nanoparticle coatings can enhance degradation processes on stainless steel surfaces. This study aims to further investigate this hypothesis, comparing spray coatings with electrophoretically deposited TiO_2.

Materials and Methods

AISI 316 steel plates were cut, sanded, and cleaned with water, acetone, isopropyl alcohol, and deionized water. A mixture of 2.5 g TiO_2 nanoparticles (Degussa P25), 15 ml deionized water, and varying methanol quantities were sprayed onto the plates and calcinated at 400 °C for 1 hour. Photocatalytic methylene blue (MB) degradation was assessed for degradation kinetics. Catalyst adsorption and photocatalytic degradation were used to evaluate ciprofloxacin (CIP) degradation rates.

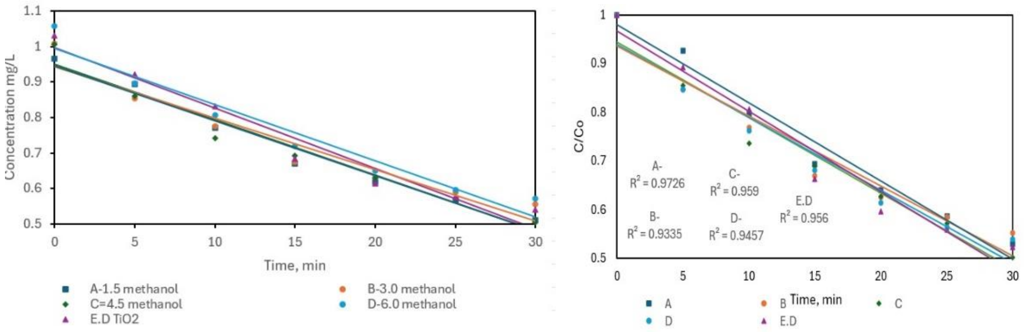

Figure 2: Degradation mechanics and rate mechanics of synthesized TiO_2 nanotubes

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DEPENDENCE OF ORGANIC CARBON CONCENTRATION ON TEMPERATURE AND METHOD IN EXTRACTS OF DIGESTATE

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Intensive and chemical-laden agriculture has significantly damaged natural ecosystems, raising concerns about soil conservation, soil quality improvement, human health protection, and environmental preservation. This necessitates the exploration of alternative agricultural methods such as organic farming, which avoids mineral fertilizers and uses biological substances of organic origin to provide plant nutrients. One such substance is digestate, a high-organic matter waste product used as fertilizer. Scientific research supports the use of biogas digestate as a soil fertilizer, a biological agent against diseases and pests, and a substrate in hydroponics [1-3].

In order to create liquid or bulk products suitable for fertilizing plants or for use as a plant growing base, it is necessary not only to know the chemical composition, but also to evaluate the physical properties, the crystalline constitution of materials, their homogeneity, surface, and resistance to temperature effects. Therefore, X-ray diffraction (XRD), and simultaneous thermal analysis (VTA) were performed. In this study, a chemical analysis of digestate samples from three companies (UAB "Agaro riešutas", UAB "Tvari energija" and UAB "Kurana") was carried out. There digestate is produced as waste. Results showed that the digestates contain primary and secondary nutrients (N, P2O5, K2O, CaO, MgO, C). It was found that the digestate is rich in organic matter and organic carbon. The content of organic matter in samples ranges from 11.0 to 16.5 % and organic carbon ranges from 39.0 to 50.0 %. The curves of X-ray structural spectroscopic analysis allow us to say that digestate samples 1 and 2 do not have a crystalline structure. After simultaneous thermal analysis (VTA) studies, it was found that in all three digestate samples, the remaining water is removed at a temperature of 100 ℃ and this process is represented by a small endothermic effect, and the corresponding 7-8% mass loss. After evaluating the results of the chemical and instrumental analysis, it was decided to produce liquid extracts from the digestate, which would be used to fertilize plants directly or as additives in liquid fertilizers. To obtain the high concentration of organic carbon from the digestates, extracts were produced using water and potassium alkali under various conditions. The samples were stored for 3, 6, and 9 hours at different temperatures, while being mixed with a stirrer. Additionally, the samples were heated to temperatures of 50°C, 70°C, and 90°C. The results showed that potassium-based extracts had higher concentrations of organic carbon, and increasing the temperature positively affected the organic carbon concentration in the samples.

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PRODUCTION OF POTASSIUM DIHYDROGEN PHOSPHATE WITH BIOACTIVE ADDITIVES

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Fertilizers are an integral part of modern agriculture. By purposefully using fertilizers, plants are provided with the necessary nutrients, thus increasing crop yield and resistance to diseases. Growing concern for sustainable agriculture has led to a growing interest in organic and mineral fertilizers with organic additives [1, 2].

One of the fertilizers with the highest concentrations of potassium and phosphorus is potassium dihydrogen phosphate. However, this material is rarely used in agriculture due to its high cost. One of the new production technologies that can significantly reduce the cost of potassium dihydrogen phosphate is the conversion reaction between potassium chloride and ammonium dihydrogen phosphate in aqueous solutions. The potassium dihydrogen phosphate obtained during the synthesis can be used for the production of complex granular fertilizers, and the remaining crystallization solution for the production of liquid complex fertilizers.

This project dealt with the synthesis of potassium dihydrogen phosphate (Figure 1) by converting potassium chloride and ammonium dihydrogen phosphate in aqueous solutions. Chemical and instrumental analysis methods were used to determine the optimal ratio of starting materials and the most economical synthesis conditions. The solid phase obtained with the best properties was used to produce granular NPK fertilizers. In the production of NPK fertilizers, lupins (*Lupinus polyphyllus*) were used as organic nitrogen additives, after examining the nitrogen content in their aqueous and alkaline extracts.

In the production of NPK fertilizers, microcrystalline cellulose and calcium lignosulfonate [3] were used as additives to improve granulation. The chemical and physical properties of the obtained fertilizers were analyzed. The influence of the used organic additives on the concentration of plant nutrients in the final product, as well as the influence of granulation additives on the physical properties of the obtained granules, were evaluated.

Fig. 1. Synthesized crystalline material

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SPRAY-DRYING TECHNIQUES FOR ENCAPSULATION OF MICROALGAE EXTRACT IN MODIFIED STARCH

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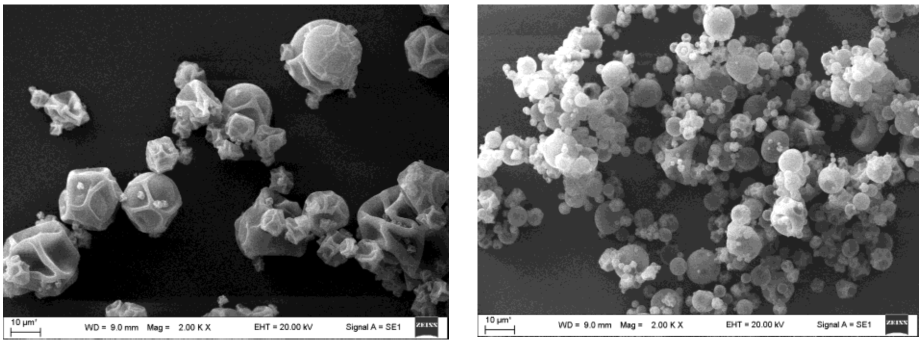
Microalgae are regarded by many in the scientific community as food of the future. However, most bioactive compounds are intrinsically unstable when exposed to unfavorable environmental factors during processing and storage, including extreme pH, high temperatures and oxygen. As a strategy to overcome these limitations, encapsulation technology is considered to be a promising approach to maintaining the stability and functionality of bioactive compounds of microalgae.

In this study, the encapsulation of microalgae extract *(Arthrospira platensis)* using modified starch and by employing spray-drying techniques has been investigated. In the first stage, the aqueous formulations containing 10 % (w/w) starch sodium octenylsuccinate and 1, 2, 3 and 5 % w/w of microalgae extract were prepared by using rotor-stator homogenizer. In the second stage, the encapsulated microalgae extract was obtained in the form of solid particles using spray drying. Two different techniques, i.e. conventional and nano spray drying were used to obtain micro- and submicroparticles.

Colour measurements, thermogravimetric and SEM analyses were performed for prepared solid samples. According to the SEM results, the smallest diameter of the capsules obtained was characteristic for the nanospray-dried samples (see Fig. 1).



Spray-dried encapsulated microalgae extract **(S-CHF-SNE-1%)**

Nanospray-dried encapsulated microalgae extract **(NS-CHF-SNE-1%)**

Fig. 1. SEM micrographs of spray-dried and nanospray-dried encapsulated microalgae extract

The amount of phycocyanins (PC) released from the capsules into distilled water and simulated saliva medium were determined. The Trolox equivalent antioxidant capacity (TEAC) of the samples was assessed by using cupric reducing antioxidant capacity (CUPRAC) and 2,2′-azinobis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) methods.

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FORMATION OF CALCIUM SILICATE HYDRATES WITH INTERCALATED Cr^{3+} IONS UNDER HYDROTHERMAL CONDITIONS

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Calcium silicate hydrates and calcium silicates play important roles in cement chemistry, catalysis, medicine, adsorption, and other innovative fields. There are over 40 known crystalline calcium silicate hydrate phases, with molar CaO/SiO_2 ratios varying from 0.44 to 3 [1, 2]. Each calcium silicate hydrate possesses distinct properties that can be modified by intercalating metal ions into their structure. For example, sodium ions influence pore shape, lithium ions reduce surface area and porosity, iron ions decrease crystallinity [3-6]. Recently, calcium silicate hydrates (CaO/SiO_2=1.0-1.5) with intercalated Cr^{3+} ions have been successfully applied for the catalytic oxidation of volatile organic compounds [7]. However, the literature lacks data on the synthesis and properties of calcium silicate hydrates with intercalated Cr^{3+} ions. Therefore, this work aims to determine the influence of hydrothermal synthesis conditions (temperature and duration) on the formation and properties of calcium silicate hydrates with intercalated Cr^{3+} ions.

For the hydrothermal synthesis, a mixture of amorphous silicon dioxide and calcium oxide with the molar ratio of CaO/SiO_2 equal to 1 was used. The solid raw materials were mixed with chromium nitrate solutions (c= 1.0, 2.5, 5.0, 10.0 Cr^{3+} g/l) to reach a water-to-solid ratio of 10.0:1.0. The mixtures were treated under hydrothermal conditions at 200 °C and 220 °C for 0-72 h. It was determined that increased temperature and prolonged duration of hydrothermal synthesis positively affected the formation of crystalline calcium silicate hydrates, while an increase in Cr^{3+} ions concentration negatively affected their formation. However, despite the synthesis conditions, more than 98% of chromium ions intercalated into the structure of the synthesis products. Thermal analysis and nitrogen adsorption-desorption analysis showed that intercalated ions strongly affected the thermal stability and porosity of products.

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THERMOLYSIS OF CYPROTERONE ACETATE AND PROGESTERONE: ISOCONVERSIONAL KINETIC ANALYSIS BASED ON THERMOGRAVIMETRIC DATA

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Despite the current growing interest in thermal stability of pharmaceuticals and their behavior under thermal stress, there is a lack of kinetic studies that present the decomposition of these molecules when heating in an oxidative/inert atmosphere. For this reason, the main objective of this study was to evaluate the kinetic of decomposition of two hormone compounds, namely cyproterone acetate (CYPA, structure in Fig.1a) [1] and progesterone (PROG, structure in Fig.1b) [2] as pure active ingredients vs. two commercialized pharmaceutical solid formulations that contains these compounds in a complex matrix of excipients.

