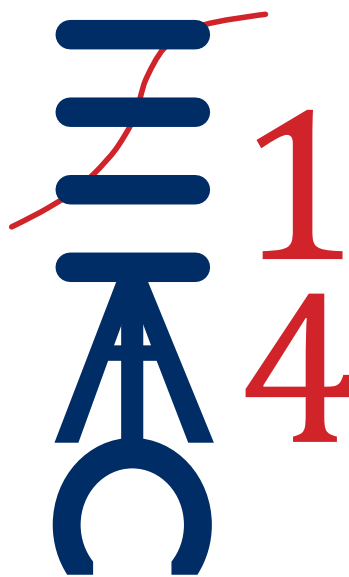




ESTAC 14

14th European Symposium on
Thermal Analysis and Calorimetry
SPLIT • CROATIA • SEPTEMBER 15–18, 2026



BOOK OF ABSTRACTS

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ESTAC14

BOOK OF ABSTRACTS

Editors:

Matko Erceg

Nataša Stipanelov Vrandečić

Branka Andričić

Andrei Rotaru



14th European Symposium on Thermal
Analysis and Calorimetry

September 15–18, 2026 • Split • Croatia

**14th European Symposium on Thermal Analysis
and Calorimetry (ESTAC14)**

**15-18 September 2026
Split, Croatia**

Book of abstracts of the 14th European Symposium on Thermal Analysis and Calorimetry (ESTAC14).

14th European Symposium on Thermal Analysis and Calorimetry (ESTAC14).
15-18 September 2026
Split
Croatia

Editors:

Matko Erceg, Nataša Stipanelov Vrandečić, Branka Andričić, Andrei Rotaru

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Organizers

The 14th European Symposium on Thermal Analysis and Calorimetry

ESTAC14

15-18 September 2026 – Split, Croatia

is organized by the:

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**Croatian Committee for Thermal Analysis and Calorimetry
(CCTCA)**

University of Split

**Central and Eastern European Committee for
Thermal Analysis and Calorimetry (CEEC-TAC)**



**14th European Symposium on Thermal
Analysis and Calorimetry**

September 15–18, 2026 • Split • Croatia

The 14th European Symposium on Thermal Analysis and Calorimetry

ESTAC 14

15-18 September 2026 – Split, Croatia

is organized by the:



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Foreword

Dear Participants of ESTAC14,

It is our great pleasure to welcome you to Split, Croatia, for the 14th European Symposium on Thermal Analysis and Calorimetry (ESTAC14), which will take place from 15 to 18 September 2026.

This edition of ESTAC is particularly special, as we are celebrating the **50th anniversary of the European Symposium on Thermal Analysis and Calorimetry**. Fifty years of ESTAC represent an important milestone in the development of our scientific community and provide an opportunity to celebrate the history of the symposium while looking towards the future of thermal analysis and calorimetry. Let us remember that the first ESTAC was held in 1976 in Salford, United Kingdom, followed by meetings in Aberdeen (UK, 1981), Interlaken (Switzerland, 1984), Jena (Germany, 1987), Nice (France, 1991), Grado (Italy, 1994), Balatonfüred (Hungary, 1998), Barcelona (Spain, 2002), Kraków (Poland, 2006), Rotterdam (Netherlands, 2010), Espoo (Finland, 2014), Braşov (Romania, 2018) and Palermo (Italy, 2022). We are honoured to host the 14th edition of ESTAC on its 50th anniversary, in the beautiful city of Split, Croatia. We hope that ESTAC 14 will continue this outstanding tradition and environment for scientific interaction. Participants will have the opportunity to engage in plenary and invited lectures, poster presentations, sponsors workshops, exhibitions by leading instrumentation companies and various social events.

The ESTAC14 conference gathers 208 registered participants from 32 countries, presenting a total of 192 contributions. The programme includes 6 Plenary Lectures, 12 Invited Lectures, 48 Oral Presentations, including the Rigaku–ICTAC Promising Researcher Award 2026 Lecture, 6 Technical Lectures and 120 Poster Presentations. The programme covers a broad range of topics in thermal analysis and calorimetry, including thermal characterization and behaviour of advanced materials, thermal kinetics and thermochemistry, polymers, sustainable and functional materials, as well as applications of thermal analysis in chemistry, materials science, energy, batteries, environmental science, pharmaceuticals and related fields. The scientific programme has been designed to provide a balance between Plenary and Invited Lectures, Oral and Poster Presentations, as well as opportunities for discussion, networking and informal exchange. In addition to the scientific sessions, the programme will include meetings of ESTAC and ICTAC boards and committees. We have also tried to offer opportunities to explore the city and enjoy the social programme as well.

At ESTAC14, we will also have the pleasure of recognizing outstanding contributions to the field through the ESTAC Awards. The award recipients will be recognized during the conference programme, adding another special dimension to this anniversary edition of the symposium. The recipients of the ESTAC14 Awards are: Prof. Dr. Peter ŠIMON (David Dollimore ESTAC Award), Prof. Dr. Nicolas SBIRRAZZUOLI (Eugen Segal ESTAC Award), Dr. Gabriela ZELENKOVÁ (Judit Simon ESTAC Award) and Prof. Dr. Ignazio BLANCO (Jose Criado Award for Innovation and Promotion of Thermal Analysis). The Rigaku–ICTAC Promising Researcher Award 2026 will be awarded to Dr. Juan Ivorra-Martinez.

We would like to express our sincere thanks to the people, institutions and associations who contributed and supported the organization of this event, especially to the members of the Honorary Committee, Scientific Committee, International Organizing Committee and National Organizing Committee, Central and Eastern European Committee for Thermal Analysis and Calorimetry, National Associations for Thermal Analysis and Calorimetry from European countries, Croatia Society of Chemical Engineers, University of Split, Faculty of Chemistry and Technology, Faculty of Science and Faculty of Maritime Sciences. We acknowledge the great support of our Sponsors: Superlab/LabExpert/PerkinElmer (Platinum Sponsors), Netzsch/Altium (Gold Sponsors), Waters Materials Science/Labtim Adria (Gold Sponsor), AstrineXLab/Hitachi (Silver Sponsors), Ru-Ve/IKA (Silver Sponsors), Rigaku (Silver Sponsor), Mettler-Toledo (Silver Sponsor), Anton Paar (Bronze

Sponsor), Linseis/Ansar Analitika (Bronze Sponsors) and KEP Technologies EMEA – SETARAM (Bronze Sponsor). A special acknowledgement has to be addressed to the *Journal of Thermal Analysis and Calorimetry* and *Thermal Advances*, where special issues will be dedicated to research papers of our conference.

ESTAC14 is hosted by the Faculty of Chemistry and Technology, University of Split, in cooperation with the Croatian Committee for Thermal Analysis and Calorimetry of the Croatian Society of Chemical Engineers and Technologists (HDKI) and the Central and Eastern European Committee for Thermal Analysis and Calorimetry (CEEC-TAC).

Split, situated on the Adriatic coast of Croatia, is a city where more than seventeen centuries of history meet contemporary Mediterranean life. At its heart lies the historic centre built around the Palace of the Roman Emperor Diocletian, a UNESCO World Heritage Site. Split is an inspiring blend of ancient heritage, Mediterranean culture and modern research infrastructure. We hope that you will take some time during your stay to discover Split, its historical heritage, Mediterranean atmosphere and beautiful surroundings.

We have tried to do our best so that ESTAC14 will provide you with an inspiring scientific experience, valuable discussions and new professional contacts, and that you will leave Split with pleasant memories of both the conference and the city.

We look forward to welcoming you to Split and to celebrating **50 years of ESTAC together with you in Split!**

With best regards,
Matko Erceg
Chair of ESTAC14

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General Information

The “14th European Symposium on Thermal Analysis and Calorimetry (ESTAC14)” has gathered **208 registered participants** from **32 countries**, presenting a total number of **192 contributions**. Of those, 4 are Award Plenary Lectures (**APL**), 2 are Plenary Lectures (**PL**), 12 are Invited Lectures (**IL**), 3 Parallel Sessions of Oral Presentations – 48 contributions (**OP**) including the Rigaku–ICTAC Promising Researcher Award 2026 Lecture, 6 Technical Lectures (**TL**) & 3 Sessions of Poster Presentation – 120 contributions (**PS**). Each oral session is comprised of 16 presentations, while each poster presentations includes 40 presentations.

List of Award Plenary, Plenary and Invited Lecturers

Award Plenary Lectures

- *Nicolas Sbirrazzuoli* (Université Côte d'Azur, France) - **Eugen Segal ESTAC Award**
- *Ignazio Blanco* (University of Catania, Italy) - **Jose Criado Award for Innovation and Promotion of Thermal Analysis**
- *Peter Šimon* (Slovak University of Technology, Slovakia) - **David Dollimore ESTAC Award**
- *Gabriela Zelenková* (University of Ostrava, Czech Republic) - **Judit Simon ESTAC Award**

Plenary Lectures

- *Wenbing Hu* (Nanjing University, China)
- *Kinga Pielichowska* (AGH University of Krakow, Poland)

Invited Lectures

- *Málek Jiří* (University of Pardubice, Czech Republic)
- *Katoh Katsumi* (Fukuoka University, Japan)
- *Petuhov Oleg* (Moldova State University, Moldova)
- *Papuga Saša* (University of Banja Luka, Bosnia and Herzegovina)
- *Duce Celia* (University of Pisa, Italy)
- *Tony Spassov* (University of Sofia "St. Kliment Ohridski", Bulgaria)
- *Farjas Jordi* (University of Girona, Spain)
- *Dudek Magdalena* (AGH University of Science and Technology, Poland)
- *Holjevac Grgurić Tamara* (Catholic University of Croatia, Croatia)
- *Čelan Koroščin Nataša* (University of Ljubljana, Slovenia)
- *López Beceiro Jorge J.* (University of A Coruña, Spain)
- *Brebu Mihai* (Petru Poni Institute of Macromolecular Chemistry, Romania)

The Rigaku–ICTAC Promising Researcher Award 2026

- *Ivorra-Martínez Juan* (Polytechnic University of Valencia, Spain)

Tuesday, 15 th of September		Wednesday, 16 th of September		Thursday, 17 th of September		Friday, 18 th of September							
9 ⁰⁰ -16 ³⁰	Sponsors Workshops on TA&C (program available HERE)	9 ⁰⁰ -9 ⁴⁵	Wenbing Hu, PL1	9 ⁰⁰ -9 ⁴⁵	Kinga Pielichowska, PL2	9 ⁰⁰ -9 ⁴⁵	Gabriela Zelenková, APL4						
		9 ⁴⁵ -10 ¹⁵	Platinum Sponsor, TL1 Superlab/PerkinElmer	9 ⁴⁵ -10 ⁰⁵	Gold Sponsor, TL3 Waters Materials Science	9 ⁴⁵ -9 ⁵⁵	Silver Sponsor, TL5 Rigaku						
		10 ¹⁵ -10 ⁴⁵	IL1	10 ¹⁵ -10 ⁴⁵		IL2	IL5	IL6	IL9	IL10			
		10 ⁴⁵ -12 ⁰⁰	Poster Session 1 Coffee break		10 ⁴⁵ -12 ⁰⁰	Poster Session 2 Coffee break		10 ⁴⁵ -12 ⁰⁰	Poster Session 3 Coffee break				
14 ⁰⁰ -16 ⁰⁰	ESTAC14 General Assembly	12 ⁰⁰ -12 ²⁰	OP1.01	OP2.01	OP3.01	12 ⁰⁰ -12 ²⁰	OP1.09	OP2.09	OP3.09	12 ⁰⁰ -12 ²⁰	OP1.13	OP2.13	OP3.13
		12 ²⁰ -12 ⁴⁰	OP1.02	OP2.02	OP3.02	12 ²⁰ -12 ⁴⁰	OP1.10	OP2.10	OP3.10	12 ²⁰ -12 ⁴⁰	OP1.14	OP2.14	OP3.14
		12 ⁴⁰ -13 ⁰⁰	OP1.03	OP2.03	OP3.03	12 ⁴⁰ -13 ⁰⁰	OP1.11	OP2.11	OP3.11	12 ⁴⁰ -13 ⁰⁰	OP1.15	OP2.15	OP3.15
		13 ⁰⁰ -13 ²⁰	OP1.04	OP2.04	OP3.04	13 ⁰⁰ -13 ²⁰	OP1.12	OP2.12	OP3.12	13 ⁰⁰ -13 ²⁰	OP1.16	OP2.16	OP3.16
		13 ²⁰ -14 ⁴⁵	Lunch			13 ²⁰ -14 ⁴⁵	Lunch			13 ²⁰ -14 ⁴⁵	Lunch		
		14 ⁴⁵ -15 ³⁰	Ignazio Blanco, APL2			14 ⁴⁵ -15 ³⁰	Peter Šimon, APL3			14 ⁴⁵ -15 ¹⁵	IL11	IL12	
		15 ³⁰ -15 ⁵⁰	Gold Sponsor, TL2 Netzsch/Altium International			15 ³⁰ -15 ⁴⁰	Silver Sponsor, TL4 AstrinexLab/Hitachi			15 ²⁰ -15 ⁴⁵	ESTAC15 & Forthcoming conferences		
		15 ⁵⁰ -16 ²⁰	IL3	IL4	15 ⁴⁵ -16 ¹⁵	IL7	IL8	15 ⁴⁵ -16 ⁰⁰	Closing Ceremony				
		16 ²⁰ -17 ⁰⁰	Coffee break			16 ¹⁵ -19 ⁰⁰	Afternoon break	16 ⁰⁰ -19 ⁰⁰	Afternoon break				
		17 ⁰⁰ -17 ²⁰	OP1.05	OP2.05	OP3.05								
17 ²⁰ -17 ⁴⁰	OP1.06	OP2.06	OP3.06										
17 ⁴⁰ -18 ⁰⁰	OP1.07	OP2.07	OP3.07										
18 ⁰⁰ -19 ⁴⁵	Nicolas Sbirrazzuoli, APL1	OP1.08	OP2.08	OP3.08	19 ⁰⁰ -23 ³⁰	Conference Dinner	19 ⁰⁰ -20 ⁰⁰	City tour					
20 ⁰⁰ -23 ³⁰	Welcome Cocktail												
1. Lecture hall A at Faculty of Chemistry and Technology (A-02, Level 0)													
2. Lecture hall B at Faculty of Chemistry and Technology (A-01, Level 0)													
3. Lecture hall C at Faculty of Chemistry and Technology (A-03, Level 0)													
4. Square in front of the Faculty of Chemistry and Technology													
5. Space next to the A-02, Level 0, at the Faculty of Chemistry and Technology													
6. Canteen at the Student Center, University of Split													
7. The Mediterranean Institute for Life Sciences, Meštrovićevo šetalište 45, 21000 Split https://maps.app.goo.gl/ZHWDPQhCS2R3wd1R9													
8. Starting place: https://maps.app.goo.gl/ahHd5u71rYcvgft8													

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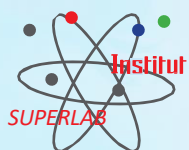
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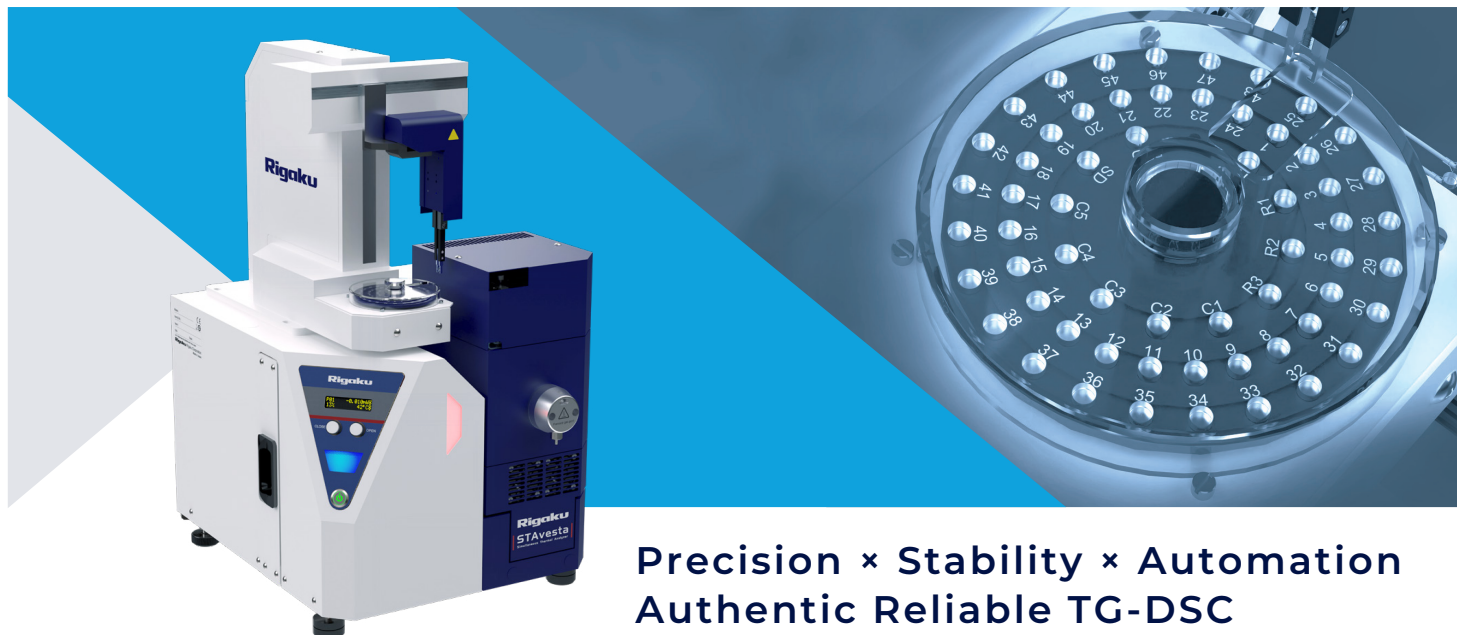
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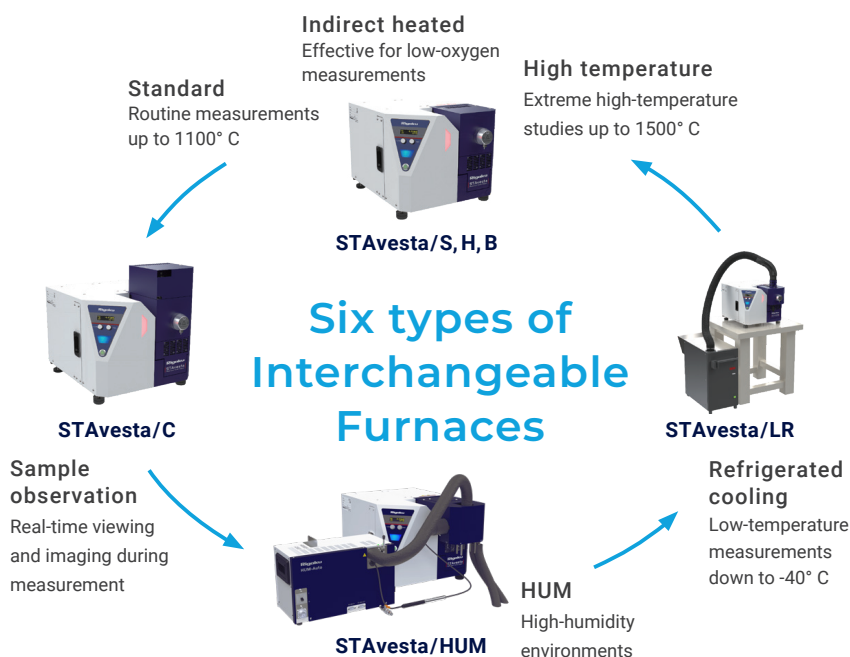
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Award Plenary Lectures

ADVANCED KINETIC ANALYSIS OF POLYMERS MECHANISTIC INSIGHTS, SIMULATION BREAKTHROUGHS, AND FUTURE DIRECTIONS

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Advanced isoconversional kinetic analysis (AIKA) has undergone major advances over the past decade, leading to significant methodological improvements and a substantial expansion of its application domain [1-18]. These “model-free” approaches allow determination of the effective activation energy (E_a) and pre-exponential factor (A_a) without assuming any specific reaction model. They are particularly valuable for systems governed by multi-step kinetics. For such complex processes, however, the most rigorous strategy consists in explicitly writing the rate equations for each elementary step and solving the resulting system of differential equations through nonlinear optimization. These model-based kinetic analysis (MBKA) or model-fitting methods are theoretically sound, but they raise practical difficulties: selecting an appropriate mechanistic scheme, choosing physically meaningful initial parameter values, and avoiding convergence toward mathematically acceptable yet physically unrealistic solutions [12]. A promising direction is the combined use of model-free and model-based approaches. Advanced isoconversional kinetic analysis focuses on the evolution of kinetic parameters with temperature or conversion providing insight into changes in the rate-limiting steps, an aspect that fundamentally distinguishes it from purely model-based methods. The combination of advanced isoconversional kinetic analysis with multi-step-multi-temperature-program model-fitting strategies, yield a far more comprehensive description of reaction kinetics and of the underlying transformation mechanisms. Several successful case studies will be presented, illustrating how physically meaningful kinetic information can be extracted from thermoanalytical data in diverse fields such as biobased polymers and nanocomposites, thermosets polymerization, crystallization, glass transition, and physical aging of polymers. It will be shown how kinetic parameters obtained by combining AIKA and MBKA can be interpreted in terms of reaction mechanisms or changes in the rate-limiting step for complex chemical reactions and physical transitions, and how this new approach can guide the extraction of reliable kinetic parameters. This combined approach thus offers a powerful framework for elucidating multi-step kinetics and modeling complex processes [14].

Keywords: Advanced kinetic analysis, polymerization, crystallization, glass transition, biobased polymers.

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BEHIND THE DATA: THOROUGHNESS, ACCURACY, AND INNOVATION IN THERMAL AND CALORIMETRIC MEASUREMENTS

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During thermal and calorimetric measurements, part of the research focuses on standardizing measurement procedures to best exploit the potential of the used techniques (DSC, TGA, DMA, LFA) by minimizing errors and maximizing measurement accuracy [1]. Specifically, beyond the use of these techniques for the characterization of the most diverse materials, three aspects of my research activity can be identified as being aimed at achieving the above-mentioned purposes: calorimetric measurements, lifetime prediction, and thermal methods in additive manufacturing (AM).

Using a differential scanning calorimeter to measure heat capacity and thermodynamic parameters requires special attention, both in instrument and measurement calibration and in the measurement procedure itself, from the calibration to the choice of the mass and shape of the sample, suggesting the most suitable instrumental and operating conditions to correctly measure the heat capacity (C_p), Energy (U), Gibbs Energy (G), Free Energy (F), Enthalpy (H) and Entropy (S) [2,3].

Thermogravimetric analysis (TGA) is widely used to test the degradation stability of materials, with a strong focus on polymers and their composites, to make reliable lifetime predictions [4]. The TG parameters, obtained at high temperatures, are thus processed with kinetic equations, but no matter how accurate and precise the kinetic method adopted is, there remains a significant gap between the prediction and the real degradation of the material [5]. Starting from this consideration, the reliability of the lifetime predictions is faced with comparing measurements obtained at low temperature with those carried out at high temperatures, in the molten state [6].

Anyway, over the past 20 years, additive manufacturing has completely revolutionized the design and production of materials, and, from the simple thermal characterization of the early days [7] to the calculation of thermal conductivity by combining different TA techniques [8], thermal analysis is a powerful tool in the AM service. Specifically, this activity highlights the potential of using thermal conductivity tests as a faster and more resource-efficient proxy for mechanical testing in initial assessments. Such tests are particularly valuable in applications where thermal conductivity is a critical requirement (e.g., battery thermal management systems, heat exchangers, cooling systems in electronics).

Keywords: Differential Scanning Calorimetry, Lifetime prediction, Additive Manufacturing, Thermal Conductivity

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GENERAL RATE EQUATION – APPLICABILITY AND LIMITATIONS

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Thermoanalytical techniques are widely applied to study the kinetics of processes occurring in condensed state. These processes are very often complex; their mechanisms are frequently unknown or too complicated to be characterized by a simple kinetic model since they tend to occur in multiple elementary steps with different rates.

For the description of the kinetics of such complex processes, methods based on the general rate equation (GRE) are the most widely applied in thermoanalytical kinetics. Single-curve, Kissinger, isoconversional, model-fitting and non-parametric kinetics methods belong to the methods based on GRE [1].

Application of GRE assumes that kinetics of the process under study can be described as a product of the temperature and conversion functions. This holds perfectly for the elementary processes. Validity of the assumption is more an exception than a rule in the case of complex processes. For the complex processes comprising two or more elementary steps, GRE becomes just a formal mathematical tool approximating the kinetic hypersurface. The kinetic parameters hence do not possess any clear physical meaning; however, modelling of the process is feasible without knowing the detailed mechanism [2]. The kinetic parameters obtained from kinetic analysis do not describe the mechanism of the process; they describe the rate of heat evolution/consumption for DSC, or the rate of mass loss for TG. The kinetic parameters obtained from DSC and TG measurements thus may be different [3].

The Arrhenius equation is applied as the temperature function practically exclusively [2]. Since GRE is just a formal mathematical tool in the case of complex mechanisms, also other temperature functions can be employed. The Berthelot-Hood equation describes the experimental data equivalently well as the Arrhenius equation; moreover, extrapolation based on the Berthelot-Hood equation provides reasonable assessment of the lifetime of materials. Extrapolations based on the Arrhenius equation mostly overestimate the lifetime estimations [4].

Isoconversional principle is used for the determination of activation energy. Improper application of the principle leads to the kinetic compensation effect [2]. The isoconversional methods as mostly applied are over-parameterized so that the kinetic parameters obtained carry great errors and they are mutually correlated. The unified isoconversional method removes this drawback [5].

Keywords: general rate equation, temperature function, isoconversional methods.

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THERMOPOROMETRY: POROSITY CHARACTERIZATION OF CARBON MATERIALS FROM THE WET STATE

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Reliable characterization of porosity is essential for understanding and optimizing carbon materials used in adsorption, separation, catalysis, and environmental remediation. Conventional techniques, particularly gas physisorption and mercury intrusion porosimetry, generally require completely dried samples and may therefore provide limited information on materials operating under liquid-phase conditions. Thermoporometry (TPM), based on differential scanning calorimetry, offers a complementary approach by relating the freezing or melting point depression of a probe confined within pores to pore size through the Gibbs–Thomson relationship. This enables porous materials to be characterized directly in the wet state.

This contribution presents the systematic development of TPM for carbon materials, focusing on the experimental and computational factors that control the reliability of the resulting pore size distributions. Water as the most widely used probe because of its availability, safety, and high melting enthalpy. Nevertheless, its application requires careful consideration of the non-freezable interfacial water layer (called as delta layer, δ). Studies of carbons with systematically varied pore structures and surface chemistries demonstrated that δ is not a universal constant. In oxidized carbons, δ increased from 0.46 to 2.72 nm as the oxygen content increased from approximately 15 to 25 wt%.

Because experimental determination of δ is time-consuming, two complementary strategies were explored: empirical calibration of the DSC response and prediction of δ from readily available material properties. The calibration-based approach was compared with a physically based method incorporating experimentally determined or predicted δ values. Both yielded reasonable pore size distributions and consistent porosity trends. Calibration was advantageous for rapid routine analysis and generally produced sharper distributions, whereas the δ based method provided a more physically meaningful interpretation when interfacial effects were significant. A broader study of carbons with varied pore structures and oxygen contents further showed that textural properties, particularly BET area, influence δ more strongly than oxygen content alone. A simple logarithmic relationship between δ and BET area was therefore proposed, enabling rapid δ estimation while preserving reliable pore size distributions and mesopore volumes.

A second major challenge is the limited pore-size range accessible with water, especially when the melting signals of pore-confined and bulk water overlap. Mathematical peak separation was introduced to isolate the pore contribution from complex DSC curves, while alternative probes extended the measurable range. Octamethylcyclotetrasiloxane enabled characterization across a broader mesoporous range after optimization of the temperature programme, inclusion of a pre-freezing step, and deconvolution of overlapping transitions. Menthol was subsequently introduced as a solid probe for large mesopores and macropores. After optimization of sample drying, pore-filling time, and heating rate, menthol-based TPM detected pores from approximately 20 to 1500 nm and agreed well with nitrogen physisorption, mercury intrusion porosimetry, and electron microscopy.

These results show that reliable TPM requires appropriate probe selection, controlled sample preparation, suitable thermal programming, and careful signal processing. Rather than replacing gas physisorption, TPM provides complementary insight into the functional porosity of carbon materials under conditions closer to practical use.

Keywords: thermoporometry, porous carbon, non-freezable layer; porosity; macropores

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Plenary Lectures

CHIP-CALORIMETRY CHARACTERIZATION OF MICROMETER THERMAL CONDUCTIVITY

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With the rapid development of highly integrated micro-electronics, their thermal management challenges the proper characterization of thermal conductivity in micro-scale materials. We recently developed chip-calorimetry measurements (Flash DSC, Mettler-Toledo AG) to characterize the cross-plane and in-plane thermal conductivity of micro-meter-thin polymer films, as well as their surface and interface thermal contact resistances. The results could reproduce the reasonable observations and demonstrated the chip-calorimetry approach as promising for the proper characterization of thermal conduction performances in micro-scale materials.

Keywords: Thermal conductivity; Thermal contact resistance; Flash DSC; Polymer

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HYBRID PHOTOTHERMAL PHASE CHANGE MATERIALS FOR NEXT-GENERATION THERMAL ENERGY STORAGE TECHNOLOGIES

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The development of multifunctional phase change materials (PCMs) with enhanced heat management capabilities has accelerated due to the increasing demand for efficient and sustainable thermal energy storage (TES) systems. Consequently, PCMs have attracted growing attention not only for thermal energy storage applications but also for use in composites with photothermal conversion capabilities. These hybrid systems combine latent heat storage functionality with photothermal conversion through the incorporation of nanostructured light-absorbing additives, enabling simultaneous solar energy harvesting and thermal storage within a single material platform.

Literature data indicate that the highest solar radiation conversion efficiencies in PCMs can be achieved through the incorporation of transition metal nitrides, including MXenes, as well as carbon nanomaterials. At the same time, carbon nanomaterials can be used for PCM shape stabilization, preventing leakage above the PCM melting point, while also improving the thermal and electrical conductivity of the resulting materials. Additionally, in recent years, carbon aerogels have emerged as one of the most promising systems for PCM shape stabilization.

In this work, a sustainable method for converting waste citrus biomass into ultralight porous carbon aerogel composites using polysaccharides such as starch, chitosan, gellan gum, and sodium alginate as binders was developed. The obtained results confirmed the suitability of citrus biomass-derived aerogels as effective supporting materials for PCM shape stabilization. Subsequently, organic PCMs modified with titanium-based MXenes were incorporated into the carbon aerogels. The influence of the hybrid architecture and MXene additives on PCM-based systems was investigated using thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and other techniques. The experimental results revealed that the developed hybrid PCMs exhibit enhanced photothermal conversion efficiency and good long-term thermal reliability compared with conventional PCMs.

These findings highlight the potential of hybrid photothermal PCMs as promising candidates for advanced TES technologies in renewable energy systems, smart buildings, electronics thermal management, and industrial heat recovery applications.

Keywords: phase change materials, aerogels, MXenes, photothermal conversion

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Invited Lectures

UNVEILING GLASS TRANSITION KINETICS VIA ADVANCED THERMAL ANALYSIS

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Studying glass transition kinetics is crucial, as this universal phenomenon governs not only inorganic glasses and polymers but also pharmaceutical formulations and complex foods. Understanding how amorphous materials transition from a brittle glass to a rubbery state over time and temperature allows scientists to predict long-term stability and optimize manufacturing processes. In the food industry, managing these kinetics prevents ice crystal growth in ice cream and sugar crystallization in chocolate, preserving product texture. In pharmaceuticals, it prevents the crystallization of amorphous drugs, thereby enhancing their solubility and bioavailability. Ultimately, mastering this behavior optimizes the mechanical strength of synthetic polymers and addresses fundamental questions regarding the physics of the liquid-to-glass crossover.

This communication utilizes thermal analysis methods to investigate these structural relaxation processes. Particular attention is paid to estimating kinetic parameters that describe the inherent non-exponentiality and non-linearity of the glass transition. The analysis builds upon two recently published frameworks: the first distinguishes between non-exponentiality and non-linearity during isothermal structural relaxation [1], while the second analyzes the glass transition width and its complex dependence on material fragility, non-exponentiality, and non-linearity [2]. By applying these theoretical approaches to experimental thermal data, we demonstrate advanced methodologies for precisely characterizing the structural relaxation behavior of diverse glass-forming systems.

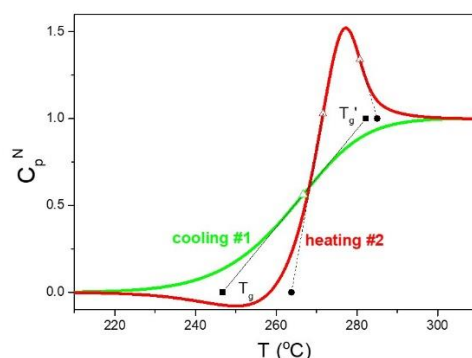


Figure: Normalized heat capacity in the glass transition region at the same cooling and heating rate

Keywords: Glass transition kinetics, Thermal analysis, Nonexponentiality, Nonlinearity, Structural relaxation

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THERMAL DECOMPOSITION BEHAVIOR OF NITROCELLULOSE MEMBRANES FOR LATERAL FLOW IMMUNOASSAY

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Lateral flow immunoassay (LFIA) is a rapid diagnostic method widely used for the detection of infectious diseases such as influenza and COVID-19 [1]. In LFIA, a liquid sample migrates along a nitrocellulose (NC) membrane by capillary action. If the target antigen is present, it is captured by antibodies immobilized on the membrane, producing a colored test line that is visually inspected to determine the test result. NC, which is also a major component of explosives, is susceptible to spontaneous decomposition even at room temperature, generating nitrogen oxides (NO_x) and nitric acid [2]. These decomposition products may react with antigens or antibodies, potentially affecting the analytical performance of LFIA. Therefore, the purpose of this study was to investigate the degradation behavior of NC membranes and its influence on LFIA performance. Two commercial LFIA membranes from different manufacturers and modified membranes containing different additives were examined by thermal analysis and isothermal aging tests under dry-air and humid-air conditions. Thermal analysis using a Calvet-type reaction calorimeter at a heating rate of 0.05 K min⁻¹ revealed that the onset temperatures of all NC membranes were lower under humid conditions than under dry conditions. Consistent with the thermal analysis results, the isothermal aging tests showed that humid conditions promoted membrane degradation, as evidenced by increased NO_x release and residual acid content together with decreased surface pH during storage. More severe degradation was observed for membranes containing glycerin. LFIA tests using the aged membranes under humid conditions revealed reduced color intensity of the test line, suggesting an increased risk of false-negative results. This tendency was particularly evident for one of the commercial membranes. After prolonged aging, however, the color intensity occasionally increased, possibly due to nonspecific coloration. These findings demonstrate that moisture and membrane additives strongly influence the degradation behavior of NC membranes, and that the resulting degradation can adversely affect the reliability of LFIA.

Keywords: Immunochromatography, Nitrocellulose, Stability, Thermal degradation

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MICROWAVE HEATING OF CARBON MATERIALS: FUNDAMENTALS AND APPLICATIONS

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Microwave heating is routinely described as volumetric. Carbon particles, however, reveal a different picture: in the beginning, the hottest events may not occur inside the particles, but rather at the interface, *i.e.* at the contact between particles. The heating mechanism was examined using a carbonization series prepared from walnut shells (350-800 °C), combining microwave heating curves, electrical-resistivity measurements and direct observation of particle contacts. A sharp transition in microwave response accompanies a four-order-of-magnitude decrease in resistivity between 550 and 650 °C. Low-temperature chars remain almost transparent to 2.45 GHz irradiation, whereas more conductive samples spark and reach incandescence within 30 s. For intermediate chars, heating becomes autocatalytic: an initially slow temperature rise lowers the semiconductor-like resistivity, after which microwave coupling accelerates abruptly. The decisive experiment separates otherwise identical particles into contacting and non-contacting arrangements. Incandescence and sparks appear selectively at the contacts; isolated particles show no comparable response. These observations support an interfacial mechanism in which the oscillating field drives charge accumulation on conductive carbon domains separated by resistive surface layers or gas gaps. Local electrical breakdown then converts stored electromagnetic energy into intense contact heating. The resulting resistor-capacitor network explains why electrical resistivity, particle size, packing and surface chemistry jointly control ignition, hot-spot propagation and apparent heating uniformity. This contact-first picture reconciles rapid heating with pronounced heterogeneity and provides practical design rules for microwave synthesis and regeneration of activated carbons: engineer the resistivity window and the contact network, rather than treating the carbon bed as a homogeneous dielectric.

Keywords: microwave heating, activated carbon, interfacial polarization, electrical breakdown, electrical resistivity

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RESPONSE SURFACE METHODOLOGY IN THE RESEARCH OF WASTE PLASTIC PYROLYSIS

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Pyrolysis can be defined as the thermally driven decomposition of organic materials, including biomass and plastics, into a range of useful products, in an oxygen-free or oxygen-limited environment such that no significant gasification occurs. The pyrolysis processes of waste plastics have become a subject of significant interest recently, as they represent the basis for the production of second-generation biofuels, materials and chemicals. Process parameters such as temperature, heating rate, residence time, along with the composition of the feedstock, the presence or absence of catalysts, reactor configuration and other variables, determine the yield and quality of the product. Therefore, investigating every possible combination of influencing parameters can be a rather complex and time-consuming task, especially if the process is carried out in a pilot scale or fixed-bed reactor, which requires hours just to set up and clean the reaction system, including the condensation system. Faced with similar problems in other fields of research, scientists resort to a specific choice of certain combinations of influential variables, so that by applying appropriate statistical tools to the obtained results, a reliable mathematical description of the event space can be developed, that is, a description of all combinations of influential variables. This is usually called Design of experiment (DOE) and Response Surface Methodology (RSM). Recently, researchers have started to apply such methodologies in the research of the pyrolysis process. By examining the influence of combined temperature/input mass variables in one example, and combined temperature/composition of plastic mixture variables, in another example, on the yield and quality of pyrolysis products, such an application of RSM in process optimization was demonstrated as part of our own research.

Keywords: pyrolysis, waste plastic, Response Surface Methodology, RSM, fixed-bed reactor

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EVOLUTION OF ANIMAL GLUES TOWARD NEXT-GENERATION COLLAGEN-BASED BIOADHESIVES: A SYSTEMATIC STUDY OF THE GLUING PROCESS

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Gelatin-based adhesives, also known as animal glues, are reversible materials obtained by partial hydrolysis of collagen from animal-derived sources. Although their use declined with the introduction of synthetic adhesives, they are currently attracting renewed attention, especially in conservation science and sustainable materials development [1][2].

In this work, we performed a systematic study of traditional animal glues obtained from a wide range of animal sources, with the aim of understanding the gluing mechanism and identifying key parameters that can be tuned for the design of next-generation collagen-based bioadhesives. Both European commercial glues (from bone and hide) and traditional Japanese glues used in Urushi lacquer were investigated. We evaluated the role of several physicochemical parameters governing adhesive performance. Measurements were carried out both in solution and in the solid state. In solution, rheology, contact angle, surface tension and adhesion tests were performed to assess viscosity, wettability and bonding ability. In the solid state, differential scanning calorimetry (DSC), transmission and scanning electron microscopy (TEM, SEM), atomic force microscopy (AFM), and Fourier-transform infrared spectroscopy (FTIR) were used to investigate thermal behavior, morphology and structural features. While our results highlight the importance of physicochemical parameters in determining adhesive performance, the role of the molecular and supramolecular structure of these materials remains an open question. In particular, whether a specific organization (e.g. helix-like structures or other arrangements) governs the macroscopic properties of the glues is still unresolved and represents a key point for future investigations.



Figure 1: From traditional animal glues to next-generation bioadhesives: a systematic study of sources, properties and tunable parameters.

Keywords: hydrolysed collagen, DSC, TGA-FTIR, ATR-FTIR, XRD

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SOME APPLICATIONS OF THERMAL METHODS IN PHASE DIAGRAM CONSTRUCTION AND THERMODYNAMIC CHARACTERIZATION OF BINARY ORGANIC SYSTEMS

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Thermal methods, particularly differential scanning calorimetry and related calorimetric techniques, are powerful tools for constructing phase diagrams and evaluating the thermodynamic properties of binary organic systems. In this work, representative binary mixtures are considered to illustrate how thermal analysis can be used to identify eutectic compositions, co-crystals, determine melting temperatures, and estimate associated thermodynamic parameters. In the naproxen-citric acid system, thermal and structural characterization showed that mechanochemical treatment did not lead to complete eutectic or cocrystal formation, but instead produced a microcrystalline two-phase mixture with improved interparticle contact, which significantly enhanced dissolution behavior.

Thermal analysis is also a versatile approach for constructing phase diagrams and assessing the thermodynamic properties of binary organic systems relevant to separation and extraction processes. Binary mixture of decanoic acid (DA) and 1-dodecanol (DD) was investigated as the liquid phase in polymer inclusion membranes (PIMs) for the extraction of Dy³⁺ and Y³⁺. Temperature-modulated differential scanning calorimetry (TMDSC) was used to determine the phase behavior of the DA-DD system, revealing a eutectic composition at approximately 70 wt% DD and 30 wt% DA, as well as a peritectic point near 40 wt% DD and 60 wt% DA. The resulting phase diagram identified composition ranges that remain liquid above 20 °C, supporting their use as membrane liquid phases. In the second system, the binary mixture of tri-*n*-octylphosphine oxide (TOPO) and 4,4,4-trifluoro-1-phenyl-1,3-butanedione (HBTA) was studied by differential scanning calorimetry to establish its phase diagram, melting behavior, and eutectic composition. The eutectic point was determined at a HBTA:TOPO mass ratio of 60:40, and a Tammann diagram was constructed to validate the thermal analysis results and quantify the melting enthalpy contributions across composition range. Both systems demonstrate how thermal methods provide reliable experimental access to phase equilibria and thermodynamic properties, enabling the rational design of low-melting binary mixtures for applications in rare earth separation and lithium extraction.

Keywords: DSC, DTA, TG, phase diagrams, binary organic systems

Acknowledgements:

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KINETIC ANALYSIS APPLIED TO THE PROCESSING OF POLYMER MATRIX COMPOSITES FOR STRUCTURAL APPLICATIONS

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In the manufacturing of polymer-based structural materials, every processing step such as curing, vitrification, degradation, stress relaxation, or crystallisation is governed by underlying transformation kinetics. Understanding and modelling these kinetics is therefore essential for defining and optimizing processing conditions, anticipating long-term behaviour, and constructing reliable process design tools such as processability maps.

In this talk, we will show that isoconversional methods are suitable for analysing the kinetics of the processes that are relevant for polymer processing because they are versatile, robust, and stringboard to implement. While well established for curing and thermal degradation, we show that isoconversional analysis extends well beyond these classical cases. Specifically, we demonstrate its successful application to: (i) the crystallisation kinetics of thermoplastics; (ii) the stress relaxation of vitrimers; (iii) curing kinetics under diffusion control, three scenarios not traditionally addressed by model-free approaches and self-healing kinetics in vitrimers [1–3].

In each case, the kinetic descriptors derived from isoconversional analysis feed directly into TTT diagrams and processability charts, enabling quantitative prediction of processing windows. Experimental validation confirms the reliability and breadth of the approach, establishing isoconversional methods as a unified framework for kinetic modelling across a wide range of polymer systems and transformation phenomena.

Keywords: isoconversional analysis; curing kinetics; crystallization kinetics; TTT diagrams; stress relaxation

Acknowledgements:

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SYNERGISTIC MODIFICATION ENGINEERING FOR OPTIMISING THE PERFORMANCE OF SOLID OXIDE FUEL CELLS SUPPLIED BY WASTE ORGANIC MATERIALS

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Hydrogen is currently the primary fuel used to power fuel cells. Owing to its rapid oxidation kinetics and very low polarisation losses, hydrogen–oxygen fuel cells achieve the highest current density and electrical power density. However, the logistics of hydrogen supply, storage and utilisation still require improvement from both technical and economic perspectives. Recently, increasing attention has been paid to the use of chemical hydrogen carriers and waste organic materials as fuels for solid oxide fuel cells (SOFCs). These cells can be powered by gaseous, liquid or solid fuels from various sources. An interesting option is the use of organic waste materials from the agri-food processing industry to fuel SOFCs. In SOFCs fuelled by carbon-bearing materials, these materials undergo direct conversion into electricity and waste heat.

Thermal analysis methods play a crucial role in the selection of waste materials, enabling the assessment of their thermal stability and chemical reactivity. One of the main factors determining the efficiency of the carbon gasification process in the Boudouard reaction is the physicochemical and thermal properties of waste -based carbon materials. Carbon gasification with carbon dioxide to produce carbon monoxide (i.e. the reverse Boudouard reaction) is one of the most important routes for the electrochemical performance of direct carbon SOFCs (DC-SOFCs). This simple reaction, which proceeds according to the following scheme, $C + CO_2 = 2CO$, is endothermic and requires a significant amount of heat. To improve the efficiency of carbon gasification, resulting in an increased CO concentration in the reaction products, the Boudouard process must be optimised. A crucial role is played by catalyst materials, which are natural components of organic waste matter.

This paper presents research findings on the selection of synergistic carbon-based fuels derived from organic materials and on the correlation between the physicochemical properties of the electrode materials and data obtained via differential thermal analysis (DTA), differential scanning calorimetry (DSC), thermogravimetry (TG) and thermogravimetry in atmospheres of varying composition (CO_2 , Ar- CO_2 and Ar- H_2). Results regarding chemical reactivity, determined through thermal analysis, are correlated with electrochemical reactivity. Optimised electrode materials, specifically perovskite oxides and cermets, are also identified. These enable high current densities and electrical power outputs in the range of ~ 100 to ~ 350 mW/cm² at temperatures of 600°C–850°C.

A major hurdle in the successful commercialisation of SOFC systems is the degradation of the solid oxide cell and stack components during prolonged exposure to fuel contaminants at high operating temperatures. An SOFCs system is expected to operate for several thousand hours without significant degradation in output power. The major factors influencing the degradation of SOFCs are elevated temperature, thermal cycling, redox cycling, load cycling and poisoning effects from fuel contaminants such as sulphur and carbon compounds. The stability of such a mobile power source involving a small SOFC stack supplied by biochars will be presented and discussed.

Keywords : solid oxide fuel cells, organic waste, biocarbon, electrochemistry, power sources

COPPER-MODIFIED TI-6AL-4V ALLOY FOR BIOMEDICAL APPLICATION

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Copper alloying of Ti-6Al-4V is gaining attention for orthopedic applications, as it may offer both mechanical strengthening and inherent antibacterial behavior. This work examines the microstructural, thermal, and mechanical characteristics of Ti-6Al-4V-Cu alloys to assess their potential as implant materials.

Specimens with varying copper content were produced by vacuum induction centrifugal casting. The microstructural analysis was carried out using optical microscopy (OM) and scanning electron microscopy (SEM-EDS). Phase precipitation was confirmed by X-ray diffraction (XRD) analysis. Phase transformations were determined by Differential scanning calorimetry (DSC), while Vickers microhardness testing was used to evaluate mechanical response. Both SEM-EDS and XRD confirmed the presence of the Ti₂Cu intermetallic phase alongside the α/β titanium matrix. This finding is significant because Ti₂Cu has been reported in the literature to exhibit both antibacterial activity and precipitation-hardening effects. A marked increase in microhardness was observed compared to standard Ti-6Al-4V, pointing to an effective strengthening contribution from Cu addition and Ti₂Cu precipitation — a property expected to translate into improved wear resistance under long-term implant service.

Taken together, these findings position Ti-6Al-4V-Cu as a promising material for biomedical applications.

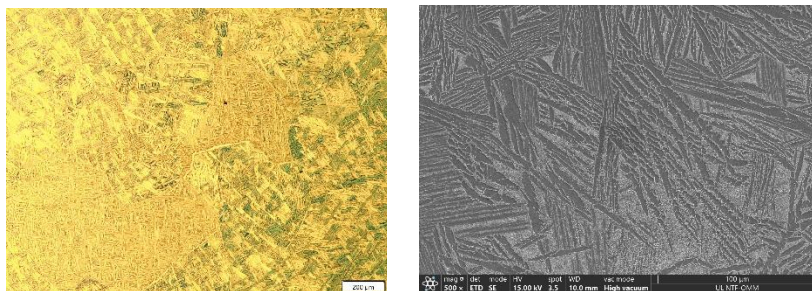


Figure 1. OM and SEM micrographs of Ti-6Al-4V-xCu alloy

Keywords: Ti-based alloys, microstructure, orthopedic application, intermetallic phases

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THERMAL DECOMPOSITION OF A COPPER(II) OXALATE COORDINATION COMPOUND: INTEGRATING TGA-MS WITH COMPLEMENTARY SOLID-STATE TECHNIQUES

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The thermal behaviour of coordination compounds is governed by a sequence of coupled processes involving dehydration, decomposition of the coordination sphere, formation of intermediate solid phases and release of gaseous products. In this work, the thermal decomposition of potassium bis(oxalato)cuprate(II) dihydrate, $K_2[Cu(C_2O_4)_2] \cdot 2H_2O$, is examined as a model system using thermogravimetric analysis coupled with mass spectrometry (TGA-MS) and complementary solid-state techniques, including X-ray powder diffraction (XRPD) and Fourier-transform infrared spectroscopy (FT-IR). Thermogravimetric measurements were performed under inert and oxidising conditions, enabling comparison of the decomposition behaviour and gaseous products evolved in different atmospheres. In argon, the thermal treatment revealed a sequence of distinct mass-loss events and the formation of intermediate solid phases at selected temperatures. These intermediates were examined further by XRPD and FT-IR to correlate changes in solid-state composition with the observed thermal effects and to follow the progression of the decomposition process. The results indicate complete dehydration in the initial stage, followed by progressive decomposition of the oxalate-containing coordination sphere and the formation of potassium- and copper-containing inorganic phases, including oxalate, carbonate and oxide species. TGA-MS confirmed the evolution of H_2O , CO and CO_2 during thermal treatment, while the final composition of the residue depends on the applied thermal conditions and the behaviour of volatile potassium-containing species at elevated temperature. Attention is also given to experimental and interpretative aspects relevant to coupled thermal analysis. The results demonstrate that the measurement environment may influence the composition of evolved gases and that thermally treated residues may undergo post-measurement alteration. In particular, rehydration of residues after thermal analysis and the detection of unexpected hydrogen signals indicate that the conditions within the TGA cell may contribute to secondary processes that need to be considered when interpreting TGA-MS data. These observations highlight the importance of distinguishing between processes intrinsic to the investigated material and those arising from the experimental environment.

Keywords: thermal analysis, TGA-MS, coordination compounds, thermal decomposition, complementary techniques

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BRIDGING THE LOGISTIC AND CLASSICAL KINETICS: AN ARRHENIUS-ANCHORED SHAPE APPROACH FOR SOLID-STATE REACTIONS

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Identifying the reaction model $f(\alpha)$ remains the bottleneck of kinetic analysis. We construct a 3D morphological classifier by fitting simulated curves of all classical solid-state models (F, A, R, D families [1]) with a generalized derivative logistic function (B-dual) whose shape parameters capture the full peak profile. Three descriptors—peak asymmetry w , thermal-width deviation $\ln(m)$ ($m = b/b_{\text{Kiss}}$, $b_{\text{Kiss}} = E_a\beta/RT_p^2$), and conversion at peak α_p —define a feature space where each model occupies a characteristic region. For an unknown sample, E_a is obtained from the slope of $\ln(b)$ vs. $1/T_p$ across heating rates (a Kissinger-type relationship applied to the fitted width parameter [2]), the model is identified via Mahalanobis distance to the reference database, and $\ln(A)$ is calculated analytically from the exact peak-rate relationship [3]. For overlapping processes, a hybrid Arrhenius-anchored deconvolution separates components, each independently classified by the reference morphological map. Validation on 20 synthetic curves with known triplets across 9 Arrhenius-based models (A2–A4, D2, D4, F1–F3, R2) recovered E_a within 0.84% error (mean 0.38%) and $\ln(A)$ within 5.5% (mean 2.1%), with $R^2 > 0.9998$ in all cases [4]. Preliminary analysis suggests that certain logistic descriptors could act as mechanism-specific invariants even for complex and/or non-Arrhenius kinetics.

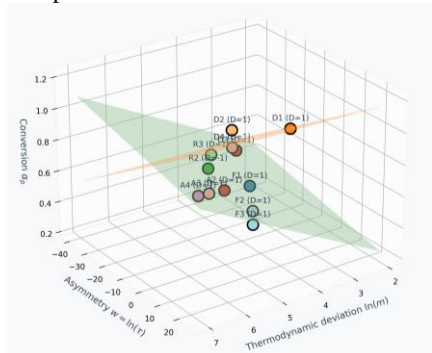


Figure 1: 3D morphological classification space mapping theoretical ICTAC models by peak asymmetry (w), thermodynamic deviation ($\ln m$), and peak conversion (α_p). Translucent planes segregate kinetic families: orange bounds diffusion (D-series) and green groups contraction/nucleation (R- and F-series).