(a) (b)

Fig. 1. Structure of analyzed compounds: (a) CYPA and (b) PROG

Thermogravimetric data were recorded in the temperature range of 40–800 °C in dynamic air atmosphere, at five heating rates, namely 2, 4, 6, 8 and 10 $°\text{C}\cdot\text{min}^{-1}$. For kinetic analysis [3, 4], isoconversional methods of Friedman, Kissinger-Akahira-Sunose and modified non-parametric kinetics were applied, to obtain the best kinetic model that describes the decomposition of the analyzed samples.

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OXIDATIVE DEGRADATION OF PREGNENOLONE AND PRASTERONE – A KINETIC ANALYSIS

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Pregnenolone (abbreviated PREG, Fig.1a), is a naturally occurring endogenous steroid used as active ingredient mainly in over-the-counter supplements, currently being investigated for possible therapeutic applications, but its poor bioavailability limits it's utilization in medicine [1]. PREG can be administered *per os*, as a topical formulation, or by injection into muscle [2].

Prasterone (abbreviated PRAS, Fig.1b) is incorporated in several medications, but is also available as an over-the-counter dietary supplement used to correct dehydroepiandrosterone deficiency. PRAS is used for menopausal hormone therapy, in the treatment of vaginal atrophy, and for cervix preparation during childbirth. PRAS is designed for oral or intramuscular administration, skin and vaginal application [3].

(a) (b)

Fig. 1. Structure of analyzed compounds: (a) PREG and (b) PRAS

Since the development of over-the-counter supplements is not subjected to strict regulations as pharmaceutical drugs, the necessity of analyzing the formulations is evident. According to this, in this study we set our aim into comparative characterization of PREG and PRAS as pure ingredients by instrumental methods (TG/DTG, DSC, FTIR, PXRD) versus two commercial supplements present on the Romanian market. Also, to evaluate the effect of excipients over the stability of formulations vs. pure ingredients, an in-depth kinetic analysis was carried out.

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KINETICS OF Cr^{3+} ADSORPTION ON SYNTHETIC HYDROXYAPATITE

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Chrome ions are known to be as major waste water toxic contaminants, which can be found in surface waters and ground water [1]. Due to high removal efficiency, simplicity and cost effectiveness, adsorption is a promising approach to remove this heavy metal from wastewaters [2]. Since hydroxyapatite is cheap, stable and insoluble in water compound, characteristic to large specific surface area as well as strong adsorption properties, the latter material can potentially be used as synthetic adsorbent to adsorb heavy metals [3]. Therefore, the aim of this work was to apply synthetic hydroxyapatite as an adsorbent to remove Cr^{3+} ions from solution and to determine the kinetic parameters of adsorption.

Synthetic hydroxyapatite was produced in a mixture of calcium carbonate and phosphoric acid (Ca and P molar ratio equal to 1.67) during 2 h of microwave assisted hydrothermal synthesis at 180 ºC. Synthesized products were characterized by analysis methods of XRD, FT-IR and BET.

Time depended adsorption capacity was measured in 25-250 mg/L concentrations of Cr^{3+} ion. In a solution of 25 mg/L of Cr^{3+} ion, the highest adsorption capacity was achieved after 5 min of adsorption removing 81% of ion. Moreover, in a solution of 100 mg/L of Cr^{3+} ion, the highest adsorption capacity was achieved after 15 min of adsorption removing 51 % of ion. Lastly, in a solution of 250 mg/L of Cr^{3+} ion, the highest adsorption capacity was achieved after 10 min of adsorption removing 55 % of ion. It is also important to note that the synthesized samples showed high capacity of stability, since the synthetic adsorbent did not release any of Cr^{3+} ions during the desorption experiments.

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KINETIC STUDY OF THE ANATASE TO RUTILE PHASE TRANSITION IN TITANIUM DIOXIDE

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Titanium dioxide, a material with extensive applications in catalysis, solar cell production, and as a white pigment, is extracted from various natural ores that undergo specific treatments to modify their physical and chemical properties for industrial use. In industry, titanium dioxide is mainly found in two crystalline forms: anatase and rutile. The phase ratio and the impurities present significantly influence the properties of the end product. Anatase, the metastable form of titanium dioxide found in nature, changes into the stable form rutile at higher temperatures. This phase transition is crucial for optimizing production processes and improving the quality of the end product.

The study investigates this transition using methods such as thermogravimetric analysis coupled with mass spectroscopy (TGA-MS) to detect water and sulphur dioxide. In addition, X-ray powder diffraction (XRD) and the Rietveld method are used to quantify the phase composition. The crystalline size of the material is calculated using the Scherrer formula. The Avrami method and the linearization method, a linearized form of the Avrami equation, were utilized. The kinetic parameters of phase transition were determined by identifying the function that best fits the experimental data according to either method.

These analytical methods provide important insights into the phase transitions of titanium dioxide, which contribute to process improvements and increased product quality in industrial applications.

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KINETIC STUDY AND CATALYTIC ACTIVITY OF Fe^{3+} CATALYST SUPPORTED ON GYROLITE FOR VOC OXIDATION

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Volatile organic compounds (VOCs) are among the main anthropogenic air pollutants and are typically removed through catalytic oxidation, a process for which kinetic research is still limited, particularly for newly synthesized materials. In this study, gyrolite intercalated with Fe^{3+} ions was investigated in catalytic oxidation experiments using propyl acetate as a model VOC. A total of 0.2 grams of catalyst was placed in a fixed-bed quartz reactor and supplied with air flows ranging from 200 to 370 mL/min, saturated with propyl acetate. These flow rates, along with the reactor volume and void fraction, were used to calculate the contact times of propyl acetate, which ranged from 3.79 to 7.44 seconds. GC/MS analysis of the inlet and outlet streams indicated that the catalyst is active at temperatures above 300 °C, with longer contact times required to achieve significant oxidation.

According to the law of mass action, the catalytic oxidation should follow pseudo-second-order reaction, given that oxygen is present in large excess. In this case, the kinetic data can be modeled in semi-logarithmic coordinates, as presented in Figure 1.

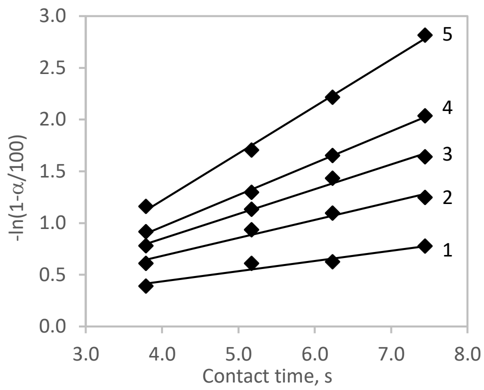

Fig. 1. Propyl acetate oxidation kinetics in semi logarithmic coordinates at various temperatures: 1 – 260 °C; 2 – 280 °C; 3 – 300 °C; 4 – 320 °C, 5 – 340 °C

The slopes of received straight lines indicate catalytic reaction rate constants, that increase with increasing temperature. Reaction rate constants vary between 0.099 and 0.454 s^{-1} depending on temperature and are lower when compared to calcium silicate hydrate with intercalated Cr^{3+} [1]. When plotted in Arrhenius coordinates, reaction rate constants yielded comparably lower activation energy of 49385 J/mol. Even though reaction is more sensitive to temperature it does not translate to higher rate constants because of low Arrhenius constant of 7333 s^{-1}.

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CRYSTALIZATION PROPERTIES OF ZIRKONIA AND BISMUT-ADDED $MgO-Al_2O_3-SiO_2$-GLASS CERAMICS SYSTEM

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Reports have been published regarding the composition of $MgO-Al_2O_3-SiO_2$(MAS). MAS glass ceramics are materials with excellent mechanical properties such as high strength, high young modulus and low expansion coefficients. Cordierite phase is the main phase of MAS glass ceramics and provides high strength properties to this type of glass ceramics [1,2]. In this study, excess ZrO_2 and Bi_2O_3 were added to the MAS composition prepared stoichiometrically from magnesite waste, quartz, kaolin and alumina. The crystallization properties of the prepared compositions were investigated by Kissenger, Ozawa and Augustine-Bennet methods. The mechanical properties of the material were also investigated in the study.

Table 1. Glass transition temperature (T_g) and crystallization temperature (T_c) of samples with different heating rate.

Sample Code	**Tg (^{0}C)**			**Tp (^{0}C)**		
	1^{o}C/min	2.5^{o}C/min	5^{o}C/min	1^{o}C/min	2.5^{o}C/min	5^{o}C/min
MASB	919	920	929	975	996	1028
MASBZ	904	927	929	1000	1027	1050

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DIELECTRIC AND KINETIC PROPERTIES OF SAMARIUM-DOPED BARIUM STRONTIUM TITANATE GLASS CERAMICS

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Reports have been published regarding the composition of $BaO-TiO_2-Al_2O_3-SiO_2$ (BST). BST is a dielectric material with excellent dielectric properties, including a high dielectric constant, small dielectric loss, low leakage current, and large dielectric breakdown strength. BST is a solid solution of $BaTiO_3$ and $SrTiO_3$ with enhanced dielectric properties that has been utilized in various electronic, electrooptical, optical, acoustic, and microwave devices [1,2]. In this research doping of Sm_2O_3 in the $BaO-TiO_2-Al_2O_3-SiO_2$ system affects the crystallization of the glass and the dielectric properties of the glass ceramics. The dielectric constant of these glass ceramics is significantly higher compared to glass ceramics made without Sm_2O_3. The glasses were subjected to various heat-treatment schedules for crystallization. Differential thermal analysis (DTA) patterns show exothermic crystallization sharp peak which shift towards lower temperature with doping of Sm_2O_3. Also, the dielectric constant and tan (δ) of glass ceramics BST and BST-Sm crystallized at 800 ˚C for 1 hour obtain as 17 and 2.45, respectively. These glass ceramics may be used for capacitor applications.

Table 1. Glass transition temperature (T_g) and crystallization temperature (T_c) of samples with different heating rate.

Sample Code	**Tg (^{0}C)**			**Tp (^{0}C)**		
	1°C/min	2.5°C/min	5°C/min	1°C/min	2.5°C/min	5°C/min
BST	580	594	604	712	750	757
BST-Sm	525	559	562	622	655	692

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THERMAL EXPANSION, BULK DENSITY, AND YOUNG'S MODULUS OF ILLITIC CLAY AND $CACO_3$ MIXTURES UP TO 1200 °C

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Nowadays, the production of ceramic materials is focused on the addition of various waste materials. Usually, these materials are characterized by a high content of calcium oxide (CaO). The addition of CaO into the clays often causes the formation of anorthite ($CaO\cdot Al_2O_3\cdot 2SiO_2$), which can improve the material properties, such as the mechanical strength, the porosity, and the firing shrinkage.

In this study the mass changes, thermal expansion, bulk density, and Young's modulus of ceramic samples made from illite-smectite and $CaCO_3$ are investigated up to 1200 °C. The samples are prepared with different $CaCO_3$ contents, namely 17.6 wt.%, 19.6 wt.%, 21.6 wt.%, 23.6 wt.%, and 25.6 wt.%, and are made by dry pressing with 6 wt.% addition of polyvinyl alcohol in the form of 3 wt.% solution. The measurements are performed on rectangular samples at room temperature after their firing at different temperatures up to 1200 °C. The results show that the mass loss of studied samples increases with the amount of $CaCO_3$ in the samples. The shrinkage, the bulk density, and Young's modulus decrease with $CaCO_3$ content in studied sample fired at 1200 °C. After firing at 1200 °C, Young's modulus is higher about eight times than for raw samples.