Keywords: morphological oracle, kinetic model identification, B-dual function, isoconversional analysis, kinetic deconvolution

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PY-MS AND PY-GC-MS FOR ANALYSIS OF VOLATILE BIOACTIVE NATURAL COMPOUNDS IN VEGETAL MATERIALS

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Bioactive compounds are the nature defence system against decay or biological attack. Nowadays the bioactive natural compounds are largely used in food preservation, nutraceuticals or medicine, to replace the synthetic counterparts which proved to have side effects or loose efficacy due to microbial adaptability. Qualitative and quantitative evaluation of compounds with potential antioxidant/antimicrobial values present in plant species is, therefore, of high scientific interest. Analytical pyrolysis coupled with mass spectrometry (Py-MS) or with gas chromatography-mass spectrometry (Py-GC-MS) is a valuable tool to determine the composition of volatile compounds in plant materials, using limited amount of samples (~ 2 mg). This facilitates the differentiation of volatile profiles across diverse sources.

Py-GC-MS was used to evaluate the composition of terpenes/terpenoides from fruits and leaves of *Baccaurea*, a wild fruit tree naturally growing in Borneo rainforest, a popular healing plant. This is a valuable screening tool to determine the spectrum of volatile compounds of plant materials before extracting them as essential oils. The thermal profile showing the sequential dehydration, release of volatiles and torrefaction / pyrolysis as well as temperature dependent formation of terpenes, alkyl aromatic, alkyl phenols and of guaiacol/syringol derivatives was determined by Py-MS – Figure 1.a. Then, sequential Py-GC-MS at different temperatures strengthened the Py-MS observation.

Besides, Py-MS technique was successfully used to distinguish the evolution of α -agarofuran and of γ -eudesmol from agarwood, this becoming a valuable tool for quality assessment – Figure 1.b.

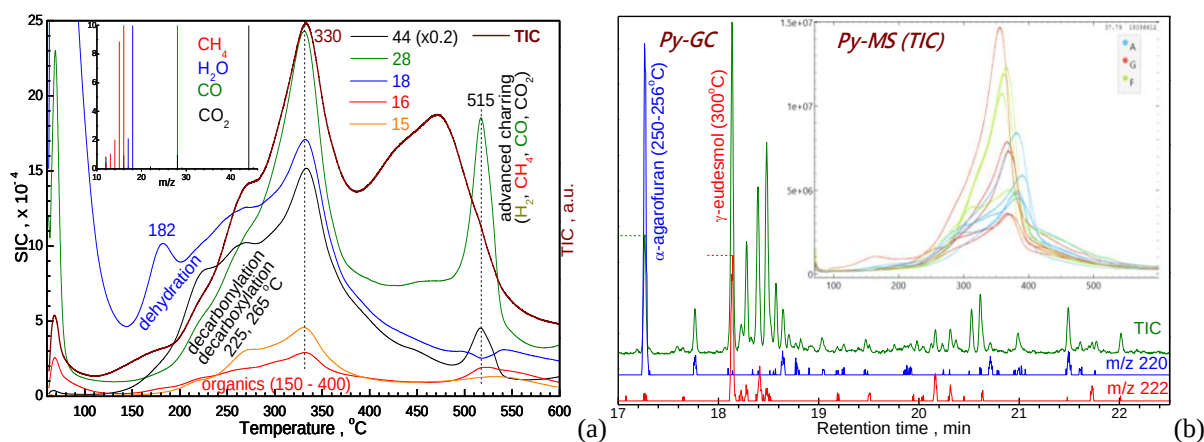


Figure 1. Step-by-step formation of volatiles from the fruits of yellow *Baccaurea macrocarpa* (a) and formation of α -agarofuran and of γ -eudesmol from pyrolysis of agarwood (b).

Keywords: PY-MS, PY-GC-MS, EGA, bioactive volatiles

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Oral Presentations 1

THERMAL CHARACTERIZATION AS A TOOL TO UNDERSTAND THE PROCESSING–STRUCTURE RELATIONSHIP IN SUSTAINABLE POLYMER-BASED MATERIALS

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The development of sustainable polymer-based materials requires a detailed understanding of their thermal behaviour. In this context, thermal characterization becomes a key tool to evaluate the relationship between processing conditions, structural evolution and final material performance. This work presents a general approach to the thermal analysis of biobased and biodegradable polymer systems, including aliphatic polyesters, plasticized formulations and polymer composites containing functional fillers [1, 2].

Differential scanning calorimetry was used to study the main thermal transitions, including glass transition, cold crystallization and melting behaviour, with special attention to the influence of thermal history, cooling conditions and post-processing treatments. Thermogravimetric analysis provided information about thermal stability, degradation onset and the effect of repeated processing cycles on polymer degradation. Regarding dynamic mechanical thermal analysis was also considered to relate temperature dependent stiffness with molecular mobility.

The results highlight the relevance of thermal characterization for defining suitable processing windows, identifying crystallization phenomena and evaluating the effect of additives, fillers or thermal treatments on material stability. Overall, this approach provides a versatile framework for the design and optimization of sustainable polymer materials, allowing a better understanding of how thermal events govern their processability and potential application in sectors such as packaging, biomedical devices and additive manufacturing.

Keywords: thermal characterization, biopolymers, DSC, DMA

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UNIFIED KINETIC THEORY AND ITS APPLICATION FOR REVERSIBLE THERMAL DEHYDRATION AND HYDRATION PROCESSES IN SOLID–GAS SYSTEMS

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The reversible thermal dehydration of a salt hydrate and hydration of its anhydride extend the potential application of the thermochemical energy storage (TCES) systems. The kinetics of the individual processes determine the practical operation conditions of the TCES system. The kinetic descriptions of the reversible thermal dehydration and hydration processes are advanced kinetic procedures that extend beyond the conventional kinetic analysis for the reaction in the solid state. This phenomenon can be attributed to the substantial influence of atmospheric water vapor on the kinetics of both processes. Achievements of universal kinetic description across varying temperatures and partial pressure of water vapor ($p(\text{H}_2\text{O})$) are desired to provide further insight into the kinetic characteristics of the processes. Moreover, the establishment of a unified theory for the universal kinetic description applicable to both the thermal dehydration and hydration processes is imperative for the development of a useful kinetic tool for characterizing the kinetics of the TCES system. In this context, the present study focused on the universal kinetic descriptions of the thermal dehydration and hydration processes based on the unified kinetic theory. The reversible reaction process is characterized by the separation of temperature and $p(\text{H}_2\text{O})$ ranges for the thermal dehydration and hydration with the equilibrium pressure ($P_{\text{eq}}(T)$) versus T curve as the boundary. An extended kinetic equation accompanied by an accommodation function (AF; $h(p(\text{H}_2\text{O}), P_{\text{eq}}(T))$) is applicable to universally describe the kinetics of both processes as a function of T , degree of reaction (α), and $p(\text{H}_2\text{O})$ [1-4].

$$\frac{d\alpha}{dt} = A \exp\left(-\frac{E_a}{RT}\right) f(\alpha) h\left(p(\text{H}_2\text{O}), P_{\text{eq}}(T)\right), \quad (1)$$

where A , E_a , R , and $f(\alpha)$ are the Arrhenius preexponential factor, apparent activation energy, gas constant, and kinetic model function, respectively. The practical functional forms of the AF for the thermal dehydration and hydration processes can be formulated based on the classical surface nucleation and interfacial reaction processes comprising a series of elementary steps, yielding an analytical form as [1-4]:

$$h\left(p(\text{H}_2\text{O}), P_{\text{eq}}(T)\right) = \left(\frac{1}{p(\text{H}_2\text{O})}\right)^a \left(1 - \left(\frac{p(\text{H}_2\text{O})}{P_{\text{eq}}(T)}\right)^b\right), \quad (2)$$

where exponents (a , b) are correlated to the rate-determining step for the surface nucleation and interfacial reaction processes. It is noteworthy that exponents (a , b) possess positive values for the thermal dehydration process [1,2] and negative values for the hydration process [3,4]. This presentation will demonstrate the realization of the universal kinetic descriptions of both thermal dehydration and hydration processes across varying T and $p(\text{H}_2\text{O})$ values, as exemplified by several reversible reactions in solid–gas systems.

Keywords: Kinetics, Thermal dehydration, Hydration, Extended kinetic equation, Accommodation function

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EFFECT OF WATER VAPOR ON THE KINETICS OF REVERSIBLE THERMAL DEHYDRATION OF COPPER(II) CHLORIDE DIHYDRATE AND HYDRATION OF ITS ANHYDRIDE

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The reversible thermal dehydration of copper (II) chloride dihydrate (CC-DH) and the hydration of its anhydride (CC-AH) have been listed for a potential application to the thermochemical energy storage (TCES) system [1]. For application to the TCES system, it is imperative that the intrinsic kinetic behaviors of both the thermal dehydration and hydration processes, as well as the thermodynamic characteristics, be taken into consideration. However, the kinetics of both the processes have not been fully elucidated. This phenomenon is attributed to the complex kinetic behaviors of the processes, which are significantly influenced by the atmospheric water vapor.

In this study, the effects of water vapor on the kinetics of reversible reactions in the CC-DH–CC-AH–water vapor system were systematically investigated to reveal the intrinsic kinetic behaviors. A humidity-controlled thermogravimetry (TG) was utilized for collecting the kinetic data for both the thermal dehydration of CC-DH and the hydration of CC-AH at various partial pressure of water vapor ($p(\text{H}_2\text{O})$) values. The thermal dehydration process was systematically traced under linear heating and isothermal conditions in a stream of wet N_2 with varying $p(\text{H}_2\text{O})$ values (0.1–10.8 kPa). To monitor the hydration of CC-AH, the CC-AH sample was obtained by subjecting CC-DH to linear heating in a stream of dry N_2 in the TG instrument. Subsequently, the hydration of CC-AH was systematically traced under linear cooling and isothermal conditions in a stream of wet N_2 with varying $p(\text{H}_2\text{O})$ values (1.3–10.8 kPa).

The thermal dehydration of CC-DH and the hydration of CC-AH both exhibited a single-step mass change process to complete each reaction, accompanied by changes in the sample color between blue-green (CC-DH) and yellowish-brown (CC-AH) and in the crystalline phases between those of CC-DH and CC-AH. The temperature (T) and $p(\text{H}_2\text{O})$ regions of the thermal dehydration and hydration processes were thoroughly characterized with reference to the equilibrium pressure of the reaction ($P_{\text{eq}}(T)$) versus T curve. At a constant T , the thermal dehydration of CC-DH was systematically retarded with the increase in the $p(\text{H}_2\text{O})$ value, while the opposite was observed for the hydration of CC-AH. Conversely, at a constant $p(\text{H}_2\text{O})$, the thermal dehydration of CC-DH was accelerated with increasing T , while the reverse was observed for the hydration of CC-AH. The kinetics of the individual reaction processes were analyzed based on the extended kinetic equation that describes the reaction rate as a function of T , degree of reaction (α), and $p(\text{H}_2\text{O})$ [2–5].

$$\frac{d\alpha}{dt} = A \exp\left(-\frac{E_a}{RT}\right) f(\alpha) h\left(p(\text{H}_2\text{O}), P_{\text{eq}}(T)\right),$$

where A , E_a , and R are the Arrhenius preexponential factor, apparent activation energy, and gas constant, respectively. The functions of $f(\alpha)$ and $h(p(\text{H}_2\text{O}), P_{\text{eq}}(T))$ are accommodation functions with regard to α and $p(\text{H}_2\text{O})$, respectively. In this presentation, the results of the kinetic analysis will be reported, yielding the intrinsic kinetic parameters for the individual reaction processes across varying T and $p(\text{H}_2\text{O})$ values.

Keywords: Copper(II) chloride dihydrate, Thermal dehydration, Copper(II) chloride, Hydration, Kinetics

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PREDICTION OF NUCLEATION EFFICIENCY BASED ON MATCHING LATTICE DIMENSIONS

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Our work reports a quantitative correlation between the lattice dimensions and nucleating efficiency of additive materials in crystalline polymers. Isotactic polypropylene was used as model material, since the most nucleating agents are applied in this polymer. The aim is to build an empirical model, which can predict the possible nucleation efficiency of a material with known crystalline lattice dimensions in the studied polymers with reasonably good accuracy. The efficiency of the nucleation is characterized by differential scanning calorimetry (DSC) and thermo-optical microscopy (TOM). The crystalline lattice dimensions were collected from existing databases:

- <https://www.ccdc.cam.ac.uk/structures/>
- <https://crystallography.io/search/unitcell>

To describe the structural matching, best fit to the crystallographic sizes between the unit cell of the polymer and that of the additive are investigated. The selected dimensions with the best match are handled as independent parameters and the prediction model is built up by nonlinear regression analysis on the correlation between the relative dimensional matching and the nucleation efficiency. The nucleating efficiency was characterized by a relative efficiency scale worked out by Thierry et al [1]. Our results indicate clear correlation between the dimensional matching and nucleating efficiency of the additives, and the prediction of efficiency also can be done with good accuracy [2]. Although, only few crystalline polymers and additives were investigated the good agreement hints that our empirical model is generalisable to other polymers and additives as well. Accordingly, such kind of modeling can be very useful in the development of nucleating agents for crystalline polymers.

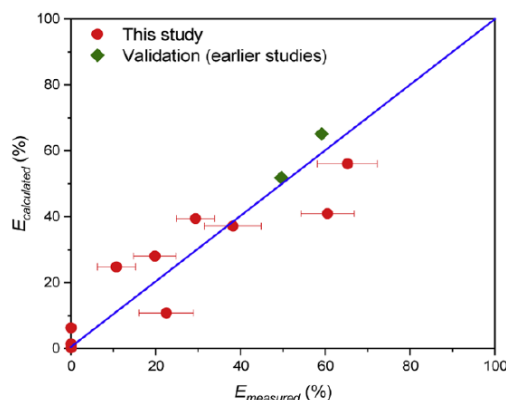


Figure 1. Good correlation between predicted and measured nucleating efficiency

Keywords: crystalline polymers, nucleating agents, matching lattice dimensions, empirical prediction

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CRITICAL ASSESSMENT OF CRYSTALLIZATION KINETICS PREDICTION FROM ROUTINE DSC

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Predicting crystallization behavior and phase stability in glass-forming systems remains a paramount challenge in materials science. In current practice, the standard approach to evaluating crystallization kinetics relies heavily on differential scanning calorimetry (DSC) data, typically obtained under non-isothermal heating regimes. This contribution critically assesses the fundamental limitations and pitfalls of this methodology, demonstrating why kinetic models applied to routine DSC curves frequently yield physically inconsistent results.

Based on an experimental dataset for chosen chalcogenide glassess, we highlight the profound discrepancy between conventional kinetic models derived from DSC data and the actual physical mechanisms governing crystallization. Routine DSC analyses are inherently compromised by several critical factors: the material's thermal history (e.g., the presence of quenched-in nuclei) [1], transient nucleation during heating, and the frequently dominant role of surface crystallization [2]. In practice, these phenomena preclude the accurate determination of the critical crystallization onset and invalidate the extrapolation of kinetic behavior to broader temperature ranges.

Furthermore, routine evaluations often default to the indiscriminate application of classical frameworks, such as the Johnson-Mehl-Avrami (JMA) model [3-5]. Based on independently measured kinetic parameters—encompassing viscosity, crystal growth rates, and true nucleation rates—this study reveals the fundamental flaws of such an approach. We emphasize the critical importance of the nucleation stage and the necessity of strictly distinguishing volume from surface crystallization, where the assumptions of conventional bulk models inherently break down. Our findings underscore that without explicitly accounting for these distinct mechanisms and the prior thermal history, physically meaningful predictions cannot be reliably extracted from conventional DSC curves.

Keywords: glass, crystallization kinetics, DSC, JMA model

Acknowledgements:

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THE MULTIPLE MODULE CALORIMETER (MMC)

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The Multiple Module Calorimeter (MMC) consists of a base unit that can be equipped with interchangeable measuring units. This allows the operator to decide whether to apply an ARC-type module for worst-case-scenario testing, a scanning module to screen samples for detecting endothermic or exothermic transitions in an adiabatic environment or a coin-cell-module for isothermal charging and discharging cycles of batteries. The latter allows for the detection of independent heat signatures for forming, charging and discharging processes on coin cell type batteries. The detected heat is lost for the capacity of the charged battery. The ability to quantify these heat losses makes it possible to calculate the efficiency of charging and discharging cycles independently from each other to optimise battery development.

Designed as a twin-cell sensor, but significantly larger in size compared with the well-known DSC setup, the sample carrier of the coin cell module (MMC CMod) is capable to test larger sample amounts. This can be beneficial for inhomogeneous samples or samples containing little amounts of impurities. When sample homogeneity or preparation is difficult in a 10 mg range, the larger volume of coin-type containers (2032) can take sample amounts in the range of 200 mg.

The newly developed heat flow calibration not only allows the use of melting standards, like it is well-known from DSC, but also an electrical in-situ *TrueJoule* calibration which is much better suited to isothermal battery testing. Due to the possibility of selecting any temperature and any enthalpy within the accessible range, the electrical *TrueJoule* calibration does not only overcome the limited number of available melting standards but also breaks the limitation of being fixed to distinct temperatures and distinct amounts of heat.

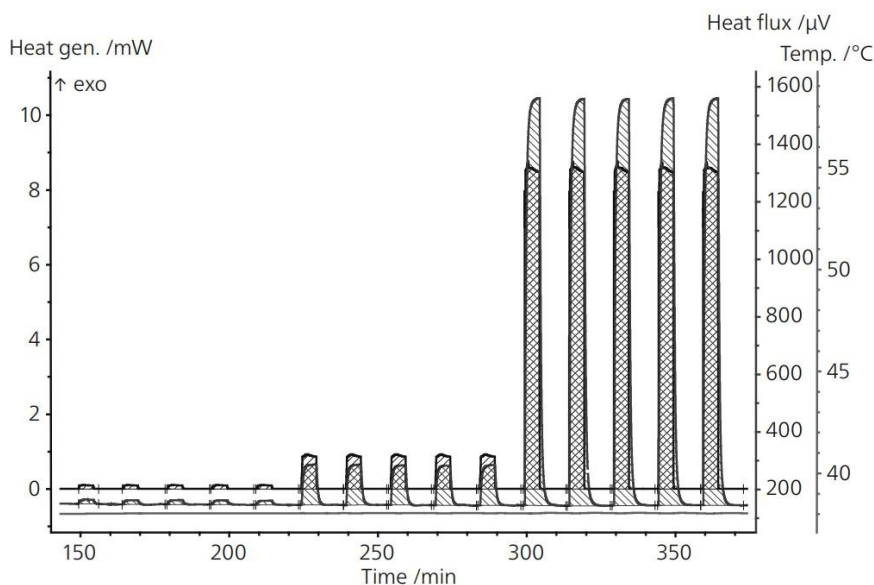


Figure 1. *TrueJoule* calibration at 35°C showing five pulses and sensor response at 0.1, 1.0 and 10 mW

Keywords: battery testing, heat signature, charging and discharging, electrical calibration

THERMAL DECOMPOSITION STUDY OF SODIUM, MANGANESE AND NICKEL ACETATE: FROM PRECURSORS TO FUNCTIONAL OXIDES

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Metal acetates are inexpensive, highly soluble precursors that are widely used to prepare transition-metal oxides for catalysis and energy storage, including the layered Na–Mn–Ni oxides currently investigated as sodium- and lithium-ion cathode materials [1]. Whether an acetate yields a single oxide, a basic carbonate or a partially reduced metal is governed by many variables, such as the metal cation, the crystal water and the surrounding atmosphere, so that the thermal-decomposition step is a crucial part of the synthesis. The thermal behaviour of the sodium [2], manganese [3,4] and nickel [5,6] salts has been reported individually. These previous studies have characterized these salts using several instruments, atmospheres, and heating rates. As a result, it is difficult to isolate the individual roles of the metal cation, crystallization water, and atmosphere during the occurrence of thermal decomposition. This inconsistency has left the exact nature of the intermediates and final oxides under debate. For example, reported residues in air range from Mn_2O_3 [4] to Mn_3O_4 [3] for manganese, and from pure NiO [6] to NiO – Ni⁰ mixtures [5] for nickel. Since the stoichiometry and purity of the final oxide strictly govern its performance in practical applications, resolution of these discrepancies is of paramount importance.

In this work, anhydrous sodium acetate together with manganese(II) and nickel(II) acetate tetrahydrates are studied under a single protocol: simultaneous thermogravimetry–differential thermal analysis (TG/DTA) is carried out under flowing synthetic air and argon at 10 °C min⁻¹; the solid residues recovered at successive stages of decomposition are characterised by capillary X-ray diffraction and attenuated total reflectance Fourier-transform infrared (ATR-FTIR) spectroscopy, in order to study the crystalline phases and the functional groups of the precursors and their decomposition products.

The thermogravimetric curves reveal clearly distinct decomposition routes. The two tetrahydrates first lose their crystal water endothermically at low temperature and then decompose through a sharp and strong exothermic step under flowing air, obtaining a transition-metal oxide as a residue, whereas anhydrous sodium acetate shows no dehydration: it remains stable up to higher temperature, melts at about 330 °C and only then decomposes. Comparing the TG/DTA curves under static conditions, oxidising (air), and inert (argon) atmospheres, together with the structural and spectroscopic analysis of the residues, the intermediates and the final products were identified to rationalise the role of the metal cation and the different atmosphere in affecting each decomposition pathways. The study aims to provide a single and consistent reference for the controlled use of sodium, manganese and nickel acetates as oxide precursors for application involving lithium- and sodium-ion cathode materials and catalysis.

Keywords: metal acetates, thermal decomposition, simultaneous TG/DTA, ATR-FTIR, oxide precursors

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THERMAL BEHAVIOR OF CESIUM IODIDE AND SODIUM IODIDE FOR GASEOUS IODINE FORMATION IN SFR

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Since the severe accident of Fukushima Daiichi Nuclear Power plant in 2011, many studies have been carried out on the source term, such as fission products released from nuclear fuel to the external environment. In light of this situation, research of the source term is also being focused on advanced nuclear power plant, such as a sodium-cooled fast reactor (SFR). The primary concern pertains to the quantity of radioactive nuclides that would be released into external environment from the reactor core in a nuclear reactor. Iodine is a pivotal radioactive nuclide in the context of source term evaluation. The formation of gaseous iodine is crucial event, because the behavior of aerosols, such as agglomeration and sedimentation, is not expected. Moreover, the formation of gaseous iodine has the potential to increase the risk of not being able to retain the fission products within the containment vessel of nuclear reactor. However, the details of the formation behavior of gaseous iodine, such as formation conditions and formation rate, are not well understood. Considering the severe accidental condition of SFR, it is assumed that gaseous iodine would be formed from two iodides: cesium iodide (CsI) as fission product of nuclear fuel [1] and sodium iodide (NaI) as reaction product in liquid sodium [2]. Thus, it is very important to understand the thermal behavior of CsI and NaI under various atmospheric conditions. For the first step, thermal behavior of CsI and NaI in an inert atmosphere was investigated using TG–DSC (STA 449 F3 Jupiter, NETZSCH) equipped with a platinum–rhodium heating furnace. Chemical reagents of CsI and NaI with a purity of approximately 99.5% (Fujifilm Wako Pure Chemical) were used as sample without further purification. Each sample, weighed into an alumina crucible (internal diameter: 4 mm, height: 6.8 mm), was heated from 300 to 1273 K at different heating rates under 200 cm³ min^{−1} argon flow. In both samples, two DSC endothermic peaks were observed, in which the latter peak was accompanied by the mass loss process of consumption of the entire samples. The first endothermic DSC peaks for CsI and NaI were observed at 903 and 931 K, respectively, corresponding to the fusion [3–4]. The enthalpy changes of the fusion for both samples was found to be comparable, with a value of approximately 24 kJ mol^{−1}. The second DSC endothermic peak accompanied by the complete mass loss indicated the enthalpy changes of approximately 123 and 111 kJ mol^{−1} for CsI and NaI, respectively, which are corresponded to those of evaporation [5–6]. The application of the Kissinger plot to the peak top temperature of the DTG curves at varying heating rates revealed the apparent Arrhenius parameters for the vaporization process (E_a /kJ mol^{−1}, $\ln(A/s^{-1})$), which were determined to be (147 ± 14, 8.9 ± 1.6) and (144 ± 11, 8.6 ± 1.3) for CsI and NaI, respectively. The present results indicated that, in an inert gas atmosphere, the thermal behavior of CsI is analogous to that of NaI, particularly with regard to the phase transition behavior and the vaporization kinetics.

Keywords: Severe accident, Source term, Gaseous iodine formation, Cesium iodide, Sodium iodide

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THERMAL STABILITY PREDICTION OF SUGAR ALCOHOLS

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This study deals with the thermal stability of selected sugar alcohols as promising medium-temperature phase change materials (PCMs) for latent heat thermal energy storage. To address this, four representative sugar alcohols were chosen (D-dulcitol, D-mannitol, erythritol, and *myo*-inositol) and isothermal stress tests were conducted at temperatures above their respective melting points. Subsequently, differential scanning calorimetry (DSC) was employed to quantify the changes in the melting enthalpy compared to unstressed materials. Supplementary Fourier-transform infrared spectroscopy (FTIR) and thermogravimetry (TG) measurements were carried out in order to provide insight into the degradation reactions. FTIR and TG analyses suggest that the degradation of the studied sugar alcohols proceeds primarily via dehydration and condensation reactions, as evidenced by the absence of C=O bands and significant mass loss below 200 °C likely due to water evaporation. The DSC results demonstrate that polymorphism is a primary challenge in stability evaluation of PCMs by this technique. This is especially the case of D-dulcitol and D-mannitol, where unpredictable variations in crystalline phase were observed and even made the kinetic analysis unfeasible for the latter. DSC also revealed secondary exothermic events across all materials, attributed to cold crystallization of amorphous phases in erythritol and D-dulcitol, and a solid–solid transition in *myo*-inositol. Decrease in melting enthalpy with respect to heating time for D-dulcitol and erythritol followed first-order kinetics

$$\frac{\Delta_{\text{fus}}h_s}{\Delta_{\text{fus}}h_0} = \exp(-k_1t),$$

while *myo*-inositol required to employ models based on Avrami equation or n^{th} -order kinetics model

$$\frac{\Delta_{\text{fus}}h_s}{\Delta_{\text{fus}}h_0} = [(n-1)k_nt + 1]^{\frac{1}{1-n}}.$$

From the enthalpy–time curves, apparent rate constants of degradation were determined at various temperatures. Further kinetic analysis was carried out to obtain the parameters of the Arrhenius equation in order to predict the stability of the studied alcohols. Predicted stability curves highlight the exceptionally high relative stability of erythritol compared to the other studied sugar alcohols. Our findings emphasize that the choice of the conversion function as well as of the temperature function is critical for developing accurate long-term prediction models for PCMs.

Keywords: differential scanning calorimetry; melting enthalpy; kinetic analysis; thermal degradation

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DUAL INHIBITION EFFECT OF DEEP EUTECTIC SOLVENTS, RELINE, MALINE, AND GLYCELIN ON METHANE HYDRATE NUCLEATION AND FORMATION

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The search for environmentally friendly hydrate inhibitors is indispensable for flow assurance and the prevention of hydrate formation in gas or oil pipelines. Due to their non-toxic and biodegradable properties, deep eutectic solvents (DESs) are classified as new green solvents. In this work, the dissociation conditions of methane hydrates influenced by three DESs: reline [1], maline, and glycelin [2] were experimentally determined. Reline was derived from urea and choline chloride (ChCl) at a molar ratio of 2:1. Glycelin was formed by combining ChCl and glycerol at a molar ratio of 1:2. In contrast, maline was formed by combining ChCl and malonic acid at a 1:1 molar ratio. For the kinetic studies, a high-pressure micro-differential scanning calorimeter was used to measure the induction time for hydrate formation. Compared with pure water, all the studied samples containing DES demonstrated the potential to prolong the induction time for hydrate nucleation. The isochoric method was applied to determine the hydrate-liquid water–vapor-three-phase-coexisting phase boundary. All three additives consistently inhibited methane hydrate formation. Three DESs, reline, maline, and glycelin, are identified as both kinetic and thermodynamic inhibitors, exhibiting dual (kinetic and thermodynamic) inhibition of nucleation and methane hydrate formation. With their environmentally friendly and cost-effective properties, DESs hold great potential for promoting greener flow assurance applications in the oil and gas industry.

Keywords: DSC, deep eutectic solvents, methane hydrate, kinetic hydrate inhibitor, flow assurance

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INVESTIGATING THE ROLE OF COAL RANK IN COMBUSTION BEHAVIOR: KINETIC AND THERMODYNAMIC EVALUATION

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In this study, the combustion behaviors of coal samples of different rank values, obtained from the Soma and Zonguldak basins, were investigated. At the beginning of the study, the physical and chemical properties of the coals were determined through ultimate, proximate, carbon-sulfur, and calorific analyses. Subsequently, thermal analyses of the coals were performed under a dynamic oxygen atmosphere (at a flow rate of 100 mL/min) utilizing a coupled TG/DTG/DSC technique, and their combustion kinetics and thermodynamic parameters were calculated.

While Zonguldak coal represents a high-rank bituminous structure, Soma coal exhibits an intermediate-rank characteristic within the sub-bituminous range. Consequently, the ash and moisture contents of Zonguldak coal are lower, while its carbon percentage and net calorific value are higher. These parameters are presented in Table 1.

Table 1. The proximate analysis results and calorific values of coals (I.M: Inherent Moisture, V.M: Volatile Matter, F.C: Fixed Carbon and L.H.V: Lower Heating Value)

Coal Type	I.M (%)	Ash (%)	V.M (%)	F. C (%)	L.H.V (kcal.kg ⁻¹)
Zonguldak	0.28±0.15	37.7±0.32	24.9±0.06	37.5±0.36	4663
Soma	2.24±0.10	44.2±0.21	39.2±0.17	16.6±0.19	1996

The high volatile matter content of Soma coal reduces its ignition temperature while simultaneously lowering its calorific value. Furthermore, its high moisture content contributes to a decrease in its calorific value. However, combustion efficiency is not solely dependent on the calorific value; it is also determined by the low activation energy required to initiate this combustion. Therefore, the combustion activation energies of both coals were investigated using the Kissinger-Akahira-Sunose (KAS) and Flynn-Wall-Ozawa (FWO) methods, which belong to the class of integral methods [1]. Soma coal exhibits an average combustion activation energy of 116.64 kJ.mol⁻¹, a value lower than the average activation energy of Zonguldak coal, which stands at 139.76 kJ.mol⁻¹. Additionally, thermodynamic parameters of transition complexes ($\Delta H^\ddagger$, $\Delta G^\ddagger$, and $\Delta S^\ddagger$) were evaluated to elucidate the thermal behavior changes across combustion. Improvement efforts targeting the removal of moisture and inorganic matter from Soma coal will raise its heating value and subsequently improve combustion efficiency, ensuring a more viable and efficient use of this energy source.

Keywords: Coal, Combustion kinetics, FWO, KAS.

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FROM FUNDAMENTAL REACTIVITY TO 3D-PRINTED ARCHITECTURES: ADVANCES IN CAO-BASED MATERIALS FOR THERMOCHEMICAL ENERGY STORAGE

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The transition toward carbon neutrality requires efficient and scalable solutions for high-temperature energy storage. Thermochemical energy storage (TCES) based on the reversible $\text{CaCO}_3 \leftrightarrow \text{CaO} + \text{CO}_2$ reaction (Calcium Looping, CaL) is a promising approach due to the low cost, abundance, and high theoretical energy density of CaO-based materials, as well as their compatibility with concentrated solar power and industrial waste-heat recovery. However, practical deployment is hindered by rapid sorbent deactivation due to sintering, limited gas–solid contact, and solids-handling challenges. Significant efforts have therefore focused on improving multicycle stability through microstructural control, optimized synthesis routes, and the incorporation of stabilizing dopants to mitigate sintering and preserve reactivity.

Recent advances move beyond conventional powders toward structured CaO-based architectures that enhance heat and mass transfer and facilitate reactor integration. In this work, we present the design and fabrication of three-dimensional CaCO_3/CaO structures using additive manufacturing techniques, including direct ink writing and stereolithography. Low-cost limestone-derived formulations were optimized to produce self-supporting monoliths with tailored porosity and channel geometries. The 3D-printed materials were evaluated under multicycle calcination/carbonation conditions. They retain structural integrity and porosity after repeated cycling, showing improved gas accessibility, reduced transport limitations, and enhanced cyclic performance compared to powdered references. Structural characterization (XRD, SEM, X-ray tomography) confirms slower sintering and preserved pore connectivity. Additionally, composite formulations containing stabilizing additives (e.g., MgO , SiO_2) further improve durability.

Overall, this work demonstrates that combining fundamental understanding with advanced manufacturing enables the development of next-generation CaL materials. The transition toward engineered 3D architectures opens new pathways for compact and efficient TCES systems.

Keywords: Thermochemical energy storage, Calcium Looping, Additive Manufacturing

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BACKBONE CONFORMATIONAL ENTROPY CHANGE IN HELIX FOLDING DETERMINED BY DIFFERENTIAL SCANNING CALORIMETRY

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The stability of the native protein structure reflects a balance of thermodynamic forces, dominated by the backbone conformational entropy loss (ΔS_{BB}) upon folding. Despite decades of study, ΔS_{BB} estimates span 3–7 cal mol^{−1} K^{−1} per residue, with even a 1 cal mol^{−1} K^{−1} error translating to ± 30 kcal mol^{−1} for a 100-residue-long protein. Helix-coil transition theory relates ΔS_{BB} to the nucleation parameter ν , but independent experimental determination has proven difficult: most studies rely on a fixed, decades-old estimate ($\nu = 0.036$ – 0.048), largely because spectroscopic methods require auxiliary parameters that obscure ν through correlations. We show that differential scanning calorimetry (DSC) overcomes this by directly measuring absolute heat capacity, bypassing spectroscopic assumptions. We measured DSC profiles (0–120 °C) of alanine-rich peptides Ac-(AAKAA)_n-GY-NH₂ ($n = 1$ – 6), revealing broad, weakly cooperative transitions that shift systematically with length. Fitting a helix-coil transition theory (Lifson-Roig model) derived heat capacity model function to all profiles within a Bayesian framework showed that nucleation constant is well-determined. This yields a unified thermodynamic parameter set: $\nu = 0.08 \pm 0.01$, propagation enthalpy $\Delta H = -1.2 \pm 0.1$ kcal mol^{−1}, and $\Delta C_p = 3 \pm 1$ cal mol^{−1} K^{−1} per residue - giving $\Delta S_{BB} = (5.2 \pm 0.3)$ cal mol^{−1} K^{−1}, nearly double prior estimates but consistent with recent MD simulations (~ 5.0 cal mol^{−1} K^{−1}). These results establish DSC as a powerful tool for quantifying helix-coil thermodynamics, with implications for protein stability and folding-upon-binding in intrinsically disordered proteins.

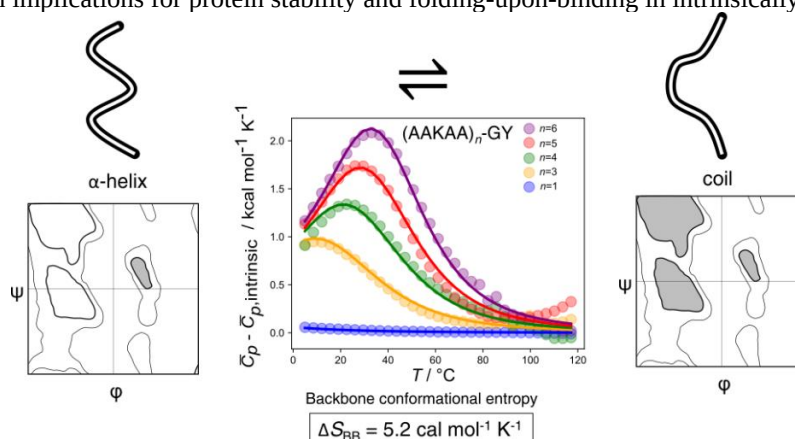


Figure 1. Calorimetric determination of backbone conformational entropy change in α -helix folding.

Keywords: α -helix, protein folding, conformational entropy, calorimetry, backbone entropy

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THERMODYNAMIC INSIGHTS ON THE STRUCTURE-PROPERTY RELATIONSHIPS IN TRI-SUBSTITUTED BENZENES

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An extensive experimental and computational thermodynamic study of nine methyl-substituted nitrobenzoic acids was carried out with the aim of characterizing the phase transition and formation thermodynamics¹.

The combustion energies were measured using high-precision combustion calorimetry, and the enthalpies of formation of the crystal phase were derived. The sublimation enthalpies were obtained from the vapor pressure-temperature dependencies measured using the classic Knudsen effusion mass loss and the transpiration methods. The standard molar enthalpies of vaporisation were derived from the temperature dependence of the mass-loss rates measured using non-isothermal thermogravimetry. The thermal behaviour, including melting temperatures and standard molar enthalpies of fusion, was investigated by differential scanning calorimetry. The consistent set of experimental properties at the reference temperature $T=298$ K was evaluated and recommended for thermochemical calculations.

The pairwise interactions of the substituents on the benzene ring were derived from nitro-toluenes, methyl-benzoic acids and nitro-benzoic acids available in the literature, and the additivity of the contributions when three substituents are placed simultaneously in the benzene ring was discussed.

Keywords: benzoic acid derivatives, thermochemistry, phase transitions, structure-property relationships

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EXPLOITING STATISTICAL AND CLASSICAL THERMODYNAMICS FOR MODELLING THE MULTIMERIC PROTEIN DENATURATION/DISSOCIATION PROCESSES

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A large cut of proteins with known structure (about 30-50%) has a natural tendency to self-associate into multimers, mostly existing as homomers, i.e., homo-oligomeric protein complexes composed of two or more identical subunits. In addition, even roughly 45% and 60% of eukaryotic and prokaryotic proteins, respectively, that are deposited in data banks as single polypeptide chains also exist as homomers [1].

The potential advantages of being a multimeric protein in living systems have been mainly identified as: genetic saving (less prone to translational errors), functional gain intended as any enhancement in the efficiency of the biological action upon oligomerization; structural advantage, that, among others, is related to the ability of finely tuning the protein (often enzyme) activity through subtle conformational changes more easily allowed in multimers [2].

In this context, in the recent years, many biochemical, spectroscopic, and computational methods were developed to assess the protein oligomer structural features, while much less theoretical progress has been experienced concerning the description of multimeric proteins that undergo thermal denaturation that, in any case, can be traditionally assessed by DSC [3].

Indeed, even in the simplest case of a multimeric protein undergoing denaturation with a concomitant dissociation, the intersection of equilibrium thermodynamics and statistical mechanics is no longer so simple as in the monomeric proteins since, apparently, the total number of molecules changes when moving from the native state to the denatured one. To overcome this difficulty, the most applied quantitative methods through the years have been based on an approach proposed by Privalov and Potekhin in the 1980's [4], which, however, is just a validation method and not a method that permits an independent simulation of the calorimetric curve for a fair fitting of the experimental data.

To fulfil this gap, in this work we present a methodology and strategy that link statistical mechanics concepts (canonical partition function) with the classical thermodynamics' equilibrium constant that permit the developed of independent thermodynamic models suitable to assess the stoichiometry, the stability, and the peculiar dissociation/denaturation mechanisms of such multimeric proteins for the most common cases according to the literature [5].

In any case, the methodology and strategy are general and pave the way for the development of models that may reflect more complex scenarios than those considered in this work, if the case.

Keywords: DSC, Protein dissociation, Thermodynamic models

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NON-ISOTHERMAL CRYSTALLIZATION KINETICS AND THERMAL STABILITY OF BOROTELLURITE GLASSES INVESTIGATED BY DIFFERENTIAL THERMAL ANALYSIS

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Borotellurite glasses (BTGs) have attracted considerable attention owing to their high density, optical transparency, and potential applicability in radiation shielding technologies. Following our previous investigation on high-density BTGs [1], the present study focuses on elucidating their non-isothermal crystallization kinetics using differential thermal analysis (DTA) and various kinetic models. Accordingly, the crystallization behavior and thermal stability of $x\text{B}_2\text{O}_3$ – $(100-x)\text{TeO}_2$ glasses (x : 5-20 mol%) were investigated by DTA at heating rates of 5, 10, and 20 °C.min⁻¹. The glass transition temperature (T_g) and crystallization peak temperature (T_p) were determined and subsequently used to calculate the activation energy for crystallization employing the Ozawa, Kissinger, and Augis-Bennett methods. The results showed that increasing both the heating rate and B_2O_3 concentration shifted T_g and T_p toward higher temperatures, indicating enhanced thermal stability and increased resistance against crystallization. The primary crystallization peak appeared between 360 and 505 °C, depending on composition and heating rate, while a secondary exothermic peak was observed for the BT10 composition. Heat-treatment experiments combined with X-ray diffraction analysis revealed that the crystallization process was associated with the precipitation of $\alpha\text{-TeO}_2$. The activation energies obtained from the three kinetic approaches were in good agreement and generally increased with increasing B_2O_3 content. The highest activation energy values were obtained for the BT15 composition, suggesting improved structural stability and a reduced tendency for crystallization. A slight decrease in activation energy at higher B_2O_3 concentrations indicates the onset of structural rearrangement and an increased propensity for crystallization. The present findings provide new insights into the composition-dependent crystallization behavior of BTGs and contribute to optimizing their thermal processing and long-term performance in advanced functional applications.

Keywords: Borotellurite glass; Differential thermal analysis; Non-isothermal crystallization kinetics; Activation energy; Thermal stability.

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Oral Presentations 2

HIGH-PERFORMANCE THERMOCHROMIC COATINGS WITH W-DOPED VO₂ NANOPARTICLES IN SiO₂ MATRIX SYNTHESIZED USING A FAST THERMAL OXIDATION

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Global warming and energy crisis drive a focus on energy-saving materials. Buildings have been estimated to produce about 20% of all anthropogenic greenhouse gas emissions and are responsible for up to 40% of the primary energy consumption in the world. Approximately 50% of the total building energy is consumed for compensating the heat gains and losses via windows and glass facades, which are the most energy-inefficient components of buildings. It is evident that energy-saving smart windows with adjustable throughput of solar energy can lower the energy expenditure. Vanadium dioxide (VO₂) exhibits a reversible phase transition from a low-temperature monoclinic VO₂(M1) semiconducting phase to a high-temperature tetragonal VO₂(R) metallic phase at a transition temperature of approximately 68 °C for the bulk material. The automatic response to temperature and the abrupt decrease of infrared transmittance with almost the same luminous transmittance at the transition into the metallic state make VO₂-based coatings a promising candidate for thermochromic smart windows reducing the energy consumption of buildings. We report high-performance thermochromic VO₂-based coatings with a decreased transition temperature to 30 °C, an integral luminous transmittance of coated glass above 60%, a modulation of the solar energy transmittance up to 10%, and enhanced environmental protection. They have been prepared on standard soda-lime glass and quartz glass by using a three-step process, consisting of magnetron sputter depositions of SiO₂ films and V-W films without substrate heating and their post-deposition oxidation in pure oxygen (solid-state dewetting) at the temperatures of 400 °C and 450 °C. We present and explain the design, structure and optical properties of thermochromic coatings with two layers of W-doped VO₂ nanoparticles dispersed in SiO₂ matrix, which are produced by a fast solid-state dewetting (5 min) from thin (9 nm and 11 nm) V-W films. Our main objective has been to investigate the effect of the solid-state dewetting temperature and the V-W film thickness on stoichiometry, crystallinity and morphology (note the conditions for the localized surface plasmon resonance) of the W-doped VO₂ nanoparticle layers, which determine, together with a top antireflection SiO₂ layer, thermochromic performance of the coatings.

Keywords: Thermochromic coatings, VO₂-based coatings, smart windows, low deposition temperature, fast thermal oxidation, decreased transition temperature

A COMPLETE TIME-TEMPERATURE-TRANSFORMATION DIAGRAM FOR THE AEROSPACE EPOXY RESIN RTM6: INTEGRATING CURE KINETICS, THERMAL DEGRADATION AND CURING OVERHEATING

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RTM6 is a monocomponent epoxy resin widely used in the aerospace industry, where it is processed by resin transfer moulding and related liquid-composite-moulding routes to manufacture high-performance structural parts. The time–temperature–transformation (TTT) diagram is one of the most useful tools for designing and optimizing the cure cycle of such thermosets, as it maps, as a function of cure temperature and time, the boundaries between the different physical states of the material. The cure kinetics of RTM6, including the slowdown of the reaction caused by diffusion as the resin approaches vitrification, has been characterized in a previous work by means of isoconversional methods, which provided the gelation and vitrification boundaries of the diagram. In the present work we complete the TTT diagram of RTM6 by adding the two boundaries that delimit its safe processing window at high temperature: thermal degradation and curing overheating. The degradation kinetics is determined from thermogravimetric measurements performed under both inert and oxidative atmospheres and analysed with isoconversional methods, since the degradation pathway — and therefore the temperature at which the resin can still be used — depends strongly on the surrounding atmosphere. The curing overheating is evaluated by means of a recently developed analytical model that predicts the maximum temperature difference within a curing laminate and the critical thickness above which a thermal runaway occurs. Degradation sets the upper temperature limit of the usable range, whereas overheating becomes a critical constraint when curing thick parts, in which the heat released by the exothermic cure reaction cannot be efficiently dissipated. By combining the cure, degradation and overheating boundaries, the resulting complete TTT diagram defines the full processing window of RTM6 and provides a practical tool for selecting safe cure temperatures and part thicknesses.

Keywords: RTM6 epoxy resin, Time–temperature–transformation diagram, Thermal degradation, Curing overheating, Isoconversional methods.

METASTABLE W–Zr THIN-FILM ALLOYS: EFFECT OF COMPOSITION ON PROPERTIES, THERMAL STABILITY AND OXIDATION RESISTANCE

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One of the practical advantages of the magnetron sputtering technique is the ability to mix metal elements atom by atom and thus prepare thin-film alloys relatively easily with a diverse composition. Since magnetron sputtering is also highly non-equilibrium low-temperature deposition process with high cooling rates (≥ 106 K/s) on the atomic scale at the substrate surface, kinetics plays an important role and governs the nucleation and thin-film growth. As a result, metastable structures or phases far from the equilibrium state may evolve and novel materials with unexplored properties may be synthesized. Thin-film metallic glasses (TFMGs) with short- and medium-range ordering of atoms are an example of this type of materials. Thanks to the disordered structure, they provide a combination of exceptional physical and functional properties.

Therefore, the talk will focus on the composition and properties of the binary W-Zr system with up to 83 at.% Zr, deposited onto unheated, unbiased and rotating substrates using magnetron co-sputtering of W and Zr targets in the argon atmosphere [1]. Special attention will be paid to their thermal stability and oxidation behavior [2,3], which are critical factors influencing the long-term performance and durability of materials under elevated temperature conditions.

The metastable structure of the W-Zr films with 0-24 at.% Zr is thermally stable in argon up to 1200°C and their oxidation in air proceeds via formation of protective surface oxide layer [2].

On the other hand, thermal stability of the W-Zr TFMGs with 33-83 at.% Zr extends to 1400°C. Additionally, controlled post-deposition oxidation of these films in air leads to the formation of hard amorphous substoichiometric W-Zr-O films with tunable optical and electrical properties [3]. This is a consequence of a unique oxidation process driven by thermodynamic factors, enabling gradual oxygen incorporation throughout the film volume.

Keywords: W-Zr, Metallic glass, magnetron sputtering

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KINETIC ANALYSIS OF METAL OXIDE THIN FILM PREPARED BY AEROSOL WRITING

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Thermal treatment provides an effective route for controlling the nucleation and crystallinity, and thereof phase composition and structural properties of various metal oxide systems. The approach has been considerably straightforward for three-dimensional particle systems, even nanoparticle-based powders, but not so much for thin films. Moreover, the thermal evolution of even more specific thin films derived by aerosol writing can differ from that of conventional thin films due to their confined geometry, heterogeneous microstructure, and high defect density. This work investigates the thermal evolution and crystallization behaviour of metal oxide thin films prepared by high-energy aerosol deposition (SPAD).

Nanoparticles based on Co, In, Mo, and W oxides were deposited as thin films in initially amorphous or poorly crystalline states and subsequently subjected to controlled thermal treatments. The evolution of crystallinity was monitored using time-resolved grazing-incidence X-ray diffraction (GIXRD), enabling the determination of the transformed fraction and evaluation of crystallization kinetics under isothermal conditions.

The results demonstrate that thermal treatment effectively modifies the structural state of aerosol-written films, while the crystallization behaviour depends strongly on the oxide composition and deposition-induced microstructure. Compared with three-dimensional nanoparticle powders, the thin films exhibit deviations from conventional crystallization behaviour, which can be associated with restricted growth, heterogeneous nucleation, interfacial effects, and structural defects. These findings highlight the importance of material-specific thermal treatment protocols and demonstrate the potential of aerosol writing combined with controlled annealing for engineering metal oxide thin films with tailored structural properties.

Keywords: aerosol writing, metal oxide thin films, thermal treatment, nucleation, crystallization, GIXRD

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THERMAL STABILITY OF MIXED STRUCTURES OF WOLFRAMITE AND SCHEELITE

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Metal tungstates crystallize in two main structural types, namely wolframite and scheelite. The wolframite structure is based on chains of edge-sharing octahedra (MO_6 and WO_6), which results in a relatively flexible and anisotropic framework that becomes mechanically less stable at elevated temperatures [1]. In contrast, the scheelite structure consists of isolated WO_4 tetrahedra in a rigid three-dimensional network, leading to higher structural stability and resistance to deformation [2]. These structural differences strongly influence the thermal and mechanical behavior of tungstate materials.

In this work, pure CaWO_4 (scheelite structure) and MnWO_4 (wolframite structure) were prepared using dry mechanoactivation and precipitation synthesis methods. In addition, mixed $\text{Ca}_{1-x}\text{Mn}_x\text{WO}_4$ compositions were prepared via the same two synthesis methods. The aim was to investigate how the substitution between Ca^{2+} and Mn^{2+} affects phase formation, thermal stability, and high-temperature behavior. Particular attention was paid to mixed compositions, where both cations are present, in order to understand the transition between scheelite- and wolframite-type structures.

Thermal analysis (DSC/TG) and heat microscopy reveal that the evolution of $\text{Ca}_{1-x}\text{Mn}_x\text{WO}_4$ is highly non-linear with respect to composition. While Ca-rich samples exhibit relatively simple crystallization and gradual densification typical of the scheelite structure, Mn-rich compositions show complex multi-step behavior associated with defect-rich precursors and structural reorganization. Interestingly, even intermediate compositions display features closer to MnWO_4 , especially at high temperatures, where partial viscous deformation is observed. This indicates that the wolframite phase is mechanically less stable and therefore has a greater influence on the overall behavior of the mixture. Overall, the results suggest that the transition between scheelite and wolframite is not continuous, but proceeds through heterogeneous, multiphase states strongly influenced by Mn incorporation.

Keywords: tungstate, wolframite, scheelite

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SURFACE MOBILITY IN AMORPHOUS CHALCOGENIDE FILMS PREPARED BY THERMAL EVAPORATION AND SOLUTION PROCESSING

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Understanding molecular transport and surface mobility in amorphous chalcogenides is essential for the development of advanced optical, photonic, and electronic devices. Surface-related phenomena significantly influence stability, mechanical response, diffusion processes, and processes related to changes in structure, such as structural relaxation and crystallization (nucleation and crystal growth).

This contribution focuses on the investigation of surface mobility in amorphous chalcogenide films prepared by thermal evaporation and solution processing techniques. The study compares molecular transport in bulk materials, thermally evaporated thin films, and spin-coated thin films. The surface mobility is explored using surface grating decay experiments followed by atomic force microscopy, as well as using nanomechanical characterization methods (nanoindentation). The combination of these methods allows us to reveal near-surface viscosity and self-diffusion coefficients in amorphous thin films.[1-3]

The results demonstrate that preparation technology has a substantial influence on the near-surface transport properties of amorphous chalcogenides. Thermally evaporated films exhibit surface mobility behavior relatively close to bulk materials. In contrast, solution-processed films show significantly enhanced surface mobility and faster grating decay kinetics. These findings highlight the importance of considering deposition-induced structural heterogeneity when interpreting diffusion, relaxation, and mechanical behavior in amorphous chalcogenide thin films.

Keywords: chalcogenide films, nanoindentation, atomic force microscopy, surface mobility

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MAXIMIZING THE VALUE OF THERMAL ANALYSIS FOR DIFFICULT AND ONE-SHOT SAMPLES

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Thermogravimetry (TG) and differential scanning calorimetry (DSC) benefit from very mature instrumentation and well-established calibration/validation procedures. However, measurement reliability is frequently governed by circumstances and decisions made before measurement even begins: initial sample state, sample handling, sample geometry and mass, pan selection, and all of the thermal analysis 'knobs'. These parameters become particularly important for compositionally labile and poorly conducting samples, or when there is a very limited amount of sample available. This contribution presents a practical guideline for sample preparation and method design addressing these recurring problems, particularly in fast-paced R&D settings.

For compositionally labile materials, *i.e.* weakly bound hydrates/solvates, emphasis is placed on stabilization of the prepared sample for the introduction to dry atmosphere, along with tuning the measurement conditions for the intended initial state. [1] For samples with poor thermal conductivity, programmed and sensed temperature may differ significantly from local sample temperatures because of external thermal resistance and slow internal equilibration. [2] Preparation of sample compact and deliberate changes of sample mass, heating rate and gas flow are discussed as practical method improvements. Additionally, a simple visual model will be used to illustrate the behavior of the poorly conductive samples in TG/DSC experiments. The model motivates method parameter selection from an allowable temperature error and conductivity estimates, and shows how thermal lag can diagnose contact, transport and reaction effects [2, 3]. Finally, a challenge of making an informative one-shot TG/DSC run is discussed, with emphasis on explicit information priorities and estimates of pre-decomposition thermal behavior. Representative case studies and examples of poor vs. good results will be used to support the guidance for maximizing the value of thermal analysis experiments.