THE MONITORING OF THE MELTING PROCESS OF GLASS-CERAMIC SAMPLES

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The requirements for electrically insulating materials have appeared naturally with the rapid development of electrical engineering. Usually, these insulating materials are porcelain, which must withstand weather fluctuations for many years. The properties of porcelain insulators are further improved by the application of a suitable surface layer. This layer is a smooth glassy material called glaze. Classical (traditional) glazes could be replaced by glass-ceramic glazes, which achieve better mechanical and electrical properties. Nevertheless, they have not been used for the surface treatment of high-voltage insulators in the meantime. For the production of glass-ceramic glazes, it is necessary to melt the precursor mixture, and then quickly cool it down to form the so-called frits. The frits are the starting material for future glazes. Therefore, it is important, that the chemical composition of glazes is consistent with the chemical composition of the future glass-ceramic glaze. A one-step firing is used, where the suspension is applied to the raw ceramic body of the insulator, and then the insulator is fired together with the glaze for the reduction of the energy of applying the glaze. The aim of this paper is the observation of the melting progress of ceramic samples with different initial compositions, which effectively initiating the formation of mineralogical phases cordierite, mullite, anorthite, spodumene, albite, spinel, and diopside during devitrification (the thermal treatment).

Acknowledgements. This work was supported by Constantine the Philosopher University in Nitra, Grant No. VII/10/2024.

EFFECTS OF ADDING Cr AND Al ON CORROSION RESISTANCE OF NbTiCrAl HIGH ENTROPY ALLOYS

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High Entropy Alloys (HEAs), a new class of metallic materials, have attracted attention due to their unique properties, including high mechanical and corrosion resistance [1]. Corrosion resistance is particularly important in the oil and gas industry, where economic losses are significant [2]. In this context, HEAs have shown excellent corrosion resistance to various aggressive media, whether in structural or biomedical applications, due to the formation of passive protective stable and compact films [3,4]. Therefore, this work investigates the effects of the addition of Cr and Al to the binary equimolar system Nb-Ti with the aim of optimizing the corrosion resistance of the high entropy alloy of the NbTiCrAl system. The alloys 7Nb7Ti, 7Nb7Ti2Cr, 7Nb7Ti2Al, 7Nb7Ti1Cr1Al, 3Nb3Ti1Cr1Al, were produced by electric arc melting furnace and characterized by scanning electron microscopy (SEM/EDS) and X-ray diffraction (XRD). Corrosion tests were also carried out by potentiodynamic polarization (PP) and impedance (EIS), in 3.5% NaCl solution. XRD results showed the presence of only BCC (B2) phase, while SEM/EDS analysis revealed a monophase solid solution microstructure with dendritic morphology. PPL results showed that the addition of Cr and Al significantly improved the corrosion resistance. The addition of Cr resulted in a significant reduction in corrosion current density (Icorr) and a shift to more positive corrosion potentials (Ecorr). By adding Al also promoted improvements, but the combination of both elements was more effective, providing a lower Icorr and the Ecorr values more positive. In addition, the alloys with Cr and Al showed lower passivation densities, indicating the formation of more protective and stable passive films. The EIS results corroborated with the obtained results by PP. The Nyquist plots showed larger capacitive arcs for the alloys containing Cr and Al. The Bode plots show a higher phase angle, suggesting a more ideal capacitive behavior and a better protection against corrosion. Thus, the combined addition of Cr and Al elements in the Nb-Ti system resulted in HEAS with significantly improved corrosion resistance, showing the potential for application in aggressive environments.

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DEVELOPMENT OF SUSTAINABLE LIME-CEMENT PLASTER/PHASE CHANGE MATERIAL COMPOSITES AS ENERGY-EFFICIENT CONSTRUCTION MATERIAL

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This study presents a detailed investigation of phase change material (PCM) composites suitable for incorporation into lime-cement plasters for cooling and heating purposes in interior walls and ceilings. PCMs are microcapsules that absorb and release thermal energy at certain temperatures during the melting and solidification process. In order to achieve a sufficient cooling effect during the day, it is important that the PCM solidifies completely at night and vice versa during the day.

Three plasters are compared in the study: the reference material (lime-cement plaster without PCM additive) and two PCM composite materials (with a PCM additive of 20 and 30 % by volume). The latent heat capacity of the PCM supported the phase transition of the material within a narrow temperature range, increasing thermal comfort without compromising structural integrity. The thermal properties were evaluated by repeated heating and cooling cycles and showed consistent results indicating a reversible phase change of the PCM during melting and solidification. Examination of the microstructure by microscopic techniques showed a uniform distribution of the PCM material in the matrix. The average size of the PCM paraffine wax microcapsules with the melamine-formaldehyde membrane was determined to be about 5 μm in diameter. The work also focuses on the definition of parameters that measure living comfort. We found that the addition of PCMs increases the feeling of comfort on warm and cold days of the day throughout the year.

These results highlight the potential of PCM incorporation into plasters mixes to improve thermal properties while maintaining mechanical strength and provide valuable insights into the development of sustainable and energy-efficient building materials.

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ASSESSING ALTERNATIVE BIOMASS FUEL DERIVED FROM INSECT FARMING WASTE

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The use of fossil fuels greatly increases carbon dioxide emissions, and this encourages the search for alternative sources that are renewable or by-products of other industries and have a lower environmental impact. Various biomass raw materials are used for this purpose. This can be oil from food crops like sugarcane, corn, algae etc. or biomass from agriculture, forest residues, municipal waste etc. [1–3]. This study was intended to analyze the properties of another alternative biomass (insect farming waste – Frass) in order to establish the possibilities of using it as biofuel.

At the beginning, a simultaneous thermal analysis of the used raw materials was carried out, the data of which showed that the Frass samples contained moisture. After it is removed and the temperature reaches ~200 °C, exothermic processes begin, accompanied by the release of heat of ~10000 J/g.

Using frass, coffee grounds and micro cellulose, biomass fuel pellets were formed in laboratory conditions in three different ways: tablets by piston press (Frass tablets – diameter ~200 mm and height ~5–15 mm); cylinders by screw press (Frass cylinders1 – diameter ~5 mm, length ~10–30 mm), and cylinders by compaction matrix (Frass cylinders2 – diameter ~5 mm, length ~10–30 mm).

The properties of the produced granules were investigated: granule stiffness, hygroscopicity, ash content, heat capacity, ash melting point, ash chemical composition. The chemical composition of the gases produced during the burning of such pellets was also analyzed using a Perkin Elmer Clarus 500 chromatograph and the obtained results are presented in Table 1.

Table 1. Combustion gas composition of Frass-biofuel samples formed in different ways

Frass-biofuel sample	**Composition, %**			
	O_2	**CO_2**	**N_2**	**SO_2**
Loose Frass	79,36	19,91	0,39	0,34
Frass tablets	79,26	20,14	0,26	0,34
Frass cylinders1	81,67	17,29	0,48	0,55
Frass cylinders2	79,14	20,11	0,33	0,41

The data show that, regardless of the physical form of the sample, the combustion gases are mostly oxygen (79.14-81.67%), about one-fifth (17.29-20.14%) carbon dioxide, and only trace amounts of nitrogen and sulfur dioxide, as these components make up less than 0.5%.

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SELENIUM BIOFORTIFICATION EFFECT ON GLUCOSINOLATE CONTENT OF *BRASSICA OLERACEA* VAR. *ITALIC* AND *ERUCA VESICARIA*

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Plants serve as important sources of organic Se, possessing the capability to accumulate inorganic Se or its metabolites and store them as organic Se forms. In the environment, selenium occurs in various species of which selenates (SeO_4^{2-}) and selenites (SeO_3^{2-}) are the most significant inorganic forms of Se that are readily taken by plants [1]. As Se is an important micronutrient essential for most organisms, biofortification represents an important strategy to increase Se in plant edible parts. Broccoli (*Brassica oleracea* var. *italic*) and rocket (*Eruca vesicaria* L.), are among popular vegetables consumed by humans. Belonging to the Brassicaceae family, these plants are characterized by glucosinolates (GSLs), specialized metabolites involved in plant responses to both biotic and abiotic stress factors [2].

The first goal of this study involved the cultivation of broccoli and rocket sprouts in hydroponic systems using different concentrations of selenium in HNO_3: 1, 2.5, and 5 ppm. The qualitative and quantitative analysis of GSLs in different plant parts was performed by their desulfo-counterparts using UHPLC-DAD-MS/MS technique. Increased levels of Se had adverse impacts on the growth and biomass of both broccoli and rocket sprouts. Methionine-derived GSLs were the primary type found in both. In broccoli sprouts, gluconapin and glucobrassicanapin were the predominant GSLs, with their levels decreasing at lower Se concentrations compared to the reference. Rocket sprouts mainly contained glucoerucin as the major GSL, and similarly, reduced GSL levels were noted. Additionally, young broccoli and rocket plants, cultivated in soil, were irrigated with sodium selenate solutions at concentrations of 1, 2, and 5 mM. Notably, the adverse effects of the selenate solution on the growth of both aerial parts and roots were evident in comparison to reference plants. The main GSLs detected in broccoli where glucoerucin in the roots, glucoraphanin in stems and 4-hydroxyglucobrassicin in leaves. Rocket plants predominantly contained glucoerucin in the roots and 4-methoxyglucobrassicin in the aerial parts. Under Se-induced stress, the total GSL content generally increased in both plants. Remarkably, the roots and aerial parts of rocket plants treated with high concentrations of sodium selenate exhibited the presence of glucoselenoerucin. This study highlighted that the plant's capacity to tolerate Se stress and its regulation through selenoglucosinolate production are dependent on species, its growth stage, and the form of selenium used for fertilization.

Acknowledgements. We are thankful for the scientific-research equipment financed by EU grant (KK.01.1.1.02.0018).

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OPTIMISATION OF ASSISTED SOLVENT EXTRACTION FOR ISOLATION OF SEA FENNEL PHENOLICS

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In recent decades, new green analytical methods have been developed to produce higher quality plant extracts and optimize the time, yield and cost of their production. Their application also minimizes the harmful effects on the environment and conserves natural resources, which are two main goals of green chemistry. Assisted solvent extraction (ASE) is a relatively new green method that minimizes solvent and sample consumption while increasing the yield of desired analytes using high temperatures and pressures in a short time.

The aim of this work was to find the optimal ASE parameters for the extraction of phenolic compounds, namely chlorogenic acids, from sea fennel. Sea fennel is an edible, perennial halophytic plant that grows along the coasts of the Mediterranean, Black Sea and European Atlantic and is recognized as a good source of bioactive phytochemicals. For this purpose, hydroethanolic mixtures (20, 40, 60 and 80% ethanol) were used as solvents and the extraction was carried out at different temperatures (20, 60, and 100°C). The extractions were performed for 5 minutes under a pressure of 100 bar/1500 psi. From the results obtained, it can be concluded that the composition of the solvent significantly affects the extraction of phenolics and that increasing the ethanol content in the mixture leads to a higher yield of these compounds (from 52 to 63%). The effects of temperature can also be observed and the applied temperature of 60°C resulted in the richest extracts (total phenolics of 673 µg gallic acid equivalents/g). The profile and content of the individual phenolic compounds in the extracts, especially the dominant chlorogenic acids, were analyzed by high-performance liquid chromatography and the results obtained were in agreement with those obtained by the spectrophotometric method. Again, the extract obtained at 60°C with 80% ethanol as solvent contained the highest concentration of chlorogenic acids (33.56 µg/g) so these parameters can be selected as optimal for the extraction of phenolics from sea fennel samples.