Keywords: sample preparation, difficult samples, method development

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VALORISATION OF KAOLINITIC CLAYS, NATURAL POZZOLANS AND CONSTRUCTION AND DEMOLITION WASTE MIXTURES AS PORTLAND CEMENT SUBSTITUTES

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Portland cement production represents one of today's greatest environmental challenges, as its manufacturing generates a significant amount of CO₂. Given the need to achieve climate neutrality by 2050, one of the priority strategies is the reduction of the clinker factor through the incorporation of supplementary cementitious materials (SCM). In this context, this study evaluates the potential of various mineral resources and construction and demolition waste (CDW) to be used as SCM. The kaolinitic clays used came from San José (Bolivia) and Pinell de Brai, Spain. Natural pozzolanes of dacitic composition from Bolivia also were tested. To this end, various binary and ternary mixtures of pastes and mortars were produced; standard calcareous sand and a CEM I 52.5 R Portland cement were used. Chemical composition was determined by X-ray fluorescence. X-ray powder diffraction and differential thermal and thermogravimetric analysis were used to obtain information about the reactivity of the components and to quantify the carbonates and clays present in the materials used. Mineral phases and thermal properties of clays were previously being obtained [1,2]. Those of pozzolanes and pastes were determined in the present study. The addition of clays and construction and demolition waste has a significant effect on rheological behavior, as it increases the amount of water required to achieve a normal consistency; this, in turn, limits the degree of Portland cement replacement to proportions not exceeding 30%. Ternary mixtures with substitution of 10 and 20% of clays and 10-20 % CDW were tested. The results show best mechanical performance using the san José clays than those from Pinell de Brai. Both have similar chemical and mineralogical composition but those from San José present an amorphous phase, which increase their reactivity. Notable formulations include 20% san José kaolin and 10% CDW) and 10% Pinell de Brai clay and 20% waste, which achieved flexural strengths between 11 and 12 MPa and compressive strengths of 54-55 MPa at 56 days—values comparable to the reference mortar. These preliminary results validate the technical feasibility of recovering these resources into sustainable binders.

Keywords: Construction and Demolition Waste, kaolinitic clays, Recycling, mortar

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SUSTAINABLE UTILIZATION OF İZMIT GULF MARINE SEDIMENTS FOR GLASS AND ENAMEL PRODUCTION

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Bottom sediments accumulating in coastal environments are generally considered environmental waste; however, they can also serve as a valuable secondary raw material for glass and ceramic products. In this study, the potential use of bottom sediments collected from the İzmit Gulf (Kocaeli, Türkiye) in glass and enamel production was investigated. Two glass formulations were prepared for glass production: one containing 100% bottom sediment, and the other containing bottom sediment with added sodium carbonate as a plasticizer. Both compositions were melted in an elevator melting furnace, poured into water, ground, and sieved below 33 microns for the enameling process. The surfaces of low-carbon 1040 steel plates, selected as the substrate material for enameling, were mechanically sanded, cleaned with alcohol, and sandblasted to ensure adequate surface preparation. After preheating the substrate samples at 200 °C for 20 minutes, glass powders were applied to the top surfaces by sprinkling, then the samples were fired at 900 °C for 10 minutes. Microstructural and phase analyses were conducted to evaluate the properties of the produced glass powders and enamel coatings. The results show that the glass and enamels obtained from the bottom sediments of İzmit Gulf possess suitable structural and surface properties for artistic and decorative applications. These findings demonstrate that marine sediment waste can be effectively used as a raw material for glass and enamel production, providing a sustainable approach to both environmental management and artistic material development.

Keywords: İzmit Gulf, marine sediment, glass material, enamel coating, waste valorization

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APPLICATION OF DIFFERENTIAL SCANNING CALORIMETRY FOR THE DETERMINATION OF QUARTZ CONTENT IN MINERAL MATRIX MATERIALS

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Quartz (SiO_2) is commonly used as a component of mineral fillers for paints and coatings, where its content influences the mechanical properties of the final material. Differential scanning calorimetry (DSC) enables the quantification of quartz through the reversible α -quartz $\leftrightarrow$ β -quartz phase transition, using the associated solid-state transition enthalpy as the analytical signal.

In this study, quartz content was determined using the Julia DSC system (Anton Paar), equipped with a Peltier-based cooling system that enables controlled cooling to temperatures as low as -35°C . Binary mixtures of quartz with alumina, talc, and leucophyllite were investigated over a range of compositions. During the first heating cycle, dehydration of chlorite-containing phases in some matrix materials produced thermal effects that overlapped the comparatively weak $\alpha \rightarrow \beta$ quartz transition. In contrast, the subsequent cooling segment exhibited a distinct $\beta \rightarrow \alpha$ quartz transition, which was used for evaluation. Measurements of quartz–alumina reference mixtures showed a linear relationship between the transition enthalpy and the nominal quartz content. This calibration was subsequently applied to determine the quartz content in talc- and leucophyllite-based mixtures.

The results demonstrate that the $\beta \rightarrow \alpha$ quartz transition measured during the cooling cycle can be used to determine quartz content in different mineral matrix materials by DSC.

Keywords: differential scanning calorimetry, quartz

THERMODYNAMIC AND THERMAL CHARACTERIZATION OF IONIC LIQUIDS AND IONOGELS FOR SUSTAINABLE ENERGY STORAGE

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The development of safe and efficient electrochemical energy storage devices requires a deep understanding of the thermodynamic behavior of electrolytes. Ionic liquids (ILs), particularly Ethylammonium Nitrate (EAN) and its mixtures with inorganic salts (Li^+ , Mg^{2+} , Ca^{2+} , Al^{3+}), are prominent candidates due to their low volatility and wide liquid range. However, for practical applications, their confinement into solid-like structures such as ionogels via sol-gel synthesis (formic acid and ethanol routes) is increasingly relevant.

This work presents a comprehensive thermal and calorimetric study of EAN-based systems. Differential Scanning Calorimetry (DSC) was employed to determine transition temperatures and phase behavior, revealing how salt concentration and silica confinement influence the glass transition (T_g) and crystallization processes. The thermal stability of the synthesized ionogels was validated through Thermogravimetric Analysis (TGA), ensuring their suitability for high-temperature operations.

A key thermodynamic highlight is the determination of solution enthalpies using high-precision isoperibol calorimetry (LKB 8700), providing insights into the ion-solvent interactions and the solvation energy of multivalent cations in EAN. Furthermore, the environmental impact of these electrolytes was assessed via isothermal microcalorimetry (TAM III), monitoring the metabolic heat flow of soil microorganisms to evaluate ecotoxicity at different trophic levels. These calorimetric insights, combined with transport property analysis, establish a robust pathway for the design of sustainable and thermally stable electrolytes for the next generation of batteries and supercapacitors.

Keywords: Differential scanning calorimetry, thermal stability, ionic liquids

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SUSTAINABLE AQUEOUS ELECTROCHEMICAL ENERGY-STORAGE SYSTEMS BASED ON PRUSSIAN BLUE/BIOCHAR COMPOSITE ELECTRODES AND INORGANIC FUNCTIONAL ADDITIVES

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The development of safe, low-cost, and environmentally sustainable energy-storage technologies requires new electrode materials and aqueous electrochemical systems with reduced environmental impact. In this work, a zero-waste aqueous electrochemical system based on Prussian blue, wheat-grain-derived biochar, and inorganic electroactive additives was investigated. Composite electrodes were prepared using biochar as a carbonaceous component and Prussian blue combined with MnO_2 or FeS_2 as cathode materials. A sodium alginate/sodium sulfate aqueous electrolyte–binder phase was applied to provide a sustainable alternative to conventional organic-based electrode processing.

The influence of cathode composition and electrolyte modification using glycerol or natural halloysite was evaluated through complementary electrochemical and spectroscopic techniques, including galvanostatic charge–discharge measurements, cyclic voltammetry, Tafel polarization analysis, electrochemical impedance spectroscopy, and FT-IR spectroscopy. The results demonstrated that the FeS_2 -containing system without additional electrolyte modifiers exhibited the most favorable electrochemical performance, combining the highest discharge capacity, stable cycling behavior, and moderate charge-transfer resistance. In contrast, glycerol addition increased polarization and impedance, leading to reduced cycling stability. Halloysite showed a stabilizing effect by limiting voltage increase during operation, although it decreased the achievable capacity.

Among the investigated materials, the FeS_2 -modified Prussian blue electrode exhibited the highest electrochemical performance, reaching a maximum discharge capacity of approximately 0.157 mAh and maintaining approximately 0.148 mAh after 21 cycles. The improved behavior was associated with favorable charge-transfer characteristics and stable voltage profiles. The incorporation of glycerol negatively affected electrochemical kinetics by increasing impedance and limiting ion transport. Halloysite, acting as a structural mineral modifier, improved voltage stability but introduced additional limitations in low-frequency charge transport.

The obtained results confirm the potential of combining bio-derived carbon materials, Prussian blue, and abundant inorganic compounds for the development of sustainable aqueous energy-storage systems. The study highlights the importance of controlling electrode composition and electrolyte structure to optimize charge transport and operational stability.

Keywords: sustainable energy storage; Prussian blue; biochar; aqueous battery; FeS_2 ; MnO_2 ; electrochemical characterization

STRUCTURAL CHANGES OF ACID GEOPOLYMERS INDUCED BY THERMAL LOAD

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Phosphoric acid-activated metakaolin (or another aluminosilicate precursor) belongs to a growing class of aluminophosphate binders, hereafter referred to as silico-alumino-phosphate geopolymers (SAP), which combine phosphate species with aluminosilicate precursors to form mixed inorganic networks. These materials are supposed to be chemically and thermally stable [1, 2] however there is lack of systematic knowledge in this field [3]. To fulfill this gap, two types of thermally activated clays (kaolin and mixed clay with content of kaolinite, illite and quartz) were used as SAP precursors. The behaviour of these two SAP under thermal load (up to 1200 °C) was studied firstly by thermogravimetry and DSC. There the dehydration taking place at about 150 °C was observed as just a single process with mass loss. The freshly prepared SAP are generally amorphous matters, composed from hydrated Al-O-P and Si-O-Si networks. The dehydration takes place spontaneously and slowly even at ambient temperature, but the 150 °C heating has accelerated this process. The dehydration led to crystallization of SiO₂ polymorphs (cristobalite, tridymite). Further the residual mechanical properties were tested at 200, 400, 600, 800, 1000 and 1200 °C and the effect of thermal loading on structural evolution was examined by XRD and solid state MAS NMR. The material retained its shape and integrity but the strength gradually decreased, while structure of the material rearranged towards higher content of crystalline phases, isostructural with SiO₂ polymorphs.

Keywords: acid activated geopolymer; silico-alumino-phosphate geopolymer; thermal load; NMR spectroscopy

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INFLUENCE OF AMORPHOUS TiO₂ ADDITIVE ON THE SYNTHETIC GYROLITE STABILITY UNDER ISOTHERMAL CURING AT 200 °C TEMPERATURE

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Due to the large interplanar distance between the [SiO₄]⁴⁻ tetrahedra forming its chains, ions of many chemical elements can be incorporated into the crystal structure of gyrolite. This property allows for the modification of its properties and broadening of its application possibilities [1-3]. Previous studies have shown that the additive of amorphous TiO₂ affects the formation of the intermediates and their recrystallisation into the final synthesis product, gyrolite [4]. Therefore, it remains unclear whether this additive can directly penetrate into the synthetic gyrolite structure.

In order to assess this, the aim of this work was to determine the influence of amorphous TiO₂ additive on the stability of the synthetic gyrolite structure under hydrothermal conditions

The raw mixture corresponding to the gyrolite stoichiometry (CaO and SiO₂ in a molar ratio of 0.66) was mixed with water and hydrothermally treated for 48 h at a temperature of 200 °C. From 5 to 20% of the additive was added to the obtained synthesis products and the samples were additionally hydrothermally treated for 24 h.

Compared to the products of the single-stage 72 h synthesis, the crystallinity of the gyrolite synthesized under the conditions of this study was higher. The results of XRD analysis were confirmed by STA data: in the DSC curves of the products with the additive do not show any effects characteristic of amorphous TiO₂ crystallization into anatase (~450 °C) or rutile (~600-700 °C). In the synthesis products with the additive, an exothermic effect was observed in the range of 830-860 °C, which characteristic of crystallization processes. To determine the nature of the final thermal effect and to evaluate the composition of the products formed during the heat treatment, the synthetic samples were calcined at 900 °C for 1 hour.

Keywords: gyrolite, thermal stability, amorphous TiO₂.

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TEXTILES, PLASTICS, BIOBASED POLYMERS AND CHEMICAL RECYCLING

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Sustainable and high-performance polymers can be produced by combining and utilizing both bio-based and recycled materials. By carefully choosing the monomers (bio-based or from recycled plastics and textile fibres), the properties of these polyesters and polyamides can be tuned for applications ranging from textile fibres to packaging applications, thereby reducing our dependence on fossil-based polymers. Plastics are an integral part of modern life. The annual polymer production in the world reached 430 Mt in 2024 and is expected to increase to 1230 Mt by 2060.^{1,2} Polyolefins make for roughly 58 % of the annual production; whereas poly(ethylene terephthalate) (polyester, PET) is roughly 6 %; and polyamides, polyurethanes, and polystyrene account together for the rest 26%. It should be noted, that polyesters makes for 57% of the annual textile fiber production.³

Substituting existing fossil-based polymers and plastics with biobased alternatives is very attractive and necessary for future sustainability. However, incompatibilities of biobased polymers and materials with existing recycling streams are of concern. Mechanical recycling is currently the main recycling method of plastics. However, the materials degrade as they are processed into new products. In chemical recycling, e.g. polyesters are depolymerized into monomers or oligomers by hydrolytic reactions. The obtained monomers and oligomers can then be repolymerized.

2,5-furandicarboxylic acid (FDCA) is a versatile bio-based monomer. The bio-based polyester poly(ethylene furanoate) (PEF) is compatible with PET⁴ and they can be processed and copolymerized together. PET and PEF may also be chemically recycled together, yielding in materials with similar properties to virgin materials.⁵ Soft and 3D-printable copolyesters from PEF can be obtained by copolymerization with long-chain aliphatic diols.⁶ Similarly copolyamides can be obtained from FDCA by copolymerization of diamines with varied long-chain diamines.⁷

Keywords: fibers, plastics, biobased polymers

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INVESTIGATION OF THERMOMECHANICAL ANALYSIS OF COMPOSITE SOLID ROCKET PROPELLANT

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Composite solid rocket propellants are widely used in modern types of military munitions as well as civilian amateur rocketry. In case of military applications, the rocket motors are required to maintain their properties during long-term storage. Investigating thermo-mechanical properties of solid rocket propellants is essential in terms of their safe use. The presented research concentrates on testing composite solid rocket propellant obtained from sustainer motor of anti-aircraft missile with the use of dynamic mechanical analysis (DMA) and thermo-mechanical analysis (TMA). The selected samples of the propellant were additionally subjected to artificial ageing. The glass transition temperature region was determined with the use of Netzsch DMA 303 Eplexor device in the temperature range from -120°C to 100°C and heating rate of 2K/min. Different frequencies of applied force were used to present the shift in mechanical properties results. Relative expansion and coefficient of thermal linear expansion, were determined with the use of Netzsch TMA 402 F3 Hyperion in the temperature range from -100°C to 100°C with heating rate 2K/min. The experimental procedures with the use of DMA and TMA methods were conducted on the basis of relevant NATO STANAG agreements. The work outlines the importance of glass transition and thermal expansion analysis of solid rocket propellants in terms of their safe use in military and civilian applications and preventing serious accidents during extended service life.

Keywords: double-base solid rocket propellants, thermo-mechanical analysis, glass transition

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Oral Presentations 3

FROM VISCOSITY TO CRYSTAL GROWTH: KINETIC PROCESSES IN CHALCOGENIDE GLASSES

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Chalcogenide glasses represent an important class of amorphous materials owing to their unique optical and electrical properties, including high infrared transparency and high refractive index. These properties make them promising candidates for applications in infrared optics, photonics, optical fibers, and phase-change memory technologies. Since transport and crystallization processes strongly influence the performance and long-term stability of such materials, understanding the relationship between viscosity, diffusion, and crystal growth kinetics is essential for both fundamental research and technological applications.

This work focuses on the investigation of viscosity behavior, crystallization kinetics, and self-diffusion processes in both bulk and thin-film samples of several chalcogenide glass systems¹⁻³. Viscosity measurements were performed using thermomechanical analysis, nanoindentation, and the pressure-assisted melt filling technique, covering a broad viscosity range from the glass-transition region to the melt.⁴ The obtained viscosity data were described using phenomenological models such as the Mauro–Yue–Ellison–Gupta–Allan (MYEGA).⁵

Crystal growth kinetics was investigated using ex situ and in situ optical microscopy techniques, particularly infrared microscopy, which enabled direct observation of crystal growth in chalcogenide glasses. The measured crystal growth rates were analyzed using interface-controlled kinetic models, together with the decoupling parameter ξ , which describes deviations from the Stokes–Einstein relation between viscosity and diffusion.⁶ The analysis revealed significant differences in crystallization behavior among the studied systems, including polymorphic crystal growth, competitive crystallization processes, and varying degrees of decoupling between viscous flow and diffusion-controlled crystal growth.

The presented results provide a broader understanding of transport and crystallization phenomena in chalcogenide glasses and demonstrate how direct crystal growth measurements can be used to gain insight into the self-diffusion mechanisms governing structural rearrangements in amorphous materials.

Keywords: Chalcogenides, Crystallization, Kinetics, Viscosity, Diffusion

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DIELS-ALDER-BASED POLYMER NETWORK DESIGN: STRUCTURE, REACTIVITY, PROCESSING AND PROPERTY RELATIONS

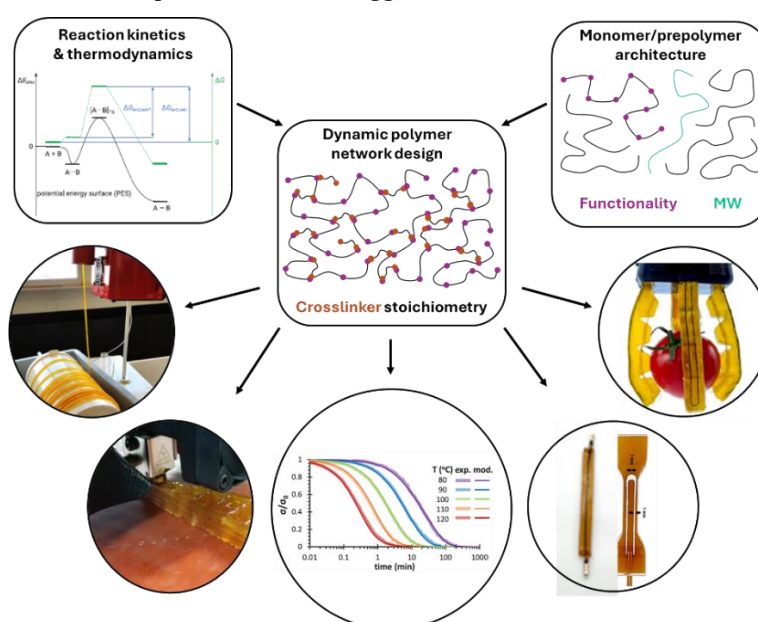
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The macroscopic behaviour of dynamic polymer networks can be designed very versatile through in-depth knowledge of the dynamic character of the dynamic covalent chemistries and design of the polymer network structure. The choice of the dynamic chemistry and the concentration of the reactive groups determine the chemical reaction kinetics, while the prepolymer architecture, molecular weight and functionality determine how the chemical dynamics translate into macroscopic physical behaviour in response of external triggers.



The combination of structure-reactivity [1] and structure-property [2] relations enables simulating the chemical and physical behaviour under processing conditions and during application. Mechanistic models relate the gel point and gel transition temperature to the reaction kinetics and thermodynamics, and the viscoelastic behaviour as a function of temperature and shear conditions to the prepolymer architectures. Several orders of magnitude difference in shear behaviour and stress relaxation [3] can be achieved by changing the prepolymer molecular weight, functionality and architecture, or the addition of fillers [4] without significant changes in mechanical stiffness and self-healing performance.

Keywords: Diels-Alder, reversible cycloaddition, self-healing polymers, dynamic covalent chemistry

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EXPLORING HIDDEN RELATIONSHIPS IN DSC DATA OF RECYCLED POLYETHYLENE TEREPHTHALATE THROUGH MULTIVARIATE STATISTICAL ANALYSIS

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The increasing demand for sustainable plastic waste management highlights the need for reliable methods for assessing the quality of recycled plastics. Recycled polyethylene terephthalate (rPET) exhibits considerable variability in its properties due to differences in feedstock composition, processing history, and degradation level. Differential scanning calorimetry (DSC) is widely used to characterise the thermal behaviour of these materials; however, interpretation often relies on individual thermal parameters, which may overlook complex relationships within the dataset. In this study, DSC was used to characterise a set of industrially recycled PET samples. Thermal parameters associated with glass transition, melting, and crystallisation were extracted from the DSC curves, and after appropriate data preprocessing, subjected to multivariate statistical analysis. Pearson correlation analysis was used to evaluate relationships among selected thermal parameters, while principal component analysis (PCA) was applied to identify the main sources of variability and reveal latent structures within the dataset. The results showed that several DSC parameters are strongly interrelated and form distinct groups associated with crystallisation and melting behaviour. PCA reduced the dimensionality of the dataset and enabled clear visualisation of sample similarities and differences, revealing clustering of samples according to their estimated degree of degradation and highlighting thermal characteristics relevant to material quality assessment. The proposed approach provides a more comprehensive interpretation of DSC data than evaluation of individual parameters alone and offers a practical framework for classifying unknown rPET samples. This study demonstrates how multivariate statistical tools can enhance the interpretation of DSC datasets by transforming multiple thermal descriptors into a comprehensive basis for material classification and quality assessment.

Keywords: recycled polyethylene terephthalate, differential scanning calorimetry, multivariate statistical analysis, principal component analysis, quality assessment

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PROCESSING-INDUCED THERMAL AND MOLECULAR CHANGES IN RECYCLED POLYSTYRENE/LIGNIN SYSTEMS

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Repeated melt processing of recycled polystyrene (rPS) can induce molecular degradation and consequently alter its thermal behaviour. [1] The incorporation of lignin as a bio-based additive may additionally influence the response of rPS during melt mixing. [2] In this work, the combined effects of processing conditions and lignin content on the molecular and thermal properties of rPS/lignin systems were investigated. Recycled polystyrene (rPS) and rPS/1 mass% lignin blends were prepared under various melt-mixing conditions, including different screw speeds and mixing times, while blends containing higher lignin contents (5 and 10 mass%) were prepared under selected processing conditions. Neat rPS processed under identical conditions was used as a reference to distinguish the effects of processing from the effect of lignin addition. The materials were characterized using gel permeation chromatography (GPC), differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and dynamic mechanical analysis (DMA) in order to correlate molecular degradation with thermal and thermomechanical behaviour. The results showed that prolonged melt mixing, and higher speed led to a reduction in molecular weight accompanied by changes in thermal response, indicating processing-induced degradation of rPS. Results indicate that lignin may partly mitigate molecular degradation during melt processing, while its addition also increases chain mobility and stiffness, as reflected in lower glass transition values and higher storage modulus. The study highlights the strong interdependence between melt-processing conditions, molecular degradation, and thermal properties in recycled polystyrene/lignin systems.

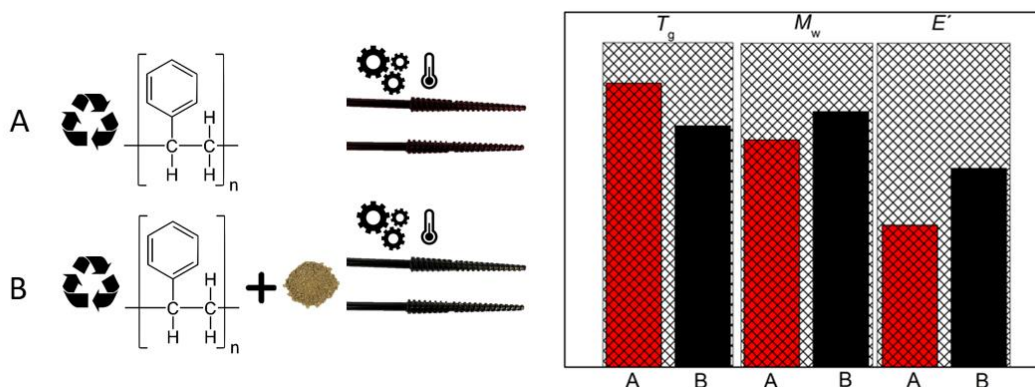


Figure 1. Schematic overview of changes in thermal, molecular, and thermomechanical properties in recycled polystyrene (A) and recycled polystyrene/lignin (B) systems.

Keywords: thermal analysis, processing, recycled polymers, lignin, molecular changes

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COMPREHENSIVE CHARACTERIZATION OF ENVIRONMENTAL MICROPLASTICS AND THEIR DEGRADATION PRODUCTS: A COMBINED PYROLYSIS AND UHPLC APPROACH

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The accelerating accumulation of plastic debris in the global biosphere demands advanced analytical methodologies capable of tracking both the physical presence and the complex chemical transformation of these ubiquitous contaminants. Once released into natural ecosystems, microplastics undergo continuous weathering driven by solar radiation, mechanical stress, and microbial activity. This environmental aging leads to severe fragmentation and the subsequent generation of intricate degradation products, including structural oligomers, oxidized fragments, and leaching chemical additives. Conventional analytical techniques often fall short in monitoring these processes, as they typically focus either on bulk polymer identification or small-molecule profiling, thereby failing to capture the full spectrum of plastic degradation and its dynamic interaction with the surrounding matrix.

This study addresses this critical methodological gap by establishing a powerful, complementary analytical framework that seamlessly integrates analytical pyrolysis with ultra-high performance liquid chromatography (UHPLC). The innovative combination of these two distinct techniques allows for a parallel investigation: pyrolysis destabilizes the macromolecular polymer core via controlled thermal degradation to identify the parent material, while UHPLC offers molecular-level profiling of soluble degradation products, chemical additives, and mobile transformation pathways. By analyzing both the solid polymer matrix and its liquid-soluble degradation fractions, this dual-platform approach overcomes the traditional limitations caused by sample weathering, biofouling, and complex environmental interferences that usually obscure spectral data.

The overarching significance of this research lies in its unique ability to provide a comprehensive, holistic overview of the entire lifecycle and chemical evolution of environmental microplastics. While pyrolysis ensures the highly reliable identification of heavily degraded, severely weathered, and structurally obscured plastic matrices, the UHPLC platform offers critical, unprecedented insights into the specific chemical signatures of polymer aging and the simultaneous release of potentially toxic compounds into ecosystems. Applying this integrated methodology to environmental samples successfully demonstrates its utility in distinguishing between intact primary polymer structures and secondary transformation products formed in situ. Ultimately, this research bridges the historical gap between material characterization and environmental toxicology. By providing a deeper, more accurate understanding of how plastics degrade, transform, and migrate through natural systems, this newly established framework substantially enhances the precision of environmental monitoring and provides essential, comprehensive data to support robust ecological risk assessments and future global regulatory policies.

Keywords: microplastics degradation, analytical pyrolysis, UHPLC-MS, ecological risk assessment

Acknowledgements:

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FROM GREEN CHEMISTRY TO SUSTAINABLE ENERGY: PHYSICOCHEMICAL PERSPECTIVES ON ADVANCED MATERIALS AND ENVIRONMENTAL SYSTEMS

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The continuous development of the global energy market, the depletion of conventional natural resources (including crude oil and natural gas), and the increasing demand for energy require the exploration of new, sustainable and environmentally friendly energy sources. Among the most promising alternatives are biofuels and hydrogen, which are widely considered as key energy carriers for future low-carbon technologies.

A new approach in environmental engineering is based on the integration of green chemistry principles with the design of environmentally compatible chemical systems and advanced functional materials. This interdisciplinary strategy enables the development of innovative solutions aimed at sustainable energy production, storage, and environmental protection.

A literature survey conducted using the SciFinder Scholar database demonstrated significant scientific interest in these emerging research areas. Searches performed using keywords such as “catalytic decomposition of biomass”, “biofuels”, “hydrogen storage” and “green batteries” identified more than 5000 scientific publications, including approximately 200 studies focused specifically on environmentally friendly materials and sustainable chemical systems.

The presentation will summarize the main research directions developed by our group within the Environmental and New Materials Research Laboratory, focusing on the physicochemical characterization and application of advanced materials for environmental and energy-related technologies. Current research activities are concentrated on four major areas:

1. Innovative applications of starch- and cereal-based materials in environmental engineering, including their use as collectors of heavy metal ions, soil stabilizers, components of drilling fluids, and renewable resources for biofuel production.
2. Development of catalytic ceramic materials containing lithium and transition metal ions and investigation of their catalytic properties in the thermal decomposition and conversion of biomass-derived systems.
3. Functionalization of carbon-based nanomaterials, including multi-walled carbon nanotubes, fullerenes, and graphene, with organophosphorus anions derived from selenium- and sulfur-containing acids as well as organic disulfide derivatives, with the aim of developing advanced electrode materials for lithium-ion batteries.
4. Design of advanced hydrogen storage systems based on novel ultra-light lithium alloys and hybrid materials combining carbon nanotubes with lightweight lithium-based materials.

The presented research highlights the role of physicochemical studies, green chemistry concepts, and advanced materials engineering in developing innovative technologies for sustainable energy generation, storage, and environmental applications.

Keywords: Multi-walled carbon nanotubes, hydrogen storage system, environmental engineering processes, electrode materials, thermal analysis of biomass.

TEMPERATURE DEPENDENT RHEOLOGY AS A VERSATILE TOOL FOR THE ASSESSMENT OF PROFESSIONAL DETERGENTS

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There is increasing interest in biodegradable professional washing formulations which are skin safe and don't accumulate in the ecosystem [1]. One of the main components of washing formulations are surfactants, whose class representatives are sodium dodecyl sulphate and sodium benzenesulphonate. Despite their cleaning power, these chemicals are being replaced by more biodegradable and eco-friendly alternatives based on sulphonates, sarcosinates and glucosides which all possess different surface chemistry and are formulated differently based on their physical properties and cleaning power [2].

The aim of this study is to systematically analyse and compare formulations based on three types of anionic surfactants: sarcosinate, glucoside and sulphonate. Temperature dependent rheology was employed for formulations consisting mainly of anionic surfactants, non-ionic surfactants, hydrotropes, humectants, chelators and water in order to determine the flow type (Newtonian or non-Newtonian) and the dependence of viscosity on the formulation temperature which has significant use in application. Furthermore, dilute water solutions of anionic surfactants were also examined in order to study the molecular interactions of different types of surfactants. Critical micellar concentration was determined as a function of viscosity and is also varied with temperature and shear rate in order to fully assess the effect of external factors such as temperature and shear stress/rate on application parameters (contact surface temperature and jet ejection parameters). Moreover, further efforts have been made to characterize the micelles each surfactant forms relative to their molecular structure based on experimental data (Huggins constant, intrinsic viscosity, linear viscoelasticity). Such a broad examination of surfactant systems via rheology, where multidimensional results can be obtained, displays the versatility of this method has for the assessment of detergent formulations.

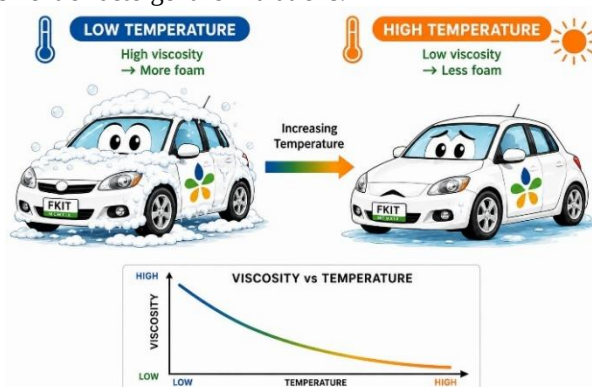


Figure 1. Schematic representation of the influence of viscosity on washing parameters.

Keywords: temperature dependent rheology, surfactant, micelles

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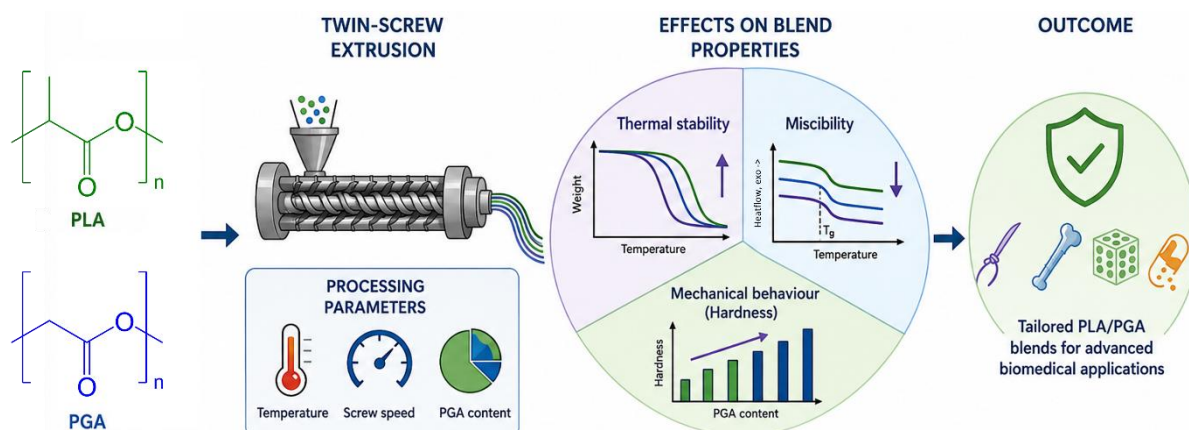
EFFECT OF PROCESSING CONDITIONS ON THE DEGRADATION, MISCIBILITY, AND MECHANICAL BEHAVIOUR OF PLA/PGA BLENDS PREPARED BY TWIN-SCREW EXTRUSION

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Poly(lactide) (PLA) and poly(glycolide) (PGA) are biocompatible polyesters extensively used in medical applications, including resorbable sutures, orthopaedic implants, tissue engineering scaffolds, and drug delivery systems [1,2]. In this work, the effects of twin-screw extrusion parameters, namely temperature, screw speed, and blend composition, on the thermal properties and hardness of PLA/PGA blends were systematically examined. A Box–Behnken design of experiments was employed, and the resulting materials were analysed using a combination of thermal, mechanical, imaging, and chromatographic techniques. The findings indicate that processing conditions (screw speed and mixing temperature) significantly affect thermal stability and degradation. While the hardness of pure PLA is unaffected across processing conditions, blends with higher PGA content showed increased sensitivity to these parameters. The addition of PGA to the PLA matrix improved thermal stability, enthalpy, and hardness, while reducing the glass transition temperature. Overall, this study establishes guidelines for tailoring PLA/PGA blend properties through controlled processing for targeted biomedical applications.



Keywords: polyglycolide, polylactide, differential scanning calorimetry, thermogravimetric analysis, Box–Behnken design

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FROM PHASE-CHANGE TO STIMULI-RESPONSIVE SYSTEMS: THE ROLE OF THERMAL ANALYSIS AND CALORIMETRY IN ENGINEERING PICKERING WAX-BASED MICROPARTICLES

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Pickering-engineered wax microparticles have recently emerged as versatile platforms for the development of multifunctional soft materials, combining structural integrity, interfacial stability and advanced physicochemical functionalities. In this contribution, we present the evolution of a research activity devoted to the design of wax-based microparticles stabilized by inorganic nanoparticles, highlighting how thermal analysis and calorimetric techniques have provided the fundamental tools for understanding, optimizing and tailoring their properties for different applications.¹⁻³

The first generation of these materials consisted of beeswax microparticles stabilized by halloysite nanotubes through a solvent-free Pickering emulsification strategy. Their incorporation into geopolymeric matrices generated sustainable composite materials exhibiting enhanced thermo-mechanical performances and latent heat storage capabilities. More recently, the concept has evolved toward stimuli-responsive microparticles in which hematite nanoparticles replace halloysite as Pickering stabilizers, yielding hybrid wax/hematite microparticles capable of magnetic guidance and visible-light-triggered catalytic activity.

Differential Scanning Calorimetry (DSC) has represented the key technique throughout the entire development process. Beyond the determination of melting and crystallization temperatures and latent heat, DSC has been employed to investigate the influence of nanoparticle adsorption on wax crystallization pathways and phase-transition reversibility.

Thermogravimetric Analysis (TGA) has complemented calorimetric investigations by providing quantitative information on the organic/inorganic composition of the microparticles, nanoparticle loading, degradation pathways and thermal resistance.

The combined thermal approach further provides valuable information for processing and long-term reliability. Thermal cycling experiments assess the durability of repeated phase transitions, while kinetic analysis of degradation contributes to defining safe operating windows for processing and application. Such information is essential for translating laboratory-scale Pickering formulations into technologically relevant materials.

Overall, this work demonstrates that calorimetry and thermal analysis are not merely characterization tools but constitute fundamental design methodologies for engineering multifunctional Pickering microparticles. The correlation between phase transitions, thermal stability, interfacial organization and functional performance opens new perspectives for the rational design of responsive materials for energy storage, smart coatings, catalysis, and environmental remediation.

Keywords: Wax, Microparticles, Remediation, Cultural Heritage, Smart nanomaterials

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BIOACTIVE PCL COMPOSITES WITH EUCALYPTUS LIGNOCELLULOSIC RESIDUES FOR HORTICULTURAL APPLICATIONS

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The extensive use of non-biodegradable plastics in agricultural applications has raised increasing environmental concerns due to their persistence in soil, accumulation of plastic waste, and generation of microplastic contaminants that may negatively affect soil health and ecosystem functions [1–3]. In this context, biodegradable composites represent a promising strategy for the development of sustainable materials with reduced environmental impact and added functionality [4].

In this work, bioactive composites based on poly(ϵ -caprolactone) (PCL) and eucalyptus-derived lignocellulosic residues were developed for potential horticultural applications, such as biodegradable inserts and seedlings pots. Composites containing 20, 40, and 60 wt% lignocellulosic biomass were prepared using both untreated eucalyptus residues and biomass after essential oil extraction with the aim to evaluate the role of natural extractives (e.g., essential oil) on both the composite material properties and bioactivity.

The structural, thermal, and mechanical properties of the composites were investigated by thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), tensile testing, and micro-computed tomography (μ -CT). Moreover, the materials were subjected to preliminary soil biodegradation tests in order to simulate real horticultural application conditions and to assess the possible effect of residual eucalyptus essential oils on the biodegradation behavior of the materials. At different time of soil exposure, the samples were analysed in term of morphology, molecular weight by gel permeation chromatography (GPC), and thermal stability.

The obtained results provide useful insights into the development of sustainable bioactive composites derived from agroforestry residues, for the production of biodegradable and environmentally friendly plastic items to be used in variety of horticultural applications.

Keywords: biodegradable composites, poly(ϵ -caprolactone), eucalyptus lignocellulosic residues, soil biodegradation, horticultural applications.

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GEOPOLYMERIC MATERIALS BASED ON HALLOYSITE NANOTUBES FOR ENVIRONMENTAL APPLICATIONS

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Geopolymers are promising inorganic binders and alternative cementitious materials due to their high mechanical performance, durability, and reduced environmental impact in comparison with Portland cement.

In this work, we studied geopolymeric materials obtained by the alkaline activation of halloysite clay nanotubes (HNTs) through sodium hydroxide as well as wood ash lye, which represents a sustainable and low-cost alternative for the synthesis of geopolymers. The effects of the activator (wood ash lye or sodium hydroxide) and curing temperature (25 and 50 °C) on the structural, textural, mechanical, and durability-related properties of the geopolymeric materials have been investigated. The curing temperature in the range of 25-50 °C did not induce marked structural changes of the geopolymeric materials. Nevertheless, geopolymers cured at 50 °C exhibited better properties, suggesting that moderate thermal curing plays a key role in improving geopolymer performances. Textural and morphological analyses revealed that geopolymerization leads to a densification of the halloysite structure, accompanied by a reduction of specific surface area and pore volume. As compared with the conventional NaOH-activated geopolymers, the materials prepared by ash lye possess different pore size distribution and macroporosity.

Geopolymeric films based on activated HNTs were investigated by several thermal methods (TGA, DMA, DSC) allowing to control their efficiencies for heat barrier action and remediation purposes [1]. Porous geopolymers exhibited excellent CO₂ capture capacity that was detected by thermogravimetric experiments under CO₂ atmosphere. Composite geopolymeric films can be obtained by alkaline activation of halloysite mixed with hydroxypropyl cellulose [2]. Fire exposure tests evidenced that composite geopolymers are flame resistant, while the presence of HNTs (before geopolymerization) retard the flame propagation reducing the burning rate in comparison with pristine biopolymer.

Geopolymerized beads formed by halloysite and alginate revealed high adsorption capacity for CO₂ and volatile organic compounds [3]. The actual geopolymerization of the gel beads was proved by TGA, while DMA experiments were conducted to evaluate the mechanical resistance of the beads under compression force.

The attained knowledge on geopolymers (films, beads, bricks) evidenced that the alkaline activation of halloysite nanotubes represents an efficient tool to obtain functional materials useful for environmental applications.

Keywords: Halloysite nanotubes, Geopolymers, Composite, Alkaline Activation, Environmental applications.

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THERMAL CHARACTERIZATION OF MONOSACCHARIDES-AMINO AIDS MODEL SYSTEMS

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The Maillard reaction is a highly complex network of thermally induced transformations between reducing sugars and amino compounds, responsible for the formation of desirable flavour and colour compounds as well as undesirable contaminants such as acrylamide and advanced glycation end products.^{1,2} Although numerous factors governing the reaction have been identified, the influence of organic acids and antioxidants on the thermal behaviour of low-moisture model systems needs to be further investigated. Thermal analysis offers a powerful approach for investigating these interactions by directly monitoring heat flow and mass changes associated with reaction progression.

In this work, model systems composed of reducing monosaccharides (glucose, fructose and mannose), amino acids (asparagine, alanine, cysteine and phenylalanine), gluten, selected organic acids (citric, malic and tartaric acid), ascorbic acid and Trolox were investigated under low water activity conditions. Thermogravimetric analysis (TGA) was employed to monitor temperature-dependent mass changes, while differential scanning calorimetry (DSC) was used to characterize heat-flow profiles during scanning and isothermal treatment. Fourier-transform infrared spectroscopy (FTIR) complemented the thermal analyses by monitoring structural changes induced during heating.

The combined application of TGA, DSC and FTIR proved to be an effective strategy for detecting subtle differences in reaction onset, thermal stability and reaction progression within complex Maillard model systems. The results demonstrate that complementary thermal analysis provides mechanistic insight into the influence of additives on thermally induced reactions and represents a valuable tool for designing food-processing strategies that minimize the formation of undesirable Maillard reaction products while preserving technological and sensory quality.

Keywords: Maillard reaction, sugar-amino acid model systems, DSC, TGA

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THERMODYNAMIC AND ENZYMATIC ASSESSMENT OF SOIL MICROBIAL COMMUNITIES UNDER LONG-TERM IRRIGATION WITH PLASMA ACTIVATED WATER

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With the increasing global challenges in agricultural production, innovative methods such as Plasma Agriculture are gaining significant attention. Plasma Activated Water (PAW), characterized by the presence of reactive oxygen and nitrogen species (RONS), has shown potential in stimulating plant growth and enhancing seed germination. However, the long-term impact of PAW on the soil environment – specifically on the functional state of microbial communities – remains poorly understood [1-3].

In this study, we investigated the complex response of soil microbial communities across four different soil types following a 90-day irrigation period of *Allium schoenoprasum* and *Solanum lycopersicum* with PAW. The PAW was generated using a high-frequency pin-hole discharge with air bubbling (2-minute exposure). To assess microbial status, soil enzymatic activities (dehydrogenase and urease) were measured and complemented by isothermal microcalorimetry (IMC), a powerful tool for monitoring microbial metabolic heat flow.

From the measured power-time curves, key thermodynamic and kinetic parameters were determined, including maximum normalized heat flow (P_{max}), the time to peak (t_{max}), total heat dissipation (Q), and the thermodynamic parameter (X_0). Furthermore, the microbial growth rate constant (k) was calculated by fitting an exponential growth model to the normalized heat flow data.

Preliminary results indicate that while PAW irrigation maintains baseline soil functions, the microbial growth kinetics (k and t_{max}) exhibit soil-specific responses to RONS accumulation, highlighting the importance of soil buffering capacity. This study demonstrates that isothermal microcalorimetry provides a more sensitive and dynamic perspective on PAW-soil interactions compared to static enzymatic assays, offering essential data for the safe implementation of plasma technologies in sustainable agriculture.

Keywords: microcalorimetry, plasma activated water, soil enzymes

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DIFFERENTIAL SCANNING CALORIMETRY FOR MONITORING TREATMENT RESPONSE IN MULTIPLE MYELOMA

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Differential scanning calorimetry (DSC) has emerged as a promising technique for characterizing the thermal stability of complex protein mixtures in biological fluids. Plasma thermograms reflect the cumulative thermal denaturation of the major plasma proteins and are altered by changes in protein composition and intermolecular interactions associated with disease. These disease-specific thermal signatures suggest that DSC may complement conventional laboratory methods for monitoring treatment response.^{1,2}

The objective of this study was to evaluate whether serial DSC measurements of blood plasma can be used to monitor treatment efficacy in patients with multiple myeloma (MM). Blood plasma samples were collected from newly diagnosed and relapsed MM patients before and during intensive therapy. DSC measurements were performed at regular intervals throughout treatment. Thermogram evolution was compared with routinely used biochemical markers, including monoclonal immunoglobulin (M-protein) and serum protein electrophoresis.

The results demonstrate that DSC is capable of detecting dynamic alterations in plasma thermal stability during treatment of multiple myeloma. The strong agreement between DSC thermograms and conventional biochemical markers indicates that thermal profiling may provide a rapid, label-free and minimally invasive complementary approach for monitoring therapeutic response. The study further highlights the potential of DSC for longitudinal characterization of complex biological systems and supports its broader application in clinical thermal analysis

Keywords: Differential scanning calorimetry, Blood plasma, Multiple myeloma, Thermal profiling

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CALORIMETRIC & PREDICTIVE METHODS TO CHARACTERISE PHARMACEUTICAL CO-AMORPHOUS MIXTURES

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Up to 90% of drugs in development are classified with poor aqueous solubility [1] which can negatively affect therapeutic efficacy. Amorphous drug formulations are appealing to enhance kinetic solubility due to their disordered structure and resulting higher free energy. However, the amorphous phase is unstable and tends to recrystallise into a lower energy state.

Co-amorphous mixtures (CAMs) are a homogeneous, single-phase of a drug and one or more low molecular weight coformers, which can be another drug or an excipient. The amorphous state can be stabilised by reducing molecular mobility (antiplasticisation), and/or increasing physical separation of alike components, and/or hetero-intermolecular interactions which need to break before rearrangement and neat component crystallisation [2].

Differential Scanning Calorimetry (DSC) is widely used to determine CAM glass transition temperatures (T_g) and assess stabilisation mechanisms. Traditionally, Gordon-Taylor (GT) modelling provides predicted values of CAM T_g from its neat components. Deviation from predictions suggest non-ideal mixing which can be a result of hetero-intermolecular interactions. Furthermore, the Schroeder van Laar equation can be used to predict the eutectic composition, where all components which form a CAM melt simultaneously at a lower temperature than their neat counterparts.

To this extent, three main topics will be discussed:

1. Mapping T_g , eutectic behaviour, and GT model deviation using ternary plots.
2. Design of experiments to screen the optimal ternary CAM ratio for physical stability.
3. Reverse modelling. Using stable CAM T_g values to predict the T_g of unstable amorphous drugs [3].

These topics push the boundary for what a typical CAM study entails and delivers a novel perspective on the use of predictive modelling beyond simple binary CAM T_g predictions. Experiences from method development of the CAM mixing and quench-cooling process will be discussed to open conversations with other scientists who are interested in pursuing similar work.

Keywords: Co-Amorphous Mixture(s), Differential Scanning Calorimetry, Phase Diagram(s), Gordon-Taylor, Schroeder van Laar.

Acknowledgements:

We are grateful to Osama Bin Abu Bakar and Harsh for conducting an initial study as a part of their MSc Thesis', which were foundational to aspects of this work. We express thanks to Sophie Janbon, Samuel Owusu-Ware, David Watson, and Daniela Carta for contributing their expertise as PhD co-supervisors. Parts of this work was funded by the Engineering and Physical Sciences Research Council (EPSRC) and AstraZeneca.

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DETERMINATION OF NUCLEATION TEMPERATURE OF PHARMACEUTICAL GLASSES

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Solid pharmaceutical dosage forms usually include active ingredients in a crystalline state. However, amorphous state is sometimes used for improving solubility of the compound, of which low physical stability can be an issue. As crystallization is initiated by nucleation, understanding and controlling the nucleation step is important for assuring physical stability of amorphous pharmaceuticals. Because direct experimental detection of nuclei is difficult, nucleation is typically assessed using indirect methods using microscopic observation or thermal analysis. Microscopic observation provides direct information on the overall crystallization process, whereas thermal analysis gives quantitative information on crystallization under precise environmental control.

We have identified nucleation temperatures of more than 20 compounds to find that they were located slightly above the glass transition temperature and/or well below that¹. Ibuprofen glass has a glass transition temperature at -45 °C. It does not crystallize during heating in differential scanning calorimeter; however, cold crystallization to form I was observed if it is annealed at nucleation temperature beforehand. The cold crystallization was the most enhanced when it was annealed at -15 °C, which was regarded as the optimum nucleation temperature². On the other hand, characteristic length of cooperatively rearranging region was determined as a function of temperature using relaxation data obtained by broadband dielectric spectroscopy. It was compared with calculated critical size of nuclei to find that they agree at ca. -15 °C. This result indicated that nucleation is enhanced, when sizes of critical nuclei and cooperatively rearranging region coincide². The same assumption was applied to celecoxib glass to find that the nucleation temperature to form I was ca. 60 °C. It agreed with the observation that storage of celecoxib glass at 60 °C caused crystallization to form I. Both compounds had another temperature range well below their glass transition temperatures to enhance crystallization to a different crystal form^{1,3}. It was likely to be controlled by local molecular mobility, i.e., based on a different mechanism of nucleation. These findings should be helpful to assume nucleation temperature of new candidate compounds.

Keywords: Pharmaceutical glass, nucleation temperature, crystallization, cooperatively rearranging region, molecular mobility

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Technical Lectures

USING A NOVEL MODE OF OPERATION OF TGA-GC/MS FOR TIRE WEAR PARTICLES (TWPs) ANALYSIS

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Detecting and quantifying tire wear particles (TWPs) in the environment poses a unique environmental challenge due to their chemical complexity. There are emerging concerns around TWPs due to their potential high numbers of particles released, outnumbering microplastics, as well as the leaching of toxic additives such as 6-PPD which has been linked to the death of salmon, even when present at very low levels ($<0.1\mu\text{g/L}$). [1,2] Conventional analytical techniques such as pyrolysis (Py-GC/MS) or thermal extraction-desorption (TED-GC/MS) gas chromatography have been used but pose limitations on sample mass and throughput with additional complexity to characterisation and quantification procedures. [3,4] The work presented here aims to demonstrate the use of the new approach that overcame the challenges above by utilising a coupling between thermogravimetric analysis (TGA) and gas chromatography-mass spectroscopy (GC/MS). The novel mode of operation, PyroTGA-GC/MS, uses fast heating to enable robust quantitative analysis of TWPs in road dust samples. [5] We are introducing a technique that provides an economically viable solution for large-scale commercial analysis of the tire rubber and particles.

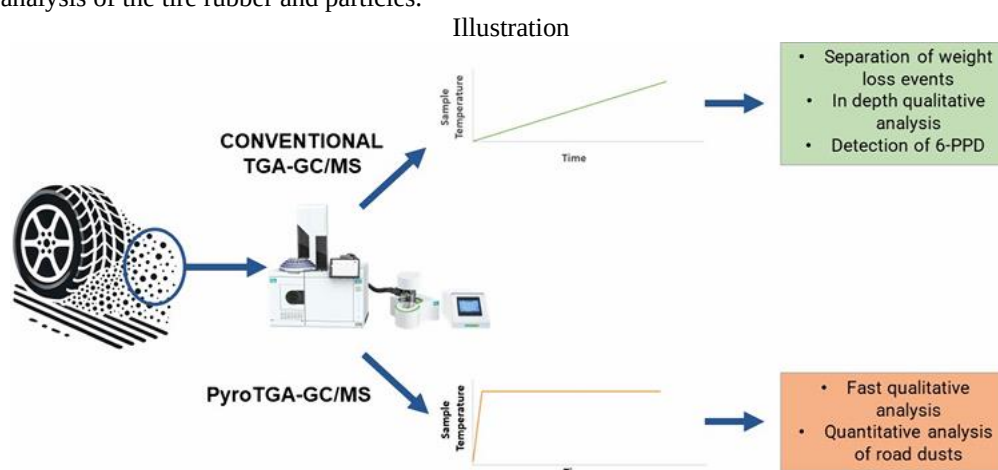


Illustration – Conventional vs. New novel approach to analysis and its benefits.

Keywords: tire wear particles, analytical chemistry, evolved gas analysis, TGA-GC/MS

Acknowledgements:

We thank Kieran Evans for the bulk of the work, Prof. Neil Ward for supplying the road dust samples, and various PerkinElmer colleagues (Nicholas Lancaster, Pamela Dickson & Tim Mann) for their valuable support and advice. We acknowledge the support of the UK Natural Environment Research Council (NERC) through the Capital Award for the Applied-RadioIsotope & Environmental Laboratory (ARIEL) (grant no. NE/V017616/1).

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RHEOMETRY AS A COMPLEMENTARY TECHNIQUE TO THERMAL ANALYSIS: A JOURNEY THROUGH TYPICAL APPLICATIONS

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Rotational and High Pressure Capillary Rheometry are widely used to study the flow and deformation behaviour of materials across a wide range of outer applied forces. These methods help to analyse processing behaviour, predict shelf life, monitor structural changes from a macroscopic point of view etc. However, the boundaries with thermal analysis are fluid [1]. Thermal degradation measured with Thermogravimetric Analysis TGA can also be analysed with Capillary Rheometry to monitor the same effect under real processing conditions and determine the additional impact of applied pressures or speeds. A phase transition measured with Differential Scanning Calorimetry DSC can also be analysed by oscillatory rheometry, accessing the mechanical properties of the material at rest [2]. For example, with DSC a glass transition of a Hotmelt adhesive can be determined, but in addition a temperature sweep in oscillatory shear using a rotational rheometer detects the same glass transition from a mechanical point of view and gives additional insights into elasticity and viscous damping of the polymer structure, see Figure 1. Aside of comparing results using thermal analysis and rheometry, the talk will provide a journey around the manifold applications of rotational and capillary rheometry for different types of materials.

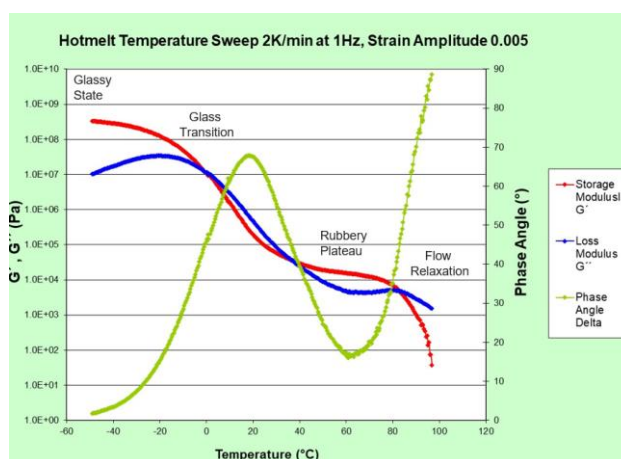


Figure 1. Temperature Sweep on a Hotmelt Adhesive measured in Oscillatory Shear

Keywords: Rotational Rheometry, Oscillatory Rheometry, Capillary Rheometry, Complementary Techniques, Thermal Analysis

References:

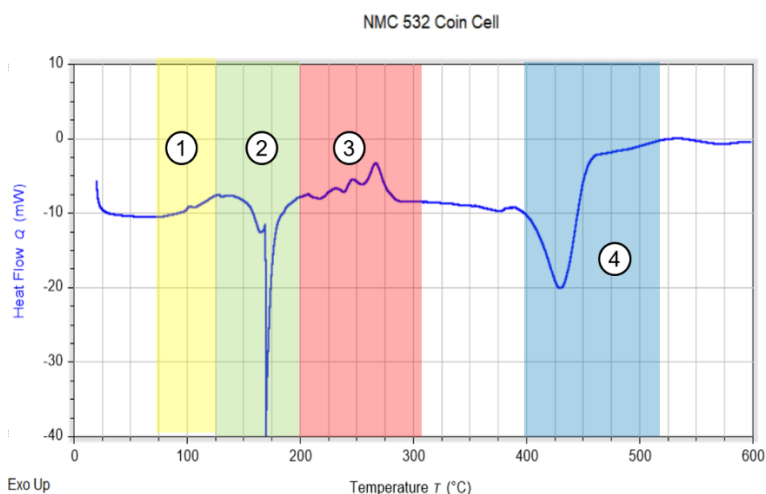
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CALORIMETRY AS A TOOL TO ASSESS BATTERY RUNAWAY REACTIONS

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The study of Thermal Runaway in Lithium-Ion Batteries (LIB's) especially as a function of the State Of Charge (SOC) is a fundamental step in LIB's development¹. Since most of the processes involved are strongly exothermic, Differential Scanning Calorimetry in sealed pans is a common technique for assessing the main parameters in terms of temperature of decomposition of the Solid Electrolyte Interphase (SEI) and heat associated to anode and cathode reactions². The sealed pans however prevent the possibility to study the process on a fully assembled system (coin shaped LIB) and to analyze the composition of the gases developed during the runaway. In this work, we present preliminary studies on a modified DSC system that can test coin shaped LIB's through the whole runaway with different possibilities of Evolved Gas Analysis and Electrochemical Analysis.



Figure/Table (if any)

Figure 1. The heat flux measured during different stages of the runaway process, namely: (1) SEI Reorganization and Decomposition (2) coin cell gasket and separator melting (3) electrolyte and cathode reactions (4) decomposition and combustion.

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FROM INSTRUMENT TO INSIGHT: APPLIED EXCELLENCE IN THERMAL ANALYSIS WITH HITACHI THERMAL ANALYZERS

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Thermal analysis has become an indispensable tool for material characterization, enabling the correlation of thermal behavior with composition, structure, processing, and performance across the entire product lifecycle. While analytical instrumentation continues to advance, the greatest value is achieved when technology is combined with application expertise and practical implementation.

This presentation introduces the AstrineXLab representation program, highlighting the capabilities of the Hitachi Thermal Analyzers portfolio through an application-driven perspective. An important part of the presentation is the Applied Excellence Laboratory concept, which combines advanced instrumentation with application-focused workshops, practical demonstrations, and collaborative knowledge transfer.

Selected case studies from polymer and pharmaceutical applications will illustrate the complementary use of differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and dynamic mechanical analysis (DMA) for comprehensive material characterization, thermal stability evaluation, formulation support, quality control, and routine analytical decision-making. Attention will be given to Dynamic mechanical analysis which provides additional insight into viscoelastic properties and temperature-dependent mechanical performance, offering valuable information for material selection, product development, and performance assessment.

The presentation aims to demonstrate how close collaboration between technology providers, application specialists, and laboratory users creates an effective pathway from instrumentation to practical analytical excellence, supporting academic innovation and sustainable laboratory performance across industrial sectors.

Keywords: thermal analysis, material characterization, Hitachi Thermal Analyzers, polymers, pharmaceuticals

Acknowledgements:

The support of RoVi Consultancy and Hitachi High-Tech Analytical Science, Europe, is gratefully acknowledged.

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AN APPROACH TO CHROMATIC THERMAL ANALYSIS (CHROMTA™) USING SAMPLE-OBSERVATION THERMAL ANALYSIS

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Visual changes in materials—such as variations in color, transparency, gloss, or turbidity—often precede or occur independently of distinct thermal event signals detected by conventional methods. In this application, we investigate Chromatic Thermal Analysis (ChromTA™), a complementary analytical approach that quantitatively integrates sample-observation thermal methods combined with image-based color evaluation, with particular emphasis on polymeric, organic, and pharmaceutical and formulated materials.

During thermal analysis measurements, time-resolved observation images are continuously acquired and converted into quantitative color representations, including RGB and CMYK, and, where appropriate, HSV and L*a*b*. The resulting color parameters are treated as temperature- and time-dependent numerical data and analyzed in full synchronization with the thermal characteristics. By defining multiple regions of interest on the sample surface, spatial non-uniformity and localized progression of changes can be evaluated in a reproducible and operator-independent manner.

Representative applications demonstrate characteristic chromatic responses associated with phenomena such as glass transition, early-stage crystallization, melting, phase transitions, decomposition, drying or desolvation, and discoloration related to degradation. Notably, continuous trends in color parameters are observed in temperature regions where conventional thermal signals remain indistinct, indicating that chromatic metrics can sensitively capture the onset and progression of underlying structural or physical changes.

Rigaku ChromTA is not intended to replace conventional thermal analysis; rather, it enhances interpretability by transforming visible changes into quantitative, synchronized data. This provides an additional evaluation axis directly linked to temperature, offering practical value in materials research, formulation development, process optimization, and quality evaluation. These examples highlight the versatility and effectiveness of ChromTA across different material types and its ability to detect early-stage transformations that may not be identified by conventional techniques.

Keywords: sample-observation thermal analysis, chromatic thermal analysis, polymers, pharmaceuticals, image analysis

REVOLUTIONARY DSC 5+ SETS NEW STANDARD IN RESEARCH WITH DIFFERENTIAL SCANNING CALORIMETRY

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Material science and market need to develop new materials is fast growing chemistry field and therefore also Thermal analysis have become important field for research. Differential Scanning Calorimetry is one of techniques which provide us relevant results. Mettler-Toledo developed new modern Differential Scanning Calorimeter DSC 5+ to measure, detect, evaluate and interpretate thermal events when material is expose to different temperature programs. Instrument allow to heat or cool samples with different heating rates or keep samples at isothermal conditions. New ceramic sensor which is built in DSC 5+ holds two technologies: technology based on Heat Flux or Power Compensation principle. We can easy detect very small effects with HeatFlux technology where are 136 Thermocouples built in¹ or with use of Power Compensation technology with 2 heaters and Pt sensors which keep temperature on a sample and reference side equal we are capable to determine cp values with an error less than 1%². This presentation will highlight technical details of new DSC 5+ and provide some application.

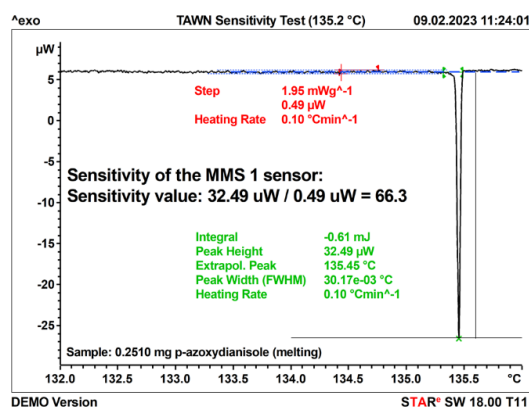


Figure 1. TAWN sensitivity of new DSC 5+.¹

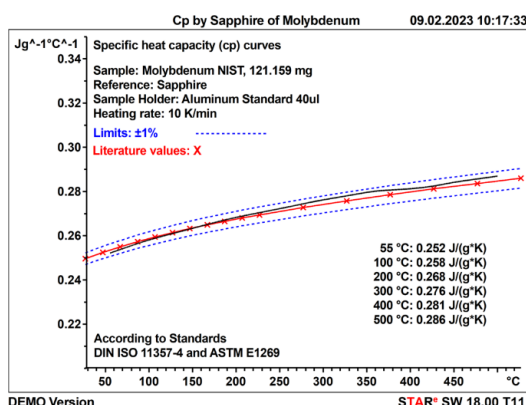


Figure 2. cp determination of Molybdenum with new DSC 5+.²

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Poster Session 1

THERMAL ANALYSIS OF THE PHASE BEHAVIOR IN THE HBTA:TOPO BINARY SYSTEM

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The development of deep eutectic solvents (DESSs) is a key issue for the realization of green and efficient metal extraction processes. The present study aims to experimentally construct the phase diagram of the binary system consisting of tri-n-octylphosphine oxide (TOPO) and 4,4,4-trifluoro-1-phenyl-1,3-butanedione (HBTA) and, thus, determine its eutectic composition for the solvent extraction of Li⁺. Differential scanning calorimetry was used to characterize the phase transitions (melting temperatures and enthalpies) over the entire composition range of the binary mixture. Its eutectic composition was established at HBTA:TOPO mass ratio of 60:40. For further validation of the eutectic composition from the experimentally measured thermal effects for melting of different HBTA:TOPO mass ratios, a Tammann diagram was also constructed. Only mixtures with HBTA:TOPO mass ratios of 70:30, 60:40 (eutectic composition), and 50:50 were liquids at 30 °C, while at room temperature of 25 °C, the 70:30 mixture formed crystals. All three mixtures, which were liquids at 30 °C, were found to extract Li⁺ effectively. However, at a room temperature of 25 °C, only the eutectic mixture (60:40 mass ratio) extracted Li⁺ effectively, while the mixture with HBTA:TOPO mass ratio of 50:50 formed crystals when mechanically agitated and, therefore, was deemed as unsuitable for Li⁺ extraction.

Keywords: deep eutectic solvent (DES); 4,4,4-trifluoro-1-phenyl-1,3-butanedione (HBTA); tri-n-octylphosphine oxide (TOPO); binary phase diagram; Li⁺ extraction

Acknowledgements:

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PYROLYSIS OF A MIXTURE OF BIOMASS AND PLASTIC IN A FIXED BED REACTOR

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Pyrolysis as a thermally driven decomposition of organic substances can be applied to variable waste materials. Research is usually focused on individual materials, but can also focus on mixtures of different materials to optimize yields and product quality. In order to investigate the possible synergistic effect of the joint pyrolysis of plastic and biomass, co-pyrolysis of these material mixtures was carried out in a laboratory-scale fixed-bed reactor. All experiments were carried out under previously defined fixed conditions (500 °C, 0.1 L/min; 45 min), i.e. conditions that contributed to the maximum yield of the liquid phase (pyrolytic oil) during the pyrolysis of pure plastic mixture. Blend of waste polypropylene 45%, low-density polyethylene 35%, and high-density polyethylene 25% by weight served as the plastic sample, while blend of spruce and beech sawdust served as the biomass sample, in the examined plastic/biomass mixtures. The following mass ratios in the mixtures were tested: plastic:biomass = 1:3; 1:1; 3:1. Recorded yields of liquid, gases and solid products were compared with the theoretically expected values. It was found that pyrolysis of a 3:1 mixture of plastics nad biomass leads to a synergetic effect in terms of increasing the yield of pyrolysis oil, while with other mixtures there was no synergistic effect observed, and the yield of both pyrolysis oil and the gas have been lower than the theoretical expected values. The mechanism of the synergistic effect that occurs during the copyrolysis of biomass and plastic has not been fully elucidated. However, it can be assumed that in the conditions of co-pyrolysis, the thermal craking of plastic takes place at a slightly lower temperature, due to the catalytic effect of the coke formed from the biomass and the carbon's participation in the radical mechanism of plastic degradation. In general, it can be concluded that the conditions of the co-pyrolysis process (both the appropriate choice of process parameters and the type of reactor in which the process is carried out), that is, the conditions of heat and mass transfer in the system, play a significant role in achieving a synergistic effect in the form of an increase in the yield of condensable or gaseous pyrolysis products.

Keywords: pyrolysis, co-pyrolysis, plastic/biomass mixture, sinergetic effect, fixed-bed reactor

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INFLUENCE OF MINERAL ADDITIVES AND WATER-REDUCING ADMIXTURE ON THE MICROSTRUCTURAL AND THERMAL PROPERTIES OF SELF-COMPACTING CEMENT MORTARS

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Along with satisfying aesthetic and decorative requirements for cement mortars, limitations are placed upon the use of expensive raw materials, which are reduced with the use of cement replacement materials. Inorganic inert materials or technogenic waste are used as such. Marble powder was used in this work as the inert materials. In addition, the water reducer as a polycarboxylate-based admixture is used to reduce the large volumes of water needed for the hydration of cement mortars. Due to superior water-reduction capabilities of these admixtures, the workability of the fresh mixes is improved.

The aim of this work is to study the influence of additives and fillers on the microstructure of cement mortars using a complex of analytical methods. The density of their structure at 28 and 120 days of water curing were evaluated by measurement of some physical-mechanical properties such as density, compressive strength and porosity. Powder XRD, FT-IR, SEM and Thermal analysis (TG-DTG-DSC-MS) were used to identify the crystal structures, morphology and thermal decomposition. The advantages of these analytical methods were used for determining the content of sulphate/carbonate-containing phases, the dehydration, decarbonation and dehydroxylation steps that define the thermal decomposition reaction mechanism and reveal the influence of additives, aggregates, and water-to-cement ratio.

Keywords: White Portland cement, self-compacting mortars, cement hydration, mineral additive

Funding:

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MODIFICATION OF THE STRUCTURAL CHARACTERISTICS OF NANOSIZED PHOSPHORITE VIA LONG-TIME MECHANOCHEMICAL PROCESSING

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Phosphorite is a phosphate-rich rock that is often used in agriculture as fertilizer, activator, and soil improver. Its value as a raw material and its diverse applications necessitate more effective processing methods, one among which is mechanochemical activation. This work studies the changes occurring at the structural level within the phosphorite ore upon prolonged mechanochemical processing. Their effect on the phosphorite's end properties is also investigated. Building on previous findings that structural defects require processing times exceeding 30 min, this study extends activation to 150 min. The activation time is kept the same for all samples, while the size of the Fe grinding bodies used during activation varies between 3, 5, 10, and 20 mm.

The results indicate the occurrence of both anionic and cationic isomorphism, leading to the formation of multiple apatite minerals - A-B type carbonate-apatites and hydroxyl-fluorapatite from the initial francolite in the raw ore. Solid-phase synthesis of pyrophosphates and tricalcium phosphates was also observed, along with a reduction in the crystallite size of the initial minerals to nanosize. The changes in the chemical activity ($P_2O_5^{ass}/P_2O_5^{total}$) and constituent element contents (F/Ca/P) were subsequently analyzed. The chemical analysis results present the increase in phosphorus mobility (as P_2O_5 in assimilable form) in a 2% citric acid from 44% to 50%, calculated as $P_2O_5^{ass}/P_2O_5^{total}$, compared to 21.46% in untreated phosphate rock. The samples were studied via chemical analysis, powder X-ray diffraction (PXRD) and Rietveld refinement, Wavelength Dispersive X-ray fluorescence (WD-XRF) analysis, Fourier transform infrared spectroscopy (FTIR), and thermal analysis coupled with mass spectrometry of evolved gases (TG/DTG-DSC-MS).

The induced structural changes enhance the phosphorite's potential as a raw material due to the increased solubility of the newly-formed phases and improved chemical reactivity.

Keywords: phosphorite ore; activation time; apatite isomorphism, solid-phase synthesis; crystallite size

Funding:

This work was supported by European Regional Development Fund under "Research Innovation and Digitization for Smart Transformation" program 2021-2027 under the Project BG16RFPR002-1.014-0006 "National Centre of Excellence Mechatronics and Clean Technologies"

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SYNTHESIS AND CHARACTERIZATION OF Cr⁴⁺ DOPED Li₂ZnGeO₄ GLASS-CERAMIC

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The subject of this work is Cr⁴⁺ doped Li₂ZnGeO₄ transparent glass-ceramics preparation with wide emission in the near IR region. Up to now Cr⁴⁺ doped Li₂ZnGeO₄ was synthesized only by solid state reaction. Li₂ZnGeO₄: Cr⁴⁺ glass-ceramic was synthesized for a first time. A systematic investigation was made in the system Li₂O-ZnO-GeO₂-B₂O₃ (B₂O₃ was 20 mol %) to select the optimal initial glass composition and the optimal thermal treatment conditions for obtaining the required glass-ceramic. The results of the nucleation and crystallization of nanophases in this multicomponent system are presented and discussed. XRD, DSC, TEM and other methods were used for characterization of the obtained glass-ceramics. The conducted systematic studies have shown that to obtain glass-ceramics with desired characteristics is necessary to study different compositions of starting glasses and choose the optimal one among them, which will allow suitable conditions for homogenization of the amorphous glass, obtaining the nanophase with the desired composition with the highest possible concentration of the nanophase. Optimization of the conditions of subsequent thermal treatment on the base of DSC analysis allow obtaining evenly distributed nanoparticles with sizes below 30 nm, so as to preserve the transparency of the glass-ceramics to the highest possible concentration of the nanophase. The mean size of the nanoparticles as the weight ratio between the glass and nanophase into the glass-ceramic was determined by XRD analysis. The distribution of the particles into the glass and the structure of the phase was confirmed by HRTEM analysis.

The obtained Cr⁴⁺ doped Li₂ZnGeO₄ glass-ceramics has a phase concentration of up to about 50% while preserving the transparency of up to 60% with a nanoparticle size of up to 20 nm. The glass-ceramics shows a wide emission range from 1000 to 1500 nm with a center around 1220 nm and a lifetime of 79-96 ns. This desired combination of a broad emission spectrum and a long lifetime is due to the structural features of the phase representing a crystal field with a Dq/B close to the cross-current of the ³T₂ and 1E energy levels being 1.7. Such good characteristic possesses a few silicates, but preparing of glass-ceramics from them is enough more complicated. The suitable conditions for preparation, and obtained characteristic make this ceramic perspective for application.

Keywords: Cr⁴⁺ doped Li₂ZnGeO₄ glass-ceramics, thermal behavior, characterization

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THERMAL AND CHEMICAL CONDITIONS FOR $\text{Li}_2\text{CaGeO}_4$ GLASS-CERAMIC SYNTHESIS

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The main goal of this research was to find a suitable starting glass composition and suitable conditions for its thermal treatment for preparing of Cr^{4+} doped $\text{Li}_2\text{CaGeO}_4$ glass-ceramics as an active medium for laser radiation in the range 1.1-1.6 μm . We studied the crystallization of glasses from the four-component system $\text{Li}_2\text{O}-\text{CaO}-\text{GeO}_2-\text{B}_2\text{O}_3$ after thermal treatment and determined the crystallization area of $\text{Li}_2\text{CaGeO}_4$ for three sections of the system with B_2O_3 contents of 5.0, 7.5, and 10.0 mol.%. The crystallization kinetics in the glass and the formation of different phases were investigated experimentally, depending not only on the composition of the initial glass, but also on its thermal treatment regime. X-ray powder diffraction, DSC, EPR, TEM and UV-VIS analysis were used for characterization of the glass-ceramics. From the systematic studies carried out in the four-component system $\text{Li}_2\text{O}-\text{CaO}-\text{GeO}_2-\text{B}_2\text{O}_3$, several conclusions can be drawn:

1. The crystallization region of $\text{Li}_2\text{CaGeO}_4$ is relatively narrow, localized around the stoichiometric composition of the $\text{Li}_2\text{CaGeO}_4$ compound.
2. The presence of the glass former B_2O_3 strongly supports the formation of homogeneous glasses at relatively low temperatures. However, it is also the reason for the crystallization of borate phases.
3. The crystallization of glasses during thermal treatment is related to the production of more than one phase and it is correct to speak for crystallization of a main phase and accompanying crystallization of other phases.

As a result of the conducted research, an optimal composition of the starting glass and an optimal mode of thermal treatment were selected. Glass-ceramic with a main phase $\text{Li}_2\text{CaGeO}_4$ doped by Cr^{4+} was obtained. The obtained glass-ceramic contains up to 60 mass% of $\text{Li}_2\text{CaGeO}_4$, with an average nanoparticles size of 20 nm and with transparency about 70 % relative to the transparency of the parent glass. The glass-ceramic show wide region of luminescence from 1000 to 1500 nm with centred at 1220 and 36 ms lifetime which are very good parameters for laser applications. Up to now only two silicate glass ceramic are known with such characteristic

Keywords: glass-ceramics, $\text{Li}_2\text{CaGeO}_4$, laser radiation

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SYNTHESIS, STRUCTURES, THERMAL DECOMPOSITION MECHANISM, AND LUMINESCENCE PROPERTIES OF SM(III), PR(III), ND(III), AND LA(III) COMPLEXES WITH DIVERSE CARBOXYLATE COORDINATION MODES

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A series of four novel lanthanide complexes was obtained: $(\text{Ln}(\text{3-MBA})_3(5,5'\text{-DM-2,2'-bipy}))_2 \cdot (\text{H}_2\text{O})$ ($\text{Ln} = \text{Sm}$ (1), Pr (2), Nd (3)) and $(\text{La}(\text{3-MBA})_4(5,5'\text{-DM-2,2'-bipy}))_2$ (4) (3-MBA = 3-methylbenzoate 5,5'-DM-2,2'-bipy = 5,5'-dimethyl-2,2'-bipyridine). Single-crystal X-ray diffraction structural studies established that complexes **1-3** crystallize in the triclinic system with the space group $P\bar{1}$ and the metal center adopts an nine coordinate contiguration. In contrast, complex **4** crystallizes in the monoclinic with space group $P2_1/n$. Detailed crystallographic data indicate that in complexes **1-3**, the 3-MBA ligands bind to the Ln^{3+} ions through diverse coordination modes, specifically exhibiting chelating bidentate, bridging bidentate, and bridging tridentate modes. Surprisingly, there are eight 3-MBA ligands in the structure of complex **4**, which adopt monodentate, bridging bidentate, and bridging tridentate coordination modes, which is very rare in the study of similar complex structures. A suite of complementary techniques, including elemental analysis, IR and Raman spectroscopy, and PXRD, was employed to characterize the complexes. The thermal decomposition behavior of all four complexes was studied using a coupled simultaneous TG-DSC/FTIR/MS system. The results indicate that the complexes initially lose crystal water or neutral ligand molecules at relatively low temperatures, followed by the stepwise elimination of organic carboxylate ligands at higher temperatures. The final thermal decomposition products were identified as the corresponding lanthanide metal oxides. Additionally, the fluorescence spectrum of complex **1** was measured. DFT calculations reveal an energy transfer mechanism between the triplet states of both ligands and the Sm^{3+} ion.

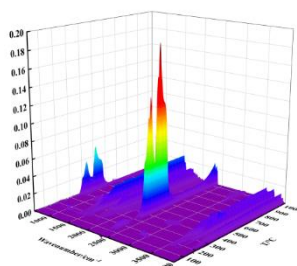


Figure 1. 3D infrared spectra of evolved gases from complex **1**

Keywords: Crystal structure, lanthanide complexes, thermal analysis, fluorescence, TG-DSC/FTIR/MS

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HIGH-ENTROPY ALLOY AS COUNTER ELECTRODE FOR TRIIODIDE REDUCTION IN DYE-SENSITIZED SOLAR CELLS

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Abstract: The CoFeNiCuZn high-entropy alloy(HEA) as counter electrode (CE) catalysts of dye-sensitized solar cells (DSSCs) were synthesized through a pycnolysis-alloying process. This process utilized a precisely controlled metal precursor composition and an atomically ratio-regulated Co-metal-organic framework (MOF) as both structural template and sacrificial precursor. Used the same preparation strategy, quaternary (CoNiCuZn), ternary (CoNiCu), and binary (CoCu) alloy systems were successfully synthesized, which can be an effective strategy for improving the power conversion efficiency (PCE). In this study, DSSCs with the four different alloy catalysts yield PCEs of 7.44% (CoFeNiCuZn), 6.18 % (CoNiCuZn), 5.85 % (CoNiCu), and 5.26 % (CoCu). The excellent performance can be ascribed to the high entropy and the “cocktail” effect of HEA, resulting from the generation of a larger number of active sites due to changes in the microelectronic and geometric structures.

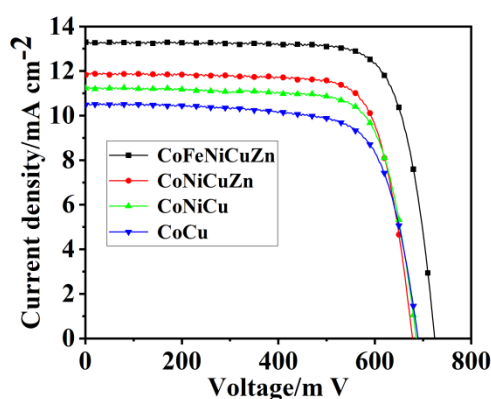


Figure 1. J-V curves of DSSCs using four different catalysts as CEs

Keywords: High-entropy alloy; Counter electrode; Dye-sensitized solar cells; Power conversion efficiency

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FIRE-INDUCED THERMAL HAZARDS IN ELONGATED RESTRICTED ENCLOSURES: FLAME IMPINGEMENT, SMOKE TRANSPORT, AND THERMAL STRATIFICATION

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Fire-induced thermal hazards in elongated restricted enclosures differ fundamentally from those in open or weakly confined spaces. When a buoyant fire plume interacts with the nearby ceiling and sidewalls, geometric confinement suppresses ambient air entrainment, promotes flame extension and ceiling impingement, and channels hot gases along the enclosure, resulting in intensified flame-boundary interaction, elevated ceiling temperatures, and persistent thermal stratification.

A series of Froude-scaled experiments was conducted in elongated enclosures with systematically varied heights, widths, cross-sectional aspect ratios, and opening conditions. Within this experimental matrix, the fire source was varied in elevation and in its transverse and longitudinal positions, while enclosures open at both ends and those closed at one end were examined over a broad range of heat release rates. The experiments characterized flame height and ceiling flame extension, temperature distributions, and smoke propagation under three characteristic regimes: a buoyant plume without ceiling impingement, intermittent ceiling flame impingement, and continuous ceiling flame impingement.

The results showed that restricted air entrainment increased flame extension and the probability of ceiling impingement, thereby intensifying local thermal exposure. Once ceiling flame impingement occurred, sidewall confinement promoted the longitudinal spread of flames and hot gases, producing a longer flame extension and slower temperature decay than in the transverse direction. The longitudinal flame extension exceeded the transverse extension by 43% for centerline sources and by 27% for wall-adjacent sources. An effective heat release rate accounting for fire source elevation, transverse location, and ceiling clearance collapsed the directional flame-length data onto a unified $2/5$ -power scaling. By introducing an effective heat release rate that accounts for fire source elevation, transverse location, and ceiling clearance, a unified correlation was established, showing that the directional flame length is proportional to the $2/5$ power of the dimensionless effective heat release rate.

Enclosure geometry also exerted a pronounced influence on thermal stratification. In the respective test series, reducing the enclosure width or increasing the cross-sectional width-to-height ratio resulted in a thicker thermal layer. Normalization by the Gaussian thermal thickness collapsed the vertical temperature distributions obtained under different geometries and heat release rates onto a unified dimensionless profile. Opening asymmetry further altered the plume and ceiling-temperature response. In one-end-closed configurations, asymmetric air supply induced a unidirectional flow, deflected the plume toward the closed end, and produced three distinct thermal-response regimes: end-unaffected, end-affected, and end-wall-dominated.

These results provide a quantitative basis for estimating ceiling flame extension, thermal-layer development, and peak thermal exposure in elongated restricted enclosures, with direct relevance to thermal-hazard assessment and fire-protection design.

Keywords: Geometric confinement, Flame impingement, Ceiling flame extension, Thermal stratification, Fire safety

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THERMODYNAMIC INSIGHTS INTO INTERFACIAL REACTIONS IN THE TiO₂ NANOPARTICLE/NaCl/POLYELECTROLYTE SYSTEM

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Calorimetry is a powerful experimental method for investigating solid/liquid interfacial processes; however, it requires a meticulously designed experimental setup [1,2]. In suspensions of metal oxide colloidal particles, various surface processes occur upon altering the pH and ionic strength, or by adding inorganic salts, small organic molecules, or macromolecules like polyelectrolytes [3]. Understanding the protonation and deprotonation of charged surface groups, as well as the adsorption of ions or molecules, is crucial for diverse applications related to the surface properties of colloids [4].

Titanium dioxide nanoparticles are commonly utilized as additives in various consumer goods, ranging from dietary products to personal care cosmetics, and are frequently modified with various polyelectrolytes. This study investigates the interaction between TiO₂ nanoparticles and polyelectrolytes to enhance the application of TiO₂ particles across various fields, including pharmaceutical, cosmetic, and food industry. In this work, calorimetric experiments were designed and conducted to examine the processes at the TiO₂/aqueous sodium chloride solution interface, alongside the adsorption of the polyelectrolytes PSS and PDADMAC onto TiO₂ nanoparticles. In addition to calorimetry, acid-base potentiometric titrations and adsorption measurements were performed. The effects of pH and ionic strength on the enthalpies of surface reactions of both TiO₂ nanoparticles and polyelectrolytes were examined. The resulting data were analysed to obtain valuable insights into the interactions within this complex system, which critically influence the stability of TiO₂ nanoparticle suspensions.

Keywords: TiO₂, polyelectrolytes, calorimetry, surface reaction, adsorption

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MOLECULARLY IMPRINTED ZnO TETRAPOD PHOTOCATALYST: SYNTHESIS AND SELECTIVE DICLOFENAC DEGRADATION

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The increasing presence of pharmaceutical residues in aquatic environments has become a major environmental concern due to their persistence and potential adverse effects on ecosystems and human health. Among these contaminants, diclofenac (DIC) is one of the most frequently detected pharmaceuticals in wastewater treatment plant effluents because of its widespread consumption and limited removal by conventional treatment technologies [1]. In this study, molecularly imprinted polymer (MIP)-modified ZnO tetrapods were developed as selective photocatalytic materials for the degradation of diclofenac in aqueous solutions [2]. To evaluate the specific contribution of molecular imprinting, non-imprinted polymer (NIP)-coated ZnO tetrapods were also synthesized and used as control materials, while pristine ZnO tetrapods served as a reference photocatalyst.

The prepared materials were comprehensively characterized in terms of their structural, morphological, surface, and optical properties using field-emission scanning electron microscopy (FE-SEM), Brunauer–Emmett–Teller (BET) surface area analysis, X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and diffuse reflectance spectroscopy (DRS).

Photocatalytic degradation experiments were conducted in a batch reactor using DIC as a model pharmaceutical pollutant under UV-A and simulated solar irradiation. The performance of MIP-coated, NIP-coated, and pristine ZnO tetrapods was systematically compared to evaluate the effects of molecular imprinting on adsorption behavior, target selectivity, and photocatalytic degradation efficiency. To determine the photocatalytic degradation mechanism, scavenger tests were performed using selective radical quenchers. The results provide insight into the synergistic effects of molecular imprinting and ZnO tetrapod photocatalysis and highlight their potential for the selective removal of pharmaceutical contaminants from water under both ultraviolet and solar irradiation conditions.

Keywords: photocatalysis, ZnO tetrapods, molecularly imprinted polymer, wastewater treatment

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WATER DIFFUSION BEHAVIOR IN GLASS FIBER-REINFORCED POLYESTER HYBRID COMPOSITES CONTAINING JAROSITE WASTE PARTICLES

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Industrial metallurgical activities generate substantial quantities of hazardous by-products that pose significant environmental risks. One such waste material is jarosite, a heavy-metal-bearing residue produced during the hydrometallurgical extraction of zinc from sphalerite ore. Jarosite contains potentially leachable elements, including toxic heavy metals such as Pb, Zn, Cd, and As, as well as elevated concentrations of iron, all of which may contribute to environmental contamination. The utilization of jarosite waste as a filler in polymer composites represents a sustainable approach to waste valorization and resource recovery.

The aim of this study was to investigate the water absorption and diffusion behavior of glass fiber-reinforced polyester hybrid composites containing jarosite particles. Prior to composite fabrication, a detailed physicochemical characterization of the jarosite waste was performed to determine its chemical composition, phase composition, morphology, and particle characteristics. Four composite laminates were fabricated using the hand lay-up technique with an unsaturated polyester resin matrix reinforced by five layers of woven glass fiber fabric and filled with 0, 5, 10, and 20 wt.% jarosite particles. The composite without jarosite was used as the reference material.

Water diffusion coefficients were determined through long-term immersion tests conducted in deionized water at 25 °C [1,2]. Moisture uptake was monitored gravimetrically as a function of the square root of immersion time until equilibrium saturation was reached. The experimental sorption curves showed good agreement with the Fickian diffusion model during the initial stage of water uptake for all composite formulations. The results indicated that the diffusion coefficient decreased with increasing jarosite content, suggesting that the incorporation of jarosite particles influences the moisture transport behavior of the composites.

Keywords: jarosite waste, hybrid composites, water diffusion, diffusion coefficient, Fickian diffusion

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EFFECT OF HEATING AND COOLING RATES ON THE SOLIDIFICATION BEHAVIOUR OF AN Al-Li-Mg ALLOY

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Microstructure development in hypoeutectic aluminium-lithium-magnesium (Al-Li-Mg) alloys involves the solidification of a primary α_{Al} dendritic network, the formation of the metastable Al_3Li (δ') phase and the subsequent development of Li- and Mg-containing intermetallic phases. Depending on the Li/Mg ratio and thermodynamic conditions, the expected phase development may include AlLi (δ), Al_2LiMg (T), $\text{Al}_{12}\text{Mg}_{17}$ (γ), and Al_8Mg_5 (β) phases. High Li/Mg ratios promote the formation of AlLi (δ), while lower Li/Mg ratios favour the formation of the Al_2LiMg (T) phase, the peritectic $\text{Al}_{12}\text{Mg}_{17}$ (γ) phase and the eutectic (α_{Al} + Al_8Mg_5 (β)) phase [1]. Previous calorimetric studies of Al-Li-Mg alloys have shown that increasing heating and cooling rates broaden the temperature intervals associated with the formation and dissolution of Al_3Li (δ') and AlLi (δ) phases [2]. At lower cooling rates, the complete solidification sequence can occur. Cooling rates above approximately 12 °C/s suppress the precipitation of the Al_2LiMg (T) phase in Al-Li-Mg alloys [3].

This study evaluates the influence of heating and cooling rates on phase formation in a hypoeutectic Al-Li-Mg alloy with a high Li/Mg ratio. The chemical composition was determined by inductively coupled plasma mass spectrometry (ICP-MS). Equilibrium and non-equilibrium calculations were performed using Thermo-Calc 2022a to predict the solidification sequence. Differential scanning calorimetry (DSC) was used to investigate the influence of heating and cooling rates of 2, 10, 20, 30, 40, and 50 K/min on the solidification behaviour of the alloy. The alloy contained 2.16 wt.% Li and 0.38 wt.% Mg (Li/Mg = 5.68). The hypoeutectic composition promotes primary α_{Al} dendrite solidification, followed by Al_3Li (δ') and AlLi (δ) phase formation [1]. Equilibrium and non-equilibrium calculations predicted similar solidification sequences, with primary α_{Al} dendritic network development at approximately 653 °C, followed by AlLi (δ) phase formation. Non-equilibrium solidification calculations indicated a shift in the formation temperature of AlLi (δ) to higher values compared to equilibrium conditions. DSC analyses showed that heating and cooling rates influence solidification behaviour through changes in the solidification interval and characteristic temperatures. Increasing the heating rate raised the temperatures associated with the dissolution of Li-rich phases from approximately 314–357 °C to 417–434 °C. The heating rate also increased the melting interval from approximately 620–651 °C to 698–704 °C. Increasing the cooling rate shifted the onset of solidification to lower temperatures and broadened the solidification interval from 618–644 °C to 541–640 °C, indicating increased undercooling. The increased undercooling observed at higher cooling rates indicates a higher driving force for nucleation compared to lower cooling rates. These observations are consistent with previous studies [2,3] and confirm that heating and cooling rates affect the solidification behaviour of Al-Li-Mg alloys with high Li/Mg ratio.

Keywords: Al-Li-Mg alloy, solidification sequence, heating and cooling rates, phase formation temperatures

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FRAGILITY IN GE-SE GLASS-FORMING SYSTEM

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The concept of normalized viscosity data, which forms the basis for the definition of fragility, was first introduced by Oldekop [1] almost 70 years ago. Since its introduction, this concept has been widely adopted and further developed in materials science, particularly through the work of Professor Austen Angell [2]. The most frequently used fragility parameter is the steepness index m , defined as the slope of the dependence in the normalized Arrhenius (also called Angell) plot. This plot shows the dependence of the logarithm of viscosity on the reduced temperature (the viscosity glass transition temperature T_{12} divided by temperature). Despite the widespread use of the steepness index, its limitations, stemming from the complex nature of viscosity behavior and the simplifications inherent in its determination, have also been pointed out [3].

One of the widely discussed phenomena in materials science related to fragility is the fragile-to-strong crossover (abbreviated as FTS or F-S). This phenomenon, first identified by Ito et al. [4] based on calorimetric measurements, is typically defined as the manifestation of dual fragility in a given material. In essence, a glass-forming system exhibiting an FTS crossover behaves as a strong liquid near the glass transition but displays significantly more fragile characteristics at higher temperatures in the melt. The detection of the FTS crossover is often based on indirect evidence. This is primarily because systems exhibiting such transitions are typically poor glass formers, which prevents the preparation of bulk samples and makes it difficult to determine their viscosities in the glass and undercooled melt region using conventional techniques. Nevertheless, the FTS crossover has also been reported in several sufficiently good glass-forming systems. One of them is the Ge-Se system. Reports describing the FTS crossover in this system are supported by direct viscosity measurements performed in the undercooled melt region using the penetration method and in the melt region using the oscillating cup (vessel) method. Nevertheless, it has been pointed out that the oscillating cup method may potentially yield inaccurate viscosity values due to inadequate accounting for the wetting behavior of the measured liquid on the crucible walls [5]. Based on these possible discrepancies, Zhu et al. [6] questioned the presence of the FTS crossover in the Ge-Se system based on their own viscosity measurements. Our viscosity measurements [7], performed over a broad viscosity range spanning 13 orders of magnitude using the penetration, parallel-plate, and PAMFT methods, confirmed that the previously reported FTS crossover in the Ge-Se glass-forming system was likely the result of inaccuracies introduced by the oscillating viscometry method.

Keywords: viscosity, fragility, chalcogenides, FTS crossover

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EFFECT OF PYROLYSIS CONDITIONS ON PORE STRUCTURE EVOLUTION IN SOFT-TEMPLATED CARBON XEROGELS

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Hierarchically porous resorcinol-formaldehyde xerogels prepared by sol-gel polycondensation in the presence of soft template Pluronic F127 are promising precursors for porous carbon materials. During pyrolysis, decomposition of the soft template, carbonization of the polymer network and framework shrinkage modify the resulting pore structure, particularly within the mesopore range. The aim of this contribution was to investigate the influence of pyrolysis temperature and heating rate on the evolution of the pore structure in soft-templated carbon xerogels using thermogravimetric analysis and DSC thermoporometry.

Originally developed materials [1] were pyrolyzed under a nitrogen atmosphere (100 mL min^{-1}) at final temperatures of 400, 500, 700 and 900 °C using heating rates of 1, 5, 10 and 20 K min⁻¹ with an isothermal step for 1 hour. Thermal decomposition during pyrolysis was monitored using TG analysis, while the porosity of the resulting carbon xerogels was investigated by DSC thermoporometry and nitrogen physisorption.

Thermogravimetric analysis showed that increasing the heating rate shifted the maximum decomposition rate towards higher temperatures and resulted in sharper DTG peaks, reflecting the kinetic nature of the thermal decomposition process. The major mass loss event occurred between approximately 340 and 410 °C for all heating rates, whereas gradual mass loss continued up to the final pyrolysis temperature. DSC thermoporometry revealed systematic changes in the melting behaviour of confined water in carbon xerogels prepared under different pyrolysis conditions, indicating modifications of the pore structure, particularly in the mesopore range.

The results indicate that both pyrolysis temperature and heating rate influence the evolution of the pore structure in soft-templated carbon xerogels, with the final pyrolysis temperature exerting the more pronounced effect under the investigated conditions.

Keywords: soft-templated xerogels, porosity, heating rate, pyrolysis temperature

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APPLICABILITY OF CZECH KAOLINITE-ILLITE CLAYS IN THE DESIGN OF THE LC3 BINDER

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This paper investigates the applicability of Czech kaolinite–illite clay in LC3 (Limestone Calcined Clay Cement) binder systems. LC3 materials have attracted considerable interest as a low-carbon alternative to traditional Portland cement, since up to 50% of Portland clinker can be replaced by a blend of calcined clay and limestone. An additional advantage of LC3 technology is the potential utilisation not only of pure kaolinitic clays, but also of lower-grade and mixed clays. [1, 2]

Within the experimental program, the calcination conditions of the clay were first optimised. Subsequently, several mixtures containing varying amounts of calcined clay (LC), corresponding reference mixtures with inert fine silica sand (LS), and a pure Portland cement mixture (PC) were prepared. Preliminary mechanical testing revealed that mixtures designed using an equal-consistency approach exhibited superior performance, whereas mixtures prepared with equal water dosage showed approximately 36% lower compressive strength. The mixtures with equal consistency were further characterised in terms of heat evolution during hydration, as well as phase composition using simultaneous thermal analysis (STA) and X-ray diffraction (XRD). The heat evolution curves indicated secondary reactions associated with the presence of calcined clay, together with a more continuous progression of hydration reactions. Furthermore, STA and XRD analyses confirmed the reactivity of the studied clay and revealed the formation of carboaluminate phases accompanied by an increase in amorphous content.

Overall, the results confirm that the studied Czech kaolinite–illite clay is suitable for use in LC3 binder systems.

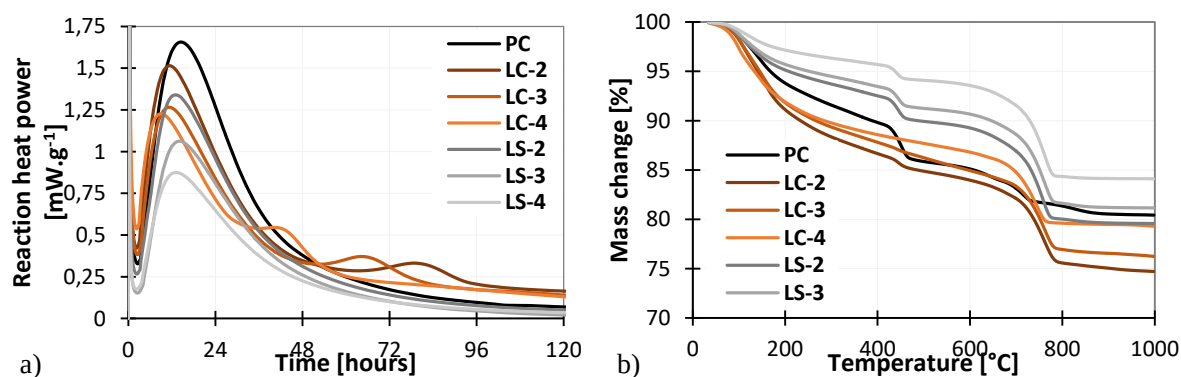


Figure 1: a) results of isothermal calorimetry, b) results of thermogravimetry

Keywords: kaolinite-illite clay, thermal activation, LC3 binder

Acknowledgements:

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KINETICS OF CO₂ ADSORPTION ON SURFACE-MODIFIED POROUS CARBONS I – ISOTHERMAL EXPERIMENTS

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The development of efficient adsorbents for carbon dioxide capture requires a comprehensive understanding of the effect of surface chemistry on adsorption kinetics. In this work, the kinetics of CO₂ adsorption on several surface-modified hierarchically porous carbons were investigated using isothermal thermogravimetry at temperatures of 15, 25, 35, and 45 °C.

A porous carbon exhibiting a hierarchical pore structure composed of micropores, mesopores (approx. 4 - 10 nm), and macropores [1] was employed as the parent material. Besides the pristine carbon (MMM), five surface-modified variants were prepared to tailor the surface chemistry using melamine treatment (MMM-mel), nitration (MMM-nitr), nitration followed by reduction (MMM-nitr-red), oxidation (MMM-ox), and oxidation followed by amination (MMM-APTES). The prepared materials were characterized by nitrogen physisorption to determine their textural properties and by zeta potential measurements to assess surface charge characteristics.

CO₂ adsorption experiments were subsequently performed under isothermal conditions using thermogravimetric analysis at the selected temperatures. The obtained adsorption curves were analyzed to determine kinetic parameters. A generalized kinetic model was fitted to the experimental data using nonlinear regression implemented through the Solver utility in MS Excel, enabling the estimation of the activation energy, pre-exponential factor, and reaction model parameters.

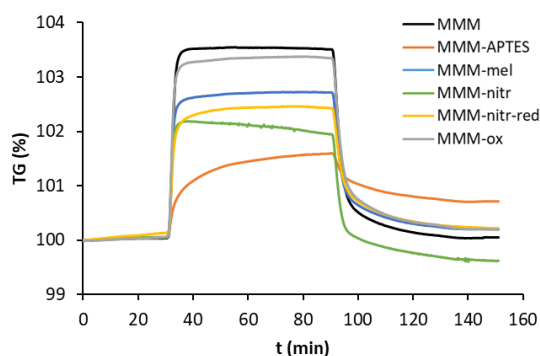


Figure 1. The experimental curves for CO₂ adsorption/desorption on pristine as well as surface-modified porous carbons obtained by isothermal thermogravimetry at 25 °C

The results provide insight into the influence of surface functionalization on CO₂ adsorption behavior and kinetics, contributing to the design of advanced carbon adsorbents for carbon capture applications.

Keywords: CO₂ adsorption kinetics, porous carbons, isothermal thermogravimetry, surface functionalization

Acknowledgements:

The Czech team was supported by the European Union under the LERCO project no CZ.10.03.01/00/22_003/0000003.

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ASSESSMENT OF A DSC-BASED NUCLEATION MODEL FOR SE-TE CHALCOGENIDE GLASSES

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Nucleation, as the initial stage of crystallization, plays a crucial role in determining the crystallization behavior and resulting properties of amorphous materials. Direct determination of the number of nuclei by microscopy is the most commonly used method. However, such analysis is usually laborious and highly time-consuming.[1]

Differential scanning calorimetry methods have been developed as an alternative tool for the study of nucleation. [2] Nevertheless, DSC-based methods are not universal, since their applicability depends strongly on the assumptions of the kinetic model used. Approaches based on the Johnson-Mehl-Avrami-Kolmogorov (JMAK) model framework are mainly suitable for systems in which the influence of surface crystallization and heterogeneous effects can be neglected. [3]

A new approach for competitive surface and volume crystallization was recently proposed by Shánělová et al. [4]. While rapid surface nucleation remains unquantified in this kinetic framework, the model evaluates time-dependent volume nucleation to estimate the average number of nuclei per powder grain from DSC data. In this work, we assessed the influence of possible sources causing discrepancies between this model and the real crystallization behavior of the Se-Te chalcogenide system.

Keywords: nucleation, kinetics, chalcogenide glass, Se-Te

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This work was supported by the Internal Grant Agency of the University of Pardubice under the SGS project (SGS_2026_006).

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CRITICAL ASSESSMENT OF CRYSTALLIZATION KINETICS PREDICTION FROM ROUTINE DSC

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Predicting crystallization behavior and phase stability in glass-forming systems remains a paramount challenge in materials science. In current practice, the standard approach to evaluating crystallization kinetics relies heavily on differential scanning calorimetry (DSC) data, typically obtained under non-isothermal heating regimes. This contribution critically assesses the fundamental limitations and pitfalls of this methodology, demonstrating why kinetic models applied to routine DSC curves frequently yield physically inconsistent results.

Based on an experimental dataset for chosen chalcogenide glasses, we highlight the profound discrepancy between conventional kinetic models derived from DSC data and the actual physical mechanisms governing crystallization. Routine DSC analyses are inherently compromised by several critical factors: the material's thermal history (e.g., the presence of quenched-in nuclei) [1], transient nucleation during heating, and the frequently dominant role of surface crystallization [2]. In practice, these phenomena preclude the accurate determination of the critical crystallization onset and invalidate the extrapolation of kinetic behavior to broader temperature ranges.

Furthermore, routine evaluations often default to the indiscriminate application of classical frameworks, such as the Johnson-Mehl-Avrami (JMA) model [3-5]. Based on independently measured kinetic parameters—encompassing viscosity, crystal growth rates, and true nucleation rates—this study reveals the fundamental flaws of such an approach. We emphasize the critical importance of the nucleation stage and the necessity of strictly distinguishing volume from surface crystallization, where the assumptions of conventional bulk models inherently break down. Our findings underscore that without explicitly accounting for these distinct mechanisms and the prior thermal history, physically meaningful predictions cannot be reliably extracted from conventional DSC curves.

Keywords: glass, crystallization kinetics, DSC, JMA model

Acknowledgements:

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KINETICS OF CO₂ ADSORPTION ON SURFACE-MODIFIED POROUS CARBONS II – NON-ISOTHERMAL KINETIC ANALYSIS

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The kinetic description of gas adsorption under non-isothermal conditions is considerably more challenging than that of conventional isothermal experiments because both the adsorption rate and adsorption equilibrium continuously change with temperature. In this work, the kinetics of CO₂ adsorption on surface-modified hierarchically porous carbons were investigated during controlled cooling using thermogravimetric analysis.

A hierarchically porous carbon (HPC) served as the parent material together with two surface-modified variants prepared by oxidation (HPC-O) and nitration followed by reduction (HPC-N). These materials exhibit comparable pore structures while differing primarily in their surface chemistry, enabling the influence of surface chemistry on adsorption kinetics to be evaluated.

CO₂ adsorption experiments were performed under non-isothermal conditions using linear cooling rates of 2, 5 and 10 K min⁻¹. The experimental adsorption curves were simultaneously analysed using a global nonlinear regression approach. A generalized kinetic model based on an Arrhenius-type temperature dependence combined with a conversion function was implemented in MS Excel Solver, allowing all cooling experiments for each material to be fitted simultaneously using a single set of kinetic parameters.

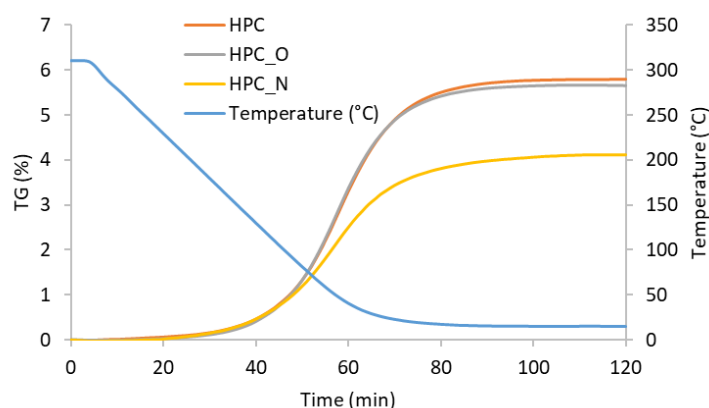


Figure 1: Representative non-isothermal CO₂ adsorption curves obtained for HPC, HPC_O and HPC_N during cooling at 5 K min⁻¹.