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METAL RECOVERY FROM GALVANIC SLUDGE

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Most chemical industry plants produce large amounts of waste every year. Surface treatment industry is no exception. The most important part of the plating process is rinsing which is necessary to remove all the excess chemicals left on the plated parts and avoid the contamination of the upcoming stages of metal finishing. To ensure the effectiveness of rinsing, the water is constantly renewed, and the contaminated water must be treated. The most used process of treatment is neutralization, using sodium hydroxide with which metal ions form insoluble metal hydroxides:

$$\boldsymbol{Ni^{2+}}_{(\boldsymbol{aq})} + \boldsymbol{2OH^{-}}_{(\boldsymbol{aq})} \rightarrow \boldsymbol{Ni(OH)_{2}}_{(\boldsymbol{s})} \tag{1}$$

$$\boldsymbol{Cu^{2+}}_{(\boldsymbol{aq})} + \boldsymbol{2OH^{-}}_{(\boldsymbol{aq})} \rightarrow \boldsymbol{Cu(OH)_{2}}_{(\boldsymbol{s})} \tag{2}$$

$$\boldsymbol{Cr^{3+}}_{(\boldsymbol{aq})} + \boldsymbol{3OH^{-}}_{(\boldsymbol{aq})} \rightarrow \boldsymbol{Cr(OH)_{3}}_{(\boldsymbol{s})} \tag{3}$$

On this case we are referring to chrome plating on plastics process which produces copper, nickel ant chromium contaminated water. After the process of neutralization, metal hydroxides are flocculated sedimented and filtrated to produce galvanic sludge. Metal-rich sludge could be a valuable raw material. In this study, we are trying to find the best possible valorization of this toxic waste.

One possible use of sludge is its pyrometallurgical treatment, which not only re-uses the toxic waste of plating process, but also the waste of coal mining – pyrite[1]. By this process, valuable metals are recovered in their stable, metal state. Another possibility is using galvanic sludge as a material for production of clay bricks[2].

Another way of valorization is to extract metals from sludge by electrowinning – a process which is based on electrodeposition of metals from their ores. In this case, instead of ores, galvanic sludge will be used as raw material. The method is complicated, but very effective. The research on recycling of galvanic sludge, shows that pure copper recovery from sludge was up to 92%[3].

On this study, we are continuing the research of electrowinning, finding the best possible ways to regain pure metals from mixed galvanic sludge efficiently.

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CLEANING OF ELECTROLESS NICKEL PLATING SOLUTION

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In search of abrasive resistance and decorative functions, scientist evolved the industry of metal finishing by electroplating. Most of the metal finishing is used on metals, but there is also a great industry of plating on plastics. Plated plastics are more resistant to environmental threats such as humidity, corrosivity and erosion. Metal plated plastic parts are heavily used by car manufacturers and sanitary equipment manufacturers.

The difference between plating on plastics and metals is that plastic is not conductive. That is why the first layer of metal is produced chemically to make the plastic conductive. For this reason, chemical nickel solution is used, which mainly consists of nickel sulfate, sodium hypophosphite, ammonia, citric acid[1]. The nickel is deposited by its reduction with hypophosphite ions:

$$Ni^{2+}{}_{(aq)} + 3H_2PO_2{}^{-}{}_{(aq)} + 4OH^{-}{}_{(aq)} \rightarrow Ni_{(s)} + 3H_2PO_3{}^{-}{}_{(aq)} + H_{2(g)} \tag{1}$$

The solution is replenished daily, so after a month of continuous additions of chemicals it becomes diluted and unusable for plating and the solution is left off as waste. We are developing a technology that will reduce the waste, by removing leftover nickel (2-3 g/L) in out off order plating bath and reusing it in electrochemical nickel plating. The removal of nickel is based on the increase of pH in the solution, making reaction 1 more rapid, by adding sodium hydroxide. The result is shown in figure 1.

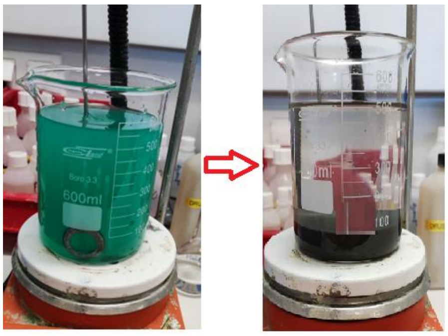

Fig. 3. The solution seconds after adding sodium hydroxide (left) and after overnight reaction (right)

After the filtration, the solution still contains 14-17 mg/L of nickel ions. They are reacted with sodium dimethylthiocarbamate and formed compound is flocculated, filtrated to make a sludge. After the second stage of cleaning, concentration of nickel ions in the solution is less than 0,005 mg/L. The cleaned solution can be discharged to sewage system, because it meets the Lithuanian government requirement of maximum 0,5 mg/L nickel concentration in sewage systems.

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NEW PLASMA METHODOLOGY FOR METALLIC NANOPARTICLE DEPOSITION

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Decreasing particle size to nano size demonstrates peculiar and improved properties such as particle size distribution and morphology, which is not shown by larger particles of the bulk material [1-4]. This unique extrinsic property of the nanoparticle's specific surface area contributes to its high value and influences different intrinsic properties, such as strong surface reactivity, which is size dependent [1-4]. Overall, these unique characteristics of nanoparticles are responsible for their multifunctional properties and the development of interest for their application in diverse areas such as energy, medicine, and nutrition [3-4]. There are many techniques used to obtain nanoparticles and they can be a simple process or a more expensive and complicated one [5]. Plasma-assisted techniques are an interesting methodology for nanoparticle deposition [5]. The main advantage is the low temperature observed in afterglow plasmas (cold plasmas), characteristic of a low-energy ion [5]. The plasma systems used in this work are a new high-density hollow cathode system with a filament surface layer plasma source, both obtained from a metallic vapor by sputtering the filament, after plasma ignition. The deposition process consists of passing a stream of volatile metal precursor vapor over a support with a high specific surface area. This research focused on depositing platinum, silver, iron, and copper nanoparticles onto p-type silicon wafers. The characterization techniques like X-ray diffraction (XRD), scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS), and atomic force microscopy (AFM) were employed. Additionally, a spectrometry technique was used to assess the absorbance of the resulting films. The results demonstrate that the particle size of different materials significantly impacts properties like the particle size distribution and morphology of the deposited film.

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CONSTRUCTING ADAPTABLE DNA TARGET LIBRARIES FOR BENCHMARKING GENE EDITING TOOLS

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Programmable nucleases, such as CRISPR-Cas9, are leading gene editing tools derived from microbial immune systems. To practically apply these programmable nucleases, researchers must thoroughly understand their processes. However, the complexity of these tools, current research methodologies, and resource constraints often extends this to process to years or even decades. Understanding specificity – a nuclease's ability to distinguish target DNA from non-target DNA – is an essential criteria for determining its value for gene editing.

We have developed plasmid-based target DNA libraries for benchmarking and characterizing the specificity of novel programmable nucleases. Each member of a library contains a changeable target sequence, flanked by a barcode sequence and a Protospacer Adjacent Motif (PAM) for cleavage pattern analysis. We use type IIS restriction endonucleases to excise the PAM, allowing us to change the PAM region while keeping the variable targets and barcodes intact. By ligating a short DNA sequence that matches the PAM of a different programmable nuclease, we achieve PAM exchange. This method quickly adapts DNA target libraries to the PAM requirements of each programmable nuclease. Subsequent rapid testing of nuclease specificity, could dramatically shorten the time between gene editing tool discovery and its implementation.

THERMAL PROPERTIES OF DIFFERENT WOOD ASH FROM PAPER INDUSTRY

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Approximately 80% of the nation's pulp, paper, and paperboard needs are met by domestic production. In order to achieve a closed life cycle for materials, integrated waste treatment is essential [1]. This research's goal is to investigate the thermal characteristics of various paper manufacturing wastes.

According to the study's findings, all samples generated as a consequence of the heat treatment and the low water content that was eventually attained are devoid of disease microorganisms and various weed seeds that might have an impact on overall soil sustainability and productivity. It is advised that the procedures and ideal circumstances identified be used in practice, and some of them already [1-4].

Some of the procedures and ideal circumstances that were identified are currently successfully applied in reality and are advised for widespread adoption. The TG-DTA experiments verify that several transformations may occur during thermal treatment, resulting in a change in the solid products' phase composition and the release of gas emissions. The pace of transformations and the degree of enthalpie changes in the system under study are determined by the weight ratio between the components. This makes it possible to calculate the ideal ratio of the components employed in order to produce products that have the appropriate physicochemical and thermal stability qualities for improving soil [1-4].

The effect is monitored by using a complex of analytical methods, such as chemical analysis, Elemental analysis, Microscopic Examination, Fourier transformed infrared (FTIR) measurements and thermal analysis (TG/DTG-DSC).

The efficacious investigations and execution of the combined handling of poultry litter and industrial waste resulting from cellulose manufacturing in Mizia demonstrate that the suggested approach has the potential to serve as a beneficial solution not just in Bulgaria but also in numerous other scenarios. It should be noted that the integrated treatment of poultry litter near additional cellulose manufacturing facilities like Svishtov, Nikopol, Stambolijski towns, etc., in Bulgaria might employ the same technique. The knowledge gained might be applied to the creation of alternative integrated waste treatment programs that aim to fully utilize a biodegradable combination of municipal and industrial wastes.

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NATURAL MINERALS BASED SENSORS

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The enormous advances in the human activities in recent decades have led to ever-improving methods of diagnostics and analyses in ambient and energy harvest. Many of these achievements have been enabled, at least in part, by advances in semiconductor detectors, and energy conversion, both based in silicon or III-V semiconductor. For consumer devices the cost is very prohibitive and reduce the development, because the dependence of the semiconductor manufacturing industry. Various studies suggest a use of alternatives for reducing the cost of semiconductor materials or consumer electronics, the main studies are in amorphous materials and polymeric semiconductor, all excellent alternatives and low-cost fabrication. In this work we porpoise a third alternative; the use of a natural minerals, residues of jewelry industry and mining in sensors production. For this study we selected some mineral residues from the jewelry industry, black, pink, and green tourmalines, and we are using them in thermal and pyrometric sensors. Tourmaline is a natural mineral and has unique pyroelectric and piezoelectric release properties. Pyroelectricity is the property of certain crystalline materials to produce a spontaneous electric polarization upon a change in temperature. Pyroelectricity occurs only in crystalline materials that both lack a center of symmetry and have a polar direction. Pyroelectric materials have a permanent electric polarization below a certain temperature, known as the Curie temperature, which is screened by surface charges in air when the temperature is held constant. A change in temperature (ΔT) causes a slight modification in the positions of the atoms within the asymmetric crystallographic structure. While powerful in their own regard, these techniques suffer from limited spatial resolution and are unable to provide information about the pyroelectric nature of minerals. Beyond mineralogy, the study of pyroelectricity has received significant interest over the last few decades, mainly motivated by its potential application in temperature sensors and infrared detectors and more recently as a possible way to harvest energy from recurrent thermal cycles are all environment-friendly and will not pollute the natural environment. In this work the natural black and colors tourmaline crystal sample was obtained from Minas Gerais State of. The crystalline phases were characterized by X-ray diffraction (with Cu Kα1) with 2θ ranging from 10° to 120° at step width of 0.01° at ambient temperatures. Besides, single-crystal Raman spectroscopy was obtained. The tourmaline sample was cut into small slices (perpendicular to *c*-axis), followed by polishing and powered was characterized in IxV analyses in frequency and thermal source. The results show a possibility of development of thermal and pyrometric sensors using a simple methodology and natural minerals.