The proposed approach provides a consistent description of CO₂ adsorption under dynamic temperature conditions and enables direct comparison of the apparent kinetic parameters of surface-modified porous carbons. The developed methodology represents a useful tool for evaluating adsorption processes under realistic non-isothermal operating conditions.

Pronounced differences in the CO₂ adsorption profiles were observed among the investigated materials, highlighting the important role of surface chemistry under non-isothermal conditions.

Keywords: CO₂ adsorption kinetics, porous carbons, non-isothermal thermogravimetry, nonlinear regression, global kinetic analysis

Acknowledgements:

This work was supported by the European Union under the LERCO project no CZ.10.03.01/00/22_003/0000003.

THERMAL ANALYSIS OF REACTION MIXTURES FOR THE PREPARATION OF MnTiO_3 BLACK PIGMENTS

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Manganese titanate (MnTiO_3) pigments represent promising ceramic pigments with potential use in high-temperature coatings and ceramic systems [1]. Their formation is strongly influenced by synthesis conditions, phase evolution, and particle development during calcination. This study investigates the effect of mineralizers on the synthesis and thermal behaviour of MnTiO_3 pigments prepared by solid state reaction, using thermal analysis combined with structural and optical characterization.

MnTiO_3 pigments were synthesized from MnO_2 (90 % purity, Sigma Aldrich, USA) and TiO_2 - anatase type AV-01 (99 % purity, Precheza, Czechia). The starting mixtures were homogenized with 5 wt.% mineralizers (KCl, NaF–NaCl 1:1, NaCl, and KCl–NaCl 1:1) and calcined at 1000–1300 °C for 4 h (heating rate 5 °C/min). The powders were subsequently washed with hot water and milled in a planetary mill [2]. Thermal behaviour was assessed using simultaneous DSC/TG analysis (Setaram LabSys Evo, France) to identify phase transformations, mass changes, and reaction pathways. High-temperature processes – sintering temperature were further monitored by heating microscopy (Hesse Instruments EM201, Germany). Phase composition was determined by XRD (Rigaku MiniFlex 600, Japan), particle size distribution by laser diffraction (Malvern Mastersizer 3000 MU, GB), and colour properties into ceramic glaze (G02891 – Glazura, Czechia) systems using CIE $L^*a^*b^*$ coordinates [3].

The results demonstrated that mineralizers significantly influenced the formation temperature of MnTiO_3 and the overall thermal evolution of the system. DSC/TG analysis revealed shifts in thermal events associated with solid-state reactions, while XRD confirmed ilmenite MnTiO_3 as the dominant crystalline phase at temperatures above 1100 °C. Mineralizers also affected particle growth and pigment colour development in glaze applications. The prepared pigments provided black to brownish-black shades with lightness (L^*) values around 34 units, while the a^* and b^* coordinates remained close to zero, indicating minimal red–green and yellow–blue colour contributions.

Keywords: inorganic pigments, thermal analysis, structure of MnTiO_3 , solid state reaction, colour properties

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THERMAL BEHAVIOR OF VINYL ACETATE MONOMER AND ITS IMPLICATIONS FOR THERMAL RUNAWAY

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Vinyl acetate monomer (VAM) is an important industrial monomer used in the production of a variety of polymeric materials. However, unexpected thermal polymerization may occur even during storage, potentially leading to thermal runaway [1]. To suppress this reaction, hydroquinone (HQ), which acts as a radical scavenger, is commonly added to VAM. In this study, the thermal behavior of HQ-containing and HQ-free VAM samples, both alone and mixed with water or the hydrolysis products of VAM (acetic acid and acetaldehyde [2]) was investigated to elucidate the factors contributing to thermal runaway. Thermal analyses were performed using a C80 high-sensitivity heat flux calorimeter (SETARAM) under both air and nitrogen atmospheres. Approximately 30 mg of sample was sealed in a stainless-steel vessel (3.7 mL) and heated from 50 to 300 °C, at a heating rate of 0.05 K min⁻¹.

Under an air atmosphere, HQ-free VAM exhibited two exothermic peaks at approximately 100 and 250 °C. The second exothermic peak at around 250 °C was attributed to the thermal polymerization of VAM, as previously reported [3]. Its heat release reached approximately 1000 J g⁻¹, indicating that this reaction could lead to thermal runaway if triggered during storage. In contrast, the first exothermic peak at around 100 °C was not observed under a nitrogen atmosphere, suggesting that it originated from the oxidation of VAM. These observations suggest that the oxidation of VAM generates reactive radical species (e.g., R· and ROO·), which subsequently initiate partial polymerization of VAM. Furthermore, the first exothermic peak of HQ-containing VAM shifted to a higher temperature, indicating that the oxidation-derived radical species were scavenged by HQ, thereby suppressing the exothermic reaction. The first exothermic peak was markedly enhanced by the presence of water or acetaldehyde, a hydrolysis product of VAM, and the maximum heat release reached approximately 680 J g⁻¹. These results suggest that contamination with water under air substantially increases the risk of thermal runaway. Furthermore, the first exothermic peak was not observed under a nitrogen atmosphere even in the presence of acetaldehyde. These findings suggest that water contamination promotes the hydrolysis of VAM, producing acetaldehyde. The acetaldehyde generated may facilitate the formation of radical species during the oxidation of VAM, thereby enhancing the first exothermic peak.

Keywords: Vinyl acetate monomer, Thermal polymerization, Acetaldehyde, Thermal runaway

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THERMAL DECOMPOSITION KINETICS OF CALCIUM CARBONATE UNDER VACUUM USING CONTROLLED-RATE EVOLVED GAS ANALYSIS WITH MASS SPECTROSCOPY

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The thermal decomposition kinetics of solids are influenced by the reaction conditions, including the gaseous environment and the self-cooling effect of endothermic processes. In order to collect kinetic data for such reactions using thermal analysis, it is imperative to reduce the effects of self-generated gases and self-cooling. The application of the controlled-rate thermal analysis (CRTA), a technique of sample controlled thermal analysis [1], under reduced pressure has been demonstrated as a promising technique to collect the kinetic data for thermal decomposition of solids [2,3]. Furthermore, the implementation of mass spectroscopy (MS) as an evolved gas analysis (EGA) enables a substantial reduction in the sample mass (m_0) for the measurements [4]. In this study, the CREGA-MS system was developed and its performance was evaluated through the thermal decomposition of calcium carbonate as a model reaction.

The CREGA-MS system is composed of an electric furnace, vacuum evacuation system, and MS. The pressure within the furnace can be regulated to achieve a vacuum less than 1.0×10^{-3} Pa. In CREGA mode, the sample temperature is controlled to maintain a constant rate of the gaseous evolution by utilizing the total pressure or partial pressure of a gas within the furnace as a feedback signal. The evolution rates of specific gaseous species during the thermal decomposition process under vacuum can be traced using a quadrupole mass spectrometer.

The CREGA-MS measurements were demonstrated using two calcium carbonate samples, CC-A and CC-B, characterized by average particle sizes of 0.47 and 8.87 μm , respectively. To collect the kinetic data of the thermal decomposition under vacuum, the respective samples were heated using the linear nonisothermal and constant transformation rate (CR) modes. For the CR modes, two series of measurement conditions were used: (1) the pressure change inside the furnace (ΔP) was varied within a range of $0.5\text{--}2.5 \times 10^{-3}$ Pa, while maintaining $m_0 = 2.0$ mg and (2) the m_0 value was varied, while maintaining ΔP to be 1.5×10^{-3} Pa. As the ΔP value increased or the m_0 value decreased, the reaction temperature increased systematically. A series of kinetic data under various measurement conditions, which were converted from the mass thermograms of $m/z = 44$ (CO_2^+), were analyzed using the Friedman method. In both the CC-A and CC-B samples, the Friedman plots represented a statistically significant linear correlation, including all data points obtained under varying measurement conditions. The CC-B sample, which was composed of the larger particle size, exhibited a constant activation energy ($E_a = 198 \pm 2$ kJ mol $^{-1}$) during the main stage of the reaction. On the other hand, the CC-A sample exhibited a larger E_a value (227 ± 6 kJ mol $^{-1}$) in the initial half of the reaction and underwent a decline in the subsequent process. The scanning electron microscopic observations of the morphological changes of the sample particles during the reaction revealed distinguishable sintering of the particles for the CC-A sample, which is expected to contribute to the kinetic behavior. The fundamental performance of the CREGA-MS instrument is discussed, as well as demonstrating the characterization of the thermal decomposition of calcium carbonate under vacuum.

Keywords: Sample controlled thermal analysis, Kinetics, Thermal decomposition, Calcium carbonate

Acknowledgements:

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THERMAL DECOMPOSITION BEHAVIOR OF STABILIZER-CONTAINING NITROCELLULOSE MEMBRANES FOR LATERAL FLOW IMMUNOASSAY

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Lateral flow immunoassay (LFIA) [1] is a rapid antigen detection method widely used for the diagnosis of infectious diseases such as influenza and COVID-19. In LFIA, a liquid sample flows through a membrane by capillary action. The target antigen in the sample is captured by antibodies immobilized on the membrane, producing a colored test line. The test result is determined by visually inspecting this test line. Nitrocellulose (NC) [2], which serves as the membrane material in LFIA and is also a major component of explosives, is prone to spontaneous decomposition even at room temperature, producing nitrogen oxides (NO_x) and nitric acid. These reactive decomposition products may interact with antigens or antibodies, potentially leading to diagnostic errors in LFIA. In NC-based explosives, decomposition is generally suppressed by the addition of phenolic stabilizers. However, this approach cannot be directly applied to LFIA membranes because conventional stabilizers react with NO_x and cause yellowing of the membrane, which may interfere with the visual interpretation of the test results. Therefore, this study explored alternative stabilizers suitable for LFIA membranes. For this purpose, NC membranes containing Tinuvin 123 (BASF, T123), a mixture of Irganox 1010 and Irgafos 168 (BASF, IRM), or the conventional stabilizer ethyl centralite (reagent grade, EC) were prepared. Their thermal stability was evaluated at a heating rate of 0.05 K min⁻¹ using a Calvet-type reaction calorimeter. In addition, NO_x release, surface pH, residual acid content, and color change were investigated during isothermal aging at 70°C under both dry-air and humid-air conditions. The results of the thermal analysis showed that the stabilizer-free NC membrane had onset temperatures of approximately 115°C under dry conditions and 110°C under humid conditions. In contrast, the addition of stabilizers increased these temperatures to 136°C and 128°C for T123, 133°C and 120°C for IRM, and 137°C and 133°C for EC, respectively. These results suggest that T123 provides a thermal stabilization effect comparable to that of the conventional stabilizer EC. In addition, the isothermal aging test revealed that the stabilizer-free membrane underwent significant degradation under humid-air conditions after 80 h of aging. This degradation was accompanied by increased NO_x release and residual acid content, a marked decrease in surface pH, and visible damage to the membrane. In contrast, the T123-containing membrane maintained similar properties under both environments for more than 330 h of aging. Furthermore, the T123-containing membrane retained its original color, whereas the EC-containing membrane exhibited slight yellowing. These findings demonstrate that T123 is a promising stabilizer for NC membranes used in LFIA, providing thermal stability comparable to that of EC while minimizing membrane yellowing.

Keywords: Immunochromatography, Nitrocellulose, Stabilizer, Stability, Thermal degradation

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SUBLIMATION AND VAPORIZATION OF SODIUM IODIDE AS A SOURCE FOR THE GENERATION OF GASEOUS IODINE

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The atmospheric dispersion of radioactive iodine constitutes a significant concern in risk assessments predicated on hypothetical severe accidents involving sodium-cooled fast reactors. Sodium iodide (NaI), a product of the reaction between radioactive iodine, a fission product, and molten sodium, a coolant, has been identified as a potential source of gaseous iodine. However, the kinetics and mechanism, by which gaseous iodine is formed from NaI, remain to be fully elucidated. The present study focused on the thermal behavior of NaI, with the objective of investigating the formation process of its gas or aerosols. Thermogravimetry–mass spectroscopy measurements of NaI in a stream of helium and simulated air (He–O₂) revealed the formation of gaseous iodine by the reaction of gaseous NaI and oxygen in the reaction atmosphere. Consequently, the thermally induced production of gaseous NaI was identified a pivotal process that governs the formation of gaseous iodine. When NaI was heated under nonisothermal conditions in a stream of dry N₂, the mass loss initiated at approximately 900 K by the sublimation. Subsequently, the mass loss rate was found to be accelerated due to a fusion occurring at 933 K, which result in a change in the mass loss mode to vaporization from the molten NaI. The enthalpy changes of the sublimation and vaporization were determined using the series of mass loss curves under isothermal conditions in the individual temperature ranges of sublimation and vaporization [1]. The enthalpy changes of the sublimation and vaporization processes were determined to be 212 ± 7 and 131 ± 7 kJ mol^{−1}, respectively. Comparisons with the previously reported enthalpy change values revealed that the value for the sublimation process determined in this study corresponded to an intermediate value between those for the formation of gaseous monomer (NaI)(g) and dimer (NaI)₂(g) [2], while that for the vaporization process exhibited a slightly smaller value than that of the previously reported value [3]. Within the framework of the mass loss kinetics, the sublimation and vaporization processes were both characterized by the zero-order rate behavior. The Arrhenius parameters (E_a /kJ mol^{−1}, A /s^{−1}) were determined to be $(215 \pm 5, (1.6 \pm 0.1) \times 10^8)$ and $(123 \pm 4, (1.7 \pm 0.3) \times 10^3)$, respectively. Under nonisothermal conditions, the contributions of the sublimation and vaporization processes to the total mass loss process were systematically varied with the heating rate. The overall kinetic behaviors of the mass loss process to form gaseous NaI were successfully described by a conjunction of the kinetic equations for the sublimation and vaporization processes, as denoted by the subscripts, 1 and 2, respectively.

$$\frac{d\alpha}{dt} = \left[A_1 \exp\left(-\frac{E_{a,1}}{RT}\right) f_1(\alpha) \right]_{T_i}^{T_m} + \left[A_2 \exp\left(-\frac{E_{a,2}}{RT}\right) f_2(\alpha) \right]_{T_m}^{T_f}, \quad (1)$$

where T_i , T_m , and T_f are the temperatures of initiation of mass loss, fusion, and completion of mass loss, respectively.

Keywords: Sodium iodide, Sublimation, Vaporization, Thermodynamics, Kinetics

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EFFECT OF POTASSIUM PERCHLORATE ADDITION ON THE COMBUSTION AND THERMAL BEHAVIOR OF GUANIDINE NITRATE/BASIC COPPER NITRATE-BASED GAS GENERANTS

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Gas generants for automotive airbags generally consist primarily of guanidine nitrate (GN) and basic copper nitrate (BCN). These compositions contain various additives to improve combustion characteristics. Potassium perchlorate (KClO₄) is one such additive used in industry and is expected to increase the combustion rate because of its strong oxidizing ability. However, empirical observations have suggested that the addition of KClO₄ can reduce ignitability under certain conditions, potentially compromising ignition reliability. Therefore, this study investigated the effects of KClO₄ on the combustion behavior of GN/BCN-based gas generants through combustion tests and thermal analysis. Stoichiometrically mixed samples of GN (25–50 μm), BCN (≤10 μm), and KClO₄ (≤50 μm) were compression-molded into cylindrical pellets (φ9.5 mm × 30 mm). Combustion tests were conducted using a chimney-type strand burner under an N₂ atmosphere at 0.01–5.0 MPaG to measure the combustion rate¹⁾. In addition, differential scanning calorimetry (DSC) was performed on 3 mg samples under an N₂ atmosphere 1.0, and 3.0 MPaG with a heating rate of 5.0 K/min.

The combustion test results showed that the KClO₄-containing samples exhibited higher combustion rates at 1–5 MPaG than the additive-free sample but a lower combustion rate at 0.1 MPaG. Combustion could not be sustained at 0.01 MPaG, suggesting that KClO₄ increases the pressure dependency on the combustion rate. Figure 1 shows the combustion behavior of the generants. The additive-free sample produced a relatively large amount of red-hot condensed matter during combustion, whereas KClO₄-containing sample produced almost none. This observation indicates that KClO₄ alters the condensed-phase reactions. Figure 2 shows the DSC results for the gas generants. Pressure had little effect on the DSC profile of the additive-free sample, whereas the KClO₄-containing sample exhibited marked pressure-dependency in its thermal behavior. Together with the combustion observations, these results suggest that KClO₄ alters condensed-phase reactions of the GN/BCN composition, thereby changing its combustion behavior under different pressure conditions.

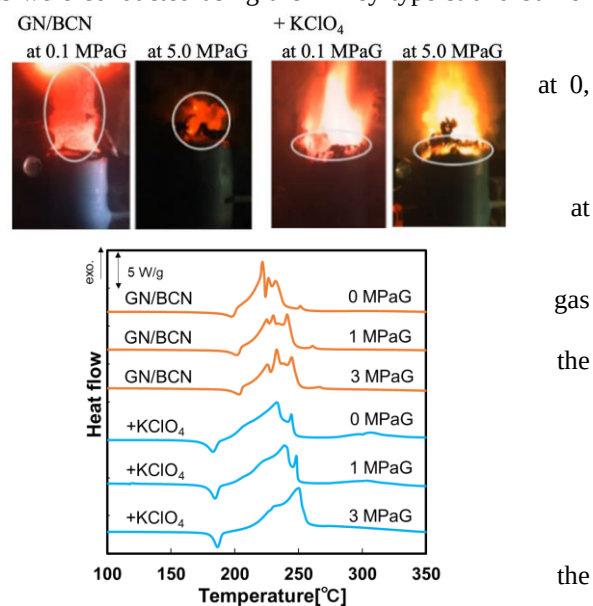


Figure 2. DSC curves of the samples

Keywords: Airbag, Combustion behavior, Thermal analysis, GN/BCN-based gas generant, Potassium perchlorate

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HYDROTHERMAL SYNTHESIS OF TOBERMORITE USING CONCRETE PRODUCTION WASTE

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Tobermorite ($\text{Ca}_5\text{Si}_6\text{O}_{16}(\text{OH})_2 \cdot 4\text{H}_2\text{O}$) is a calcium silicate hydrate that plays an important role in the production of autoclaved aerated concrete, thermal insulation materials, adsorbents, and other products [1], [2]. It can be synthesised from various natural raw materials containing calcium and silicon oxides [3]. However, to support the transition towards a circular economy, suitable industrial waste streams should be considered as alternatives to natural raw materials. It is estimated that more than 2.3 million tonnes of calcium-containing waste are generated annually in Lithuania, and these materials have considerable potential for use in the synthesis of calcium silicate hydrates [4]. Nevertheless, limited information is available on their application in the synthesis of tobermorite and related calcium silicate hydrates. Therefore, this study aims to investigate the feasibility of synthesising tobermorite using concrete production waste as a secondary raw material.

In this study, concrete production waste and quartz were used as raw materials. The concrete production waste contained quartz, calcium carbonate, residual cement phases, and cement hydration products. Before synthesis, the waste was ground in a rotary mill for 2 h, followed by additional grinding in a planetary mill at 900 rpm for 10 min and calcination at 800 °C for 2 h. The SiO_2 and CaO contents in the calcined waste were 44.6 and 37.5 wt.%, respectively. The CaO/SiO_2 molar ratio of the waste was 0.90; therefore, an additional amount of quartz was added to obtain the stoichiometric ratio of 0.83 required for tobermorite formation. The resulting mixture was hydrothermally treated at 200 °C for up to 72 h. Concrete production waste was found to be a promising secondary raw material for the synthesis of calcium silicate hydrates. During the initial stage of hydrothermal treatment, poorly crystalline calcium silicate hydrates formed and subsequently recrystallised into tobermorite, the main synthesis product. The initial calcium-containing phases reacted within 8 h, whereas unreacted quartz remained detectable even after 72 h of synthesis. Microcline, calcite, and albite were also identified in the products. The addition of sodium or potassium hydroxide could be considered to enhance quartz dissolution and promote more complete tobermorite formation.

Keywords: tobermorite, hydrothermal synthesis, industrial waste, calcium silicates.

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GYROLITE WITH INTERCALATED Sn^{4+} IONS: SYNTHESIS AND CATALYTIC ACTIVITY IN VOC OXIDATION

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Air pollution is one of the major challenges of this century, and volatile organic compounds (VOCs) are among its main contributors. Various technologies are used to reduce their adverse effects on the environment and human health. One of the most effective and economical methods is catalytic oxidation, which converts VOCs into CO_2 and H_2O [1, 2]. However, this method can be costly because it commonly relies on noble metals such as palladium and platinum [3]. Therefore, cheaper and more environmentally friendly alternatives are being sought. One potential alternative is calcium silicate hydrates intercalated with metal ions, which have already demonstrated promising performance in VOC oxidation [4]. Gyrolite is a particularly promising silicate because it has a large interplanar spacing, allowing substantial amounts of metal ions to be intercalated without disrupting its crystalline structure. However, data on the synthesis of these compounds containing metal ions and their application in oxidation reactions remain limited. Therefore, this study aimed to investigate the catalytic performance of gyrolite intercalated with tin(IV) ions in VOC oxidation.

The following raw materials were used in this study: calcium oxide, obtained by calcining calcium carbonate at 950 °C for 2 h; amorphous silicon dioxide ($\text{SiO}_2 \cdot n\text{H}_2\text{O}$); and tin(IV) chloride pentahydrate ($\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$). Calcium oxide and silicon dioxide were mixed to obtain a CaO/SiO_2 molar ratio of 0.66. To intercalate tin(IV) ions into the gyrolite structure, hydrothermal synthesis was carried out in two stages. In the first stage, 2 g of the $\text{CaO}-\text{SiO}_2$ mixture was mixed with 20 mL of water. The resulting suspension was hydrothermally treated in PTFE vessels placed in a Parr Instrument autoclave at 200 °C for 48 h. In the second stage, tin(IV) ions were added at a concentration of 100 mg per gram of the initial mixture ($c_{\text{Sn}} = 10 \text{ g/L}$), and the suspension was maintained under the same conditions for an additional 24 h. The resulting products were dried at 50 °C.

It was determined that gyrolite [$\text{Ca}_8\text{Si}_{12}\text{O}_{30}(\text{OH})_4 \cdot 7\text{H}_2\text{O}$], together with traces of xonotlite [$\text{Ca}_6\text{Si}_6\text{O}_{17}(\text{OH})_2$], formed during the first stage of synthesis at 200 °C for 48 h. The addition of tin(IV) chloride, corresponding to 100 mg of tin(IV) per gram of the initial $\text{CaO}-\text{SiO}_2$ mixture, followed by additional treatment at 200 °C for 24 h, did not affect the phase composition of the products. Analysis of the liquid phase showed that all tin(IV) ions had been intercalated into the gyrolite structure. Thermal analysis showed that gyrolite with intercalated tin ions gradually dehydrated as the temperature increased to 700 °C. At higher temperatures, it recrystallized into wollastonite, which remained stable up to 1000 °C. The tin and chlorine ions intercalated into the gyrolite structure reacted with calcium ions, resulting in the formation of calcium chlorate, megavite, and cassiterite at temperatures above 800 °C. Gyrolite with intercalated tin ions also exhibited catalytic activity in the oxidation of propyl acetate to H_2O and CO_2 . The conversion rate depended on the oxidation temperature, increasing from 34% to 68% as the temperature rose from 240 °C to 340 °C.

Keywords: gyrolite, calcium silicates hydrates, hydrothermal synthesis, thermal stability, tin(IV) ions

Acknowledgements:

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APPLICATION OF CALCIUM- AND SILICON-CONTAINING SECONDARY RAW MATERIALS FOR WOLLASTONITE SYNTHESIS

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The increasing volume of waste highlights the need to move to a circular economy by converting waste materials into valuable products. However, more than 2.2 billion tonnes of waste are generated annually in the European Union, of which nearly 30 wt.% is landfilled [1]. In Lithuania, over 2.3 million tonnes of calcium-rich waste are generated each year, while their utilisation remains limited [2]. Such wastes could be used as secondary raw materials for calcium silicate synthesis, particularly wollastonite. Wollastonite is widely used in ceramics because of its thermal stability, biocompatibility, and reactivity, and may also contribute to lower-carbon material production [3]. However, limited information is available on the use of uncured concrete waste and calcium carbonate-rich water treatment residues generated in Lithuania. Therefore, this study aims to evaluate their suitability for wollastonite production.

In this study, two industrial waste materials were used: uncured concrete waste from concrete production (BC) and calcium carbonate-rich waste from a water treatment process (CC). The waste materials were dried, crushed, and ground in a rotary mill for 2 h, followed by additional grinding in a planetary mill at 900 rpm for 10 min. After calcination at 950 °C for 2 h, BC contained 51.6 wt.% CaO and 30.2 wt.% SiO₂, whereas CC contained 73.2 wt.% CaO and 12.2 wt.% SiO₂.

For the one-step synthesis, the ground BC and CC wastes were separately mixed with quartz to prepare mixtures with a Ca/Si molar ratio of 1.0. The mixtures were subsequently calcined at temperatures ranging from 700 to 1300 °C for 1 h. Regardless of the initial mixture composition and calcination temperature, mixtures of calcium silicates and unreacted starting materials were obtained. Calcium-rich silicate phases predominated at lower temperatures, whereas increasing the calcination temperature promoted wollastonite formation.

For the two-step synthesis, the ground waste materials were first calcined at 800 °C and then separately mixed with quartz to obtain mixtures with a Ca/Si molar ratio of 1.0. Two grams of each mixture were placed in a 25 mL PTFE vessel, and 10 mL of distilled water was added. The vessels were placed in an autoclave, and hydrothermal synthesis was performed in unstirred suspensions under saturated steam pressure at 175 °C for 4–72 h. Calcium silicate hydrates, including C–S–H(I), tobermorite, and scawtite, formed during hydrothermal treatment. Subsequent calcination of the hydrothermal synthesis products at temperatures above 800 °C resulted in wollastonite formation.

Keywords: wollastonite; calcium silicates; hydrothermal synthesis; high temperature synthesis

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THERMAL ANALYSIS OF BIOCOMPOSITES CONTAINING PLANT-BASED ADDITIVES DERIVED FROM AGRICULTURAL WASTE

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In recent years, plant-based additives have attracted increasing interest in polymer processing due to their eco-friendly nature, unique functionality, and cost-effectiveness. Agro-industrial wastes containing polyphenolic compounds have been recognized as promising natural stabilizers capable of protecting polymer materials against degradation [1-4]. The aim of this study is to evaluate the effect of bio-additives derived from agro-industrial plant waste on the thermal stability of polymer materials based on biodegradable polylactide (PLA) and synthetic polymers, such as polyethylene (PE) and ethylene–norbornene copolymer (EN). The thermal stability of the agro-waste used as bio-additives was determined by thermogravimetric analysis (TG) prior to polymer processing. Subsequently, a thermal analysis of polymer compositions containing plant-based additives was performed. For polylactide-based biocomposites, measurements were performed using differential scanning calorimetry (DSC) under an air atmosphere, whereas for synthetic polyolefins containing phyto-additives, oxidation induction times (OIT) were determined. Higher oxidation temperatures (T_o) of PLA and extended oxidation induction times of polyolefins containing agro-industrial waste indicated the antioxidant activity of the natural additives and the enhanced oxidative resistance of the polymer materials. The most effective stabilizing performance in the biocomposites was exhibited by wastes containing the highest levels of polyphenolic compounds.



Figure 1. Biocomposite and raw materials used for their preparation (polymer granules and plant additives)

Keywords: bio-additives, polyphenols, polymer stabilization

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MULTIFUNCTIONAL MATERIALS BASED ON CARBON AEROGELS AND PHASE CHANGE MATERIALS MODIFIED WITH MXENES

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In the context of the intensive search for new technologies in the field of green energy, the use of citrus waste as precursors for the production of carbon matrices seems particularly promising.

Nanometric MXene materials in the form of transition group carbide and/or nitride layers are of considerable interest [1]. Their properties are connected to the surface groups such as –OH, =O, –Cl, or –F, which are accessible during synthesis. This makes them highly likely to be used in biomedicine, electronics, photothermal conversion, and microchips [2]. All available 2D carbon-based MXene materials are characterized by excellent electrochemical properties, which are achieved through their conductivity, the ability to access the surface through various functional groups, and redox monitoring. This layer is based on rapid transport and storage, resulting in consistent performance [3].

This work studies the durability and shape stability of porous carbon materials/poly(ethylene glycol) (C/PEG) composites. Biomass derived from orange waste and various polysaccharides, such as starch, chitosan, gellan gum, and sodium alginate, were used to obtain carbon aerogels [4]. The most promising systems are matrices based on albedo or a mixture of albedo and flabedo oranges. Gellan gum and sodium alginate proved to be the most effective biomass binder. To obtain an optimal microstructure, the samples were subjected to two-stage thermal treatment at temperatures of 200°C and 600°C. The resulting carbon matrices were then infiltrated with a phase change material (PCM). To improve the physicochemical properties and obtain PCM composites with photothermal conversion ability, they were modified with graphene and titanium-based MXenes. The resulting materials were characterized using the following analytical techniques: thermogravimetric analysis (TGA), infrared spectroscopy (FTIR), differential scanning calorimetry (DSC), scanning electron microscopy (SEM), and leakage testing.

Keywords: citrus biomass, carbon aerogels, carbon/polyethylene glycol composites, MXenes

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DSC EVALUATION OF DEFECT FORMATION MECHANISMS IN HYBRID MIXTURE OF PARAFFIN AND VEGETABLE OILS

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By combining the high-resolution capabilities of differential scanning calorimetry (DSC) and infrared (IR) spectroscopy, this study elucidates the complex physicochemical mechanisms driving defect formation in hybrid paraffin and vegetable oils mixtures. We systematically assessed the thermodynamic behavior and microstructural evolution of samples manufactured via two distinct technological routes, which represent the extremes of industrial processing: a low-temperature process (L43) and a high-temperature process (L8). To fully evaluate the impact of thermal history on structural relaxation and long-term stability, these materials were subsequently subjected to carefully controlled storage climates at 15°C, 25°C, and 40°C.

Our comprehensive analytical data demonstrate that a sample's specific thermal history drastically alters its underlying crystallization and melting kinetics. This is evidenced by significant shifts in transition temperatures, marked variations in melting enthalpy, and the emergence of secondary thermal events that indicate dynamic phase reorganization and the redistribution of components within the solid matrix. Notably, conditioning the materials at a reduced temperature of 15°C induced profound phase destabilization. This environment lowered the nucleation barrier, triggering rapid, heterogeneous nucleation and the formation of highly defective, metastable structures. In contrast, exposure to a 40°C environment prompted accelerated degradation processes and an advanced structural reshuffling of the lipid and paraffinic components.

Optimal microstructural coherence and thermodynamic equilibrium were achieved exclusively when the samples were maintained at 25°C. Ultimately, these findings highlight how intimately the overall phase stability, free energy distribution, and physical integrity of such complex organic matrices are tied to both the initial processing heat gradient and the subsequent environmental exposure during storage.

Keywords: paraffin; differential scanning calorimetry (DSC); structural defects

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BIO-BASED SHAPE-STABLE PHASE CHANGE MATERIALS FROM KRAFT LIGNIN AND POLY(ETHYLENE GLYCOL) PREPARED WITH DEEP EUTECTIC SOLVENTS

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Phase change materials (PCMs) based on poly(ethylene glycol) (PEG) offer adjustable melting points and high latent heat storage capacity; however, their application is limited by leakage in the molten state [1]. Kraft lignin provides a rigid aromatic structure with a glass transition temperature above 100°C, making it a promising shape-stabilising matrix [2]. Deep eutectic solvents (DES) are biodegradable media capable of dissolving and processing lignin under mild conditions [3].

In this work, bio-based shape-stable PCMs were prepared by a one-pot method: kraft lignin was dissolved in three DES variants – choline chloride/urea (reline), choline chloride/glycerol (glyceline) and choline chloride/oxalic acid (oxaline) – followed by blending with PEG of different average molar mass (4000, 6000 or 8000 g/mol). The mixtures were cast onto Petri dishes and dried to obtain solid samples. The proposed stabilisation mechanism involves a hydrogen bond network between the hydroxyl groups of lignin and oxygen atoms in PEG, within a rigid amorphous lignin matrix. Conceptually, this approach refers to the gelatinised starch/PEO composition reported previously [4], replacing water with DES and starch with lignin. The high T_g of kraft lignin restricts the macroscopic mobility of molten PEG while preserving its microscopic ability to crystallise. Optionally, gelling agents (starch or agar) were added to improve structural integrity.

The blends were characterised by differential scanning calorimetry (DSC; phase transition enthalpy, melting/crystallisation temperatures, thermal cyclic stability), thermogravimetric analysis (TGA; thermal stability) and Fourier-transform infrared spectroscopy (FTIR; hydrogen bond interactions). Leakage tests at 80°C on filter paper enabled the preservation of shape to be observed. The influence of DES type, PEG molar mass and lignin-to-PEG ratio on thermal storage capacity and form stability was evaluated. The proposed approach, based entirely on bio-derived reagents and free from aggressive solvents, offers a green alternative to conventional PCM stabilisation via chemical cross-linking or porous-carbon impregnation.

Keywords: phase change materials, kraft lignin, deep eutectic solvents, poly(ethylene glycol), thermal energy storage

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THERMAL ANALYSIS OF A NEW COORDINATION POLYMER BASED ON 4,4'-BIS(1H-IMIDAZOL-1-YLMETHYL)BENZENE

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As a highly dynamic field within crystal engineering and supramolecular chemistry, coordination polymers are widely studied for their customizable structures and unique functionalities [1]. Metal-organic frameworks (MOFs) - a prominent subclass characterized by coordination networks containing metal ions, organic ligands and potential voids - have shown significant interest [2]. Current research focuses on novel MOFs engineering with functional properties, particularly high thermal stability, to enable their use in high-temperature industrial processes. Thermal analysis serves as a primary method for evaluating these properties.

A new 1D coordination polymer with molecular formula $\{[\text{Co}(\text{C}_{10}\text{H}_{16}\text{O}_4)\text{BIX}] \cdot (\text{C}_{10}\text{H}_{18}\text{O}_4)_{0.5} \cdot \text{H}_2\text{O}\}_n$, where $\text{C}_{10}\text{H}_{18}\text{O}_4$ = sebacic acid and BIX = 4,4'-bis(1H-imidazol-1-ylmethyl)benzene has been synthesized and thermogravimetric characterized (Figure 1). The measurement has been done in air and the compound was heated up to 1000 °C with 10 °C/min rate.

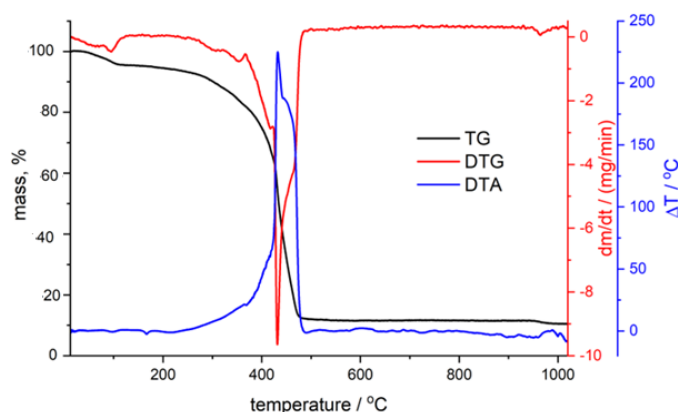


Figure 1: Thermoanalytical curves TG/DTG/DTA of 1D- $\{[\text{Co}(\text{C}_{10}\text{H}_{16}\text{O}_4)\text{BIX}] \cdot (\text{C}_{10}\text{H}_{18}\text{O}_4)_{0.5} \cdot \text{H}_2\text{O}\}_n$ polymer.

The compound loses crystallization water molecules (4,64 %) from the polymer lattice at temperature below 120 °C. Thermal decomposition of the network occurs between 160 - 500 °C (87,94 %) with competitive removal of the guest sebacic acid molecules from the external sphere together with thermolysis of the coordinated sebacic dianions and BIX ligand molecules. There is no clear removal of sebacic acid inclusion molecules from the channels, however it may occur up to 375 °C. The final residue is formed at 500 °C and corresponds to cobalt(II) oxide (12,15 %).

High thermal stability of coordination polymers can make these materials eligible for technical applications concerning processes involving heating.

Acknowledgements: This work was funded by the Research Subprogram SSMCCLP (code 010602).

Keywords: coordination polymer, cobalt(II), sebacic acid, 4,4'-bis(1H-imidazol-1-ylmethyl)benzene.

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THERMAL STABILITY AND HEAT-INDUCED PHASE EVOLUTION OF METAL/ACTIVATED CARBON COMPOSITES FOR REMOVAL OF NITRITES

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Metal-modified activated carbons combine adsorption with redox activity, but their practical use in water treatment requires knowledge of their thermal stability and heat-induced phase evolution. A series of AC-Mn1, AC-Mn2, AC-Mn1S, AC-Mn2S, AC-Co1 and AC-Cu1 composites was prepared by hydrothermal modification of activated carbon derived from walnut shells. Unmodified AC-0, commercial MnO₂ and Centaur activated carbon were used as references. Thermal behaviour was investigated in air from 20 to 700 °C by thermogravimetric analysis. Selected samples were subsequently heated at 650 °C for 10 min and characterized by X-ray diffraction. Their ability to remove nitrite ions from water was evaluated under batch conditions. The composites remained thermally stable up to 240 °C. AC-Mn1, AC-Mn2 and AC-Co1 exhibited two main degradation stages, assigned to water release and combustion of the carbonaceous component, whereas AC-Mn1S, AC-Mn2S, AC-Cu1 and AC-0 degraded in three stages. Thermal treatment promoted the formation of crystalline oxide phases. Mn₃O₄ crystallites of 5.4 nm formed in AC-Mn1; AC-Mn2 contained 91.8% Mn₃O₄ with a crystallite size of 22.0 nm; and AC-Mn1S contained 72.4% Mn₃O₄ and 27.6% α -MnO₂, with crystallite sizes of 17.3 and 18.1 nm, respectively. Co₃O₄ crystallites of 29.2 nm formed in AC-Co1. Nitrite-removal equilibrium was reached within 5-10 min, with removal efficiencies of 89-98%; AC-Cu1 showed the highest performance, while the unmodified carbon was comparable to commercial Centaur. The results demonstrate that controlled thermal treatment governs the oxide phase composition of metal/carbon composites and yields fast, efficient sorbent-catalysts for water depollution.

Keywords: thermogravimetric analysis, activated carbon, metal oxides, nitrite removal, water treatment

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This research was funded by the Republic of Moldova Ministry of Education and Research subprogram “ECOQUA” (code 010603) of the Institute of Chemistry of Moldova State University.

KINETICS STUDY OF HYDROGEN DESORPTION FROM W-DOPED MgH_2

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The influence of microstructure, morphology, and tungsten (W) addition on the hydrogen desorption behavior of MgH_2 –W composites was investigated. The composites were synthesized by high-energy ball milling with different W contents (2 and 5 wt%) and milling times ranging from 15 to 45 min. Morphological and microstructural characterization was performed using X-ray diffraction (XRD), laser particle size distribution (PSD) analysis, and FTIR-ATR (Fourier transform infrared spectroscopy–attenuated total reflection). The thermodynamic and kinetic properties of the materials were evaluated by temperature-programmed desorption (TPD). The TPD curves were successfully described using Fraser–Suzuki and Weibull functions, enabling the consideration of heterogeneous energy barriers and reaction mechanisms influenced by milling time and W content. The apparent activation energy was found to decrease with increasing temperature, whereas the pre-exponential factor exerted a strong influence on the overall desorption rate. These findings highlight the importance of considering multiple kinetic parameters, rather than apparent activation energy alone, to achieve a comprehensive understanding of hydrogen desorption. Despite the limitations of the applied methodology, the proposed modeling approach provides valuable insight into the effects of milling, tungsten addition, and microstructural modifications on hydrogen desorption from MgH_2 .

Keywords: hydrogen desorption, tungsten, kinetics, Fraser–Suzuki function, Weibull function

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EFFECT OF PMMA POLYMER MATRIX ON HYDROGEN DESORPTION PROPERTIES OF MgH_2

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MgH_2 is a promising solid-state hydrogen storage material due to its abundance, cost-effectiveness, non-toxicity, high theoretical hydrogen storage capacity, and reversible hydrogen uptake/release. However, in addition to thermodynamic and kinetic limitations, its high sensitivity to air significantly restricts practical applications. Exposure of Mg or MgH_2 to oxidizing atmospheres leads to the formation of a dense $\text{MgO}/\text{Mg}(\text{OH})_2$ surface layer, which hinders hydrogen diffusion and reduces hydrogen sorption capacity. One practical strategy for improving air stability is the use of polymeric gas-barrier coatings. In this study, MgH_2 /poly(methyl methacrylate) (PMMA) composites were synthesized by high-energy ball milling for periods ranging from 0.5 to 2 h. Morphological and microstructural characterization was performed using X-ray diffraction (XRD), laser particle size distribution (PSD) analysis, and FTIR-ATR spectroscopy. Thermodynamic and kinetic properties were evaluated by temperature-programmed desorption (TPD) and thermogravimetric analysis (TGA), while desorption kinetics were analyzed using dispersive kinetic models. The properties of the composites were assessed immediately after synthesis and after exposure to ambient conditions for 2 and 4 weeks. The results demonstrate that the PMMA-containing composites largely retained their hydrogen desorption capacity during storage, indicating improved resistance to air-induced degradation and highlighting the potential of polymer-protected MgH_2 systems for practical hydrogen storage applications.

Keywords: hydrogen desorption, hydrogen storage, MgH_2 , poly-methyl methacrylate

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This paper is supported by the Ministry of Science, Technological Development and Innovation of the Republic of Serbia, under Grant No. 451-03-136/2025-03/200017, and NATO Science for Peace Programme, grant no. 6037

THERMOOXIDATIVE STABILITY OF MICROPLASTIC-SIZED RUBBER PARTICLES FROM TIRE RECYCLING

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Recently, microplastics have attracted considerable attention as an emerging class of anthropogenic pollutants. Depending on the environment, microplastics may undergo various degradation processes driven by physical (abrasion, wear), chemical (photooxidation, thermal oxidation, hydrolysis), or biological factors (biodegradation). During these processes, microplastics gradually age and eventually decompose into small molecules such as CO₂, H₂O, and CH₄ [1]. Compared with bulk plastics, nano-sized and to a lesser extent micro-sized plastics are expected to degrade at a higher rate, mainly due to their higher specific surface area and excess surface energy. In this work, the thermooxidative stability of rubber particles originating from a tire recycling facility was studied using differential scanning calorimetry (DSC). The rubber particles were sieved into seven fractions (with particle sizes up to 1250 µm) and subjected to a series of non-isothermal DSC measurements at five different heating rates (1–10 °C min⁻¹) under a dynamic oxygen atmosphere. Oxidation onset temperatures obtained at various heating rates were then used to estimate the values of apparent kinetic parameters, from which the induction period (IP) was determined [2]. To minimize extrapolation error, a temperature close to the measured oxidation onset temperatures, 180 °C, was chosen as the basis for comparing IP values. Since the IP corresponds to the time during which no thermal oxidation is observed under the given conditions, its length can be considered a measure of material stability. Figure 1 shows an unexpected trend where stability increases with decreasing mean particle size. Since the sample mass was kept reasonably low (3–4 mg), the case of close packing of smaller particles hindering the oxygen supply to the sample can be ruled out. One possible explanation is that the surface of the smaller particles had already been partially oxidized before the measurement, so the oxidation peak observed during the DSC runs corresponds to the oxidation of more stable constituents, making the measured stability only apparently higher

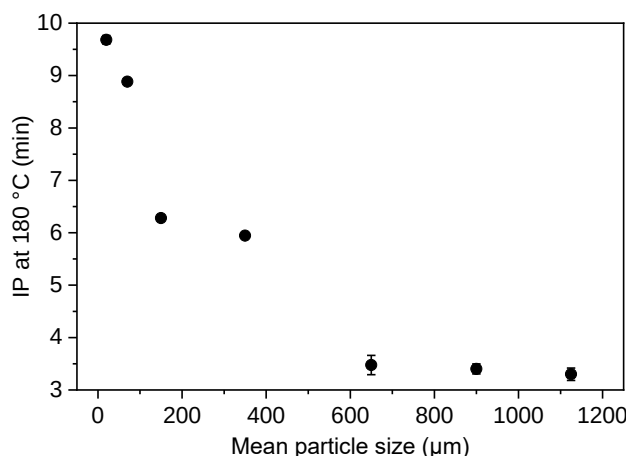


Figure 1. Induction period of thermal oxidation for samples with various mean particle sizes

Keywords: degradation, induction period, differential scanning calorimetry

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KINETIC ANALYSIS AND PREDICTIONS BASED ON THE NON-PARAMETRIC KINETICS (NPK) METHOD

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Data from thermoanalytical techniques such as thermogravimetry and differential scanning calorimetry are widely used for the kinetic analysis of complex solid-state processes, including decomposition, oxidation, curing, and crystallization. In many published studies, kinetic parameters have been evaluated using standard isoconversional methods. Since the late 1950s there is an apparently endless stream of “novel”, “advanced”, or “improved” isoconversional methods for obtaining kinetic parameters from non-isothermal data. However, it seems that the differences in the values of kinetic parameters (activation energy and pre-exponential factor) calculated using various methods are often below their uncertainties and the claimed improvement can only be perceived when working with simulated error-free datasets. This is not surprising as almost all these methods are based on the general rate equation (GRE) [1]

$$\frac{d\alpha}{dt} = k(T)f(\alpha)$$

The isoconversional methods eliminate the need for identifying the “model” characterized by the conversion function $f(\alpha)$; however, the temperature function is practically always expressed by the Arrhenius equation, $k(T) = A \exp(-E/RT)$. The methods then differ in the way in which the so-called temperature integral is approximated (in the case of integral methods) and/or the input data are transformed. The usual result of applying isoconversional methods is a set of usually conversion-dependent values of the kinetic parameters (apparent activation energy and the pre-exponential factor). The non-parametric kinetics (NPK) method [2, 3] stands out as it yields the numerical values of $k(T)$ and $f(\alpha)$ functions without assuming their functional form. In this regard, the NPK method is the only true “model-free” method which applies GRE directly without any additional assumptions.

This work focuses on the re-evaluation of selected thermoanalytical datasets previously analysed by conventional isoconversional approaches using the non-parametric kinetics method. The aim of this contribution is to compare the information obtained from standard isoconversional analysis with that provided by non-parametric kinetics. Attention is paid to kinetic predictions based on the parameters obtained using both isoconversional methods and the NPK method.

Keywords: general rate equation, isoconversional methods, kinetic parameters

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KINETIC ANALYSIS OF THE CRYSTALLIZATION OF GEHLENITE AND ANORTHITE IN AN ILLITE/SMECTITE-CACO₃ MIXTURES

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Waste materials containing CaO could be conveniently added to ceramic products to achieve better material properties while reducing some of the environmental burdens at the same time [1]. A contribution to the study of the effects of CaO on ceramic materials is presented. Namely, illite/smectite clay mixtures with the amount of CaCO₃ increasing from 17.6 wt.% to 25.6 wt.% are considered and the crystallization processes of gehlenite and anorthite in the range between 850 °C and 1050 °C are studied from heat flow curves measured in differential scanning calorimetry (DSC). The DSC peaks for the two processes, which are overlapping, are first decomposed and the activation energies are determined by the Kissinger, Flynn-Wall-Ozawa, and Friedman methods [2-4]. Based on these results, the best mixtures turn out to contain 19.6 wt.% and 21.6 wt.% of CaCO₃. Their overall activation energy varies between 404 kJ mol⁻¹ (the degree of conversion 0.1) and 332 kJ mol⁻¹ (the degree of conversion 0.9) and is equal to 360 kJ mol⁻¹ for the degree of conversion 0.5.

Keywords: kinetic analysis, gehlenite, anorthite, differential scanning calorimetry

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Poster Session 2

THERMAL CHARACTERIZATION OF STYRENE-BASED NANOCAPSULES PREPARED BY MINIEMULSION POLYMERIZATION

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Polymeric nanocarriers are promising platforms for the controlled delivery of active compounds in biomedical, cosmetic, agricultural and food applications. Miniemulsion polymerization offers a versatile route for their synthesis, allowing the formation of submicrometric polymeric particles^[1-3]. Plant oils are particularly attractive co-stabilizers due to their hydrophobicity, biocompatibility, biodegradability, and capacity to dissolve hydrophobic actives, as well as their frequent antioxidant and antiseptic properties. In addition, depending on the ratio oil/monomer that is used and the miscibility between the oil and polymer that is formed during the polymerization, phase segregation can occur inside the nanoparticles generating nanocapsules with a polymer shell and a liquid core. This work investigates the effect of oil type on the morphology and thermal behavior of polymer nanoparticles prepared by styrene miniemulsion polymerization, using sodium dodecyl sulfate as surfactant and 2,2'-Azobis(2-methylpropionitrile) AIBN as oil-soluble thermal initiator. Three co-stabilizers were evaluated: hexadecane (reference), jojoba (*Simmondsia chinensis*) oil, and andiroba (*Carapa guianensis*) oil. Transmission electron microscopy (TEM) was used to assess particle morphology and differential scanning calorimetry (DSC) to probe phase separation between the polymer shell and oil core^[3], a defining structural feature of nanocapsule morphology. For samples containing hexadecane or jojoba oil, TEM observations indicated the formation of particles with the core-shell morphology. DSC thermograms confirmed this phase separation by the presence of the melting peak of the respective oil and by the high glass transition temperature (114 °C) of high molar mass polystyrene. In contrast, particles prepared with andiroba oil exhibited a compact nanosphere morphology in TEM images, with no evidence of core-shell phase separation. DSC corroborated this finding: the melting peak of the oil was absent, and the glass transition temperature of polystyrene was considerably lower (52 °C), consistent with plasticization of the polymer matrix by the oil. This thermal behavior was attributed to in-situ compatibilization: the reactive double bonds of andiroba oil undergo covalent incorporation into the growing polystyrene chains during polymerization, as evidenced by molar mass distributions displaying a shoulder at intermediate molar masses. These findings indicate that DSC is a sensitive and discriminating technique for detecting polymer-oil phase boundaries in particulate systems, and that oil chemical composition governs the final particle morphology.

Keywords: Polymeric nanoparticles, Miniemulsion polymerization, Phase separation, Core-shell morphology Differential scanning calorimetry

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POLYMACROLACTONE NANOPARTICLES FOR BENZNIDAZOLE DELIVERY: THERMAL CHARACTERIZATION OF CROSSLINKED POLY(GLOBALIDE) NANOCARRIERS

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Polymeric nanocarriers represent a versatile strategy for enhancing the bioavailability of poorly water-soluble active pharmaceutical ingredients. In this work, an unsaturated polyester poly(globalide) (PGL), synthesized via enzymatic ring-opening polymerization (e-ROP)^[1,2], was employed to produce nanoparticles (NPs) by miniemulsification/solvent evaporation for the encapsulation of benznidazole (BNZ), a hydrophobic drug used in Chagas disease treatment^[3]. To modulate the physicochemical properties of the nanocarriers, the NPs were subsequently crosslinked via thiol-ene reaction using 2'-(ethylenedioxy)diethanethiol and azobisisobutyronitrile as initiator. The thermal behavior and crystallinity of PGL, crosslinked PGL, and BNZ-loaded NPs were investigated by differential scanning calorimetry (DSC), with complementary characterization performed by FTIR, SEM, and DLS. DSC analysis revealed that crosslinking and BNZ incorporation significantly reduced the crystallinity of the polymeric matrix, as evidenced by suppressed melting enthalpies and altered thermal transitions. These microstructural changes directly influenced drug release kinetics: crosslinked NPs exhibited accelerated BNZ release under controlled conditions, attributed to reduced crystalline order rather than purely degradation-driven mechanisms. Both PGL and crosslinked PGL NPs presented spherical morphology, mean diameters of approximately 100 nm, and narrow size distributions (PDI < 0.2). Encapsulation efficiencies of 92% and 85% were achieved for non-crosslinked and crosslinked NPs, respectively, confirming the suitability of this polyester matrix for BNZ encapsulation. These findings indicate that thermal analysis is an important tool for correlating polymer crystallinity with drug release behavior in polymacrolactone-based nanocarriers, offering a rational design pathway for hydrophobic drug delivery systems.

Keywords: Poly(globalide) nanoparticles, Differential scanning calorimetry (DSC), Thiol-ene crosslinking, Benznidazole encapsulation, Crystallinity and drug release

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CAN DSC REPLACE CONVENTIONAL PARAFFIN TESTING ACCORDING TO EN 12606-2?

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The industry is actively seeking new methods for determining the paraffin wax content of bituminous binders that are more economical, less complex, rapid, and solvent-free. Existing standard procedures, such as EN 12606-2, require extensive sample preparation and low-temperature filtration, while posing safety risks due to the high flammability of diethyl ether and the use of corrosive acids. Therefore, this study aims to develop a simple approach for paraffin determination suitable for routine laboratory use, requiring only a differential scanning calorimeter (DSC). To achieve this, we compared the standard test method with our newly developed DSC-based approach. The DSC method involves cooling to -60 °C and heating to 100 °C to quantify the fraction of paraffin wax that crystallizes during cooling and melts upon reheating. The enthalpy of the endothermic melting peak of the paraffin crystals was used to estimate the overall wax content. Consequently, this DSC approach has the potential to simplify routine analyses while reducing costs, analysis time and solvent consumption.

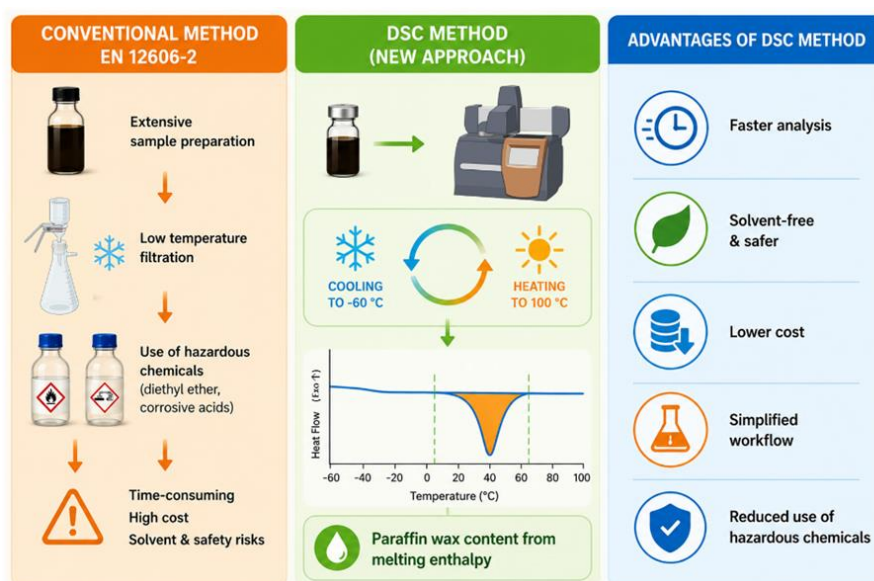


Figure 1. Comparison of standard and new DSC method

Keywords: bitumen, paraffin, differential scanning calorimetry, EN 12606-2

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METHODOLOGY FOR THERMAL CHARACTERIZATION OF CRYOGENIC FREEZING AND THAWING PROCESSES OF MULTICOMPONENT FOOD SAMPLES

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Freezing and thawing processes of food products are important parameters in determining the structural integrity and final quality of the product. Rapid cryogenic freezing using liquid nitrogen has attracted considerable attention due to its extremely high cooling capacity, enabling the formation of smaller ice crystals and reduction of cellular damage, drip loss, and textural deterioration compared to conventional freezing¹. Previous studies have shown that an increase in freezing rate results in finer ice crystal formation and improved preservation of muscle tissue in seafood products^{2,3}. To better understand these processes, precise temperature monitoring is required to investigate heat transfer mechanisms, phase transitions, and latent heat release during freezing and thawing.

In this work, a laboratory-scale experimental study was conducted to investigate cryogenic freezing and thawing of multicomponent food samples under controlled conditions. The multicomponent food samples in question were sushi products with systematically varied compositions for which a methodology was developed. Different proportions of fish, vegetables and rice, together with single-component samples were investigated to evaluate their influence on freezing and thawing behaviour. Temperature was continuously monitored using thermocouples positioned at selected locations together with infrared thermography. Freezing duration was varied as an additional processing parameter. The obtained thermal profiles showed that sushi composition affected thawing kinetics, resulting in differences in thawing time. The sample containing a higher proportion of vegetables and sauce required 14 minutes to thaw, whereas the sample with fewer vegetable and no sauce thawed in 11 minutes. Temperature evolution and heat-transfer characteristics also varied with composition, with 20 % greater heat uptake observed in the sample containing a higher proportion of vegetables and sauce. The developed methodology provides an initial platform for quantitative thermal characterization of cryogenic freezing and thawing processes in multicomponent food samples. The methodology was also coupled with calorimetric (DSC) measurements to quantify the latent heat effects associated with the observed phase transitions.

Keywords: freezing, thawing, sushi, infrared thermography, characterization

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DEVELOPMENT OF ECO-FRIENDLY ALKYL-GLUCOSIDE-BASED DETERGENT WITH IMPROVED LOW TEMPERATURE PERFORMANCE

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Sustainable cleaning products development has become one of the main challenges of today's chemical industry. Strict regulations ensuring the environmental safety necessitate a precise selection of biodegradable surfactants, chelators, hydrotropes, humectants, and solvents, in order to develop an efficient and eco-friendly detergent formulation. The detergent's performance is highly dependent on the temperature and micellar concentration. Since the micelles form only above the Krafft point temperature, it is necessary to lower it in order for a detergent to be efficient at low temperature conditions [1]. By changing the surfactant concentration and other components' contents, Krafft point temperature could be altered in a way mentioned before. In this work, alkyl-glucoside-based detergent has been developed and characterised. Several ratios of surfactant and humectant concentrations were used in formulating the detergents. The Krafft point temperatures of the formulations were determined from the alkyl-glucoside solubility curves, while differential scanning calorimetry (DSC) thermograms were used to characterize the corresponding thermal transitions. The best detergent formulation was further characterised for thermal stability via thermogravimetric analysis (TGA) and cloud point determination. The formulation's viscosity was measured at several temperatures using the Stabinger method to evaluate its ability to maintain higher retention time on the car surface, which is important for improving detergent contact time and cleaning efficiency across a wide temperature range.

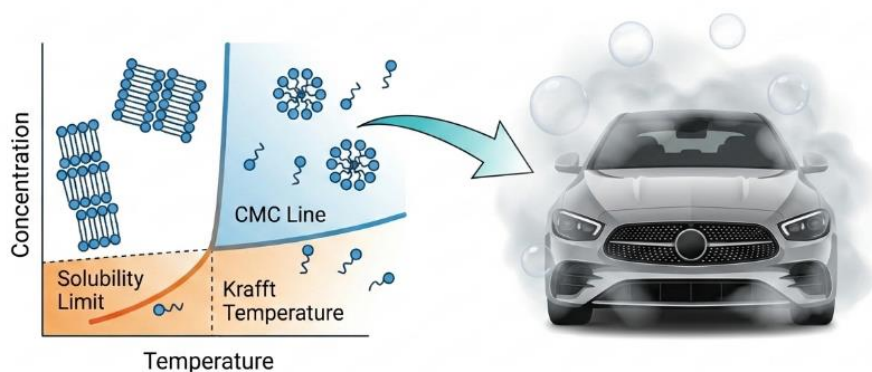


Figure 1. Solubility curve of a detergent for car maintenance.

Keywords: eco-friendly detergent, micelles, Krafft point temperature, alkyl-glucoside

Acknowledgements:

This work was carried out within the project PK.1.1.12.0124 – Smart Green WashPro – Development of Environmentally Friendly and Highly Efficient Formulations for Professional Vehicle Washing. The project was co-financed by the European Union through the European Regional Development Fund

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THERMAL, MECHANICAL, AND MORPHOLOGICAL CHARACTERIZATION OF ABS COMPOSITES REINFORCED WITH *SPARTIUM JUNCEUM* L. FIBERS: EFFECT OF FIBER CONTENT ON STRUCTURE–PROPERTY RELATIONSHIPS

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The development of sustainable fiber reinforced polymer composites depends on a systematic evaluation of the relationships between fiber incorporation, thermal stability, morphology, and mechanical performance. In this study, acrylonitrile–butadiene–styrene (ABS) composites reinforced with randomly oriented short *Spartium junceum* L. (SJL) fibers (5–20 wt.%) were prepared and systematically characterized using FTIR, DSC, TG/DTG, SEM, mechanical testing, and water absorption analysis. DSC results revealed that the glass transition of the butadiene phase was the most affected by SJL fiber incorporation, indicating changes in polymer chain mobility caused by fiber matrix interactions. FTIR analysis confirmed that the chemical structure of the ABS matrix remained largely unchanged after fiber addition, suggesting predominantly physical interfacial interactions and limited chemical compatibility between the constituents. TG/DTG analysis showed that low fiber contents slightly improved thermal stability, whereas higher fiber loadings accelerated the onset of thermal degradation due to the lower thermal stability of lignocellulosic fibers. SEM observations revealed that 10 wt.% SJL fiber loading provided the most homogeneous morphology, while higher fiber contents promoted fiber agglomeration, interfacial void formation, and reduced adhesion. Mechanical characterization demonstrated increased stiffness with fiber addition, whereas tensile strength remained relatively unchanged and elongation at break decreased due to restricted polymer chain mobility and interfacial defects. Swelling increased with increasing SJL fiber content as a consequence of the hydrophilic nature of SJL fibers. The results confirm the potential of SJL fibers as sustainable reinforcements for ABS composites, providing a favorable balance between thermal stability, mechanical performance, morphology, and water resistance.

Keywords: *Spartium junceum* L.; ABS composites; lignocellulosic fibers; thermal stability

Acknowledgements:

Parts of experiment were conducted using equipment procured within project co-financed by the Croatian Government and the European Union through the European Regional Development Fund the Competitiveness and Cohesion. Operational Program “Functional integration of the University of Split, PMF-ST, PF-ST and KTF-ST through the development of scientific research infrastructure in the building of three faculties” (KK.01.1.1.02.0018).

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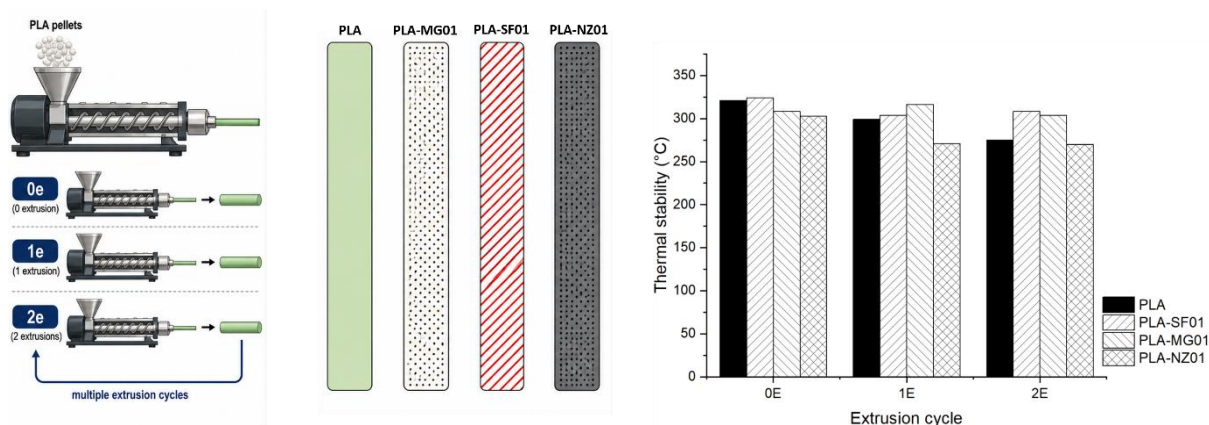
MULTIPLE EXTRUSION EFFECTS ON THERMAL PROPERTIES OF PLA/FE COMPOSITES

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Poly(lactic acid) (PLA) is a bio-based polyester widely used in packaging and material-extrusion (MEX) 3D printing, where thermal behaviour during repeated melt processing governs dimensional stability, crystallinity and recyclability. In multiple extrusion, PLA typically undergoes thermo-oxidative degradation through chain scission and transesterification reactions, which lower molecular weight and modify glass transition and melting temperatures. Previous studies on PLA reprocessing and PLA composites indicate that fillers and additives can either stabilise the matrix or catalyse its degradation, making the interplay between composition and processing history critical for filament design and recycling strategies [1,2].

In this work, neat PLA and PLA composites containing 0.1 wt.% magnetite (PLA-MG01), 0.1 wt.% strontium ferrite (PLA-SF01) and 0.1 wt.% nano zero-valent iron (PLA-NZ01) were prepared by melt mixing and subsequently subjected to one and two extrusion cycles to mimic filament production and re-extrusion. Thermal properties were evaluated by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). Main focus was on onset and maximum degradation temperatures as well as glass transition, cold crystallisation, and melting behaviour. The results show that repeated extrusion leads to a modest decrease in degradation onset and subtle shifts in T_g , T_{cc} and T_m for all materials, with the PLA-NZ01 exhibiting the most pronounced reduction in thermal stability. These observations suggest that for PLA/iron composites intended for MEX 3D printing or other applications involving multiple melt-processing steps, the choice of iron-based filler must balance added functionality with an acceptable loss of thermal stability.



Keywords: polylactide, composites, iron-based fillers, thermal analysis, multiple extrusion

Acknowledgements:

This work was supported by the project *Nanostructured Metal Oxides and Polymers for Advanced Functional Materials (NanoMOP)* (Project No. 112102).

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EFFECT OF EXTRACTION CONDITIONS ON PECTIN YIELD FROM CITRUS PEELS

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Citrus processing generates large amounts of waste including peels, seeds and pomace, accounting for 55 to 60 mass % of raw fruit weight. Lemon peels are rich in antioxidants, flavonoids, vitamin C and pectin. Pectin is a naturally occurring plant polysaccharide widely used in the food, pharmaceutical and cosmetic industries due to its gelling, thickening and stabilizing properties. Structurally, pectin consists mainly of homogalacturonan, rhamnogalacturonan I and rhamnogalacturonan II, with α -(1 $\rightarrow$ 4)-linked D-galacturonic acid forming its principal backbone.

This study investigated the effect of extraction temperature on pectin yield from citrus peels. Extraction was performed at a constant pH of 2 and at temperatures ranging from 60 to 100 °C using citrus peel particles with a size of up to 800 μ m. The recovered pectin was precipitated with ethanol, dried, and weighed to determine the pectin yield. Also the degree of esterification (DE) was determined. The results provide insight into the influence of extraction temperature on pectin recovery and contribute to the optimization of pectin extraction from citrus processing by-products, supporting their sustainable valorization.

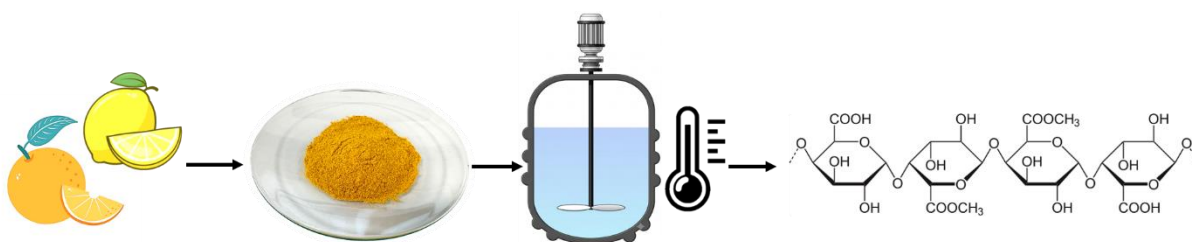


Figure 1. Shematic representation of pectin extraction from citrus peels

Keywords: citrus, extraction, pectin

Acknowledgements:

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EVALUATION OF PVC THERMAL STABILITY BY ISOTHERMAL THERMOGRAVIMETRIC ANALYSIS

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Thermal degradation of poly(vinyl chloride) (PVC) is a key factor affecting its processability. Therefore, evaluation of thermal stability is crucial for monitoring PVC quality. Thermogravimetric analysis (TGA) represents an industrially applicable method for determining the thermal stability of PVC due to the possibility of automated measurement of a series of samples. However, its potential in the isothermal mode has not yet been systematically evaluated. In this study, the thermal stability of unplasticized and plasticized PVC samples is evaluated using isothermal TGA. Given the enormous diversity of actual PVC products and the additives used in them, the study included samples stabilized with organotin, lead, and Ca/Zn compounds. For comparison, thermal stability was also determined using standard regulatory methods (Potentiometric titration, Congo Red Test). The results indicate that for unplasticized samples, thermal stability can be evaluated directly from the isothermal TGA curve. For plasticized formulations, however, the specific stabilization system plays a critical role. While the thermal stability of plasticized samples containing Ca/Zn or lead-based stabilizers can be successfully determined by taking the derivative of the TGA curve, significant evaluation complications arise in the case of organotin-stabilized systems.

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THERMOGRAVIMETRY OF COMPLEX ORGANOMINERAL SUBSTRATES: EXPERIMENT WITH THE MULTICHANNEL MACRO INSTRUMENT

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Thermogravimetric (TG) analysis of complex organo-mineral substrates, such as soils, manures, composts, and related materials, is often complicated by their inherent heterogeneity. Although sample pretreatment methods, including sieving, crushing, and grinding, can reduce variability, they may also alter the original structure of the material and thereby affect the resulting TG data [1]. An alternative approach is to increase sample mass to the gram scale, which improves representativeness but is not supported by most conventional thermogravimetric analysers. Even when larger sample masses are possible, challenges may arise, including temperature gradients within the sample, altered TG curves, and limited contact with the reactive atmosphere.

This contribution presents practical experience with a relatively new type of multichannel macro thermogravimetric analyser that enables simultaneous analysis of up to 19 samples with sample masses in the gram range. The instrument allows direct measurement of heterogeneous materials without extensive sample pretreatment, thereby preserving their natural structural characteristics and improving the representativeness of the analysis.

The presented examples focus primarily on soils, for which previous studies have demonstrated significant correlations between TG parameters and key soil properties, including soil organic carbon, total nitrogen and microbial parameters such as microbial carbon and respiration [2]. In addition, the conventional TG revealed significant number of autocorrelations among thermal mass losses [3]. Further results originate from the analysis of properties of manures prepared under various conditions and differing in their maturation degree. Results obtained with the macro TG analyzer indicate that, despite more complex data processing requirements, the technique provides a rapid and accurate method for characterizing soils and other organo-mineral substrates. The ability to analyse larger, minimally processed samples represents a substantial methodological advantage, particularly for heterogeneous environmental materials where conventional TG approaches may be limited.

Keywords: thermogravimetry; soil; manure; high sample mass

Acknowledgements:

This research was supported by the by the Technology agency of the Czech Republic project No. SQ01010201-Valorization of carbonaceous materials for reducing greenhouse gas emissions in livestock production and supporting primary production and carbon sequestration in soil.

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INTEGRATED ASSESSMENT OF SOIL MICROBIAL FUNCTIONING: COMBINING ENZYMATIC ASSAYS AND MICROCALORIMETRY TO EVALUATE THE IMPACT OF SULFAPYRIDINE

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Soil microbial activity and enzymatic functions are sensitive indicators of biogeochemical processes and overall ecosystem health. This study focuses on the assessment of key soil enzymes involved in carbon and nitrogen cycling – namely dehydrogenase, urease, and beta-glucosidase – across three contrasting soil types. While these enzymes provide insight into oxidative activity, nitrogen mineralization, and carbon turnover, a comprehensive understanding of microbial response to environmental stressors requires a thermodynamic perspective [1-3].

To complement biochemical assays, isothermal microcalorimetry was employed as a reliable method to study the thermodynamics and kinetics of microbial metabolism. From the measured power-time curves, key parameters were determined, including the maximum normalized heat flow (P_{max}), the time to peak (t_{max}), total heat dissipation (Q), and the thermodynamic parameter (X_0). Furthermore, the microbial growth rate constant (k) was calculated by fitting an exponential growth model to the normalized heat flow data [4].

These integrated methodologies were applied to evaluate the impact of the pharmaceutical compound sulfapyridine on soil microbial processes. The study aims to determine how sulfapyridine influences both specific enzymatic pathways and global metabolic heat output across different soil matrices. By analyzing variations linked to soil physicochemical properties, this work highlights the role of the soil environment in modulating chemical stress. The findings contribute to a better understanding of soil biochemical functioning and emphasize the importance of combining microcalorimetric and enzymatic tools for robust soil quality assessment and agricultural sustainability.

Keywords: Soil enzymes, Isothermal microcalorimetry, Sulfapyridine

Acknowledgements:

This work is dedicated to the memory of Professor František Krčma, whose exceptional scientific vision and kind mentorship were fundamental to the realization of this research.

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COUPLED THERMOGRAVIMETRIC TECHNIQUES FOR TEXTILES

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Thermogravimetric analysis (TGA) monitors changes in the mass of a sample, for example, in dynamic mode, where the temperature rises at a constant rate, or in isothermal mode, at a constant temperature over time. The data are usually used for determination of thermal stability, and for kinetics of thermal degradation. However, coupling with other analytical techniques can add much more information, mainly about degradation products. In this work, we describe usage of TGA and coupled TGA techniques for advanced analysis of polymeric textiles, including those based on cellulose processed using ionic liquids.

Figure 1 shows results from coupled thermogravimetric analysis – mass spectrometry for such cellulose textile. Water ($m/z = 18$) was released in two steps, firstly around 100 °C (absorbed water), and secondly around 317 °C (water from dehydration reaction). The results can be used to determine the water content and thermal stability[1].

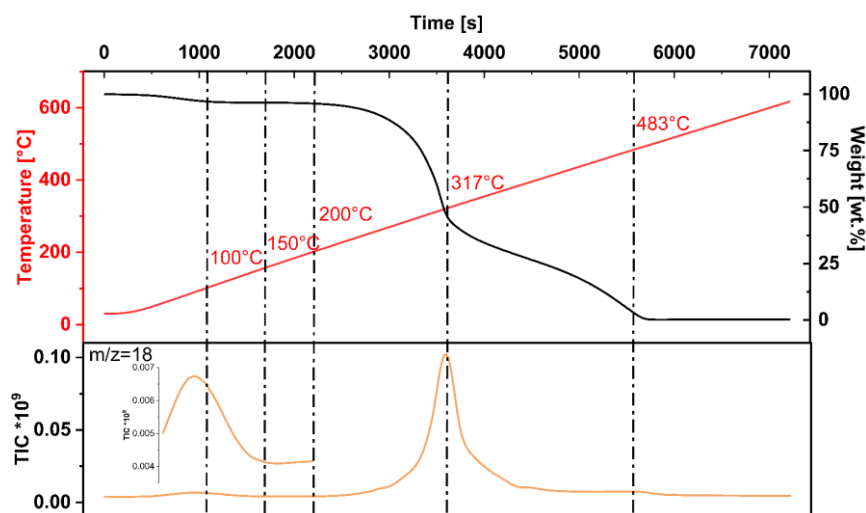


Figure 1. Thermogravimetric analysis – mass spectrometry for cellulose textile

Keywords: thermogravimetric analysis, thermal stability, cellulose, polyester

Acknowledgements:

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RECYCLING OF WASTE FISHING NETS: IDENTIFICATION OF THE POLYMER TYPE USING THERMAL ANALYSIS AND THERMOCHEMICAL RECYCLING VIA PYROLYSIS

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Marine environments are among the most polluted ecosystems from the accumulation of post-consumed plastics. Approximately 20% of marine plastic wastes originates from marine activities, such as the disposal of fishing nets. Fishing nets are typically consisted of synthetic polymers, such as polyethylene or poly(ethylene terephthalate), with nylon-6 (polyamide-6, PA-6) constituting the largest fraction. A key step towards integrating waste fishing nets into the Circular Economy of Plastics is the identification of their polymer composition. For this purpose, Differential Scanning Calorimetry (DSC), Fourier Transform Infrared Spectroscopy (FTIR) and Evolved Gas Analysis (EGA) of the pyrolysis products were employed [1,2]. The obtained FTIR spectra were compared with reference spectra of common polymers to identify matching functional group patterns. Thermal transition temperatures, such as glass transition (T_g), crystallization (T_c) and melting temperature (T_m), determined via DSC, were compared with the known values for standard polymers. Furthermore, the detection of characteristic chemical markers in pyrolysis oil, associated with the thermal degradation of specific polymers, enabled the identification of the polymer composition of fishing nets. Furthermore, for PA-6-based samples, their potential thermochemical recycling via pyrolysis was investigated in a lab-scale fixed-bed reactor. Both thermal and catalytic pyrolysis were investigated, following catalyst screening using standard PA-6 as a model feedstock for the optimal catalyst selection. The pyrolysis products were thoroughly characterized and the monomer concentration (ϵ -caprolactam) was quantified via calibrated GC-FID in order to enable an accurate evaluation of monomer recovery (Figure 1).

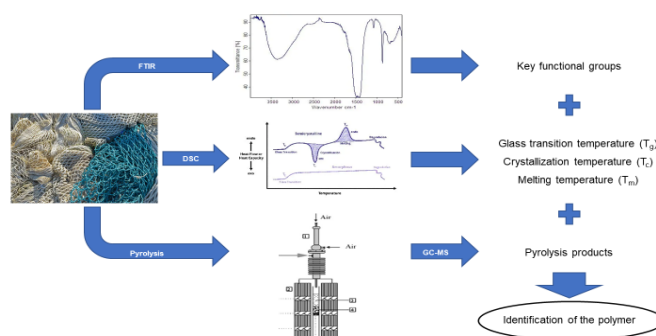


Figure 1: Flowchart of the proposed identification and recycling methods.

Keywords: Polymer recycling, waste management, waste fishing nets, pyrolysis

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FAST SCAN CALORIMETRY (FSC) AND DSC STUDY OF THE THERMAL BEHAVIOR OF POLY(PROPYLENE TEREPHTHALATE)

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Poly(propylene terephthalate) (PPT) or poly(trimethylene terephthalate) (PTT), is a biobased aliphatic aromatic polyester with favorable properties for fiber production. In this work Shell's Corterra PPT was used. A thorough study of isothermal and dynamic crystallization, as well as the multiple melting of PPT was performed using Standard Differential Scanning Calorimetry (DSC) and Fast Scanning Calorimetry (FSC) [1]. Isothermal crystallization experiments of PPT were carried out at 23 different temperatures from 169 to 209°C. The multiple melting behavior of PPT was interpreted on the basis of partial melting, recrystallization and final melting. The equilibrium melting temperature was found $T_m^0 = 281.6^\circ\text{C}$ by applying the Hoffman-Weeks method. To investigate crystallization upon cooling from the melt, experiments at 42 different cooling rates were carried out. Finally, crystallization upon heating from the glass was studied by performing experiments at 25 different heating rates following melt-quenching. Various mathematical models were elaborated for the analysis of the kinetics of PPT crystallization. The activation energy of non-isothermal crystallization was calculated using the isoconversional method of Friedmann [2]. With increasing cooling rate a larger supercooling is needed for PPT to crystallize, but finally this leads to faster crystallization rates. In case of crystallization upon heating from the glass, the crystallization peak temperature shifts strongly with the applied rate. In general, PPT could not be obtained in the fully amorphous state even by melt-quenching in standard DSC. Crystallization from the melt was prevented at cooling at rates higher than 5°C/s (300°C/min). Cold-crystallization was diminished at heating rates faster than 25°C/s (1500°C/min). From the FSC study of crystallization and melting of PPT it was concluded that recrystallization is important. We discuss the effect of heating rate on the glass-to-liquid temperature, the cold crystallization and re-crystallization process as well as the melting temperature.

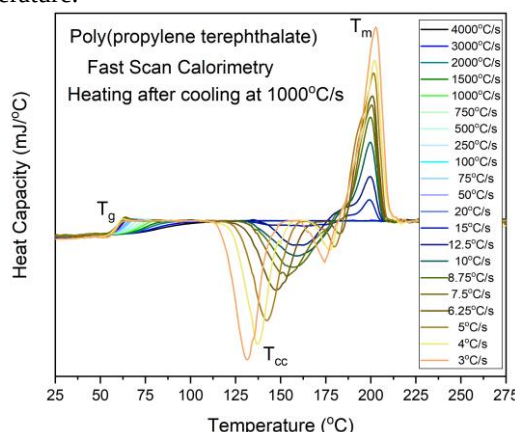


Figure 1. FSC thermograms for PPT at different heating rates after cooling at 1000°C/s from the melt.

Keywords: Poly(propylene terephthalate), DSC, Fast Scan Calorimetry, Crystallization

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THERMAL CHARACTERIZATION OF RPET NANOCOMPOSITES CONTAINING SEWAGE SLUDGE-DERIVED BIOCHAR

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- Poly(ethylene terephthalate) (PET) is a widely used thermoplastic polymer due to its excellent mechanical properties, dimensional stability and chemical resistance. Its ability to withstand exposure to a wide range of chemical agents, makes it particularly suitable for packaging applications in the food, beverage, and pharmaceutical sectors [1]. However, recycled PET (rPET) usually exhibits degraded properties compared to virgin PET. Biochar has emerged as a promising sustainable filler to enhance the thermal properties of polymer matrices [2]. In this work nanocomposites of rPET reinforced with 0.5, 1, 2.5, and 5 wt% biochar, along with neat rPET, were prepared and characterized. Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA) were employed in order to provide information on crystallization, glass transition, and thermal stability. TGA data indicate changes on thermal stability with the increase of biochar content. For quenched samples, DSC thermograms provided valuable insights into the glass transition (T_g), cold crystallization (T_{cc}), and melting (T_m) behavior of the nanocomposites. Overall, the combined DSC and TGA analyses demonstrate that sewage sludge-derived biochar can effectively modify the thermal behavior of rPET, highlighting its potential as sustainable reinforcing filler for the development of valuable recycled polymer nanocomposites.

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Keywords: Thermogravimetric Analysis (TGA), Differential Scanning Calorimetry (DSC), Polymer Nanocomposites.

Acknowledgements:

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THERMAL ANALYSIS AND CALORIMETRY FOR ASSESSING ATOMIC OXYGEN PLASMA CLEANING OF OIL PAINT SYSTEMS

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Surface cleaning is one of the most common and critical procedures in cultural heritage conservation. Within the MOXY project, a novel non-contact cleaning approach based on cold plasma-generated atomic oxygen (AO) is being developed as a sustainable alternative to conventional solvent- or mechanically-based methods [1]. Due to its high reactivity, AO is expected to oxidize carbon-based contaminants; however, its interaction with original artistic materials must be thoroughly assessed before practical application.[2]

This work investigates the effects of AO treatment on oil paint model systems prepared from linseed oil and ultramarine blue pigment. Fresh and naturally aged mock-ups were exposed to AO under controlled conditions and subsequently characterized through a multi-analytical approach. The oxidation of oil is a well-studied process involving radical chain reactions [3]. Differential Scanning Calorimetry (DSC) was employed to evaluate changes in radical activity and peroxide-related oxidation processes in the paint binder, while Thermogravimetric Analysis (TGA) provided quantitative information on mass variations and thermal stability. Evolved Gas Analysis coupled with Mass Spectrometry (EGA-MS) was used to investigate possible modifications in the polymeric network of the drying oil by monitoring characteristic pyrolysis products associated with free fatty acids, oligomers and highly cross-linked fractions. Complementary information was obtained by Solid-Phase Microextraction Gas Chromatography–Mass Spectrometry (SPME-GC/MS), used to monitor volatile organic compounds generated during oxidative degradation, and by ATR-FTIR spectroscopy for assessing surface chemical changes. [3]

The results demonstrate that thermoanalytical techniques are particularly effective in detecting AO-induced modifications in oil paint systems. In particular, EGA-MS showed high sensitivity to changes in the binder polymeric structure, while DSC and TGA provided valuable information on oxidation-related processes and mass variations. These findings highlight the potential of thermal analysis and calorimetry as powerful tools for evaluating the compatibility and safety of innovative plasma-based cleaning technologies for cultural heritage materials.

Keywords: surface cleaning, plasma-generated atomic oxygen, DSC, TGA-FTIR, EGA-MS

Acknowledgements:

The research is supported by the EU Horizon “MOXY” project, Grant 101 061 336

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<https://doi.org/10.1038/s40494-025-02067-1>

COMBINED DSC AND THERMOGRAVIMETRIC ANALYSIS OF THE CURING BEHAVIOUR OF CHINESE AND JAPANESE URUSHI LACQUERS

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- Urushi, the traditional East Asian lacquer obtained from the sap of *Toxicodendron vernicifluum* widely used in East Asia as coating on wooden and metal surfaces, cures through an enzyme-mediated oxidative process leading to the formation of a highly crosslinked polymeric network. [1] Scientific investigations on these materials remain relatively fragmented and embryonic, and the curing behaviour of urushi films as well as their long-term ageing processes are still not fully understood. This knowledge gap is largely attributed to the complexity of traditional lacquer systems, the limited availability of raw materials and the difficulty in accessing traditional preparation recipes and artisanal manufacturing protocols. Moreover, artists and conservators frequently report significant differences in both short- and long-term performance between Chinese and Japanese urushi lacquers, even when derived from the same botanical species. At the same time, lacquer films containing different pigments often exhibit distinct degradation pathways during ageing [2].
 - This work investigates the curing behaviour of commercially available Chinese and Japanese urushi lacquers and evaluates the effect of two traditional pigments, carbon black and Bengara (Fe_2O_3), using a combined thermoanalytical approach.
 - Six mock-up systems were prepared according to traditional formulations, including pure Chinese and Japanese urushi and the corresponding pigmented mixtures. Differential Scanning Calorimetry (DSC) was employed to monitor peroxide evolution during curing, while Thermogravimetric Analysis (TGA) was performed both under nitrogen to characterize the thermal decomposition profile and under isothermal conditions (80 °C, air atmosphere) to evaluate oxygen uptake associated with oxidative crosslinking. [3, 4]
 - DSC measurements revealed that peroxide concentration reaches a maximum after approximately three days of curing, followed by a progressive decrease up to 17 days. However, the exothermic signal remained detectable throughout the investigated period and increased again after 55 days, indicating that peroxide formation and consumption continue over more than one month and that the oxidative network remains dynamically evolving. The incorporation of both carbon black and Bengara reduced the thermal stability of the peroxide species, as indicated by lower onset temperatures, with the strongest effect observed for Japanese urushi.
 - Isothermal TGA showed an initial mass loss due to water evaporation followed by a mass increase associated with oxygen uptake during autooxidative crosslinking. Pure Chinese and Japanese urushi exhibited oxygen uptakes of approximately 3–4 wt%, whereas Bengara reduced oxygen incorporation, demonstrating its influence on the curing process. Moreover, oxygen uptake reached a plateau without any subsequent mass decrease during the 10,000 min experiment, indicating the absence of measurable oxidative degradation. This finding is consistent with SPME analyses, which did not reveal significant molecular evolution of volatile degradation products.
 - The combined DSC–TGA methodology provides complementary information on peroxide evolution, oxygen consumption and polymer network formation, offering a powerful approach for assessing curing kinetics and the influence of pigments in traditional urushi lacquer systems.

Keywords: Urushi, oxidative curing, DSC, thermogravimetry, oxygen uptake, cultural heritage.

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IDENTIFICATION AND COMPATIBILITY ASSESSMENT OF BLUE PIGMENTS AND CONSOLIDANTS IN HISTORICAL GLASS PAINTINGS

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Since ancient times, it has been known that the paint layers of various art and heritage items are constantly exposed to aggression from external agents (atmosphere, temperature, humidity, storage conditions, etc.). The degradation mechanisms caused by these factors can result in irreparable damage to art objects and may occur even when specific materials are used for conservation, restoration, or retouching [1].

The degradation of the pigments is directly related to the poor state of conservation of the entire work, affected by the instability of the cohesion and adhesion of the binder in the preparation layer, the eco-emission of the colour binder, or the interactions that may occur between the pigment and the binder [1,2].

The purpose of this study is to identify pigments, particularly blue pigments, from two traditional paintings dating from the 19th century (Transilvania). Both are reversed glass paintings, distemper entitled "Saint prophet Elijah" and "Great feast icon with four scenes". After identifying and characterizing the pigments, the aim is to select a consolidant with compatible visual and physico-chemical properties to ensure appropriate treatment for the specific characteristics of the polychromy.

Fourier-Transform infrared spectroscopy (FT-IR) and thermal analysis (TGA/DSC) were used to identify the chemical structure of the compounds, their thermal behaviour and compatibility between pigments and binders to be used. Particle morphology, colour coordinates, miscibility, and the appearance of the films were assessed using optical microscopy.

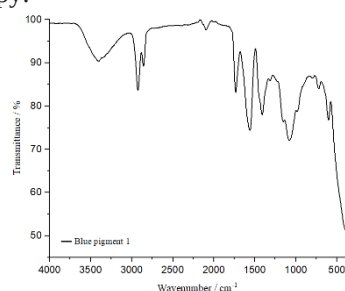


Figure 1. FT-IR spectra of blue pigment sample from "Great feast icon with four scenes"

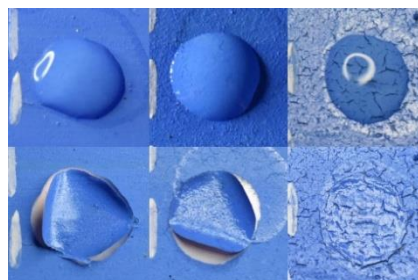


Figure 2. Pigment-binder mixture

Keywords: restoration process, pigment-binder interaction, glass paintings, physico-chemical characterization

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MULTI-TECHNIQUES ANALYSIS OF ROMAN-DACIAN STONE UNDERGOING HYDROGEL-BASED CLEANING TREATMENTS

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One of the most common and crucial task in cultural heritage conservation is cleaning [1]. Scientists in coservation field focused on the development of cleaning techniques that minimize the negative effects o conventional cleaning methods [2].

The study evaluates cleaning tests conducted on roman stone fragments of funerary and votive origin, salvaged from the Roman-Dacian settlement of Apulum, now part of the collection of the National Museum of the Union in Alba Iulia, Romania. The research focused on the mineralogical characterization of the stone, aiming to identify residues before and after cleaning, assess microstructural integrity following cleaning and to determine the efficacy of the cleaning treatements. Eight treated and untreated stone samples were analyzed via TG, FT-IR, SEM-EDX, and XRD to assess cleaning-induced changes. FT-IR was also applied to six formulations (Laponite RD, Klucel G, and agar-agar with water or ammonium carbonate) and their components. All hydrogel formulations were prepared at a concentration of 4%. Beside the hydrogel, biocide cleaning was used as a conventional method. The results of this study indicate the type of stone used for the funerary relief and the differences between untreated and treated samples regarding the interaction between the cleaning materials and the stone substrate.

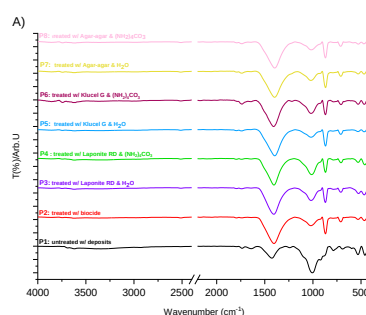


Figure 1. Overlaid spectra of stone samples (P1: untreated with deposits. P2: treated with biocide, P3: treated with Laponite RD and H₂O, P4: treated with Laponite RD & (NH₄)₂CO₃, P5: treated with Klucel G & H₂O, P6: treated with Klucel G & (NH₄)₂CO₃, P7: treated with Agar-Agar & H₂O, P8: treated with Agar-Agar & (NH₄)₂CO₃)

Keywords: stone, XRD, Infrared spectroscopy, SEM-EDX, thermal analysis

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COMPARATIVE STUDY ON THE INFLUENCE OF THERMAL, CHEMICAL, AND UV DEGRADATION FACTORS ON NATURAL AND SYNTHETIC FIBERS IN FORENSIC CONTEXT

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Fibers represent relevant forensic evidence in forensic investigations, being important tools in the investigation of crimes such as murder, sexual assault, accidents or deaths caused by a firearm, and road accidents [1,2]. Generally, fibers have been classified into two categories: natural fibers, obtained from various natural sources, such as plants or animals, and synthetic fibers, obtained from chemical processing of raw materials [3].

The research focuses on investigating how thermal stress, disinfectant, sulfuric acid and UV radiation act on the chemical structure of natural and semi-synthetic fibers. Five sets of samples were prepared in the current study: untreated natural flax fibers and semi-synthetic fibers, untreated fibers subjected to thermal stress for 14 days, treated fibers subjected to thermal stress for 14 days, fibers treated with H₂SO₄ 2N and the last set of samples was subjected to UV radiation. To analyze the action of degradation factors on the fibers, infrared spectroscopy (FT-IR), thermal analysis (TG/DTG), scanning electron microscopy with energy dispersive X-ray Spectroscopy (SEM-EDX) and comparator microscope were used. The results show that degradation factors produce changes in the chemical structures of the fibers, observed in the FT-IR spectra, but also in the thermal degradation processes, according to the TG and DTG curves, but their identification can be achieved.

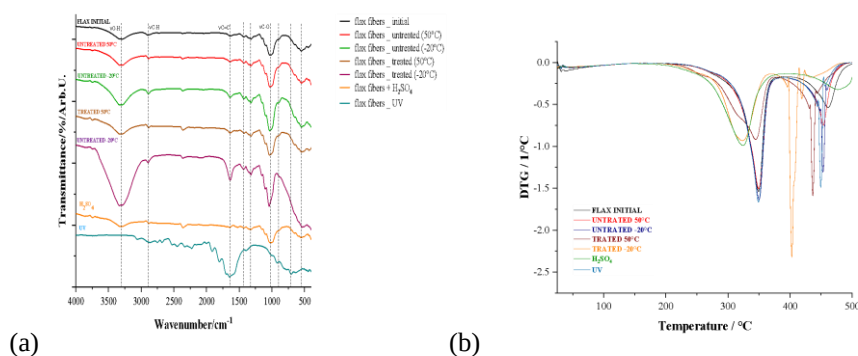


Figure 1. FT-IR spectra (a) and DTG curves (b) of flax fibers

Keywords: thermal analysis, degradation factors, fibers, forensic investigations.

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THERMAL AND COMPLEMENTARY CHARACTERIZATION OF SUSANI-GRĂMURADA HERITAGE ARTEFACTS

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Artifacts analysed in this study originate from funerary contexts investigated at the Grămurada site in Jupani (Susani, Timiș County), dated to the Late Bronze Age. The research focuses on evaluating the thermal behaviour and compositional characteristics of several metal fragments and structurally different materials to identify their technological particularities and the transformation processes they underwent within the archaeological environment. The use of thermoanalytical methods in the investigation of heritage materials enables the identification of thermal stability and structural changes associated with processing and alteration processes [1]. Combined TG/DTG, HF, and SEM-EDX analyses revealed that sample thermal stability and oxidation vary significantly, likely due to differing metallic components.

The lack of significant mass loss indicates a low amount of organic impurities and confirms the predominance of mineral and metallic phases within the analysed materials, observations consistent with findings reported in other thermoanalytical studies on archaeological materials [2]. SEM-EDX analyses reveal structural and compositional differences between the investigated artefacts, suggesting the use of distinct processing technologies and varied raw materials. The combination of microscopic and thermoanalytical data indicates the existence of differentiated manufacturing and transformation processes of materials in a funerary context. The results obtained highlight the usefulness of thermoanalytical methods in characterising archaeological artefacts and evaluating their alteration processes [3].

Keywords: TG/DTG, archaeological heritage, SEM-EDX, Bronze Age, thermal behavior

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DEVELOPMENT AND PHYSICOCHEMICAL CHARACTERIZATION OF BIOPOLYMER HYDROGELS INCORPORATING ANTIHYPERTENSIVE SARTANS

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Hydrogels represent promising pharmaceutical platforms for enhancing the administration and therapeutic efficacy of orally administered drugs, particularly in pediatric and geriatric patients with chronic cardiovascular diseases. Compared to conventional solid dosage forms, hydrogel-based systems offer several important advantages, including easier administration, improved patient compliance, flexible dosing, and the potential for controlled drug release. These characteristics are especially relevant for antihypertensive therapy, where long-term treatment adherence is essential [1].

The present study focuses on the development of natural biopolymer hydrogels based on sodium alginate and gelatin intended for the incorporation of antihypertensive sartans, namely irbesartan, losartan, and valsartan. Due to their biocompatibility, biodegradability, low toxicity, and favorable gel-forming properties, both polymers represent attractive candidates for the formulation of patient-friendly oral drug delivery systems. The selection of excipients and the assessment of drug-polymer compatibility were based on our previous systematic investigations regarding the thermally induced interactions between sartan-class active substances and various pharmaceutical additives [2].

In the preliminary phase of the study, blank hydrogel matrices were synthesized and subjected to physicochemical characterization. FT-IR-UATR analysis was employed to investigate the structural organization of the polymeric network and the intermolecular interactions between alginate carboxyl groups and gelatin amino groups, confirming the formation of stable hydrogel matrices. Thermal behavior and degradation patterns were further evaluated by TG/DSC analysis, demonstrating adequate thermal stability for potential pharmaceutical applications.

The ongoing stage of the research involves incorporating sartans into optimized hydrogel matrices and evaluating drug-polymer compatibility. Complementary analyses, including UV-Vis spectroscopy for monitoring the in vitro release profile and HPLC analysis for validating drug release and encapsulation efficiency, will provide additional insight into the controlled-release behavior of the developed formulations.

The proposed hydrogel systems may represent a promising alternative to conventional antihypertensive dosage forms, especially for long-term administration in elderly and pediatric patients. Their ability to facilitate administration, improve acceptability, and potentially provide modified drug release makes these formulations attractive candidates for future personalized pharmaceutical therapies.

Keywords: hydrogels; sartans; antihypertensive drugs; drug delivery systems; thermal analysis, biopolymers

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IDENTIFICATION AND CHARACTERIZATION OF ROAD-TRAFFIC PAINT TRACES USING FTIR SPECTROSCOPY AND THERMOGRAVIMETRIC ANALYSIS A MULTI-MODAL, DATABASE-DRIVEN APPROACH

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Road traffic, marine accidents generate a variety of physical and chemical traces that can be exploited for forensic investigation, accident reconstruction, and liability determination [1-2]. The identification and characterization of such traces, including paint fragments, rubber residues, polymeric materials, and combustion by-products requires analytical techniques capable of providing both chemical composition and thermal stability data [1-2]. In this study, Fourier-Transform Infrared Spectroscopy (FTIR) and Thermogravimetric Analysis (TGA) were used as complementary methods for the identification and characterization of trace evidence collected from real and simulated road accident scenes.

FTIR analysis was applied to identify functional groups and polymer types present in paint chips, tyre marks, and plastic debris collected from impact area. TGA was used to assess thermal decomposition profiles of the same materials, providing complementary information on filler content, polymer blends, and degradation temperatures. The combination of both techniques allowed reliable differentiation of chemically similar materials that could not be distinguished by a single method.

The results demonstrate that FTIR and TGA constitute a powerful analytical toolkit for forensic trace evidence examination in traffic accident investigations. Their non-destructive or minimally destructive nature, combined with high chemical specificity, makes them suitable for integration into standard forensic protocols. Future work will focus on building a dedicated spectral and thermogravimetric database of automotive materials to support systematic trace comparison in forensic casework.

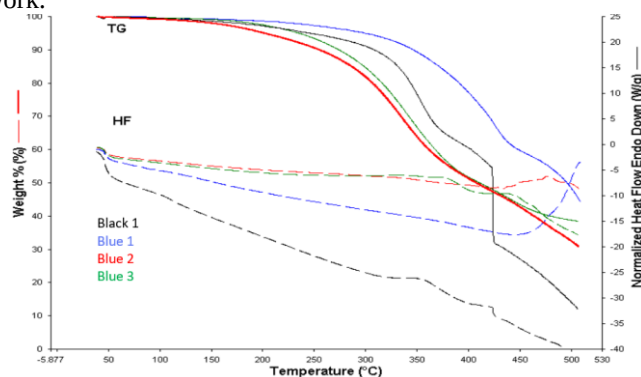


Figure 1. TGA/HF curves of a paint chip sample collected from an accident

Keywords: forensic analysis, traffic accident traces, paint evidence

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DRUG–DRUG PHYSICOCHEMICAL COMPATIBILITY ASSESSMENT OF A PARACETAMOL:AMPICILLIN:IBUPROFEN SYSTEM

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Co-administration of multiple drugs is a common clinical practice, particularly in the management of pain, fever, and infectious conditions. Paracetamol (PAR), Ampicillin (AMP), and Ibuprofen (IBU) are among the most widely prescribed active pharmaceutical ingredients worldwide, frequently encountered in simultaneous therapeutic regimens [1-2]. Despite their individual pharmacological profiles being well established, the physicochemical interactions that may arise between them, whether in formulation, storage, or combined administration remain insufficiently characterized.

This study presents a systematic compatibility investigation of binary (PAR–AMP, PAR–IBU, AMP–IBU) and ternary (PAR–AMP–IBU) mixtures at equimolar (1:1 and 1:1:1) ratios. Solid-state and solution-phase interactions were evaluated through a multimodal analytical framework combining Fourier Transform Infrared Spectroscopy (FTIR), Thermogravimetric Analysis (TGA), DSC, and Reversed-Phase High-Performance Liquid Chromatography (RP-HPLC).

The originality of this work lies in the concurrent application of three complementary techniques: spectroscopic, thermal, and chromatographic on the same set of binary and ternary mixtures, enabling cross-validation of findings and a more complete mechanistic picture of the interactions involved.

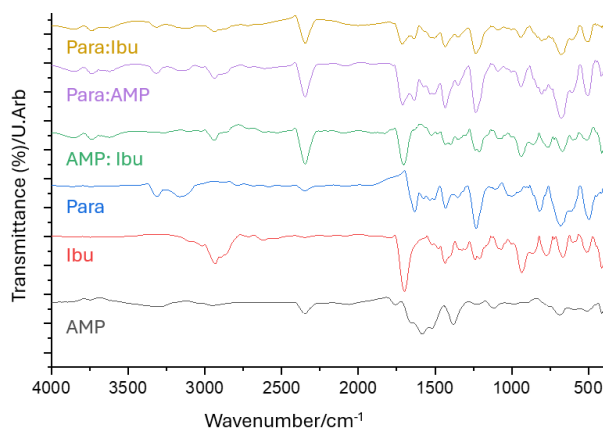


Figure 1. FTIR curves of the active substances, binary mixtures

Keywords: thermal analysis, antibiotics, chemical interactions, compatibility

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PHOSPHORUS-CONTAINING MOF–IONIC LIQUID HYBRID MATERIALS WITH ENHANCED THERMAL STABILITY AND PHENOL ADSORPTION CAPACITY

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Metal-organic frameworks (MOFs) impregnated with ionic liquids (ILs) represent promising hybrid materials for the efficient removal of phenol from wastewater due to their unique structural properties and tunable surface chemistry. Trioctylmethylammonium chloride was used to and impregnate pristine phosphorus-based MOF–IL composites containing zinc and copper centers connected with phosphonoacetic ligands (PA). Microwave-assisted and low-temperature hydrothermal synthetic methods of MOFs prove to improve both time and energy efficiency, aligned with green chemistry principles [1]. We looked into their thermal stability and adsorption capabilities. The materials' thermal stability was improved by the phosphorus-containing ligand; the anhydrous MOFs remained stable up to 350 °C [2]. The incorporation of ILs within the porous network of the MOFs improved the phenol adsorption capacity, reaching values of up to 20 mg/g under optimized conditions. Thermogravimetric analysis revealed that the MOF–IL composites exhibited excellent thermal stability, with decomposition temperatures exceeding 350 °C, indicating their thermal suitability and reuse in industrial wastewater treatment applications.

The synergistic effect between the high surface area of the MOFs and the selective affinity of the ILs toward phenol molecules contributed to enhanced adsorption kinetics and capacity. These findings demonstrate that MOF–IL composites are viable materials for phenol remediation from contaminated water, combining efficient adsorption performance with the robust thermal properties required for practical applications [3].

The presence of IL improves the adsorption capacity of MOFs as it brings to the surface of the adsorbent materials an increase in the number of functional groups, which leads to better adsorption performance, as electrostatic interactions between the positive charge of the IL and the electronegativity of oxygen molecules from phenol take place more easily. The study also emphasizes how phenol desorption and electrooxidation processes can be used to simultaneously degrade pollutants and regenerate adsorbents.

Keywords: MOFs, Phosphonates, adsorption thermodynamics, water remediation

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HIGH-PERFORMANCE DICLOFENAC REMOVAL USING AN IONIC LIQUID-MODIFIED COBALT: KINETIC, EQUILIBRIUM, AND THERMODYNAMIC STUDIES

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This study reports the synthesis, functionalization, and adsorptive application of a cobalt-based phosphonate metal-organic framework (CoMOF) for efficient removal of diclofenac (DCF), a persistent pharmaceutical contaminant frequently detected in natural water bodies. CoMOF was synthesized under environmentally benign conditions [1] and subsequently functionalized with two quaternary ionic liquids — Aliquat 336 (a quaternary ammonium-based ionic liquid) and Cyphos IL 101 (a quaternary phosphonium-based ionic liquid) — to enhance electrostatic affinity toward the anionic DCF species [2]. The physicochemical properties of the pristine and functionalized frameworks were thoroughly investigated by complementary characterization techniques, confirming successful ionic liquid grafting while preserving the structural and thermal integrity of the parent framework. Adsorption experiments demonstrated that ionic liquid functionalization markedly improved DCF uptake relative to the unmodified CoMOF, with CoMOF–Aliquat and CoMOF–Cyphos attaining maximum adsorption capacities of 124 mg/g and 116 mg/g, respectively, as determined from Langmuir isotherm modeling. The Langmuir model provided the best fit to the equilibrium data, suggesting monolayer adsorption onto a homogeneous surface with well-defined active sites. Kinetic analysis indicated rapid pollutant uptake following a pseudo-second-order rate law, implying that chemisorption governs the rate-limiting step. The dominant adsorption mechanisms were attributed to a synergistic interplay of electrostatic attraction, anion-exchange interactions, hydrogen bonding, and π – π stacking between the aromatic moieties of DCF and the framework ligands. Thermodynamic investigations conducted across multiple temperatures revealed negative values of standard Gibbs free energy (ΔG°), confirming the spontaneous nature of the adsorption process, while negative enthalpy (ΔH°) values indicated an exothermic character and positive entropy (ΔS°) values reflected increased disorder at the solid–liquid interface during DCF uptake [3]. Reusability tests confirmed efficient regeneration over multiple adsorption–desorption cycles, while groundwater studies showed high DCF removal with minimal interference from coexisting ions. These results demonstrate that ionic liquid functionalization enhances phosphonate MOF performance, making CoMOF composites promising adsorbents for pharmaceutical pollutant removal in water treatment.