ENHANCING STABILITY OF FERMENTED SPIRULINA EXTRACTS THROUGH FREEZE-DRYING ENCAPSULATION

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A. platensis (spirulina), a blue-green microalga, is highly valued for its nutritional content and potential health benefits, including antioxidant, anti-inflammatory, and immune-boosting properties [1]. Previous research has shown that fermentation can enhance spirulina's nutritional and functional properties [2]. However, its incorporation into functional products faces challenges due to issues with stability and bioavailability. Despite these benefits, fermentation and encapsulation have been less explored, particularly regarding its impact on Spirulina extracts' physicochemical properties and stability [3]. Encapsulation techniques have been developed to address these challenges, enhancing bioactive compounds' stability and controlled release. Freeze drying is particularly effective in preserving these ingredients' structural integrity and functionality. This study aimed to encapsulate fermented and non-fermented Spirulina extracts using different wall materials through freeze-drying and evaluate the resulting formulations' efficiency and properties. The encapsulation efficiency of the samples ranged from 97 to 99%, indicating successful encapsulation. The structural and technological properties were characterized through experiments measuring moisture content, solubility, hygroscopicity, bulk density, FT-IR spectroscopy, and thermogravimetric analysis. The samples exhibited moisture content below 10%, solubility ranging from 0.3 to 12% for starch-encapsulated samples and 90 to 95% for whey protein-encapsulated samples, and hygroscopicity values between 7.8 to 15.48%. Thermogravimetric analysis revealed notable thermal stability in the encapsulated Arthrospira platensis. Overall, encapsulation proved to be an efficient strategy for further utilizing fermented extracts. However, further studies are necessary to evaluate the bioactivity and safety of the obtained extracts, ensuring their efficacy for commercial use.

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SYNTHESIS AND CHARACTERIZATION OF A POTENTIAL AMORPHOUS SOLID DISPERSANT BASED ON CELLULOSE DERIVATIVES

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In recent decades, the new chemical entities (NCEs) which have a potential to be used as effective treatments against certain cancers are mostly very poorly soluble in water in their crystalline forms. Therefore, it is of interest to disrupt the formation of crystal so the compound remains amorphous [1]. One way to achieve this is to utilize amorphous solid dispersions (ASDs). By dispersing drug molecules in a stable, amorphous matrix, the crystallization is effectively blocked leading to improved solubility in water. Most commonly used matrices are polysaccharides such as cellulose derivatives and synthetic polymers [2]. Our work includes the functionalization of cellulose derivatives such as sodium carboxymethyl cellulose and hydroxypropyl methylcellulose through graft copolymerization and their characterization with infrared spectroscopy and thermal analysis using primarily differential scanning calorimetry and thermogravimetric analysis.

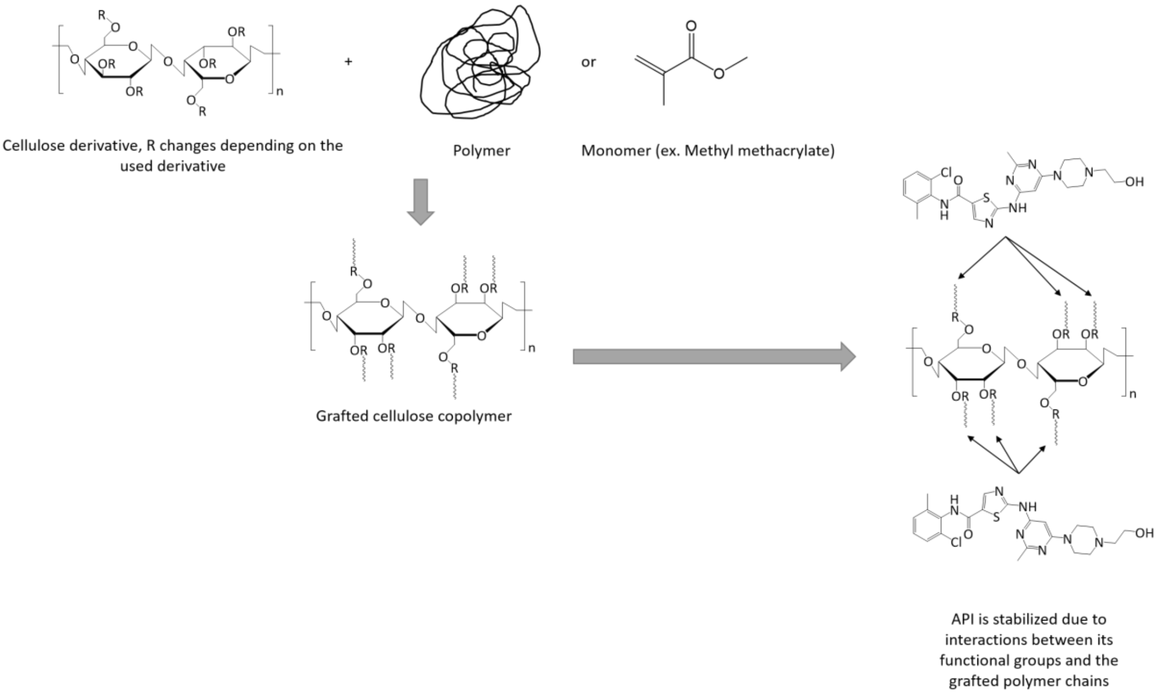

Fig. 1. Synthesis of a grafted cellulose copolymer and stabilization of an API.

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CELLULOSE ACETATE BASED BIOPLASTIC COMPOSITES WITH INORGANIC PARTICLES

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From the mid-20th century, the widespread adoption of petroleum-based plastics began across various industries. Plastic quickly gained global prominence due to its affordability, durability, lightweight properties, flexibility, and ease of manufacturing. However, the environmental ramifications of unregulated production and improper disposal of plastic products, especially plastic packaging have become increasingly apparent. Bio-based and biodegradable cellulose acetate (CA) is an excellent alternative to synthetic plastics. However, CA cannot withstand heat treatment without decomposition and plasticizers should be used to form thermally processable CA compositions. Moreover, inorganic minerals can be used as fillers in polymer composites to increase the melt fluidity and enhance mechanical, thermal and/or dielectric properties of the products.

In this study, the thermoplastic CA composites were formed from CA, various plasticizers, namely, polyethylene glycol, triacetin, triethyl citrate, and two types of inorganic filler particles derived from waste silica gel particles from aluminum fluoride production and opoka rock particles, which were hydrothermally treated and calcinated beforehand. By varying the amounts of CA, plasticizers and filler particles, a set of thermoplastic mixtures was prepared and thermally processed using twin-screw extrusion, by assessing the extrusion parameters. Additionally, the samples were formed by injection molding and their thermal, mechanical and hydrophobicity properties were evaluated for the purpose of evaluating these bioplastic composites in potential applications.

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CHEMICAL RECYCLING OF POLYETHYLENE TEREPHTHALATE BY GLYCOLYSIS USING SYNTHETIC MAYENITE CATALYSTS

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Polyethylene terephthalate (PET) is one of the world's most produced and used plastics. Due to the properties of temperature stability, clarity, chemical resistance, strong weight ratio, wide range of availability and it is not harmful for humans, PET can be extensively used in packaging processes for foodstuffs and beverages [1].

However, there is a huge problem of increasing amount of plastic wastes in the world. PET glycolysis, chemical recycling method, is one of the options to reduce PET postconsumer wastes. PET flakes dissolve in ethylene glycol (EG) in the presence of catalyst and causes formation of bis-2-hydroxyethyl terephthalate (BHET) monomer. In our research, mayenite ($Ca_{12}Al_{14}O_{33}$) was used as a catalyst for glycolysis reaction. PET is depolymerized by glycolysis reaction with EG. However, catalysts have a major impact on glycolysis reactions, as without addition of catalyst no depolymerization reaction was observed. This work reports for the first time synthetic mayenite ($Ca_{12}Al_{14}O_{33}$), obtained at different calcination temperatures (350-900°C), as an effective catalyst for PET depolymerization.

In particular, it was found that interaction between mayenite and EG can achieve high heat release in the range of 401-734 J/g (mayenite). The research results showed that efficiency of glycolysis reaction increases with calcination temperature of mayenite and the most effective catalyst is $Ca_{12}Al_{14}O_{33}$(900°C), which is characterized by big surface area (24.55 m^2/g) and pore volume (102.0 mm^3/g). It was determined that $Ca_{12}Al_{14}O_{33}$(350°C), $Ca_{12}Al_{14}O_{33}$(550°C), $Ca_{12}Al_{14}O_{33}$(900°C) optimal doses are 0.8, 0.7, 0.5 mmol/mol(PET), respectively. Yield of the reaction product bis(2-hydroxyethylterephthalate) (BHET) was 68-69 %, while the yield of the conversion of PET was 83-86 %. ^1H and ^{13}C nuclear magnetic resonance (NMR) spectra, Fourier transform infrared (FT-IR) spectroscopy, differential scanning calorimetry (DSC), thermogravimetry (TG), X-ray diffraction (XRD) and scanning electron microscopy (SEM) data analysis confirmed structure of BHET, which was obtained during depolymerization of PET while using mayenite as a catalyst.

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KINETIC ANALYSIS OF THE NON-ISOTHERMAL DECOMPOSITION OF PEO10/NaMMT NANOCOMPOSITES

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The melt intercalation method was used to prepare polymer nanocomposite based on poly(ethylene oxide) (PEO10) ($\bar{M}_v = 1.000.000$) and a nanofiller, sodium montmorillonite (NaMMT). Samples were made with different weight ratios of PEO to NaMMT – PEO10/NaMMT 100/0, 90/10, 80/20, 70/30, 60/40, 50/50, 40/60, 30/70, 20/80 and 10/90. Thermogravimetric analysis (TGA) was performed in the temperature range 50-600 °C, at four heating rates (2.5; 5, 10 and 20 Cmin^{-1}), nitrogen atmosphere. TGA showed lower thermal stability of PEO/NaMMT polymer nanocomposite compared to pure PEO. Kinetic analysis of the thermal decomposition was used to reveal the influence of NaMMT nanofiller on the thermal decomposition mechanism of PEO and to calculate the kinetic parameters of the thermal decomposition process. Calculation were performed using Netzch Professional Thermokinetic Software. The results show that nanofiller NaMMT up to weight ratio 60/40 doesn't change the simple, one-step decomposition mechanism of PEO. At higher NaMMT loadings, diffusion becomes limiting step at the beginning of the decomposition process while the main decomposition mechanism remains unchanged.

WHEN NATURE CALLS - IS THIS THE ONLY PURPOSE TRADITIONAL CAT LITTER IS GOOD FOR?

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Two types of cat litter, designated as natural bentonite and bentonite on the packaging, were analyzed to investigate their sorption ability to remove copper ions from aqueous solutions. The chemical composition and morphology of bentonite were characterized by XRF, SEM, EDS and FTIR analysis. The microstructure of the bentonite was found to be porous and nonhomogeneous with aggregates. The presence of copper in all cat litter samples before and after the sorption process was confirmed by EDS analysis. The sorption process occurred at the surface of the cat litter, and no additional peaks or changes in band positions occurred in the FTIR spectra. Kinetics and equilibrium of copper ions removal were investigated for better understanding of sorption process. Experimental equilibrium data were best fitted by the Jovanovic isotherm, which assumes mobile and monolayer localized sorption without lateral interactions. The sorption kinetics are best described by the Elovich equation and the Weber-Morris model as a function of the initial concentration of copper ions. According to the results, both types of cat litter can be used for copper ions removal from aqueous solutions, with the one designated as natural bentonite having a higher sorption capacity.

THE EFFECT OF TEMPERATURE AND P^{5+} IONS CONCENTRATION ON THE PHOSPHORUS ADSORPTION MECHANISM

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Phosphorus is an essential nutrient necessary for all living organisms' growth. The future of phosphorus rock is a topic of concern due to its finite nature and critical role in agriculture. According to projections by the International Fertilizer Development Center (IFDC), these reserves could be sufficient to meet agricultural demand for the next 50 to 100 years, assuming current rates of extraction and consumption. Nevertheless, the release of phosphorus from wastewater effluents into the environment is a significant factor contributing to eutrophication in water bodies [1, 2].

The need to control eutrophication in water bodies and the risk of supply shortage of phosphate rock has motivated the search for treatment techniques able to remove and recover P from wastewater [3]. Adsorption is recognized as one of the most effective methods due to its advantages of low cost, high efficiency, and simple operation [4,5].

In this study, adsorption experiments were carried out at 25, 35, and 45 ℃ temperatures in a thermostatic absorber by stirring 10 g of calcium silicate hydrate-based adsorbent in 1 L of KH_2PO_4 solution containing 0.2, 0.5, 1.0, 1.5 0, 2.6, and 5 g P^{5+}/L. Phosphate adsorption properties were studied by investigating experimental parameters, such as concentration and temperature—the concentration of phosphorus in the solution after adsorption was determined by the photocolorimetic method. Adsorption isotherm, kinetic, and thermodynamic parameters were estimated from the experimental results.

The results demonstrated that both concentration of initial solution and temperature of liquid medium influence the adsorbed amount of phosphorous. The optimum temperature for phosphorous ions adsorption is 35 °C and the highest removal efficiency of phosphorus ions, up to 99 %, was observed in a KH_2PO_4 solution containing 0.5 g P^{5+}/L. XRD, XRF, STA, and spectrophotometer analysis confirmed the obtained results.

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INTENSIFICATION OF THE SORPTION PROCESS: THE ROLE OF IMPELLER BLADE INCLINATION AND SIZE IN BATCH REACTOR

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Batch sorption is a useful technology for removing various contaminants from wastewater. It is influenced by a number of factors such as pH, temperature, type of sorbent, mass of sorbent, coexisting ions, and hydrodynamic conditions. Although the reaction mixture in a batch reactor must be properly mixed and there are numerous articles studying sorption, in most of these studies, factors related to hydrodynamic conditions are overlooked. Therefore, the purpose of this study was to investigate the effects of the turbine impeller blade inclination and its size on sorption efficiency and kinetics in a batch reactor. The research focused on copper (II) ions sorption on zeolite NaX particles smaller than 0.04 mm. The experiments were carried out in a unbaffled reactor with a four-blade turbine positioned close to the vessel's base (off-bottom clearance was 0.10) at 120 rpm.

To investigate the effects of impeller blade inclination and the impeller size, the impeller blade inclination was varied from 30° to 90° and the impeller to reactor diameter ratio was varied from 0.32 to 0.68. The influence of impeller size was studied with the impeller that found to be the most effective, while analyzing the effect of blade inclination.

A transient multiphase computational fluid dynamics model was developed to gain valuable insight into the system hydrodynamic behavior. The Ritchie model and the Mixed surface reaction and diffusion-controlled sorption kinetic model were used for the kinetic analysis of the experimental kinetic data [1-2].

At a constant impeller rotation speed (N = 120 rpm), the blade inclination influences the efficiency and kinetics of the process examined for the reaction conditions studied. The removal efficiency and the sorption rate increase with the inclination of the blades, i.e. the fastest sorption was achieved with a blade inclination of 90° (SBT impeller). At the same impeller speed, the influence of the SBT impeller to reactor diameter ratio, D/d_T, has no significant effect on the efficiency of the tested process if D/d_T is greater than 0.46. The sorption rate was found to vary as a function of D/d_T, which is to be expected since the critical rotation speed depends on the D/d_T ratio. The calculated AARD values show that, in most of the experiments, the surface reaction is the dominating step.

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ELECTROCOAGULATION COMBINED WITH PIROPHILITE AND ZEOLITE IN COMPOST WASTEWATER TREATMENT

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Investigation and development of efficient and economical wastewater treatment technologies are necessary to combat environmental pollution and the acute shortage of drinking water worldwide. In this paper, electrocoagulation in combination with synthetic zeolite (EC-SZ) and pyrophyllite (EC-PIR) was investigated as a hybrid treatment method for compost wastewater. The experiments were performed with aluminium electrodes, while straight blade turbine (SBT) impeller was used for mixing. The influence of sorbent addition (synthetic zeolite and pyrophyllite) and sorbent granulation (< 40 mm and 160-500 mm) on the EC was investigated with regard to the change in physico-chemical parameters in the solution (pH, temperature, COD, turbidity), the EC sludge and the collected saturated sorbent (synthetic zeolite or pyrophyllite), the changes in electrode mass and surface, voltage and power consumption, settling ability and potential difference in the suspension during settling. The chemical oxygen demand (COD) decreases by 77.98-88.76 % after 30 minutes. A slightly better collection of saturated zeolite/pyrophyllite after treatment is achieved with a smaller particle size of both sorbents. In experiments with the both sorbents with smaller particle size, good settling ability is observed and the results are consistent with a lower potential difference measured in the suspensions during settling. Among the sorbents, the settling behaviour of synthetic zeolite is better than that of pyrophyllite. In addition, corrosion damage on the electrode surface was investigated using an optical microscope. Finally, Taguchi optimization using the L4 model was applied to analyse the influence of sorbent type (synthetic zeolite and pyrophyllite), particle size (< 40 mm and 160-500 mm) and contact time (10 and 20 min) on the decrease of COD and turbidity, electrode consumption, settling velocity and voltage consumption.

QUALITATIVE AND QUANTITATIVE DETERMINATION OF SELECTED ISOTHIOCYANATES USING GC-MS/MS

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Isothiocyanates are glucosinolate hydrolysis products responsible for the aroma of these phytochemicals found mostly in plants from the cabbage family. Due to the many biological activities these compounds possess, there is a need for methods for their determination. In this study, a method for determining 2-methoxyphenyl isothiocyanate, 3-methoxyphenyl isothiocyanate and 4-methoxyphenyl isothiocyanate was developed, optimized and validated. Gas chromatography-tandem mass spectrometry (GC-MS/MS) was applied for the separation and individual qualitative and quantitative determination of these compounds. Standards of 2-, 3- and 4-methoxyphenyl isothiocyanate (MOPITC) and hexane as a solvent were used for the preparation of solutions. Gas chromatography was optimized by changing the oven temperature program rate (20 °C/min was selected). A triple quadrupole mass spectrometer, in MRM scan mode, was used for monitoring specific fragmentation reactions (quantifier and two or three qualifiers for each compound). Mass spectrometry was optimised by determining the collision energy with nitrogen for each transition. Different approaches for preparing the calibration curve solutions as well as different concentration ranges were investigated. Forward pipetting with mechanical pipettes and the range of 2.5–20.0 µg/mL was selected. Obtained calibration curves showed very good linearity ($R^2 > 0.997$) with low limits of detection and quantification. Accuracy, repeatability, intra- and inter-day precision, specificity and stability were proven satisfactory according to the ICH guideline [1]. This ensures the suitability of the method for the individual determination of 2-, 3- and 4-MOPITC in the mixture.

Table 1. MRM transitions with the optimum collision energies

MRM transition (Collision Energy)	**2-MOPITC**	**3-MOPITC**	**4-MOPITC**
Quantifier	165 → 132 (10 eV)	165 → 77 (28 eV)	165 → 122 (28 eV)
Qualifier 1	165 → 122 (28 eV)	165 → 135 (14 eV)	165 → 78 (42 eV)
Qualifier 2	122 → 95 (12 eV)	135 → 77 (14 eV)	150 → 122 (12 eV)
Qualifier 3	–	135 → 51 (40 eV)	–

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ETHYL CELLULOSE AS A HOST MATERIAL FOR THERMALLY-ACTIVATED DELAYED FLUORESCENCE EMITTERS

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Thermally-activated delayed fluorescence (TADF) emitters are usually used in OLEDs in the form of doping into a host matrix in order to prevent emission losses due to aggregation effects, leading to concentration quenching and unwanted excimer emission. As known, small molecules are usually used for the design of such host systems. In this work, we study application of ethyl cellulose (EC) as a host polymer matrix for specific TADF molecules and how it influences their photoluminescence properties in the solid-state films. Two pairs of TADF molecules were selected, which differ by the presence of sulfonyl and carbonyl group in the main chain. Fluorescence microscopy studies indicated both different morphology and emission colour of the samples, where the neat dye films were composed of aggregated species with poor emission, while the guest-host systems demonstrated smooth morphology with more even and brighter emission (Fig. 1). The mechanisms of the PL enhancement and colour changes of TADF emission in the EC matrix are discussed.

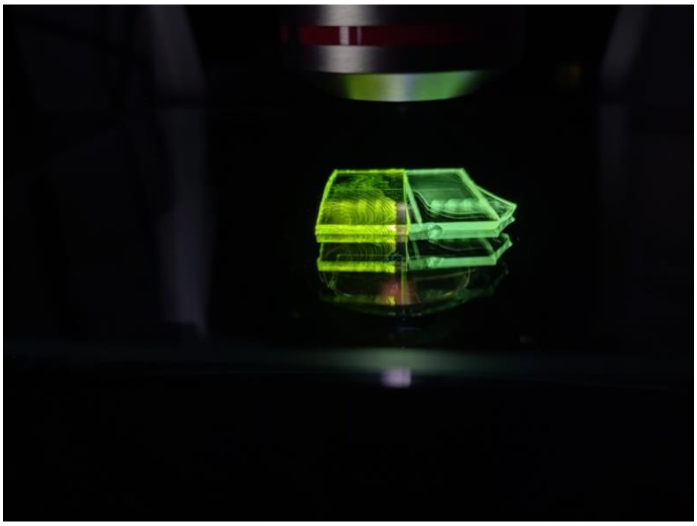

Fig. 1. Fluorescence of the neat dye film (on the left) and dye in the EC matrix (on the right) under UV light.