Keywords: Cobalt phosphonate MOF, ionic liquid, diclofenac adsorption, Aliquat 336, Cyphos IL 101

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OPTIMIZING ENZYME LOADING IN HYBRID TEOS-BIOPOLYMER XEROGELS MATERIALS

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Proteases are a critical enzymatic class representing approximately 2% of an organism's proteome, playing essential roles in nearly all cellular processes. These enzymes catalyze the hydrolysis of peptide bonds in proteins and peptides, making them invaluable for medical applications. However, their operational utility is often limited by thermal instability [1].

This study focuses on the immobilization of protease from *Bacillus licheniformis* through entrapment in hybrid gels synthesized via the sol-gel process using tetraethoxysilane (TEOS) and various biocompatible polymers (alginic acid, chitosan, and polyvinyl alcohol) [2]. The primary objective was to optimize these matrices by studying enzymatic loading and the influence of polymers on activity, specifically determining the effect of pH and temperature on both native and immobilized forms. To understand the structural-thermal relationship of these TEOS-modified hybrids, thermal analysis (TG, DTG, and DSC) and spectroscopic (FT-IR) techniques can provide vital quantitative data that supplements traditional activity assays. The integration of thermal analysis confirms that the synergy between the inorganic silica network and organic polymers is the key factor in developing robust biocatalysts for industrial and therapeutic use.

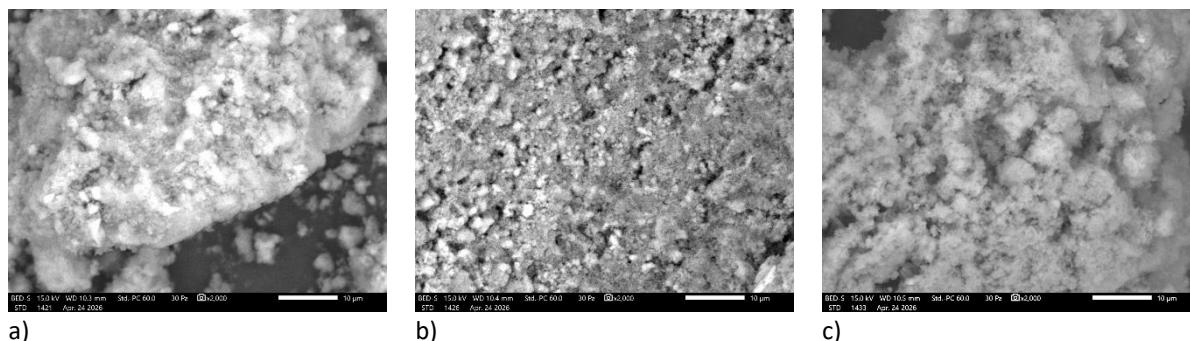


Figure 1. SEM images of modified hybrid xerogels of TEOS-alginate at **a)** 2:1, **b)** 2:4 and **c)** 6:4 ratio

Keywords: TEOS, protease, immobilization, thermal stability, sol-gel

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COMPREHENSIVE CHARACTERIZATION OF COMMERCIAL AND ILLICIT ORAL NICOTINE POUCHES ON THE ROMANIAN MARKET: VALIDATED ION-PAIR RP-HPLC DETERMINATION OF NICOTINE AND GC-MS PROFILING OF VOLATILE COMPONENTS

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The increasing popularity of nicotine pouches, including both commercially available and illicit products, has raised significant concerns regarding product composition, quality control, and the potential presence of harmful or unauthorized substances [1]. A reverse-phase high-performance liquid chromatography (RP-HPLC) method employing ion-pairing was developed and validated for the quantitative determination of nicotine. Method validation was carried out in accordance with international guidelines, evaluating specificity, linearity, accuracy, precision (including repeatability and intermediate precision), robustness, as well as the limits of detection (LOD) and quantification (LOQ). The method demonstrated excellent linearity over the tested concentration range, with satisfactory recovery and repeatability, confirming its suitability for routine analysis of nicotine in pouch matrices.

In parallel, a gas chromatography–mass spectrometry (GC–MS) method was developed for the profiling of volatile compounds [2]. This approach was designed to screen for the presence of drugs of abuse as well as flavoring agents restricted or banned under European Union regulations. The method enabled the identification of a wide range of volatile constituents, including potential contaminants and unauthorized additives, through spectral library matching (NIST). Application of the developed methods to a variety of nicotine pouch samples revealed significant variability in nicotine content, particularly among illicit products, and highlighted the presence of diverse volatile profiles. The combined RP-HPLC and GC–MS analytical framework provides a reliable and comprehensive toolset for regulatory control, product quality assessment, and public health protection related to nicotine pouches. With the addition of infrared spectroscopy and thermogravimetric analysis a comprehensive data set is provided.

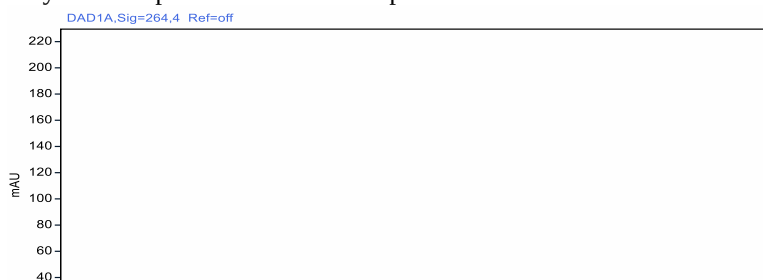


Figure 1. Representative chromatogram for nicotine

Keywords: nicotine pouches, RP-HPLC, GC–MS profiling, method validation, regulatory control

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ANALYSIS OF THE THERMAL BEHAVIOR OF BRONZE AGE CERAMICS FROM VÂNĂTORI-NEAMȚ-VALEA REA

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The ceramic fragments from the Vânători-Neamț-Valea Rea site provide relevant information on the ceramic production technology used by prehistoric communities in the eastern Carpathians. This study aims to evaluate the thermal behaviour of the ceramics and identify the structural transformations that occur during the firing process, in order to reconstruct the technological manufacturing conditions. Recent research in archaeometry highlights the importance of thermoanalytical analyses for interpreting prehistoric ceramic technologies and evaluating the relationship between ceramic composition and firing conditions [1]. Investigations combined TG/DTG, HF, FT-IR and LIBS analyses to evaluate the thermal behavior and composition of the ceramic samples. TG and DTG curves reveal successive processes of water loss and transformations of clay compounds within the 50-350 °C interval. Most samples exhibit a marked reduction of thermal effects after approximately 650-700 °C, suggesting exposure to relatively high firing temperatures, similar to those reported in other thermoanalytical studies on prehistoric ceramics [2]. FT-IR data indicate the predominance of silicate and aluminosilicate compounds, along with mineral phases specific to illitic and montmorillonite clays. LIBS analyses highlight high concentrations of iron and the presence of elements characteristic of silicate and aluminum-based ceramics. The combination of spectroscopic and thermoanalytical results suggests the use of local raw materials rich in ferrous compounds and the existence of firing processes carried out predominantly in an oxidizing atmosphere, aspects also reported in recent research on archaeological ceramics [3].

The results indicate relatively consistent control of the firing process, while also highlighting technological variations between the analysed ceramic fragments. This study contributes to a better understanding of Bronze Age ceramic production practices and emphasises the relevance of thermoanalytical methods for investigating archaeological materials.

Keywords: archaeological ceramics, TG/DTG, FT-IR, LIBS, Bronze Age

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MULTIPLE TECHNIQUES IN THE INVESTIGATION OF SOME FRAGMENTS OF DACIAN CERAMICS, DISCOVERED NEAR THE DACIAN FORTRESS OF ALUN-PIATRA ROȘIE (HUNEDOARA COUNTY)

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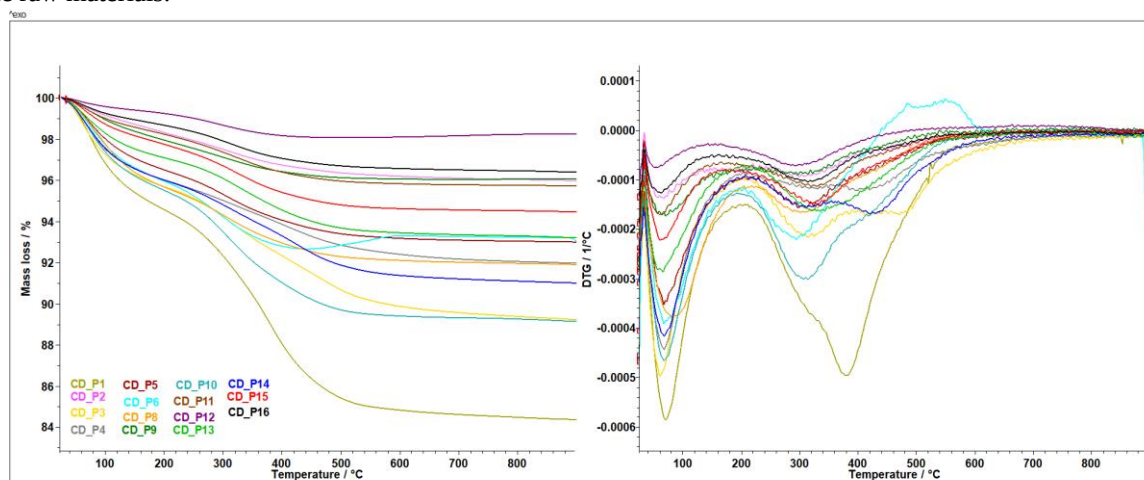
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The 16 samples presented in this work come from the research carried out on the surface of a terrace arranged on the southern slope of the Piatra Roșie hill, in the village of Alun, Boșorod commune, Hunedoara county, Romania. In that place, a construction made of wood was identified, on the surface of the ground, having the appearance of a household. The vessel fragments come from containers specific to Dacian pottery. The archaeological literature recorded the results of research, as well as some isolated discoveries, in the form of a monograph [1] and several studies, larger or smaller in size [2].

The analysis was conducted using complementary techniques TG/DTA (Figure 1), FT-IR, XRD, LIBSSEM-EDS, X-ray Computed Tomography (XCT), in order to determine details regarding production technology and origin of the raw materials.



TG curve for all pottery sample

DTG curve for all pottery sample

Figure 1. TG curves - obtained in 30-900 °C temperature range for coarse pottery samples

Keywords: Thermal stability, Pottery, X-ray Computed Tomography (XCT), FT-IR, XRD, LIBS, SEM-EDS

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KINETIC ANALYSIS OF COMPLEX HETEROGENEOUS PROCESSES

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Thermal analysis and calorimetry represent a multidisciplinary research area that integrates concepts from analytical chemistry, physical chemistry, applied physics, and materials science [1]. These techniques are powerful tools for the investigation of complex solid-state phenomena, including solid–solid transformations, gas–solid interactions, and thermal decomposition processes, which are fundamental in a wide range of technological fields, particularly in materials development and energy-related applications. Unlike homogeneous systems, solid-state reactions are intrinsically heterogeneous processes, where reaction pathways and rates are strongly influenced by structural characteristics, defects, and microstructural evolution rather than by conventional concentration-dependent effects.

A comprehensive understanding of the kinetics governing these transformations is crucial, as kinetic modelling enables the quantitative description of reaction mechanisms and allows the prediction of material behavior under conditions beyond those experimentally investigated. Nevertheless, the kinetic analysis of solid-state processes remains a challenging task due to the complexity of reaction pathways, the occurrence of simultaneous phenomena, structural rearrangements, and inherent experimental constraints.

Thermal analysis and calorimetric techniques provide valuable information on the macroscopic evolution of materials as a function of temperature and time. Their potential can be significantly enhanced through integration with complementary characterization approaches, including *ex situ* and *in situ* methodologies, which offer deeper insight into structural and mechanistic aspects of the processes under study.

Accurate mathematical modelling and appropriate treatment of experimental data are essential for obtaining reliable kinetic parameters and elucidating the factors controlling solid-state reactivity. This contribution emphasizes the relevance of thermal analysis and calorimetry as key methodologies for the kinetic investigation of complex solid-state systems and illustrates their capabilities through selected representative examples.

Keywords: Kinetics, Solid-State reactions, Heterogeneous processes, Thermal analysis, Deconvolution

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THERMAL STABILITY ASSESSMENT OF HIGH TEMPERATURE MOLTEN SALTS USING A STATIC-GAS THERMOGRAVIMETRIC SETUP

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Heat transfer fluids play a central role in high-temperature energy systems, where they enable the efficient transport, storage and recovery of thermal energy. Among the available candidates, molten salts are particularly attractive because of their high heat capacity, low vapor pressure and suitability for sensible heat storage. Potassium nitrate is especially relevant in this context, as it is one of the main components of solar salt, a molten salt mixture widely used in solar thermal technologies [1].

Despite their technological maturity, the characterization of molten salts at high temperature remains experimentally complex. Thermal stability is a critical property for defining safe operating limits. Conventional thermogravimetric analysis is commonly used for this purpose, but its open configuration and continuous gas flow may affect the measured mass loss. As a result, the apparent degradation observed during testing may include contributions from evaporation, rather than intrinsic thermal decomposition alone [2]. Creeping is another phenomenon that affects the test, as molten salts tend to migrate out of the crucible used during the experiment [3].

This work proposes a pressurized static-gas methodology to evaluate the thermal stability of potassium nitrate (KNO₃). The proposed setup operates without continuous gas flow, reducing evaporation-driven mass loss. To further mitigate salt creeping, a 3D structure fabricated by 3D printing using calcium carbonate was impregnated with the salt and placed inside the crucible to improve molten salt retention during high-temperature exposure. The thermal behavior obtained with this configuration was compared with conventional thermogravimetric analysis to identify differences in apparent mass loss and stability limits. This approach provides a more representative assessment of KNO₃ under high-temperature conditions and supports the development of improved experimental protocols for thermal energy storage applications.

Keywords: Molten salts, thermal stability, thermogravimetric analysis

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THERMAL AND THERMOMAGNETIC ANALYSIS OF MAGNETOCALORIC MATERIALS

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Magnetocaloric materials are characterized by the temperature and magnetostructural changes they undergo when subjected to an external magnetic field. Regarding their applicability, they are candidate alloys for use in magnetic refrigeration systems (cyclic process). Thermal analysis techniques allow the determination of characteristic transformation temperatures that define their operating range (and their thermal hysteresis in alloys with first-order phase transition). Furthermore, thermal and thermodynamic properties are determined [1-2]. The specific heat capacity is determined using differential scanning calorimetry, a technique that allows the determination of entropic and enthalpic changes. Recently, lock-in infrared thermography has been used to determine the adiabatic temperature change. Other characteristic parameters are the isothermal magnetic entropy change, the relative cooling power, and the refrigeration capacity, measured using thermomagnetic methods. Recent results are presented. Complementary studies have been conducted before and after several cycles, and the applicability in magnetic refrigeration devices was checked by simulation.

Keywords: Thermal analysis, magnetocaloric, refrigeration capacity, simulation, cycling

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THERMAL ANALYSIS OF SOFT MAGNETIC MATERIALS

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Thermal analysis of magnetically soft materials and composites allows the determination of the Curie temperature. It also enables determination of temperatures and thermodynamic parameters (enthalpy, entropy) associated with structural relaxation, crystallization (including nanocrystallization), and solid-state transformations (martensitic, allotropy) [1-2]. Results are presented for Fe-based and Fe-Ni-based alloys. The complementarity between calorimetric analysis and structural and magnetostructural analysis is demonstrated. Transformations associated with minor phases (such as tetragonal Fe-Ni and Fe₃Ni) are detected. Regarding magnetic materials reinforced with magnetic particles, thermomechanical studies allow us to determine the influence of the reinforcement percentage on the thermo- and magnetoelastic response of the material (conventional mixture, stereolithography). Furthermore, the rate of the polymerization reaction is modified with the magnetic material content.

Keywords: Thermal analysis, soft magnetic, magnetic composition, Fe-based alloys, Fe-Ni alloys

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INHIBITION EFFECT OF AMINO ACIDS ON METHANE HYDRATE NUCLEATION AND FORMATION EXPLORED VIA THERMAL ANALYSIS OF DSC

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The search for eco-friendly hydrate inhibitors is essential to ensure flow assurance and to prevent hydrate formation in gas or oil pipelines. Amino acids are non-toxic and biodegradable. In this study, the effects of adding amino acids on methane hydrate nucleation behavior were investigated using high-pressure micro-differential scanning calorimetry (DSC). The induction time for nucleation of methane hydrate formation in pure water and in 0.1 mol% aqueous solutions of amino acids (glycine, methionine, lysine, and isoleucine) was measured at a methane pressure of 100 bar. The average induction times of all four amino acid samples consistently exceed those in the pure water system, implying these four amino acids can act as kinetic hydrate inhibitors. The experimental results of the induction times exhibited broad distributions and large standard deviations, revealing pronounced stochastic behavior in methane hydrate nucleation. The classical nucleation theory of Toshev et al. [1] was applied to analyze the induction-time data further to evaluate the nucleation rate of hydrate nuclei.

Keywords: methane hydrates, DSC, induction time, nucleation

Acknowledgements:

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SIZE-DEPENDENT EFFECTS OF MAGNESIUM CARBONATE MICRO- AND NANOPARTICLES ON THE MECHANICAL AND THERMAL PERFORMANCE OF POLYAMIDE 12 COMPOSITES: A COMPARATIVE STUDY

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Polyamide is a widely utilized polymer material in the automotive industry, particularly in coating applications, owing to its distinct advantages. In this study, magnesium carbonate particle additives were incorporated into powdered polyamide 12 to enhance its thermal and mechanical properties. Both micro- and nano-sized magnesium carbonate particles were employed during the compounding process at loading levels of 2, 4 and 6 wt. %. To ensure a homogeneous distribution and production, the extrusion method was preferred. Tensile and flexural test specimens were subsequently fabricated via injection molding in accordance with ASTM standards. The morphological characteristics of the prepared composites were determined using scanning electron microscopy (SEM). Mechanical properties were evaluated through tensile and three-point bending tests, while the thermal behaviors were investigated using thermogravimetry (TG), derivative thermogravimetry (DTG), and differential scanning calorimetry (DSC) analyses.

The experimental results revealed that the 2 wt.% magnesium carbonate incorporation was the optimal concentration for enhancing both the thermal and mechanical properties. Higher loading levels led to particle agglomeration, as confirmed by SEM observations, which adversely affected the mechanical performance of the composites. DSC analysis demonstrated a significant increase in the crystallinity percentage, rising from 23.98% for the 2 wt.% micro-composite to 36.76% for the nano-counterpart, indicating the prominent nucleating effect of the nanoparticles. In the nano-reinforced composites, the reduction in particle size yielded a higher specific surface area, facilitating more efficient load transfer across the polymer matrix. Furthermore, the increased number of particles per unit area in the nano-filled specimens played a critical role in enhancing the mechanical properties. Specifically, the average modulus of elasticity for the nano-reinforced composite was found to be 1386.4 ± 17.8 MPa, whereas it was 1344.6 ± 34.9 MPa for the micro-counterpart. Similarly, the average tensile strength reached 42.62 ± 2.6 MPa for the nano-sized composite, compared to 40.5 ± 2.4 MPa for the micro-sized composite.

Keywords: Polyamide 12, Magnesium Carbonate, Nanocomposites, Mechanical Properties.

Acknowledgements:

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EFFECTIVE CONVECTION COEFFICIENT HEAT TRANSFER ESTIMATION OF A SINTERED MATERIAL

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In porous sintered materials and certain intermetallic systems with complex ordered microstructures, the thermal response may deviate from classical exponential decay due to interconnected porosity, internal interfaces, and heterogeneous phases, which introduce memory-dependent and nonlocal heat transfer effects commonly described as fractional thermal behaviour. To account for these anomalous characteristics, a fractional derivative-based formulation of Newton's cooling law is employed in this study to model the non-exponential temperature evolution and to estimate the effective convection heat transfer coefficient. Experimentally, the previously sintered intermetallic specimen is reheated in a sintering furnace to achieve a uniform elevated temperature, then placed on a wooden surface to approximate an adiabatic boundary condition, and its surface temperature is recorded over time using an infrared thermometer. The resulting temperature–time data are curve-fitted to obtain the analytical expression of thermal decay using an exponential function and the function found from the solution of fractional-order heat transfer function. The R² value of the exponential curve fit is 0.869 while that of the fractional solution function is 0.972. From the curve fitted function, the effective convection heat transfer parameters of the intermetallic material are determined.

Keywords: Newton's law of cooling, fractional derivative model, convection heat transfer coefficient, thermal characterization.

Acknowledgements:

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EFFECT OF Si AND PACK CHARACTERISTICS ON THE THERMAL BEHAVIOR OF ALUMINIDE-COATED Ti6Al4V ALLOY

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Ti-6Al-4V (Ti64) alloys are widely utilized due to their high specific strength, low density, excellent corrosion resistance, and superior biocompatibility. However, their relatively poor wear resistance, low thermal conductivity, and insufficient oxidation resistance at high temperatures limit their application above 400–500 °C.

To address these drawbacks, a TiAl-based intermetallic coating was developed to improve the surface performance of the alloy. According to XRD analyses, TiAl₃, TiAl₂, and TiAl phases were successfully formed on the surface through pack aluminization at 750 °C for 4 h. SEM-EDS examinations further confirmed the formation of a layered intermetallic structure. Vickers microhardness measurements showed that the coated samples reached a surface hardness of approximately 770 HV. With Si reinforcement, the coating thickness decreased from 40 µm to 20 µm, while the hardness increased to about 850 HV. Thermogravimetric and differential scanning calorimetry (TG-DSC) results indicated that the uncoated sample exhibited significantly higher weight gain compared to the coated samples, clearly demonstrating the enhanced oxidation resistance provided by the TiAl-based coating.

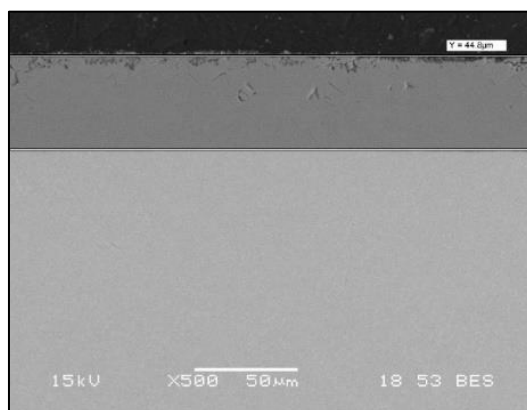


Figure 1. Pack aluminized TiAl Coated Ti6Al4V alloy at 750°C-4h

Keywords: TiAl intermetallics, DSC-Tg, Ti6Al4V Alloy, Hardness

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INFLUENCE OF PROCESSING ROUTE ON THE BIOACTIVITY AND PERFORMANCE OF LITHIUM DISILICATE–WOLLASTONITE GLASS-CERAMICS

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Lithium disilicate–wollastonite glass-ceramics with the same nominal composition (50 mol% lithium disilicate–50 mol% wollastonite) were fabricated via sol-gel and melt-derived routes. Differential thermal analysis (DTA) was employed to evaluate the thermal behaviour and crystallization characteristics of the parent glasses and to determine the appropriate sintering temperatures. Based on the DTA results, the parent glasses were sintered at 750, 800, and 850 °C for 5 h to obtain glass-ceramics. The influence of the processing route on crystallization behaviour, phase evolution, microstructure, physical properties, mechanical performance, and in vitro bioactivity was systematically investigated. Phase assemblages and microstructural features were characterized by X-ray diffraction (XRD) and scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM–EDS). Bulk density and apparent porosity were determined using the Archimedes method, while mechanical performance was evaluated by three-point bending and Vickers hardness tests. In vitro bioactivity was assessed by immersion in simulated body fluid (SBF) for 3, 7, 14, and 28 days, and the immersed samples were subsequently characterized by XRD, FTIR and SEM–EDS together with pH and weight change analyses. Increasing the sintering temperature promoted the crystallization of lithium disilicate and wollastonite, whereas quartz and lithium metasilicate gradually disappeared. The melt-derived glass-ceramics exhibited superior mechanical performance, reaching a maximum flexural strength of 185 MPa and a Vickers hardness of 705.7 HV, whereas the corresponding values for the sol-gel-derived samples were 176 MPa and 532 HV, respectively. Glass-ceramics produced by both processing routes exhibited favourable in vitro bioactivity after SBF immersion. Overall, this comparative study provides insight into how the processing route affects the crystallization behaviour and final characteristics of lithium disilicate–wollastonite glass-ceramics.

Keywords: Lithium disilicate, wollastonite, glass-ceramic, sol-gel, in vitro bioactivity

EFFECT OF PARTIAL SUBSTITUTION OF ZnO BY TiO₂ ON THE CRYSTALLIZATION BEHAVIOUR AND PROPERTIES OF ZMAS GLASS-CERAMICS

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The present study investigates the effect of partial substitution of ZnO by TiO₂ on the crystallization behaviour, phase evolution, microstructure and properties of ZnO–MgO–Al₂O₃–SiO₂ (ZMAS) glass-ceramics. Three ZMAS glass compositions with different ZnO/TiO₂ ratios were prepared by partially substituting ZnO with TiO₂ while maintaining the remaining oxide contents constant (Table 1).

The raw materials were homogenized by dry ball milling for 12 h and melted in alumina crucibles at 1500 °C for 3.5 h. The melts were cast into graphite moulds and subsequently annealed at 600 °C for 1 h. Differential thermal analysis (DTA) was performed to determine the characteristic thermal events and appropriate crystallization temperatures. In addition, non-isothermal crystallization kinetics were investigated using DTA measurements at different heating rates to determine the activation energy and crystallization mechanism. Based on the DTA results, the glasses were subjected to a single-stage heat treatment at 800–950 °C for 1 h with a heating rate of 5 °C min⁻¹ to obtain glass-ceramics.

The crystallized samples were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM), while density, microhardness, and dielectric properties were also evaluated. XRD analyses revealed the formation of gahnite, enstatite, and quartz as the major crystalline phases. The microhardness ranged from 548 to 1020 HV_{0.05}, whereas the density varied between 2.13 and 3.05 g cm⁻³ as a function of crystallization temperature. The dielectric measurements showed that decreasing the ZnO/TiO₂ ratio resulted in a lower dielectric constant and a higher dielectric loss.

Table 1. Nominal oxide compositions of the ZMAS glasses (wt.%).

Sample	ZnO	MgO	Al ₂ O ₃	SiO ₂	TiO ₂	Sb ₂ O ₃
ZMAS1	15	20.48	19.90	38.62	5	1
ZMAS2	12.5	20.48	19.90	38.62	7.5	1
ZMAS3	10	20.48	19.90	38.62	10	1

Keywords: ZnO–MgO–Al₂O₃–SiO₂, glass-ceramic, dielectric properties, crystallization kinetics

Poster Session 3

THERMOMECHANICAL STABILITY OF POLYLACTIC ACID/BIS(2-ETHYLHEXYL) PHOSPHATE POLYMER INCLUSION MEMBRANES

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Polymer inclusion membranes (PIMs) with integrated carriers offer an efficient solution for metal ions removal from wastewaters [1]. Application of biodegradable polymers presents alternative to commonly used non-biodegradable polymers and with scarce research providing great opportunity for further investigations. The main idea is to provide PIMs materials with appropriate stability, selectivity, extraction efficiency and biodegradability. In this study, PLA with different amount of bis(2-ethylhexyl) phosphate (DEHP) as an ion liquid carrier or extractant (10, 20, 30 and 40 wt%) was used as a polymer matrix for PIMs. These components were integrated through the solution casting method and obtained materials were evaluated for their potential as PIMs. Optimization of the mixing and drying conditions during preparation of the membranes has proved as very important issue. It was evident that with the variation of this conditions significant changes of the material integrity can be observed. The most stable PIMs was one with 10 wt% of DEHP. The findings of other investigations demonstrate that the inclusion of fillers within the membrane matrix enhances structural integrity of PIMs [2]. Various analytical methods, including dynamic mechanical analysis (DMA), X-ray diffraction (XRD), contact angle measurement (CA) and density measurement, were employed to explore the structure-property relationships of the PIMs. The analyses revealed development of semicrystalline membrane structures with dominant amorphous phase. It was found that the presence of the carrier altered the bulk structure of the membranes. The most promising PIMs was one with 10 wt% of DEHP, but for extraction efficiency its content has to be higher. The density and the surface properties of PIMs alters as the higher amount of DEHP is added to the membrane. Dynamic conditions in wastewater during extraction or transport experiment can lead toward changes of the membrane properties, i.e. fast functional degradation of the membranes. Stability and operational lifespan of PIMs will be limited by liquid carrier leaching into adjacent aqueous feed or receiving solutions during continuous extraction cycles [1]. Simulation of stress imposed on the PIMs during transport experiment was conducted with DMA. Mechanical properties of PIMs were analysed before and after exposure to oscillatory loading under cyclic variation of three different frequencies in water at room temperature. The results indicated that exposure of PIMs to oscillatory loading in water affected their mechanical properties compared with unexposed membranes. Further investigations should be performed in other complex conditions, as close possible to real one, to be able to predict PIMs usability for industrial applications.

Keywords: polymer inclusion membrane, polylactic acid, bis(2-ethylhexyl) phosphate

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KINETIC ANALYSIS OF POLYMER COMPOSITE FILMS BASED ON WATER-SOLUBLE POLY(VINYL ALCOHOL) WITH LITHIUM CARBONATE

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Polyvinyl alcohol (PVA) is a water-soluble synthetic biopolymer with excellent adhesive properties [1]. Properties such as biocompatibility, environmentally friendly, non-toxicity, high solubility in water, inexpensive, excellent film-forming capacity, excellent tensile strength, high dielectric constant and high thermal stability makes PVA a prospective material for the polymer electrolytes [2]. Therefore, in this work polymer composite films based on poly(vinyl alcohol) doped with different wt% of lithium carbonate (Li_2CO_3) salt were prepared by solution casting method. The degradation of prepared films was performed by the non-isothermal thermogravimetric analysis (TG) at five different heating rates (5, 7.5, 10, 15 and 20 °Cmin⁻¹) in nitrogen atmosphere. Firstly, an analysis of the general thermal characteristics related to composite films stability and thermal effects of salt addition on the thermal stability of PVA matrix was evaluated. Secondly, in order to calculate the activation energy (E) across the entire conversion range, the NETZSCH Kinetics Neo computational software was used. This software encompasses model-free methods (ASTM E698, ASTM E2890, ASTM E1641, Friedman, Ozawa-Flynn-Wall, KAS, Vyazovkin, Numeric Optimization) that allow the activation energy of the reaction process to be found without the assumption of any kinetic model [3]. Finally, the dependence of activation energy on degree of conversion revealed the kinetic complexity of thermal degradation of investigated polymer composite films.

Keywords: poly(vinyl alcohol), lithium carbonate, polymer composite, kinetic analysis

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PREPARATION AND CHARACTERIZATION OF PLA-BASED POLYMER INCLUSION MEMBRANES

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Polymer inclusion membranes (PIMs) are attracting growing scientific interest because of their potential as an efficient and environmentally friendly separation technology for a wide range of industrial applications. Although notable advances have been achieved, significant additional research is still needed to provide the material design insights required for further progress in PIMs. Therefore, the aim of this work was to provide a detailed assessment of how the carrier influences the thermal properties and stability of polymer inclusion membranes. PIMs were successfully prepared using polylactide (PLA) as the base polymer and Aliquat 336 as the carrier at different mass ratios. Their thermal behaviour was characterized by thermogravimetric analysis (TG) and differential scanning calorimetry (DSC), while membrane stability was evaluated after immersion in buffer solutions over a pH range of 1–12 for 24 h.

TG analysis revealed that the addition of Aliquat 336 significantly altered the thermal degradation behaviour of PLA-based polymer inclusion membranes, reducing their thermal stability and resulting in a two-step degradation process. DSC investigation revealed that the carrier caused the reduction of glass transition, confirming the plasticizing effect of Aliquat 336 on the PLA matrix. The membranes exhibited good stability under acidic and neutral conditions, with the lowest mass loss observed between pH 1 and 4, whereas structural degradation and increased mass loss occurred under alkaline conditions. These findings provide valuable insight into the influence of Aliquat 336 on the thermal behaviour and pH stability of PLA-based polymer inclusion membranes and contribute to the rational design of environmentally friendly membranes for separation applications.

Keywords: polymer inclusion membranes, differential scanning calorimetry, polylactide, thermogravimetric analysis

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This research is funded by the European Union – NextGenerationEU project (No. IP-UNIST-29)

STRUCTURAL AND THERMAL PROPERTIES OF A 2-D COBALT(II) COORDINATION POLYMER WITH 6-METHYLNICOTINATE

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Cobalt(II) coordination polymers with substituted nicotinate and bridging *N*-donor ligands have been reported e. g. coordination polymers based on 4,4'-bipyridine (with 2-chloronicotinate, 5-bromonicotinate and 6-hydroxynicotinate) [1-2] or on 1,2-bis(4-pyridyl)ethane (with 6-bromonicotinate and 6-aminonicotinate) [3-4]. The present work describes a 2-D cobalt(II) coordination polymer with 6-methylnicotinate ligand as the sole ligand, $\{[\text{Co}_2(\mu_3\text{-6-Menic-N,O,O}')_2(\mu\text{-6-Menic-O,O}')_2]\cdot\text{DMF}\}_n$ (**1**). It was obtained by the reaction of cobalt(II) nitrate hexahydrate and 6-methylnicotinic acid (6-MenicH) in a mixture of water and *N,N'*-dimethylformamide (DMF). The crystal structure of **1** was determined by single-crystal X-ray structure analysis. The cobalt(II) ion exhibits a square-pyramidal coordination geometry, with the basal plane defined by four carboxylate O atoms and the apical position occupied by the pyridine N atom. There are two types of 6-methylnicotinate ligands in **1**: the first one bridges two cobalt(II) ions by two bridging carboxylate O atoms, while the second one bridges three cobalt(II) atoms by two carboxylate O atoms and one pyridine N atom, resulting in the formation of a paddle-wheel dinuclear cobalt(II) cluster unit with a Co...Co contact. These paddle-wheel cobalt(II) cluster units are linked by *N,O,O'*-bridging 6-methylnicotinate ligands into a 2-D coordination network parallel to the (001) plane (Figure 1). The compound **1** was also studied by IR spectroscopy and TGA/DSC methods.

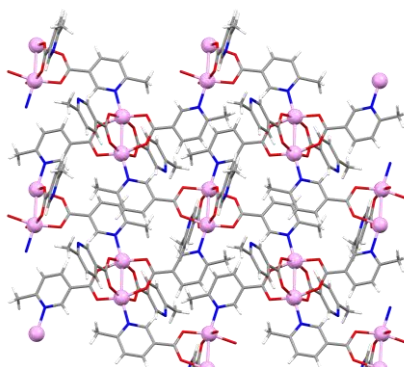


Figure 1. A view of the 2-D coordination network of **1** down the *c* crystallographic axis.

Keywords: coordination polymer, paddle-wheel cluster, cobalt(II), 6-methylnicotinate, TGA/DSC methods

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DEVELOPMENT OF ALGINATE BASED EDIBLE FILMS FOR FOOD ACTIVE PACKAGING

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Edible films or coatings help protect food from microbial contamination by providing effective barrier that extends the food's shelf life. Also, it can improve coated food organoleptic and sensory qualities [1]. Recently, there has been an increased demand for biopolymers as edible films. Polysaccharides, proteins, and lipids are the most common edible biopolymers. The main reasons are low-cost, industrial scalability, biodegradability and biocompatibility, making them ideal for delivering antioxidant and antibacterial compounds that enhance food safety [2]. The current study aims to develop bio-based edible active films or coatings, that at the same time will reduce food waste and enable by-product recycling. Onion and pomegranate generate peels as a by-product and it has been found that these peels contain flavonoids with various valuable properties, including antioxidant, antimicrobial, antibacterial etc. Onion and pomegranate peels extracts can be used to modify properties of edible biopolymers such as alginate found in seaweed. The objective of this investigation was to develop edible material based on sodium alginate (NaAlg, mark A), as major component, and pectin (mark P) or pork gelatine (mark PG) or beef gelatine (mark BG), without or with addition of onion or pomegranate peels extracts (mark OE or PE), and to investigate its potential as active edible films. The base solution for preparation of edible films is complex and beside NaAlg and pectin or pork gelatine or beef gelatine contains olive oil, Tween 80, distilled water. In these base solutions extracts are added. Processing contains multiple steps that includes heating, mixing and application of the ultrasound so that all compounds are well mixed and solution is stable and homogenous. Films are produced by solution casting technique, but with additional spraying with CaCl_2 solution. In total, nine films are produced (A/P, A/P/PE, A/P/OE, A/PG, A/PG/PE, A/PG/OE, A/BG, A/BG/PE and A/BG/OE). The structure of the films was examined using Fourier transform infrared spectroscopy (FTIR). Thermal properties and thermal stability were analysed using differential scanning calorimetry (DSC) and thermogravimetry (TG). Contact angle measurement was used to determine degree of hydrophilicity/hydrophobicity. The films with pectin show the best integrity while films with pork gelatine the worst. Addition of OE gives more elastic and stable films than PE. FTIR analysis establish existence of the secondary interactions with addition of extracts in the films. There is evidence of free and bonded water in the films, also confirmed with DSC analysis. Films with OE has equal amount of free and bonded water unlike films with PE or films without extract. This evidently has significant influence on the film's elasticity. Films shows complex degradation through the five degradation stages. The degradations kinetics of the base films (without extracts) is under the influence of the extracts. Onion peel extract shows the best thermal stabilisation of the alginate degradation stage in the prepared films.

Keywords: food by-products, pomegranate and onion peel extract, edible films

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NATURAL ANTIOXIDANTS AS SUSTAINABLE MODIFIERS OF POLY(3-HYDROXYBUTYRATE): A COMPARATIVE THERMAL ANALYSIS

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Poly(3-hydroxybutyrate) (PHB) is one of the most promising biopolymers due to its renewable origin, biodegradability, and biocompatibility. However, its wider application is limited by its high brittleness, high degree of crystallinity, and narrow processing window resulting from the small difference between its melting temperature and the onset of thermal degradation. Consequently, PHB is commonly modified with various additives to improve its processability and thermal properties. Since the use of synthetic non-biodegradable additives may diminish the environmental benefits of biopolymers, increasing attention has recently been focused to natural, renewable, and biodegradable additives as sustainable alternatives. Among these, α -tocopherol, tannic acid, and quercetin have attracted particular interest. Despite their different chemical structures, all three compounds are effective free-radical scavengers and may retard the thermal degradation of polymers. The first studies reporting the use of tannic acid as a stabilizer for PHB were published in 2015, and subsequent research has further confirmed the potential of natural antioxidants for improving the stability of biodegradable polymers. Nevertheless, a systematic comparison of the effects of α -tocopherol, tannic acid and quercetin on the thermal properties of PHB remains insufficiently explored. Therefore, the aim of this study was to investigate the influence of these natural and biodegradable modifiers on the glass transition temperature, melting temperature, and thermal stability of PHB using differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA).

Keywords: poly(3-hydroxybutyrate), thermal degradation, thermal stabilizers

ISOTHERMAL MICROCALORIMETRY FOR HIGH-THROUGHPUT SCREENING OF LACTIC ACID BACTERIAL CONSORTIA FOR VEGETABLE FERMENTATION

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The development of defined starter cultures is crucial for enhancing the consistency, safety, and functional quality of fermented vegetables [1]. Lactic acid bacteria (LAB) isolated from successful vegetable fermentations represent promising candidates for this purpose. However, efficient identification of compatible strains and microbial consortia requires robust screening methodologies.

In this study, isothermal microcalorimetry (IMC) was utilized as a high-throughput technique to evaluate LAB strains and consortia intended for kimchi and sauerkraut fermentations within the HORIZON Europe DOMINO project. Fourteen LAB isolates originating from fermented carrots, kohlrabi, sauerkraut, and kimchi were selected based on genome-scale metabolic modeling (MetaQ2Metabo) and empirical fermentation knowledge. Prior characterization confirmed their tolerance to low pH, bile salts, and NaCl, while genomic analyses verified their safety.

Individual strains and designed multi-strain consortia were cultivated in MRS medium and analyzed in triplicate using IMC. Maximum specific growth rate ($\mu_{\max}$), maximum heat-flow ($P_{\max}$), and cumulative heat production (Q_{tot}) were used to assess microbial performance and potential interactions [2].

The tested LAB isolates exhibited strain-dependent variations in metabolic activity, with $\mu_{\max}$ values ranging from 0.2 to 0.8 h⁻¹ and $P_{\max}$ values from 109 to 488 μW . Cumulative heat production varied between 6.6 and 12.0 J, indicating substantial differences in overall metabolic output. In contrast to individual strains, the designed consortia displayed more uniform calorimetric profiles, with $\mu_{\max}$ values ranging from 0.4 to 0.8 h⁻¹, $P_{\max}$ values from 283 to 455 μW , and cumulative heat production between 10.7 and 11.6 J, suggesting enhanced metabolic stability and potential metabolic interactions. Several kimchi-oriented consortia combined high growth rates, elevated heat-flow signals, and stable energy output, identifying them as promising starter cultures. These consortia were subsequently validated in kimchi and sauerkraut fermentations and characterized using ¹H-NMR, GC-MS, and HPLC. Overall, IMC proved to be a sensitive, non-invasive, and high-throughput tool for screening LAB consortia and selecting robust starter cultures for vegetable fermentations.

Keywords: isothermal microcalorimetry, lactic acid bacteria, microbial consortia, starter cultures, vegetable fermentation

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THERMAL CHARACTERIZATION OF THE MIXED HETEROGENEOUS METALLURGICAL WASTE

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The industrial waste deposit of Trepca in Kosovo was used for disposal of different metallurgy wastes including heavy metals. The metallurgical waste deposit was situated on the open space, between the tailing waste deposit Trepca and river Ibar flow. The large amounts of lead-containing wastes are produced in non-ferrous metallurgical industry, such as lead ash and lead slag generated in Pb smelting, lead anode slime, and lead sludge produced in the refining process. The results of X-ray fluorescence analysis, scanning electron microscopy (SEM), optical microscopy (LOM) and X-ray diffraction (XRD) were used for chemical and mineralogical analysis of the waste samples. Differential thermal analysis (DTA), thermo-gravimetric (TG) analysis and thermal microscopy results were used for determination of characteristic phase transformations. This pattern strongly suggests the sample contains thermally labile phases that gradually release volatiles (e.g., water, HCl/SO_x, CO₂ if carbonates exist, etc.), plus a final inorganic residue richer in oxides/silicates. The main gauge minerals were Feldspar and Diopsin-ferroan with different concentrations of PbCl₂, PbCO₃ and complex compounds containing Lead, Arsenic, Antimony, Zinc and Silver. The mixtures were exposed to weathering, and the deposit cross sections were analyzed using SEM/EDS. Chemical weathering of feldspars happens by hydrolysis and produces clay minerals, including illite, smectite, and kaolinite. The first pick shows removal of adsorbed/weakly bound water, from aluminosilicate or halite, that was observed in the XRD plots, followed by aluminosilicate and oxide framework re-arrangement, and PbCO₃ decarbonization, as well as heavy metals (Cu, Zn, Fe, Pb) oxides. In the following high temperature stage, 1252.4°C, the silicate ternary compounds recrystallization and Pb and As oxides volatilize. The final stage at 1454.2 °C Pb, As, and any remaining sulfur/chlorine-bearing trace phases show the last residual volatile loss and final phase transformation.

Keywords: heterogenous metal waste, DTA, TGA, lead compounds transformation

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INFLUENCE OF IRRADIATION ATMOSPHERE ON THERMAL PROPERTIES OF SHEEP WOOL FIBERS

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Natural fibers, such as cotton, wool and wood, have a broad application worldwide. Sheep wool fibers are used in textiles, isolation and filtering materials and have potential to be used in filtering systems, car interiors, buildings, packages and other fields [1]. However, due to complex disposal management [3] of sheep wool fibers that remain as waste material, the broadening of application possibilities is an open topic.

Pre-treatment and modification of fibers is considered to improve the properties required for specific applications [3]. In the present work, the sheep wool fibers, mainly from the local, Latvian Blackhead sheep breed, were irradiated using 9 MeV accelerated electrons with absorbed doses up to 500 kGy in vacuum and air. The characterization of non-irradiated and irradiated fibers was performed by Fourier transform infrared (FTIR) spectroscopy, while a detailed analysis of free radicals was performed with electron paramagnetic resonance (EPR) spectroscopy. The thermal behavior of sheep wool fibers before and after irradiation was characterized by thermogravimetry/differential scanning calorimetry (TG/DSC) with several heating regimes.

The obtained EPR results show that irradiation dose and atmosphere affect content of the formed free radicals, located on carbon and sulphur atoms. The FTIR spectra indicates that radiation-induced oxidation processes influence Amide I and Amide II bonds in samples, which were irradiated in air. TG/DSC curves show that irradiation and irradiation atmosphere changes decomposition temperatures of the sheep wool fibers.

Keywords: sheep wool, thermal analysis, irradiation, accelerated electrons

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EFFECTS OF POZZOLANIC PRECURSORS ON THE WOOD 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 clean energy source, biofuel energy derived from wood combustion is increasingly welcomed around the globe, especially in forestry countries like Lithuania, which generates a significant amount of wood biomass waste. According to the reports, the global yearly generation of wood biomass is approximated to be 4600 million tons, which is predicted to grow threefold by 2035. This exerts great pressure on the land resources and environmental conservation.

To explore the application of this waste in large quantities for producing alkali-activated mortar (AAM) materials, wood fly ash (WFA) and wood bottom ash (WBA) 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 (CFA) were introduced as a binary precursor to provide extra aluminosilicates at the content of 10, 20, 30, and 40% by precursor weight. The samples were subjected to 60 °C thermal treatment at the first 24 hrs. Compressive strength, water absorption, SEM-EDS, XRD and MIP were tested to analyze the mechanical properties and reaction mechanisms. In addition, thermal analysis, including exothermic profile and TG-DTA, was conducted to understand the early-age and 28-day hydration process and hydrate products.

According to the results, the addition of CFA and MK effectively improves the strength by 47 and 33%, respectively, with the highest value attaining 23 and 21 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, where higher intensity peaks aligned with calcium silicate hydroxide were identified. This indicates that adding CFA and MK accelerates the alkaline activation and enhances its degree, favouring the development of mechanical properties.

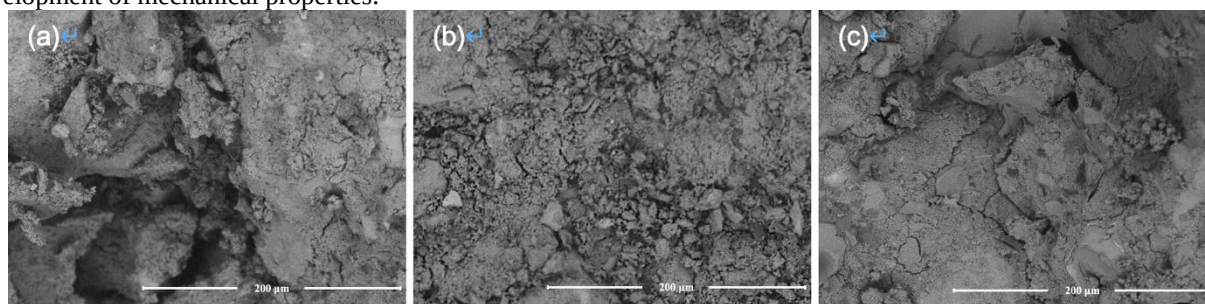


Figure 1. SEM of AAM at 500 magnification: (a) reference sample; (b) with 30% FA; (c) with 20% MK

Keywords: waste valorisation, thermal analysis, early-stage property, calcium hydroxide, hydration mechanism

ALKALI-ACTIVATED BINDERS BASED ON PROCESSING WASTE: HYDRATION KINETICS, THERMAL BEHAVIOR, AND STRENGTH DEVELOPMENT

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As a sustainable alternative to the traditional Portland cement-based binder (PCB) materials, alkali-activated binder (AAB) also has the advantage of natural resource conservation by utilising silicate-containing industrial by-products. In 1978, Davidovits proposed that binders could be produced based on the polymeric reaction between alkaline solutions and aluminate-silicate contents in the precursor materials. This greatly improves the possibilities to valorise agricultural and industrial waste, such as coal fly ash, ground granulated blast furnace slags, and rice husk ash, which were proven to be feasible for reuse for the production of AAB. In addition, it is worth noting that, compared to PCB, the carbon footprint of manufacturing AAB is reduced by a maximum of 80 % and energy consumption is lowered by 43 %. This signifies that the development of waste-based AAB meets the requirements of low environmental impact and the circular economy.

Granite is an igneous rock found throughout the Earth's crust. Its high strength, durability, and aesthetic function make it a versatile natural material with uses across many industries. It is widely used for countertops, flooring, wall cladding, and paving because of its toughness and visual appeal. Its resistance to weathering also makes it well-suited for outdoor decorative applications. Before it is marketed as slabs, granite undergoes several processing steps, including cutting, grinding, and polishing. These operations generate a great amount of granite waste, which is a source of land and water pollution. Similarly, the industrial processing of dolomite rock generates a large volume of dolomite waste (DW), and most of them are disposed of in landfills. Inadequate and inefficient disposal of waste poses a serious environmental hazard problems. Due to the consideration of minimising the environmental threats, one possible solution is to incorporate them into the production of eco-binder materials.

In this study, DW and granite stone crushing waste (GS) were used directly to fabricate AAB. 7 mol/L sodium hydroxide (SH) solution was used together with water glass (WG) as the activator solution. The solute mass of sodium silicate (SS) in the WG was maintained the same as the SH mass. In the mix design, the ratio of GS was 0, 25, 50, 75, and 100% by the total precursor mass. The samples were subjected to the thermal treatment at 90 °C for the initial 24 hrs. Compressive strength was tested to determine the mechanical properties. Thermal analysis, including exothermic profiles and TG-DTA analysis, together with XRD, was performed to understand the hydration kinetics. SEM-EDS was conducted to analyse the microstructure.

The direct incorporation of GS improves the consistency of the fresh binder and promotes the early-age hydration process. The results show that GS has higher chemical reactivity than DW in the presence of alkaline solutions, which relates to greater compressive strength. When DW was used alone, the lowest compressive strength was 15 MPa; while the 100% usage of GS contributed to the highest strength of 29 MPa. Based on the thermal analysis and microstructural tests, the enhancement of the GS ratio in the mix favours the production of hydrates and contributes to a denser microstructure.

Keywords: dolomite waste, granite slurry, thermal analysis, hydration

AMORPHOUS SiO₂ ADDITIVE INFLUENCE ON THE SYNTHETIC MAYENITE STRUCTURE PROPERTIES

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Mayenite is a hydrogarnet-group mineral found in nature [1], in alumina cements [2], [3], as a synthetic product, and as a decomposition product of calcium aluminate hydrate – katoite [4]. The specific structure and a positive electric charge per unit cell, which is balanced by free oxygen ions [5], [6], make it possible to modify the mayenite structure via various cation insertion and substitution that allows influencing its properties and application possibilities. One of the simplest ways to introduce ions is to synthesise mayenite precursors, for example, via sol-gel or hydrothermal synthesis [4], which are then thermally treated to obtain Me-substituted mayenite.

Therefore, the aim of the investigation was to determine how an amorphous SiO₂ additive influences the structural properties of synthetic mayenite.

The hydrothermal synthesis was performed at 130°C for 1 h using unstirred suspensions with CaO/(Al₂O₃+SiO₂) molar ratios of 2.8 and SiO₂ contents of 0 or 0.25. Adsorbent preparation and thermal stability of the synthetic products were evaluated by additional calcination at 350, 550, and 900°C for 1 h.

It was determined that the main product of the hydrothermal synthesis in the sample without SiO₂ was katoite, whereas with an SiO₂ additive, a Si-substituted katoite was obtained. After calcination of the synthesis products at different temperatures, pure mayenite and Si-substituted mayenite were formed. In all cases, calcination at 550°C decreases S_{BET} and pore volume. Increasing calcination temperatures (from 350 to 900°C) increased S_{BET} and pore volume. It was found that in samples without SiO₂, S_{BET} increased twice from ~11 to ~23 m²/g, and pore volume – three times (from 30 to 100 mm³/g). In the presence of the SiO₂ additive, S_{BET} are very similar (10.15 and 10.58 m²/g), but the pore volume increases by about 1.6 times (from 31 to 49 mm³/g).

Keywords: mayenite, thermal stability, specific surface area.

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THERMAL STABILITY OF DOUBLE-SALT SYSTEMS BASED ON AMMONIUM NITRATE AND MICRONUTRIENT SULFATES

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Ammonium nitrate-based fertilizers remain among the most important nitrogen sources in modern agriculture; however, their thermal stability strongly affects both processing safety and final product performance [1]. One promising strategy for the development of multifunctional materials is the incorporation of sulfate compounds into ammonium nitrate-based fertilizers [2]. In this context, double-salt systems containing ammonium nitrate and micronutrient sulfates may offer an attractive route toward the simultaneous delivery of nitrogen and essential microelements, while also modifying the thermal behavior of the fertilizer material.

This study evaluated the effect of sulfate salts on the thermal properties and the decomposition behavior of double salts with ammonium nitrate. Particular attention was paid to the effect of the cation type and the possible formation of new crystalline or semicrystalline phases that influence the material's thermal behavior. The systems under study were prepared under controlled laboratory conditions and characterized using thermal and structural analysis techniques.