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CEEC-PCMS 2

INFLUENCE OF INTERCALATED Fe^{3+} IONS ON THE THERMAL STABILITY AND POROSITY OF GYROLITE

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Calcium silicate hydrates are a group of compounds that play a crucial role in cement chemistry and are also significant in catalysis, adsorption, and medicine [1]. When modified by metal ions, these compounds can be applied in the oxidation of volatile organic compounds at lower temperatures compared to traditional catalyst [2]. While some calcium silicate hydrates modified with ions such Na, Al, Cr, Ni, and Co, etc. [3] have been investigated in detail, there is limited literature on the influence of iron ions on the synthesis and properties of calcium silicate hydrates, particularly gyrolite ($Ca_{16}Si_{24}O_{60}(OH)_8{\cdot}14H_2O$), which has unique adsorption properties [4]. For this reason, this work aims to determine the influence of intercalated iron ions on the thermal stability and porosity of hydrothermally synthesized gyrolite.

Gyrolite with intercalated iron ions were synthesized under hydrothermal synthesis (200 °C, 72 h) using stoichiometric mixture of calcium oxide and silicon dioxide (molar ratio of CaO/SiO_2 was 0.66). A 2 g of solid mixture of CaO and SiO2 was mixed with 20 ml of iron nitrate solutions of varying concentrations to achieve water-to-solid ratio of 10 and iron ions concentrations of 0, 25, 75, or 150 mg per gram of the $0.66CaO + SiO_2$ mixture.

Gyrolite with intercalated iron ions was successfully synthesized under these hydrothermal conditions. It was determined that the amount of Fe^{3+} additive and the temperature of calcination significantly influence the porosity of the products. A small amount (25 mg Fe^{3+}/g) of intercalated iron ions significantly increased the specific surface area (from 28 m^2/g to 87 m^2/g) and total pore volume (from 0.300 cm^3/g to 0.570 cm^3/g) of gyrolite. However, further increases in Fe^{3+} ions concentration led to the decreases in these values. The results were confirmed by X-ray diffraction, in-situ XRD, simultaneous thermal analysis, and Fourier-transform infrared spectroscopy. Additionally, nitrogen adsorption-desorption isotherms at 77 K were used to determine the specific surface area and total pore volume of gyrolite samples.

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SYNTHESIS OF COMPLEX CONCENTRATED SILICIDE COATINGS VIA REACTIVE MELT INFILTRATION

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New materials and coatings, primarily based on refractory high entropy alloys (RHEAS) and refractory complex concentrated alloys (RCCA), have been given much attention as alternative contenders to replace traditional superalloys allowing to increase the working-temperatures limits [1]. Silicide-based coatings are a promising solution to address the problem of poor oxidation resistance of metallic refractory alloys [2] [3]. High-entropy silicides and Complex Concentrated Silicides have recently been introduced as potential groundbreaking solutions to the challenges faced by RHEAs [4] [5]. However, the development of coating technologies using these materials is in the early stages [6] [7] [8]. This study will explore the synthesis of complex concentrated silicide coatings through a reactive melt infiltration approach. Research is being conducted using different solid RCCA substrates, which are subjected to solid/liquid interfacial experiments with various liquid Si-alloy sources at different melt temperatures and reaction times. After the high-temperature tests, the microstructure of the solidified samples will be characterized by X-ray diffraction and scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy. Subsequently, further research will be conducted to determine the oxidation behavior of these new coatings.

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BIOCIDE-POLYMER NANOCOMPOSITES: A PROMISING STRATEGY IN THE FIGHT AGAINST VIRAL INFECTIONS

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Research on biocide-containing coatings is essential for public health and to prevent the spread of diseases caused by micro-organisms [1]. The link between hygiene and disease highlights the importance of advances in surface modification and antimicrobial coatings [1]. The challenges of gram-positive and gram-negative bacteria drive the development of new disinfectants and biocidal agents [2]. Furthermore, bacteriophage ϕ6 is a useful surrogate for studying viruses such as SARS-CoV-2 in antiviral research [3].

In this work, chemically synthesized biocidal substances were combined with a PVB polymer matrix to create a biocide-polymer nanocomposite. Biocidal coatings (see Fig.1), including ampicillin sodium salt (A), benzalkonium chloride (BAC), hexadecyl trimethyl ammonium bromide (CTAB), a solution of 2-Mercaptopyridine N-oxide sodium salt (NaPT), and triclosan powder (T), were applied using a commercial slot-die coater (Ossila Ltd, UK).

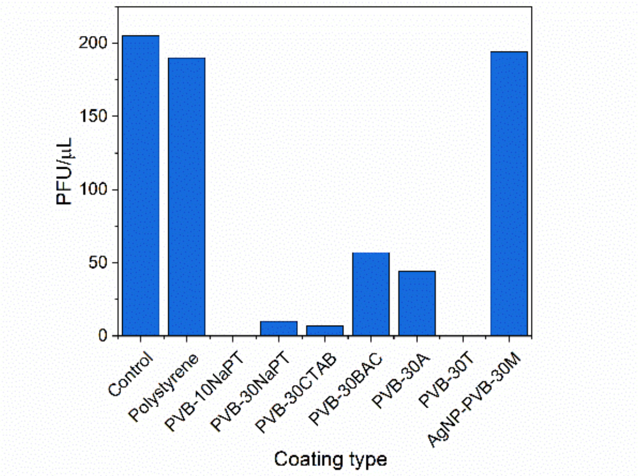

Fig. 1. The viability of phage ϕ6 was evaluated after one hour of incubation with the samples at 25 °C

The results showed that PVB-30T and PVB-10NaT samples had the highest reduction in virus infectivity, with no detectable phages. PVB-30BAX, PVB-30A, PVB-30CTAB, and PVB-30NaT also exhibited antiviral properties, halving ϕ6 virus viability. In contrast, AgNP-PVB-30M was ineffective, similar to the control (polystyrene). This study supports the development of durable antimicrobial coatings to combat infectious disease transmission.

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STUDY OF FORMATION OF CADMIUM SULFIDE LAYERS ON POLYPROPYLENE FILM

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Cadmium sulphide (CdS) is an important group II-VI semiconductor whose thin films are widely used in electronics and optoelectronic devices as photosensors [1], high-efficiency red-light emitting diodes [2] and transistors [3] due to their optical and electro-optical properties. Polycrystalline CdS thin films are one of the ideal materials for solar cells due to their good electrical properties, excellent optical transmittance and wide band gap (2,41-2,47 eV [4]). Polymers with metal chalcogenide films deposited on their surface are one of the new innovative composite materials. These composites often take on the properties of both polymers and metal chalcogenide films, which opens up significant opportunities for the development of intelligent devices. It is known that the different morphology, surface structure and stoichiometric composition of metal sulfide films have a significant influence on the electrochemical properties of materials [5]. It is therefore essential to be able to control the surface morphology and qualitative composition of metal chalcogenide films.

The aim of this study was to form a cadmium sulfide film on the surface of superhydrophobic microplastic polypropylene strips by a technologically simple and inexpensive chemical bath deposition (CBD) method. Strips of polymer 2.0×6 cm in size and 150 mm thick were used as the substrate. Cadmium acetate ammonia buffer and thiourea solutions were used as precursors for cadmium and sulfur ions respectively. The polypropylene strips were stored for up to 2 hours at 70^{o} C in a beaker containing solutions of both precursors at concentrations of 0,15-1,0 mol/L. The phase and chemical composition of the CdS layers formed on the surface of the polypropylene strip was investigated by X-ray phase analysis (XRD), inductively coupled plasma atomic emission spectroscopy (ICP-OES) and energy dispersive spectroscopy (EDS). The morphology of these layers was studied by scanning electron microscopy (SEM) and atomic force microscopy (AFM). The optical properties of the CdS films were studied by UV-Vis spectroscopy (UV-Vis). The studies confirmed that CdS films were formed on the surface of the polypropylene strips, the composition, surface morphology as well as the optical properties of which were strongly influenced by the forming conditions. It was also found that the structural order of the film formed on polypropylene becomes more uniform and homogeneous as the concentration of cadmium sulfide increases.

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HIGH ENERGY MILLED AND HIGH TEMPERATURE ACTIVATION OF APATITE-QUARTZ-CALCITE NATURAL SYSTEM

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In this study high energy milled (HEM) samples of natural phosphorites from Bulgarian and Estonian deposits were investigated. The activation was performed via planetary mill with Cr-Ni grinders with a diameter of 20 mm. This method is an ecological alternative, since it eliminates the disadvantages of conventional acid methods, namely the release of gaseous and solid technogenic products [1,2].

The aim of the study is to determine the changes in the structure to follow the solid-state transitions and the isomorphic substitutions in the anionic sub-lattice in the structure of the main mineral apatite in the samples from Bulgaria and Estonia, under the influence of HEM activation. It is also interesting to investigate the influence of HEM on structural-phase transformations on the structure of impurity minerals - free calcite/dolomite, pyrite, quartz, as well as to assess their influence on the thermal behavior of the main mineral apatite [3].

The effect of HEM is monitored by using a complex of analytical methods, such as chemical analysis, specific surface area (SSA), powder X-ray powder diffraction (PXRD), X-ray fluorescence (WD-XRF) analysis, Fourier transformed infrared (FTIR) measurements and thermal analysis (TG/DTG-DSC) coupled with Pfeiffer Omnistar Mass Spectrometer. The study of the activation effect shows that the mechanical activation has the following impacts at micro-level: (i) deformation of polyhedrons; (ii) smaller size of crystallites with an increased degree of structural defects leading to a metastable phosphorite with increased dispersity [4].The obtained results prove the correlation in the behavior of the studied samples in regards to their quartz content and bonded or non-bonded carbonate ions. After HEM activation of the raw samples, the A- and A-B type carbonate-apatites are formed. The new isomorphic phases (A- and A-B type carbonate-apatite) are decarbonized at temperatures of about 780-850 K and 1050-1100 K. In the presence of quartz, the decomposition temperature for carbonate ions in A-type is increased. In addition, the moisture and higher quartz content in the samples leads to a shift in the dehydroxylation temperature.

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STRUCTURAL CHARACTERIZATIONS OF BISMUTH SULFIDE LAYERS ON FTO GLASS

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Bismuth sulfide stands as a widely favored non-toxic semiconductor, acclaimed for its band gap energy of 1.3 eV, which is considered moderate but surpasses the band gap energy of other sulfides within the same group [1]. When coupled with FTO glass, it presents itself as the optimal selection for optoelectronic devices, especially suited for solar cells.

The present study focuses on the reaction between $Bi(NO_3)_3·5H_2O$ and L-cysteine, utilizing EDTA-Na_2 as a chelating agent. EDTA-Na_2 initially coordinates with bismuth ions to form a stable ring structure $[Bi\text{-}EDTA]^-$, thus the direct bonding with S^{2-} is prevented, making the reaction product more disperse [2]. As the temperature increases, Bi_2S_3 nuclei slowly form, leading to the formation of crystals. This reaction occurs at 90 °C and continues for a minimum of 8 hours, ultimately yielding dark Bi_2S_3.

The precipitate is collected and dispersed in a solution of glycerol and anhydrous ethanol in a 1:1 ratio. Subsequently, the suspension is introduced on to FTO glass by dip coating or applied using the spin coating method for 60 seconds, maintaining a rotation speed of 2000 RPM. Deposition varied by applying multiple layers and different concentrations. The films were then annealed at 300°C for 2 hours in order to achieve a crystalline Bi_2S_3 nanostructured thin film. All samples were analyzed by X-ray diffraction analysis on the Bruker D8 Advance diffractometer. Precipitate was scanned over the range $2\theta = 3–70°$ at a scanning speed of $1°\ min^{-1}$ using a coupled two theta/theta scan type. This test determined structural characterization of the obtained materials. The acquired results were compared and discussed.

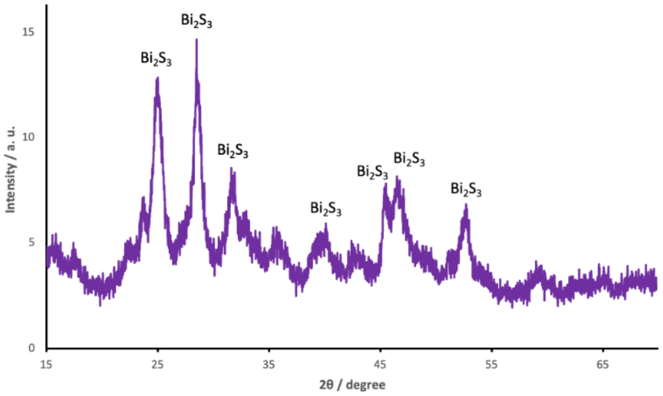

Fig. 1. XRD pattern of obtained Bi_2S_3. Compared with Bi_2S_3 JCPDS No. 84 – 279.

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HEATING MECHANISM OF CARBON MATERIALS THROUGH MICROWAVE TREATMENT

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Several technological processes utilizing microwaves have been developed and implemented at the industrial level: wood drying, chemical synthesis, various applications in food technology, while other processes are still in the research phase: sintering, welding and melting of metallic and ceramic materials, and the production and regeneration of activated carbons. The limitation of the use of these technological processes is largely due to the lack of data explaining the mechanism of microwave action on solid materials such as ceramics, metals, and carbonaceous substances.

Attempts to explain the mechanism of microwave interaction with carbonaceous substances are documented in several publications, indicating different factors that lead to heating: dipole rotation, interfacial polarization, microwave interaction with delocalized π electrons, and conduction polarization. All these mechanisms are based on the principle of dielectric heating, with differences in the polarization process: dipolar, ionic, or interfacial. Each of the aforementioned heating mechanisms has certain shortcomings that do not allow for the correlation of the structure and properties of carbonaceous materials with their behavior under microwave treatment, often adopting concepts valid only for liquids. Speaking of carbonaceous substances such as graphite, nanotubes, or activated carbon, it must be noted that these substances do not contain flexible polar groups capable of oscillating in a high-frequency field.

In this study, for the first time, some new experimental data are presented that would advance the understanding of the heating mechanism of carbonaceous substances through microwave treatment. This would allow for the understanding and implementation of the technology for the production and regeneration of activated carbons using microwave treatment. The validation of the obtained results was carried out on a wide range of activated carbons of different origins (wood, pits, shells). The proposed mechanism does not contradict the properties and structure of other carbonaceous substances and can be successfully applied to explain their behavior under microwave treatment.

The factors determining the heating dynamics are: the geometry and size of the particles (the material's capacity to accumulate electric charge), the internal electrical resistance of the material (the substance's ability to generate mobile electrons under the action of an electromagnetic field), and the presence of a dielectric layer between conductive zones (electrical resistance between particles).

THE MECHANICAL PROPERTIES OF DOUBLE-LAYER NI-BASE COATINGS PRODUCED BY THERMAL SPRAY TECHNIQUES

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Thermal spray coating is well-known for creating coatings that protect against wear and corrosion. Ni-base coatings, especially the commercial Inconel 625 and Hastelloy C-276 coatings, are widely used in the industrial sector for corrosion resistance. However, coating in the industrial sector usually involves spraying more than one layer of coating due to various advantages, such as adjusting the surface of the substrate before coating, reducing the thermal coefficient difference between the substrate and the coating material, and enhancing the adhesion strength of the coating. This research aims to study the mechanical properties of Inconel 625 and Hastelloy C-276 coatings by coating them on a Ni-20 wt%Cr bond layer. The study compares the mechanical properties of coatings applied using different coating processes, namely arc spraying and high velocity oxy-fuel (HVOF) spraying. Mechanical testing included microhardness measurements, wear tests, and nanoindentation assessments. Microstructural analysis was conducted using scanning electron microscope (SEM) and energy-dispersive X-ray spectroscopy (EDS) to examine the interfacial bonding and elemental distribution.

Results indicate that the double-layer coating exhibits excellent mechanical properties compared to single-layer coatings of either Inconel 625 or Hastelloy C-276. The microhardness of the coatings showed a significant increase, attributed to the synergistic effects of the two materials and the process used. The HVOF coating demonstrated better mechanical properties, with high resistance to crack propagation. Wear resistance tests confirmed the durability of the double-layer system, showing minimal material loss under abrasive conditions. In summary, the double-layer coating of Inconel 625 and Hastelloy C-276 is an attractive condition for applications requiring high mechanical strength and resistance to extreme conditions. This study highlights the potential benefits of combining these materials to enhance the protective performance of coatings in industrial applications.

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MICROWAVE-ASSISTED SYNTHESIS OF MAGNESIUM PHOSPHATE IN THE TEMPERATURE RANGE OF 25–200 °C

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Magnesium phosphates are inorganic salts found in living organisms and nature, which can be synthesized by reacting raw materials containing magnesium and phosphorus components. The molar ratio of magnesium to phosphorus (Mg/P) in these compounds varies from 0.5 to 1.5. Improving magnesium phosphate synthesis methods is significant in both academic and industrial fields due to their wide range of applications, especially in biomedicine and as bone substitutes. These compounds can be synthesized through various methods: precipitation, hydrothermal synthesis, sol-gel, microwave-assisted synthesis, electrochemical synthesis, and others. Each methods has its advantages and disadvantages, with the choice depending on the specific application, desired properties of the magnesium phosphate, and available resources [1-4]. However, the microwave-assisted synthesis method is the least described in the literature. Thus, this work aims to synthesize magnesium phosphate in the $MgCO_3$–H_3PO_4–H_2O system within a temperature range of 25–200 °C during microwave-assisted synthesis.

For the synthesis of magnesium phosphates, a mixture consisting of magnesium carbonate, phosphoric acid, and water was used. The molar ratio of Mg and P of the initial mixture was 1.5. The required amount of magnesium carbonate was mixed with diluted phosphoric acid to achieve liquid to solid ratio of 10. The resulting solution was left for 30 min at room temperature to remove CO_2 formed during the neutralization reaction. The prepared mixture was then placed in a microwave reactor "Monowave 300", where it was treated at temperatures ranging from 25 to 200 °C for 2 h. The reaction temperature was reached within 3 min, and the mixture was stirred at 200 rpm during the reaction.

It was determined that the phase composition and thermal properties of formed compounds strongly depend on the synthesis temperature. At temperatures of 25–60 °C, newberyite ($Mg(PO_3OH)\cdot 3H_2O$) and hydromagnesite ($Mg_5(CO_3)_4OH\cdot 4H_2O$) were formed. It should be noted that newberyite recrystallized into magnesium phosphate pentahydrate ($Mg_3(PO_4)_2\cdot 5H_2O$) when the synthesis temperature was increased to 80 °C. At higher temperatures, up to 160 °C, magnesium phosphate hydrate lost one molecule of water, forming magnesium phosphate tetrahydrate ($Mg_3(PO_4)_2\cdot 4H_2O$). During synthesis at 200 °C, along with hydromagnesite and magnesium phosphate tetrahydrate, diffraction peaks characteristic of magnesium pyrophosphate ($Mg_2P_2O_7$) was also identified.

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APPLICATION OF VARIOUS THERMAL ANALYSIS TECHNIQUES TO GAIN AN EFFECTIVE INSIGHT INTO THE THERMAL PROPERTIES OF PCM PARAFFINS AS AN ADDITIVE TO CEMENT COMPOSITES

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Phase change materials (PCMs) absorb or release heat when their aggregate state changes. The addition of PCMs to building materials such as cementitious composites changes their specific heat capacity and consequently reduces the need to temper buildings, improving their energy efficiency, which is a viable approach to harnessing solar heat and optimising energy consumption in buildings. Therefore, accurate material characterisation of thermal energy storage materials, including energy storage capacity and phase change temperature, during multiple charge and discharge cycles is important. The encapsulated PCM paraffins in the melamine-formaldehyde membrane with a solid/liquid phase change at low temperatures (20–30 °C), have been investigated [1].

DSC was used to determine the thermophysical properties of the PCMs, namely the enthalpy and specific heat capacity, the phase transition temperature interval (solid/liquid and vice versa), the latent heat of fusion and 60 heating/cooling cycles to determine the thermal stability of the material during repeated melting and curing. As PCM paraffins are flammable, TGA-MS and TGA-GC/MS were combined to verify the interval of thermal stability, analyse the released gases and investigate their potential toxicity in case of fire. The microstructure and phase transition of the PCM paraffins were observed by thermoptometry, namely by optical microscopy coupled to a hot plate and by DSC coupled to an optical microscope.

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RECOVERY OF PHOSPHORUS AND METALS FROM SEWAGE SLUDGE AND LAKE SEDIMENTS USING MAGNETIC SORBENTS

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Sewage sludge, lake, and sea sediments are substantial secondary phosphorus (P) sources that should be recycled in light of this element's increasing critical resource availability. Many common and rare metals are buried in lake and ocean sediments. This potential resource could be exploited if environmentally friendly techniques to harvest the metals can be developed. We investigated the possibility of using the electric arc furnace slag (EAF) Petrit TS blended with 10% magnetite and 10% cement as a binder. The aim of creating this sorbent was to 1) use a residual product, i.e. EAF slag, to minimize the ecological footprint, 2) enable recycling through the magnetic separation process, and 3) help close losses of P and harmful metals from the technosphere to soil and water ecosystems. Petrit TS is chemically composed of 37% CaO, 19% SiO2, 9% Al2O3, 7% FeO, and, 20% C. The material has proved to be an efficient sorbent in previous experiments, however not been amended with magnetite and cement. The set-up of a bench-scale experiment consisted of a container made of plexiglass divided into two compartments (30x30x30 cm each), filled to 15 cm height with P-rich lake sediment. The sediment surface in one compartment was covered with the magnetic Petrit sorbent (granule size 1.8−3.15 mm) corresponding to capping intensity of 1 kg m-1. The experimental system was kept at room temperature for 3 weeks until recovery and analyses of the magnetic sorbents. A short-term batch experiment on sludge collected from a basin following the effluent from a wastewater treatment plant was performed. The sludged was known to have elevated concentrations of P and certain metals. Nine 100 mL Erlenmeyer flasks were filled with 75 g of sludge. Sludge in triplicate flasks was mixed with 5 and 2 g magnetic Petrit sorbent, respectively. Three flasks were filled with only sludge. The flasks were kept at room temperature on a horizontal shaking machine for 5 days. The sorbent was freed by placing the sediment and sludge on a net with a mesh size of 0.9 mm which was washed with distilled water. Still, some organic matter was present, however, using a magnet the sorbents were released and taken for analysis. The sorbent was analyzed after digestion using ICP-MS. The wash water was dried at 50 oC until the recovered sludge was dry. Sludge and 5 cm top-sediment with and without contact with the sorbent were analyzed on the content of P and heavy metals. The results showed an average recovery rate of 89% magnetic sorbents. The removal of P from the lake sediment was highest at 77% while in the sludge system, an average removal of 37% and 51% for the 2 and 5 g sorbent amendments, respectively, was found. The metal removal varied and was more difficult to evaluate due to the virgin content of metals in the sorbent. However, higher concentrations of Zn, Cu, Mn, Co, and Cr were observed (13−27%). Experiments with longer duration and in larger set-ups, preferably under field conditions, should be performed.

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