The results allowed for a better understanding of the relationship between the composition and thermal behavior of ammonium nitrate double salts. It was found that the equimolar presence of sulfate salts affects the thermal behavior of the fertilizer matrix, increasing its thermal stability at temperatures above 300°C. These studies allow for a reevaluation of the formulation of ammonium nitrate-based compound fertilizers. The results may directly influence the design of safer and more stable fertilizer materials with improved functional properties. The findings are significant for the development of advanced compound fertilizers that combine the functions of macro- and micronutrients in a single product.

Keywords: double-salt, ammonium nitrate, thermal stability, fertilizer, micronutrient, sulfate

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THERMAL AND CHEMICAL STABILITY OF LIGNOSULFONATE–MICRONUTRIENT COMPLEXES

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The growing demand for efficient and sustainable fertilization strategies has led to increased interest in multifunctional fertilizer systems containing both macronutrients and bioavailable micronutrients [1]. In this field, lignosulfonates represent a particularly attractive group of chelating agents due to their renewable origin and their ability to bind metal ions via functional groups present in their structure [2]. Lignosulfonate-based complexes of micronutrient ions can improve the stability of micronutrients, facilitate their incorporation into fertilizer formulations, and influence the rate of nutrient release [3]. This study examines the thermal and chemical stability of lignosulfonate complexes of selected micronutrient ions in fertilizer systems.

The main objective of this study is to evaluate how the complexation of micronutrient ions by lignosulfonates affects the stability of these compounds under conditions related to the processing, storage, and application of fertilizers. Particular emphasis was placed on the role of the type of metal ions, the nature of ligand–metal interactions, and the influence of the fertilizer matrix on the stability of the resulting complexes.

The results indicate that lignosulfonates can effectively interact with micronutrient ions, forming systems with varying stability depending on composition and the chemical environment. It was found that both the thermal and chemical stability of these complexes depend on the type of micronutrient ion and the composition of the fertilizer system. This study expands current knowledge regarding the use of lignosulfonates in fertilizer technology and provides a basis for the rational design of fertilizer formulations containing stabilized micronutrients. Such systems can facilitate more efficient nutrient management, improve fertilizer functionality, and promote the wider use of renewable complexing agents in agricultural products.

Keywords: lignosulfonates, micronutrients complexes, thermal and chemical stability, metal ion complexation

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THE INFLUENCE OF LIGNIN-BASED POWDERS ON THE THERMAL PROPERTIES AND THE FLAMMABILITY OF PLA+PET BLENDS

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This study investigates the thermal behavior and flammability of polylactide (PLA) and poly(ethylene terephthalate) (PET) blends modified with bio-based fillers: calcium lignosulfonate (CLS), lignosulfonamide (SA), and lignosulfonates functionalized with tannic (BMT) or gallic acid (BMG). Differential scanning calorimetry confirmed the partial immiscibility of PLA/PET blends, revealing two glass transitions, one cold-crystallization event, and two melting peaks. The additives influenced phase transformations in distinct ways: BMT restricted the mobility of PLA chains, whereas CLS acted as a nucleating agent enhancing crystallization. Thermogravimetric analysis showed significant changes in thermal stability under oxidizing conditions—BMG reduced the onset degradation temperature, while BMT and SA increased the residual char content. Flammability tests demonstrated that BMG most effectively reduced overall combustibility, promoted char formation, and prolonged burning time. The effects of all additives were strongly dependent on the PLA:PET ratio. CLS predominantly reduced PET flammability, SA selectively inhibited PLA combustion, and BMT and BMG were particularly effective for PET-rich blends. FTIR analysis of combustion residues revealed functional groups associated with degradation products of carboxylic acids and aromatic esters. Among all systems, only blends containing BMT and BMG achieved FV-1 classification, while SA-modified blends met FV-2 requirements. BMG exhibited the highest flame-retardant potential, offering improved thermal stability, delayed ignition, and robust char formation, positioning it as a promising bio-derived additive for sustainable polyester-based materials.

Keywords: biopolymer, bio-blend, polylactide, PET, flammability, thermal properties,

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MODIFICATION OF CHITOSAN USING PHOSPHORUS SALTS TO PRODUCE A FLAME-RETARDANT AGENT

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The environmental problems resulting from fossil fuel exploitation and plastic waste accumulation have resulted in increased interest in biopolymer-based material research. Polyhydroxyalkanoates (PHAs) are naturally-derived polymers which provide an alternative solution to plastic production through their renewable and biocompatible, and biodegradable properties. Their use aligns with the concept of a circular economy and efforts to reduce the environmental footprint of modern materials.

Despite their numerous advantages, the widespread use of biopolymers in many industries – including packaging, textiles, construction materials, and composites – is still limited by their relatively low thermal stability and high flammability. This characteristic poses a significant barrier to fire safety requirements for materials used in technical applications. Consequently, intensive research is underway to find methods for modifying biopolymers that will reduce their flammability without compromising their biodegradability and environmental friendliness.

One of the potential new areas of study involves the use of phosphorus salts to modify chitosan either chemically or physicochemically. Phosphate group addition to the polysaccharide structure may result in materials that have enhanced fire resistance. Phosphorus-based flame retardants operate chemically to facilitate charring in the condensed phase which results in the development of a protective carbon layer on the surface of the material. The layer prevents the entry of air and the transfer of heat and volatile degradation products thereby reducing the speed of the burning process [1].

Modifying chitosan with phosphorus salts therefore represents an interesting strategy for designing eco-friendly flame retardants for biopolymers and biocomposites. Unlike many conventional flame retardant additives, often based on halogenated compounds, solutions based on phosphorus and natural polysaccharides can reduce the negative environmental impact and toxicity of combustion products. At the same time, the biodegradability of the base material is maintained, which is crucial for sustainable development and future materials management.

This paper presents the concept of modifying chitosan with selected phosphorus salts to obtain a material with flame retardant properties that can be used as an additive to biopolymers.

Keywords: chitosan, biopolymers, phosphorus salts, flame retardant

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INFLUENCE OF MODIFIED CELLULOSE ON PROPERTIES, MAINLY FIRE HAZARD OF POLYURETHANE COMPOSITES

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This study presents a method for the chemical modification of cotton fibers using inorganic borates. The modified cotton exhibited a significant reduction in flammability compared to untreated material. Subsequently, the modified fibers were incorporated into a flexible polyurethane (PUR) foam matrix in the presence of melamine polyphosphate as a flame-retardant additive.

The effect of chemical modification on the surface morphology of the fibers, as well as their dispersion within the PUR matrix, was investigated using scanning electron microscopy coupled with dispersive X-ray spectroscopy (SEM-EDS). Additionally, the influence of cotton incorporation on the structure of the protective boundary layer formed during combustion of PUR composites was evaluated.

The thermal properties of the resulting materials were characterized by thermogravimetric analysis (TG), differential thermal analysis (DTA), and differential scanning calorimetry (DSC). The flammability of PUR composites containing both unmodified and chemically modified cotton, including systems exhibiting synergistic interactions with melamine polyphosphate, was assessed using a cone calorimeter and a pyrolysis combustion flow calorimeter (PCFC).

Fire hazard parameters, including smoke emission and the toxicometric index, were determined using a smoke density chamber and a coupled TG–gas analysis system.

Keywords: PUR composites, modified cotton, fire hazard, toxicometric index, thermal properties

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DEVELOPMENT OF A MATHOD FOR MODIFYING AN ASPHALT MIXTURE WITH POLYMER RECYCLATES

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The study presents the development of an innovative method for modifying asphalt mixtures using polymer recyclates, with a particular focus on waste poly(ethylene terephthalate) (PET). The increasing accumulation of plastic waste and the need for more durable road materials motivate the search for sustainable solutions that combine environmental and engineering benefits. In this work, PET recyclate obtained through mechanical recycling was incorporated into asphalt binders using the wet-mixing technique. Comprehensive material characterization of PET (DSC, TG, FTIR) and systematic variation of recyclate content enabled the formulation of modified asphalt mixtures with improved performance parameters. Laboratory tests, including penetration, softening point, elastic recovery, abrasion resistance, and low-temperature cracking resistance, demonstrated that PET addition enhances stiffness, thermal stability, and resistance to mechanical degradation. Supplementary use of mineral fillers, such as waste glass fiber or basalt dust, further contributed to reinforcing the material structure. The results confirm the feasibility of using polymer recyclates as effective asphalt modifiers, showing performance improvements of up to several dozen percent compared to conventional mixes. The developed method offers a cost-effective and environmentally beneficial approach that reduces plastic waste generation and the demand for virgin asphalt, supporting the implementation of circular-economy principles in urban road infrastructure. The proposed technology shows strong potential for practical application in municipal road construction and renovation.

Keywords: asphalt modification, polymer recyclates, PET, sustainable road materials, mechanical performance

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MEBENDAZOLE INCLUSION COMPLEXES WITH FUNCTIONALIZED BETA-CYCLODEXTRINS: SOLID-STATE PREPARATION AND CHARACTERIZATION

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Mebendazole (MBZ) is a well-established broad-spectrum anthelmintic agent that has recently attracted considerable attention as a repurposed drug candidate, with emerging evidence supporting its potential in oncology, particularly against colorectal, lung and brain cancers, as well as in inflammatory and parasitic diseases beyond its original indication [1,2]. However, MBZ belongs to BCS class II, and its poor aqueous solubility remains a critical barrier limiting systemic exposure and therapeutic efficacy in these new applications [3]. Addressing this limitation through solid-state complexation with functionalized β -cyclodextrin derivatives represents a pharmaceutically viable and scalable approach [3].

In this work, inclusion complexes of MBZ with heptakis(2,6-di-O-methyl)- β -cyclodextrin (DIMEB), heptakis(2,3,6-tri-O-methyl)- β -cyclodextrin (TRIMEB) and sulfobutylether- β -cyclodextrin (SBE β CD) were prepared by kneading at a 1:1 molar ratio. The resulting systems were characterized in the solid state using ATR-FTIR spectroscopy, powder X-ray diffraction (PXRD) and thermal analysis (TG/DTG/DSC), with comparison against pure components and their physical mixtures. Modifications across spectral, diffractometric and thermoanalytical profiles confirm the formation of true inclusion complexes in all three cases. The comparative evaluation of the three functionalized derivatives provides insight into the role of substitution pattern on complexation efficiency, supporting the rational development of improved MBZ formulations for its repurposed therapeutic uses.

Keywords: mebendazole, functionalized cyclodextrins, inclusion complex, solid state, instrumental analysis

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CITALOPRAM DECOMPOSITION KINETICS: ACTIVE SUBSTANCE VS. PHARMACEUTICAL FORM

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Selective serotonin reuptake inhibitors (SSRIs) represent the first-line therapeutic class for depressive disorders, owing to their high safety and tolerability profile. Their mechanism of action involves blockade of the serotonin transporter, responsible for increasing neurotransmitter concentration at the presynaptic terminal. Inhibition of this transporter leads to elevated serotonin levels in the synaptic cleft and stimulation of postsynaptic receptors, thereby sustaining serotonergic transmission [1,2]. Among the most widely used SSRIs in clinical practice, citalopram stands out for its favorable tolerability, consistent efficacy and advantageous pharmacokinetic profile. Despite its widespread use, the literature lacks comprehensive data on the thermal stability profile of citalopram hydrobromide and on the extent to which pharmaceutical excipients may influence its thermal degradation behavior [3].

The present study investigates the thermal stability of citalopram hydrobromide as pure active substance and of a pharmaceutical formulation containing 20 mg API. The experimental protocol employed Fourier transform infrared spectroscopy (ATR-FTIR), thermogravimetric analysis (TG/DTG), and a kinetic study based on a preliminary ASTM E698 method, followed by two isoconversional approaches, the Friedman (Fr) and Flynn-Wall-Ozawa (FWO) methods.

ATR-FTIR analysis confirmed the identity and purity of the solid substance, with all major absorption bands characteristic of the functional groups of the citalopram core structure being identified, as well as the presence of the API within the pharmaceutical form. Thermal analysis revealed high thermal stability of the active substance, with an approximately 40 °C increase in thermal stability observed in the presence of excipients. Application of isoconversional kinetic methods to the first degradation process identified on the DTG curve of citalopram hydrobromide evidenced a complex decomposition mechanism, as suggested by the marked increase in activation energy values at conversion degrees above 0.70. For the pharmaceutical form, the change in degradation mechanism occurs at conversion degrees exceeding 0.50. Regarding activation energy, the active substance requires approximately 140 kJ/mol, while the pharmaceutical form requires around 160 kJ/mol, demonstrating the stabilizing effect exerted by excipients on the therapeutic formulation.

Keywords: citalopram, decomposition kinetics, isoconversional methods, pharmaceutical form, thermal analysis

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INCLUSION COMPLEX OF METHOTREXATE WITH HEPTAKIS (2,6-DI-O-METHYL)- β -CYCLODEXTRIN: COMPATIBILITY WITH EXCIPIENTS

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Methotrexate (MTX) is a potent chemotherapeutic drug, an antifolate that acts by inhibiting certain vital enzymes: dihydrofolate reductase, thymidylate synthase, and glycinamide ribonucleotide formyl transferase. MTX represents a common treatment option for solid tumors in the head, neck, and breast. It is also used to treat autoimmune diseases such as psoriasis and rheumatoid arthritis, osteosarcoma and systemic inflammatory diseases [1,2]. MTX belongs to class IV in Biopharmaceutical Classification System exhibiting poor aqueous solubility and low permeability, which together with pH-dependent dissolution, result in limited bioavailability. Complexation with cyclodextrins is a valuable approach for improving the physicochemical properties of drugs and their biopharmaceutical profile [2,3].

The present study investigates the compatibility of the inclusion complex between methotrexate (MTX) and heptakis(2,6-di-O-methyl)- β -cyclodextrin (DIMEB) with excipients in order to improve MTX pharmaceutical performance. Compatibility of the newly formed adduct with a selection of pharmaceutical excipients commonly used in solid dosage forms (like lactose, starch, talc and magnesium stearate) was investigated through a complementary set of instrumental techniques, including Fourier transform infrared spectroscopy (ATR-FTIR), powder X-ray diffraction (PXRD) and thermal analysis (TG/DTG/DSC). The absence or presence of interactions was assessed by comparing the spectral, diffractometric and thermoanalytical profiles of the complex, the pure excipients and their binary mixtures.

The results provide a rational basis for excipient selection in the preformulation of MTX/DIMEB-based solid dosage forms, contributing to the broader effort of improving the therapeutic performance of methotrexate through cyclodextrin complexation technology.

Keywords: methotrexate, excipients, DIMEB, inclusion complex, instrumental analysis

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CRYSTAL ENGINEERING APPROACH FOR IMPROVING THE SOLUBILITY OF TELMISARTAN THROUGH BINARY MULTICOMPONENT SOLID FORMS WITH SELECTED ORGANIC ACIDS

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Telmisartan, a Biopharmaceutics Classification System (BCS) class II drug, exhibits poor aqueous solubility that limits its dissolution rate and oral bioavailability [1]. The present study investigates the preparation of binary multicomponent solid forms of telmisartan with selected pharmaceutically acceptable organic acids using mechanochemical methods. The obtained systems are characterized by powder X-ray diffraction (PXRD), differential scanning calorimetry (DSC), Fourier-transform infrared spectroscopy (FTIR), and thermogravimetric analysis (TGA) in order to evaluate the formation of new solid phases and intermolecular interactions. Equilibrium solubility and dissolution studies are further performed to assess the influence of crystal engineering on the pharmaceutical performance of telmisartan. This work aims to identify novel binary solid forms with improved physicochemical properties and enhanced solubility, providing an alternative strategy for the formulation of poorly water-soluble drugs [2,3].

Keywords: telmisartan; crystal engineering; multicomponent solid forms; organic acids; solubility enhancement

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INFLUENCE OF YARN TWIST ON DRYING PERFORMANCE OF PLAIN COTTON KNITTED FABRICS

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During heavy physical activity or at high ambient temperatures, when the human body produces a large amount of heat, sweat forms on the skin, helping maintain the body's thermal balance. Consequently, the contacting clothing layer should effectively transfer perspiration away from the skin and facilitate evaporation from the outer surface. The latter is important to avoid the wearer feeling uncomfortable, wet, and chilly. Therefore, understanding the drying behaviour of textile materials is quite important for assessing the comfort potential of garments made of them. A considerable number of studies have been investigating moisture transport through textile fabrics. However, the drying ability of textile fabrics has been much less investigated, especially the effect of yarn properties on fabric drying behaviour. Therefore, this investigation aims to indicate the influence of yarn twist on the drying properties of cotton knitted fabrics. From differently twisted cotton yarns, all having the same fineness, three plain knitted fabrics were produced under controlled conditions. Cotton yarns were selected based on cotton's ability to absorb water. To assess drying behaviour, knit samples were soaked in distilled water for 2 hours. A moisture content of 30% (relative to the mass of an absolutely dry sample) was attained using a moisture analyser. The wetted sample was placed with its left side on an electronic balance pan, and sample mass was registered every 5 min for 1 h. Simultaneously, the sample surface temperature was recorded at 5-minute intervals using a portable thermal analyser. The recorded mass values of the samples allow for the calculation of the evaporation mass and construction of a time-evaporation curve. Based on this curve, the evaporation rate for each knitted fabric was determined, indicating the highest evaporation rate of the knit made from the highest-twisted cotton yarn (81 mg/h), followed by the knit composed of the lowest-twisted yarn (76 mg/h) and the knit made from the medium-twisted yarn (57 mg/h). The amount of liquid in the knits above the equilibrium moisture content is absorbed by the fibres or, to a certain extent, fills the macropores between yarn segments and capillaries between fibres. Since the surface mass of these knitted fabrics is the same, they have the same number of bonding sites for water molecules. Therefore, it can be assumed that the cotton fibres absorbed the same amount of water in all three knits. It follows that the observed differences in the evaporation rate should be attributed to free liquid distributed in macropores and capillaries. Tracking the surface temperature of the knits and the temperature difference during drying has provided a clearer understanding of their evaporation capacity and drying behaviour. Initially, the surface temperature of all samples increased over the first 5 minutes because the ambient temperature exceeded that of the samples. However, in the following 20 minutes, the surface temperature decreased, indicating that liquid evaporation was occurring. During the remaining time, varying drying behaviour of the knitted fabrics was observed, which can also be attributed to the different pore distributions in these knits. Considering that differences in the capillary and macropore geometry between knitted fabrics resulted from varying yarn twist, it can be concluded that this yarn basic characteristic can be an effective design tool for an adequate drying performance of textile materials.

Keywords: knitted fabric, yarn twist, evaporation capacity, evaporation rate, surface temperature

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THERMOOXIDATIVE STABILITY OF HAZELNUT KERNELS EVALUATED BY DIFFERENTIAL SCANNING CALORIMETRY

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The oxidative stability of lipid-rich food products is a key factor determining their nutritional quality, sensory properties, and shelf life. Nut kernels, particularly hazelnuts are highly susceptible to lipid oxidation due to their high content of unsaturated fatty acids. The aim of this study was to evaluate the thermo-oxidative stability of hazelnut kernels as a function of storage time and roasting intensity using differential scanning calorimetry (DSC).

The oxidation induction periods of fresh, stored, and thermally treated samples were determined by DSC under nonisothermal conditions. The experimental data were subsequently evaluated using the non-Arrhenian Berthelot–Hood equation to describe the temperature dependence of the induction periods and to predict the oxidative stability of the investigated samples [1]. The influence of storage duration and roasting temperatures of 125 °C and 180 °C on kernels stability was compared.

The results demonstrated a significant decrease in the oxidative stability of hazelnut kernels during storage, with the most pronounced reduction occurring between the first and second month. In contrast, roasting at 125 °C caused only minor changes in the induction periods, whereas roasting at 180 °C significantly accelerated lipid oxidation, leading to shorter induction periods.

The findings indicate that prolonged storage has a more pronounced impact on lipid degradation than moderate thermal treatment. Furthermore, the combination of DSC with kinetic modeling based on the Berthelot–Hood equation proved to be an effective approach for the rapid assessment and prediction of oxidative stability in lipid-rich food matrices. This methodology can be applied in food quality evaluation and may contribute to the optimization of storage and processing conditions for nuts and other lipid-containing bioproducts.

Keywords: differential scanning calorimetry, oxidative stability, hazelnuts, walnuts, lipid oxidation

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THE EFFECT OF SUPERCRITICAL CO₂ ON LC³ CEMENT PASTES WITH DIFFERENT LEVELS OF CEMENT REPLACEMENT

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Geothermal wells include a cement sheath that fills the annular space between the casing and the borehole wall. Its primary function is to provide structural support to the casing and protect it from the aggressive effects of groundwater [1,2]. However, cementing in geothermal wells is exposed to various aggressive factors, among which the supercritical CO₂ (scCO₂) is one of the most severe. The main objective of the study was to evaluate the effect of scCO₂ on Limestone Calcined Clay Cement (LC³) systems. LC³ materials could represent a promising eco-friendly alternative in response to CO₂-emission reduction requirements and the limited availability of conventionally used supplementary cementitious materials. Cement pastes were prepared with different levels of cement replacement (up to 65 wt.%) and exposed to scCO₂ for 7 days at 10 MPa and 70 °C. The performance of the samples under scCO₂ exposure was assessed relative to ordinary Portland cement paste using thermogravimetric analysis (TGA), Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM–EDX), and the phenolphthalein test. The combined use of TGA and FTIR enabled the detection of phase changes associated with carbonation and proved to be more sensitive than the widely used phenolphthalein test. Carbonation behaviour was influenced primarily by the availability of portlandite. The mixture containing 70 wt.% cement exhibited a similar resistance to carbonation as the reference cement paste. Carbonation of portlandite, which proceeds more rapidly than carbonation of other hydrated and clinker phases, led to densification of the surface layers, thereby limiting the penetration of CO₂ into the interior of the specimens. When the cement content was reduced to 50 wt.%, partial carbonation of sulfate-AFm phases and ettringite was confirmed in the central parts of the specimens, leading to the formation of gypsum. However, no decalcification of C-(A)-S-H was observed. Reducing the cement content further to only 35 wt.%, and the resulting limited buffering effect of portlandite, caused carbonation of both unreacted and hydrated clinker phases throughout the entire specimen. This resulted in the formation of aluminosilicate gels and an increase in porosity.

Keywords: LC³ systems, supercritical CO₂, geothermal well

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CHLORIDE BINDING CAPACITY OF CEMENT PASTES WITH VARYING METAKAOLIN CONTENTS

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NaCl-type water is among the most common geothermal waters encountered by cementitious materials used in well construction [1]. The ingress of chloride ions into the cement matrix can alter its phase composition, leading to changes in pore structure and permeability that may ultimately promote corrosion of the steel casing. Since free chlorides dissolved in the pore solution are considered the most detrimental chloride species, enhancing the durability of cementitious materials requires both low permeability and an effective capacity to immobilize penetrating chloride ions. In cement-based systems, chlorides are primarily chemically bound by AFm phases. Since metakaolin (MK) increases the availability of Al^{3+} ions and thereby promotes AFm formation, the present study investigates the resistance of blended cement pastes containing different amounts of MK to chloride attack. Attention is paid to the role of Al^{3+} availability in chloride binding and the associated changes in phase composition and microstructure. Reference cement pastes and mixtures containing silica fume (SF) and metakaolin in SF-to-MK ratios of 1:1 and 1:3 were exposed to a simulated geothermal solution and a NaCl solution with the same chloride concentration for up to 365 days under standard laboratory conditions. The ongoing changes were monitored using thermogravimetric analysis, X-ray diffraction, Fourier-transform infrared spectroscopy, ion chromatography, and scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy. As expected, the increased Al^{3+} content supplied by MK promoted the formation of sulfate-AFm phases, which subsequently transformed into chloride-AFm (hydrocalumite) via ion exchange. As a result, the content of chemically bound chlorides increased with increasing MK dosage, thereby reducing chloride ingress into the specimens. In contrast, the reference cement pastes contained a higher amount of free chlorides. The greater resistance to chloride ingress exhibited by specimens exposed to the geothermal solution, compared with those exposed to the NaCl solution, was attributed to the competitive action of CO_3^{2-} and SO_4^{2-} ions [2,3] and to accelerated hydration leading to pore structure refinement.

Keywords: geothermal water, blended cement pastes, chloride attack, metakaolin

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CHEMICAL ADMIXTURES IN ALKALI-ACTIVATED ALUMINOSILICATE MATERIALS: FRESH AND HARDENED PROPERTIES

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Alkali-activated aluminosilicate materials are promising low-carbon binders. Among the available precursors, fly ash (FA) is particularly attractive because of its economic and environmental benefits. However, its wider utilization is limited by slow setting, poor workability, and relatively low strength development. While strength can be enhanced through mechanical activation and thermal curing, workability-related issues may be addressed by chemical admixtures [1]. Their application in alkali-activated systems is complicated by the highly alkaline environment, which may affect admixture stability and performance. Nevertheless, the use of chemical admixtures in fly ash-based geopolymers remains insufficiently explored. Therefore, this study investigates low-calcium FA-based geopolymers containing a polycarboxylate ether superplasticizer, a high-molecular-weight anti-sedimentation polymer, and a hydrophobic-ester-based defoaming agent. Changes in workability, phase composition, pore structure, and compressive strength were evaluated using TG-DTA, FTIR, XRD, MIP, nitrogen adsorption, SEM-EDX, and compressive strength testing. The results highlight the importance of careful selection and combination of admixtures and provide new insights relevant to their practical application. Admixture addition generally improved workability (153–158 mm vs. 150 mm for the reference) and reduced open porosity, except for the formulation containing all three admixtures (145 mm), but these benefits were accompanied by lower compressive strength. The reference geopolymer reached 50 and 77 MPa after 7 and 28 days, respectively. A low PCE dosage (0.05 wt.%) slightly improved strength (56 and 74 MPa), whereas a higher dosage (0.40 wt.%) reduced it to 51 and 66 MPa. More pronounced reductions were observed for the anti-sedimentation agent (35 and 47 MPa) and the combined admixture system (27 and 41 MPa). Depending on the formulation, strength loss was associated with superplasticizer degradation, lower N-A-S-H gel polymerization, and weakened interfacial bonding caused by mullite particles embedded in the gel.

Keywords: Alkali-activated aluminosilicate material, superplasticizer, anti-sedimentation agent, defoaming agent

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THERMOGRAVIMETRIC CHARACTERISATION OF SORPTION PROPERTIES OF MICROPOROUS CARBON FIBERS

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Porous carbon fibers have found a wide range of practical applications. They can serve as carriers for functional substances, such as drugs, as well as for various nano- and microparticles of active solid materials. A method for producing microporous carbon fibers through controlled carbonization of cellulose has been demonstrated. The main practical advantages of this method are the availability and low cost of raw materials, as well as the feasibility of the preparation process [1–2].

This preparation approach allows for modification of the fiber structure not only by selecting different precursors but also by incorporating various additives, porogens, and extenders in different concentrations, which can be introduced into the cellulose precursor prior to carbonization.

In earlier stages of research, a series of porous carbon microfibers was prepared through the controlled carbonization of cellulose precursors. Their specific surface areas, determined by the BET method from argon adsorption isotherms, ranged from approximately 1 to nearly 1500 m²·g⁻¹. Their structure and properties were comprehensively characterized using complementary thermoanalytical and microstructural techniques [3].

In addition to previously studied water sorption behavior, the sorption of several other liquids was also examined. For this purpose, dried carbon fibers were exposed to the vapors of selected liquids in a vapor-saturated desiccator. The amount of adsorbed liquid was quantified by thermogravimetric analysis (TGA), using a TGA 7 instrument (PerkinElmer). Three liquids with different molecular sizes and geometries were chosen to provide further insight into the porous structure of the fibers: benzene (C₆H₆), hexadecane (C₁₆H₃₄), and trichlorobenzene (C₆H₃Cl₃). The results are discussed in relation to previously determined structural characteristics.

Keywords: Carbonization, Microporous materials, Thermogravimetry

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THERMAL CHARACTERIZATION OF ELECTRODEPOSITED ALGINATE HYDROGELS BY TGA/DSC-MS AND DSC

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Alginate-based hydrogels are widely used in biomedical and engineering applications owing to their high water content and tunable physicochemical properties. Since these properties are strongly influenced by the synthesis route, understanding the relationship between processing conditions, network structure, and thermal behavior is essential. In this study, alginate hydrogels were fabricated by electrodeposition using copper electrodes, with current density and deposition time systematically varied to achieve controlled ionic crosslinking. Rheological characterization demonstrated that increasing both parameters, particularly deposition time, resulted in higher shear modulus and crosslinking density, accompanied by a reduction in mesh size. The thermal behavior of the prepared hydrogels was investigated using complementary thermoanalytical techniques. Simultaneous thermogravimetric analysis and differential scanning calorimetry coupled with mass spectrometry (TGA/DSC-MS) was employed to determine the upper thermal stability limit of the hydrogels, identify the onset of polymer degradation, and monitor the evolution of gaseous decomposition products. Furthermore, differential scanning calorimetry (DSC) was used to examine the state of water within the hydrogel network. While no freezable bound water was detected in samples deposited for 5 min, its content increased to 4.60 wt.% and 6.50 wt.% in hydrogels deposited for 20 and 40 min, respectively. These findings indicate that prolonged electrodeposition produces a more densely crosslinked network capable of retaining a greater fraction of polymer-associated water. The combined rheological and thermoanalytical results provide a comprehensive insight into the influence of electrodeposition parameters on the structure, thermal stability, and water organization in alginate hydrogels.

Keywords: alginate hydrogels, electrodeposition, TGA/DSC-MS, DSC, water-polymer interactions

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TRACKING CHANGES IN THERMAL BEHAVIOUR ALONG THE POLYETHYLENE TEREPHTHALATE RECYCLING CHAIN

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The recycling of polyethylene terephthalate (PET) involves several processing steps that may alter the properties and quality of the resulting material. Understanding these changes is essential for evaluating the suitability of recycled plastic waste streams for further processing and potential applications. Differential scanning calorimetry (DSC) is widely used for polymer characterisation, as it enables rapid determination of thermal properties associated with glass transition, crystallisation, and melting. These parameters provide valuable information about the polymer's thermal history, degradation level, and crystalline structure. However, interpretation of multiple thermal parameters can be challenging when assessing differences between materials at various stages of the recycling chain. In this study, DSC was used to characterise industrially recycled PET (rPET) and PET regranulates. Thermal parameters related to glass transition, melting, and crystallisation were extracted from DSC measurements and analysed using multivariate statistical methods. Pearson correlation analysis and principal component analysis were applied to investigate relationships among selected thermal descriptors and to compare the thermal characteristics of both material groups. The multivariate approach enabled visualisation of similarities and differences between rPET and PET regranulates and revealed the thermal parameters that contribute most strongly to sample differentiation. The results demonstrate that combining DSC with multivariate statistical analysis provides a comprehensive framework for characterising PET materials and tracking changes in their thermal behaviour throughout the recycling process.

Keywords: polyethylene terephthalate recycling, regranulation, differential scanning calorimetry, multivariate statistical analysis, principal component analysis

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DESIGN AND CHARACTERIZATION OF MIXED ORGANIC-INORGANIC PEROVSKITES BASED ON METHYLAMMONIUM AND FORMAMIDINIUM ANALOGUES

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Electricity consumption has increased steadily since the Industrial Revolution, driving the transition to renewable energy sources. In the European Union, solar energy is the fastest-growing renewable source and contributes to the overall share of renewables, which accounted for 47.5% of electricity consumption in 2024. Despite this growth, solar energy remains underutilised due to cost and efficiency limitations of conventional silicon-based technologies.

Perovskite materials have emerged as promising alternatives, with efficiencies rising from 2.2% in 2006 to 25.5% in 2020. These materials, with the general formula ABX_3 , allow flexible tuning of composition and properties through substitution at the A, B, and X sites. While methylammonium lead iodide is widely studied, the toxicity of lead has motivated research into alternative metals. The suitability of perovskites for photovoltaics is commonly assessed using the tolerance and octahedral factors.

In this work, we focused on the synthesis and characterisation of mixed organic–inorganic perovskites containing iodide ions at the anionic site. Based on a systematic review and critical analysis of the relevant literature, combined with the calculation of the tolerance and octahedral factors to predict the formation of an ideal cubic structure, five different perovskites were synthesised. The synthesis was carried out in two stages: first, the organic precursors were prepared; in the second stage, they were dissolved together with inorganic iodides in selected solvents.

XRD analysis confirmed the successful synthesis of the organic precursors MAI and FAI. A detailed structural study was conducted, and the crystal structures of selected compounds were determined using single-crystal diffraction. Understanding the structural properties of hybrid organic–inorganic perovskites (HOIPs) is crucial for their application in photovoltaics and for the design of efficient solar cells.

As part of this work, particular attention was devoted to bismuth iodide-based perovskites, focusing on phase transitions in methylammonium and formamidinium analogues. Temperature-dependent infrared (IR) spectroscopy was used to investigate structural changes, revealing the existence of five phases in methylammonium bismuth iodide (MABiI) and four phases in formamidinium bismuth iodide (FABiI). These findings were further supported by differential scanning calorimetry (DSC). In addition, ultraviolet (UV) stability testing was performed to evaluate the long-term stability of the materials under light exposure.

Keywords: hybrid organic–inorganic perovskites, differential scanning calorimetry, UV stability

Acknowledgements:

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SMART PCM–LIAPOR PLASTER FOR ENERGY-EFFICIENT BUILDING RENOVATION

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The largest part of the Slovenian building stock consists of buildings constructed after the Second World War. Various studies report that buildings account for 40% of total energy consumption in the EU during their life cycle. Given the large stock of energy-inefficient buildings, energy-efficient renovation of older buildings is one of the most important measures under the Green Transition. For this reason, measures to reduce energy consumption include the use of innovative new materials.

Plaster with phase change materials (PCM) is an innovative material that can improve energy efficiency in buildings. In this study, three different plasters were prepared. The reference sample (R) is a traditional plaster made with dolomite sand, white cement, and lime (volume ratio A:C:L = 6:2:1). Sample P contains an addition of PCM slurry at 30 vol.% in the mixture, while sample PL also contains liapor. Liapor replaces sand in a proportion of XX vol.%. The properties of all investigated mixtures correspond to commercial plaster. The thermal behaviour of all three plasters was determined using differential scanning calorimetry (DSC), with repeated heating and cooling cycles performed to evaluate the stability and reproducibility of the prepared samples. The results demonstrated consistent and reproducible thermal effects, while further testing of decomposition processes – simulating fire exposure – confirmed that no harmful gases were released during heating up to 1200 °C in air.

For the study of the thermal response model, a building constructed in the 1980s was also used. The thermal performance of the PCM-enhanced plaster was investigated by installing test specimens containing phase-change material alongside reference specimens without PCM under real operating conditions. The experimental setup was located in a traditional farmhouse room equipped with a stationary solid-fuel heating system. Throughout the monitoring period, continuous measurements of indoor indoor and outdoor environmental parameters were conducted to quantitatively assess the impact of PCM incorporation on the thermal stability and dynamic thermal response of the space.

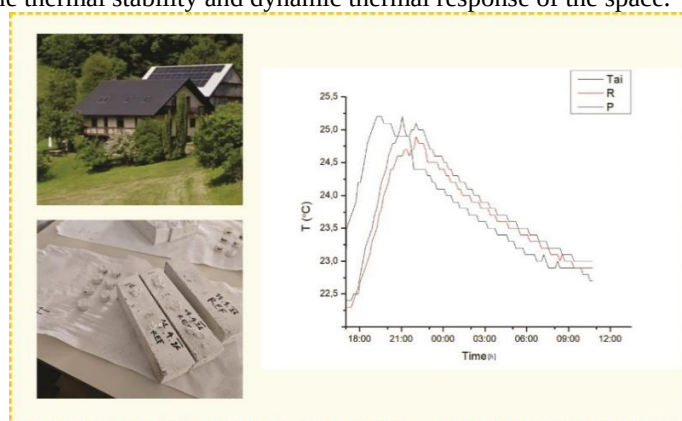


Figure 1. Images of the investigated house, samples (R and P), and their surface temperatures obtained, together with indoor air temperature (T_{ai})

Keywords: Thermal comfort, plaster with PCM, differential scanning calorimetry

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USE OF CERAMIC CONSTRUCTION AND DEMOLITION WASTE IN THE MANUFACTURE OF SUSTAINABLE TRIAXIAL CERAMICS

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Driven by the rapid increase in Construction and Demolition Waste (CDW), research into its reuse is crucial for advancing the circular economy. This study evaluates the viability of incorporating structural ceramic CDW to promote their recovery and support the circular economy. To this end, four triaxial formulations were prepared by blending kaolinitic clay from Oliete, Spain (40–55%), commercial feldspar (25–30%), and ceramic CDW (15–35%). To assess the impact of CDW on sintering and the final product properties specimens were heat-treated in an industrial electric kiln at eight temperatures, between 1025 and 1200 °C, at a heating rate of 2 °C/min and a 2-hour holding time. Characterization focused on Differential Thermal Analysis-Thermogravimetry (DTA-TG), dilatometry, gresification curves (linear shrinkage versus water absorption), and mineralogical evolution during firing. DTA-TG of all samples revealed an endothermic peak at 525–545 °C accompanied by of 4–8% weight loss due to the dehydroxylation of kaolinite into metakaolinite. Samples with higher carbonate contents exhibited a second endothermic peak at 765 °C, associated with a weight loss of ~ 2% from carbonate decomposition. An exothermic peak observed between 935 and 949 °C corresponds to mullite crystallization, which was confirmed by X-ray diffraction to increase with sintering temperature. In addition, the formation of a glassy phase is produced, which is expected to influence the final porosity of the ceramics. Dilatometric curves show an initial contraction associated with the kaolinite dehydroxylation, followed by the α - β polymorphic quartz transition at 560 °C. A second distinct contraction occurs between 840 and 920 °C, corresponding to the onset of sintering. Finally, up to 970 °C a reduction in shrinkage is observed, coinciding with mullite formation. During the cooling stage, the curve displays the reverse β - α polymorphic quartz transition. Physical parameters such as colour variation relative to firing temperature, density, and the coefficient of thermal expansion were determined for each sample. The sintering behaviour of the bodies indicated an optimal firing temperature range between 1040 and 1110 °C. Ultimately, modifying the non-plastic filler content alters the refractoriness and firing shrinkage of the material. The addition of CDW increases water absorption (porosity) and reduces linear shrinkage.

Keywords: Construction and Demolition Waste, Ceramics, Recycling, Clay materials

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ENVIRONMENTAL REMEDIATION OF TANNERY-IMPACTED SOILS THROUGH GLASS AND GLASS-CERAMIC PRODUCTION

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Soils degraded by anthropogenic contamination cannot always be restored, and alternative solutions must be implemented either to stabilize or to revalorize them. One promising strategy currently under investigation is the use of these soils in the production of glass and glass-ceramics. This study aims to evaluate the feasibility of manufacturing glass and glass-ceramics from contaminated volcanic soil sourced from an industrial area in San Jacinto de Ambato, Ecuador. This soil exhibits a high level of heavy metal contamination, particularly Cr, exceeding the permissible limit of 65 mg/kg established for agricultural soils under Ecuadorian environmental regulations (Ministerial Agreement 097-A, 2015) [1]. This contamination is associated with the discharge of effluents from local tanning industries. Soil samples were collected from two depth intervals, 0–15 cm and 15–30 cm. After drying, the samples were sieved to 2 mm to isolate and concentrate the finer fraction. Chemical analysis revealed that the composition at both depth levels is nearly identical and confirming the feasibility of sintering soda–lime silica glasses through the addition of modifying oxides. The main oxides identified in the soils were SiO₂ (57 mass%), Al₂O₃ (17 mass%), CaO (6 mass%), Na₂O (3 mass%), K₂O (1 mass%), MgO (4 mass%), Fe₂O₃ (6 mass%), and trace amounts (<1 mass%) of TiO₂, P₂O₅ and MnO. Regarding heavy metal contamination concentration varies by depth. Only Cr exceeded the regulatory threshold for agricultural land, reaching 795.9 mg/kg in the top layer (0–15 cm) and 726.7 mg/kg in the lower layer (15–30 cm). Conversely, Cu, Ni, Pb, Zn, and Co remain below the limits. The dominant mineral phase is sodic feldspar, followed by augite, illite, and <1 mass% of iron and aluminium phosphates. Based on these results, four glasses formulations were developed using different proportions of the 15–30 cm soil fraction and adding modifying oxides as carbonates. Two glasses were synthesized: one of them (G_{75/15Na/10Ca}) was composed of 75 wt% soil, 15 wt% Na₂CO₃, and 10 wt% CaCO₃, and another (G_{80/20Na}) was composed of 80 wt% soil and 20 wt% Na₂CO₃. The glass transition temperatures (T_g) were 575 and 550 °C, the dilatometric softening temperatures were 651 °C and 636 °C, and the coefficients of thermal expansion was 12×10⁻⁶ and 13×10⁻⁶ °C⁻¹, respectively. Differential thermal and thermogravimetric analysis of the glasses revealed an initial event corresponding to T_g, followed by an exothermic peak due to crystallization, and an endothermic peak associated with melting. Viscosity–temperature curves indicate a working range below 400 °C, and therefore they correspond to the so-called short-working glasses. Leachate analysis of the glasses and glass-ceramics shows that metallic elements are successfully retained within their structure. Therefore, vitrification represents a highly viable and sustainable approach for the remediation and valorisation of contaminated volcanic soils.

Keywords: Glass, Glass-ceramic, Vitrification, Remediation, Soil

Acknowledgements:

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HEAT OF HYDRATION AND SETTING TIME OF ALKALI-ACTIVATED HIGH CALCIUM FLY ASH CEMENT PASTES WITH AND WITHOUT PORTLAND CEMENT

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Heat of hydration and setting time of alkali-activated fly ash cement pastes were investigated. High calcium fly Ash (FA) was used to replace Portland cement by weight at 70%, 80%, 90% and 100%. Alkali solution of 10 Molar sodium hydroxide (NaOH) and sodium silicate solution (Na₂SiO₃) with a 0.67 ratio of Na₂SiO₃/NaOH was used. Alkali liquid/binder of 0.65 ratio was used. Setting time tests were carried out at 23°C. An Isothermal Calorimetry instrument was used for heat of hydration measurement. The results indicated that the quantity of heat produced by 100FA was significantly lower than Portland cement containing mix. This means that the heat occurred likely to come from the main Portland cement hydration which resulted in a beneficial increase in the setting time.

Keywords: High calcium fly ash, Cement, Alkali activated, Heat of hydration, Setting time

THERMAL ANALYSIS OF HYDRATED SUSTAINABLE HIGH-CALCIUM COAL BOTTOM ASH COLD-BONDED ARTIFICIAL AGGREGATES WITH LOW PORTLAND CEMENT USAGE

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This study focuses on the development of environmentally friendly cold-bonded artificial aggregates using high-calcium coal bottom ash (BA) as a supplementary binder and as the primary precursor, combined with Portland cement (PC). The objectives were to investigate the hydration products by means of thermal analysis (TG/DTG), microstructural characteristics and crushing strength of the artificial aggregates. The aggregates were prepared by partially replacing bottom ash with PC at 5-15 wt%. The water to binder ratio of 0.30 was used. TG/DTG results demonstrated that the mixture containing 10 wt% PC was optimal and significantly enhanced aggregate performance, showing higher amounts of ettringite and calcium silicate hydrate (C-S-H) phases compared to other mixtures, including the control sample (100BA). This is due to the formation of hydration products through both direct hydration of Portland cement and secondary pozzolanic reactions between the pozzolanic materials and calcium hydroxide derived from cement hydration reactions. Moreover, microstructural analysis further confirmed that artificial aggregates containing 10 wt% PC exhibited a significantly denser microstructure than the other mixtures.

Keywords: High-calcium coal bottom ash, Cold-bonded, Artificial aggregates, Thermogravimetry

HALOGEN-FREE FIRE PROTECTION: PHOSPHORUS AND BORON SYNERGY IN RIGID PU FOAMS

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Rigid polyurethane foams (RPUFs) are indispensable materials in the construction and insulation industries due to their low density and superior thermal insulation properties; however, their organic nature poses significant fire risks. This study focuses on the development of an environmentally friendly, halogen-free flame retardant (FR) system, termed "TDB," based on the synergistic effects of phosphorus, nitrogen, and boron. The TDB structure was synthesized in a two-step process involving the reaction of 1,3,5-Tris(2-hydroxyethyl)isocyanurate (THEIC) with diphenyl phosphoryl chloride (DPCP) and subsequent esterification with boric acid. [1- 3] The synthesized FR was incorporated into RPUF formulations at various concentrations (5%, 10%, and 15% by weight) using the one-shot free-rise method. Chemical structures were confirmed via $^1\text{H-NMR}$, $^{15}\text{P-NMR}$, and FT-IR spectroscopy. Morphological analysis through SEM revealed that TDB acts as a heterogeneous nucleation agent, leading to a reduction in cell size while maintaining a closed-cell structure. Flammability performance was evaluated using Limited Oxygen Index (LOI) and Cone Calorimetry tests. The LOI value increased from 18.8% for neat RPUF to 20.8% with TDB loading. Cone calorimetry results for the 10% TDB-loaded foam showed a 13% reduction in peak heat release rate (pk-HRR) and a 29% reduction in total heat release (THR) compared to neat RPUF. Furthermore, TDB significantly improved mechanical strength; the compressive strength of 10% TDB-loaded foam reached 214 kPa, a substantial increase over the 70 kPa observed for neat RPUF. Thermal conductivity was preserved, ensuring the material's primary insulation function was not compromised. These findings suggest that the TDB synergistic system is a highly effective, non-toxic alternative for enhancing the fire safety and structural integrity of rigid polyurethane foams in industrial applications.

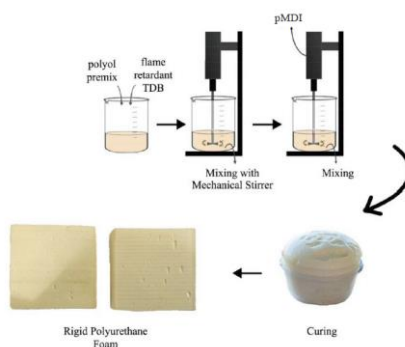


Figure1: Illustration of the scheme of preparation of RPUFs.

Keywords: rigid PU, Halogen-free flame retardant, phosphorus-boron synergy, thermal insulation, compressive strength

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INFLUENCE OF DISTINCT MECHANICAL ACTIVATION ROUTES ON THE REACTION KINETICS AND MICROSTRUCTURAL EVOLUTION OF SRZRO₃

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Strontium zirconate (SrZrO₃) is a perovskite-type ceramic of interest for high-temperature and dielectric applications because of its chemical stability, thermal resistance, and functional electrical properties. However, its conventional solid-state synthesis is often limited by slow interdiffusion between precursor particles, incomplete phase development, and the requirement for relatively high processing temperatures. In this study, the influence of different mechanical activation routes on the formation kinetics, phase development, and microstructural characteristics of SrZrO₃ powders was systematically investigated.

Stoichiometric amounts of SrCO₃ and ZrO₂ were calculated to produce approximately 100 g of SrZrO₃ according to the reaction $\text{SrCO}_3 + \text{ZrO}_2 \rightarrow \text{SrZrO}_3 + \text{CO}_2$. Three powder-processing routes were compared. In the first route, SrCO₃ and ZrO₂ powders were separately mechanically activated for 1 h, subsequently combined, and homogenized by ball milling for 24 h. In the second route, the precursors were first mixed by ball milling for 24 h and then mechanically activated together in a planetary mill for 1 h. A non-activated mixture was prepared as a reference condition. All powder mixtures were calcined using an identical thermal schedule involving a 1 h dwell at 900 °C followed by heating to 1200 °C. The calcined powders were subsequently shaped and sintered at 1400 °C.

The reaction kinetics of SrZrO₃ formation were evaluated using thermal analysis data to determine the effects of mechanical activation on decomposition behaviour, reaction temperatures, conversion characteristics, and apparent activation energy. Phase evolution and possible secondary phases were examined by X-ray diffraction, while crystallite size and microstrain variations were evaluated through peak-broadening analysis. Powder morphology, agglomeration behaviour, elemental homogeneity, and sintered microstructures were investigated by scanning electron microscopy coupled with energy-dispersive spectroscopy.

The study demonstrates how the sequence and mode of mechanical activation affect solid-state reaction kinetics and the resulting powder microstructure, providing a basis for optimizing SrZrO₃ processing routes with improved phase purity and microstructural uniformity.

Keywords: SrZrO₃; solid-state synthesis; mechanical activation; ball milling; reaction kinetics; calcination; microstructure

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SUSTAINABLE PRODUCTION OF MgAl_2O_4 SPINEL FROM WASTE MATERIALS: PROCESSING PARAMETERS AND CRYSTALLIZATION KINETICS

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Magnesium aluminate spinel is a highly important refractory material due to its very high melting point, high chemical and corrosive stability and excellent thermal shock resistance [1]. It has attracted growing interest for various applications such as structural material in fusion reactor, refractory material, capacitor, humidity sensor and catalysts [2]. It is especially evaluated as transparent ceramic [3]. However, its production from primary raw materials, namely high-purity MgO and Al_2O_3 powders, is costly. In this study, MgAl_2O_4 spinel suitable for the circular economy was produced using magnesite factory waste as a raw material, both to reduce production costs and to reduce the environmental impact of industrial waste. For this purpose, magnesite waste and Al_2O_3 with a composition determined according to the MgO - Al_2O_3 phase diagram were used as starting raw materials. The phase transformations of the spinel samples produced by solid-state synthesis at different sintering temperatures were examined by X-ray diffraction (XRD) analysis and their microstructural properties were investigated by scanning electron microscopy (SEM) analysis. The bulk density, % apparent porosity and % water absorption values were measured by the Archimedes method. In addition, studies on the crystallization kinetics of MgAl_2O_4 were carried out from differential thermal analysis (DTA) curves. For this purpose, the peak temperatures of spinel transformations were determined from DTA curves obtained at different heating rates (heating rates of 10 - 15 - 20 $^{\circ}\text{C min}^{-1}$) and the activation energy was calculated. The highest density value in the obtained MgAl_2O_4 spinel was measured as 2.95 gcm^{-3} in the sample sintered at 1550°C for 1 hour. The same sample has an apparent porosity of 0.31% and a water absorption rate of 0.16% and the measured hardness value was found to be 1680 HV .

Keywords: MgAl_2O_4 spinel, Waste recycling, Differential thermal analysis (DTA), Kinetics, Magnesite waste

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HIGH-THROUGHPUT CO-AMORPHOUS MIXTURE PREPARATION & OPTIMISATION WITH MULTI-HEATING RATE & MODULATED DSC

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Co-amorphous mixtures (CAMs) have gained widespread pharmaceutical interest owing to their ability to stabilise the amorphous state against crystallisation whilst enhancing apparent solubility. CAMs consist of low molecular weight compounds combined into a single homogenous amorphous phase. Upon mixing, crystallisation can be inhibited by noncovalent drug-excipient interactions, decreased molecular mobility, and free energy reduction between crystalline and amorphous states [1].

Differential scanning calorimetry (DSC) is considered a key technique for characterising CAMs, which can measure various properties to screen for the ‘optimal’ CAM ratio:

1. Highest glass transition temperature (T_g). Lower molecular mobility can hinder crystallisation.
2. Greatest Gordon-Taylor T_g deviation. Non-ideal mixing implies hetero-intermolecular interactions. The ratio of largest deviation of measured from predicted T_g values suggests maximised interactions.
3. Eutectic ratio. Simultaneous melting at a lower temperature also indicates maximised interactions.

In our previous work [2], binary CAMs of nifedipine (NIF) and celecoxib (CEL) exhibited three different optimal ratios, spanning 50-70 mol% CEL. To assess the ‘true’ stable optimal, different crystalline NIF-CEL mixtures were prepared simultaneously on a small scale. The solidus (T_{sol}) and liquidus (T_{liq}) temperatures of crystal mixtures were obtained by DSC. Amorphous samples were produced by automated DSC quench-cooling *in situ*, and then analysed immediately thereafter under precise, highly repeatable conditions. Variable heating rate DSC was applied to obtain T_g and dynamic fragility. Then, crystallisation was assessed through quasi-isothermal modulated temperature DSC (QiMTDSC). Annealing for different times below T_g enabled assessment of enthalpy relaxation rates using MTDSC. All data was evaluated against crystallisation behaviour within the same DSC sample, increasing comparability in a series of single 5 mg DSC experiments (Fig 1.). This enabled data-rich and automated workflows from preparation to analysis, without the usual associated costs, labour, and idle time.

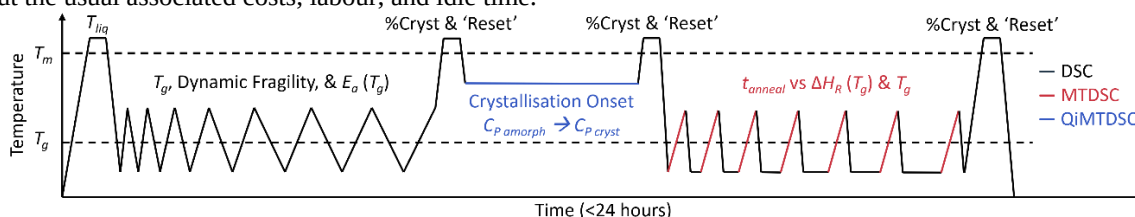


Figure 1: DSC time-temperature workflow for *in situ* CAM production and characterisation.

Keywords: Differential Scanning Calorimetry, Co-Amorphous Mixture, Eutectic Ratio, Fragility, Crystallisation.

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