21-23 MAY, 2025
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BOOK OF PROCEEDINGS

PMA & SRC CONFERENCE

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EDITORIAL

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Bratislava, 2025

21-23 MAY, 2025
SMOLENICE CASTLE

DR. H. C. ING. ŠTEFAN ROSINA AWARD

POLYMER INSTITUTE OF THE SLOVAK ACADEMY OF SCIENCES, as one of the organizers of the international conference "Polymer Materials in the Automotive Industry PMA & Slovak Rubber Conference SRC 2025 in the interest of awarding extraordinary results and outstanding works of research, development, and innovation in areas belonging to the conference topics, announces the competition

"Dr. h. c. Ing. Štefan Rosina Award"

for individuals or collectives in two categories:

INNOVATIONS WITH APPLICATION POTENTIAL

COMPETITION CRITERIA

- Innovativeness and technical level of the subject of the competition proposal
- Potential economic, business, environmental, and social benefits and protection of intellectual property rights
- Multiple proposals can be submitted to the competition, while only one application can be submitted for one subject
- The application does not have a prescribed format but must contain all the attributes described in the first two points of the competition criteria. The length of the application should be between 3 and 5 pages of A4 format
- The period for **sending applications** is from **October 1, 2024 till January 31, 2025**
- The **evaluation** of applied innovations will take place during the period from **February till April 2025**

Only individuals, research groups or companies operating in Slovakia can participate in the competition

PRESENTATION OF AWARDS

From the submitted applications, the committee will select one innovation, which will be awarded by a diploma and a financial reward in the amount of

1 000 EUR

THE AWARDS WILL BE PRESENTED DURING THE CONFERENCE

All information can also be found in the Statute of Dr. H. c. Ing. Štefan Rosina, which is available on the home page of the organizer of the competition and the webpage of the PMA & SRC 2025 conference.

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Polymeric Materials in Automotive
PMA

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Slovak Rubber Conference

BOOK
OF
PROCEEDINGS

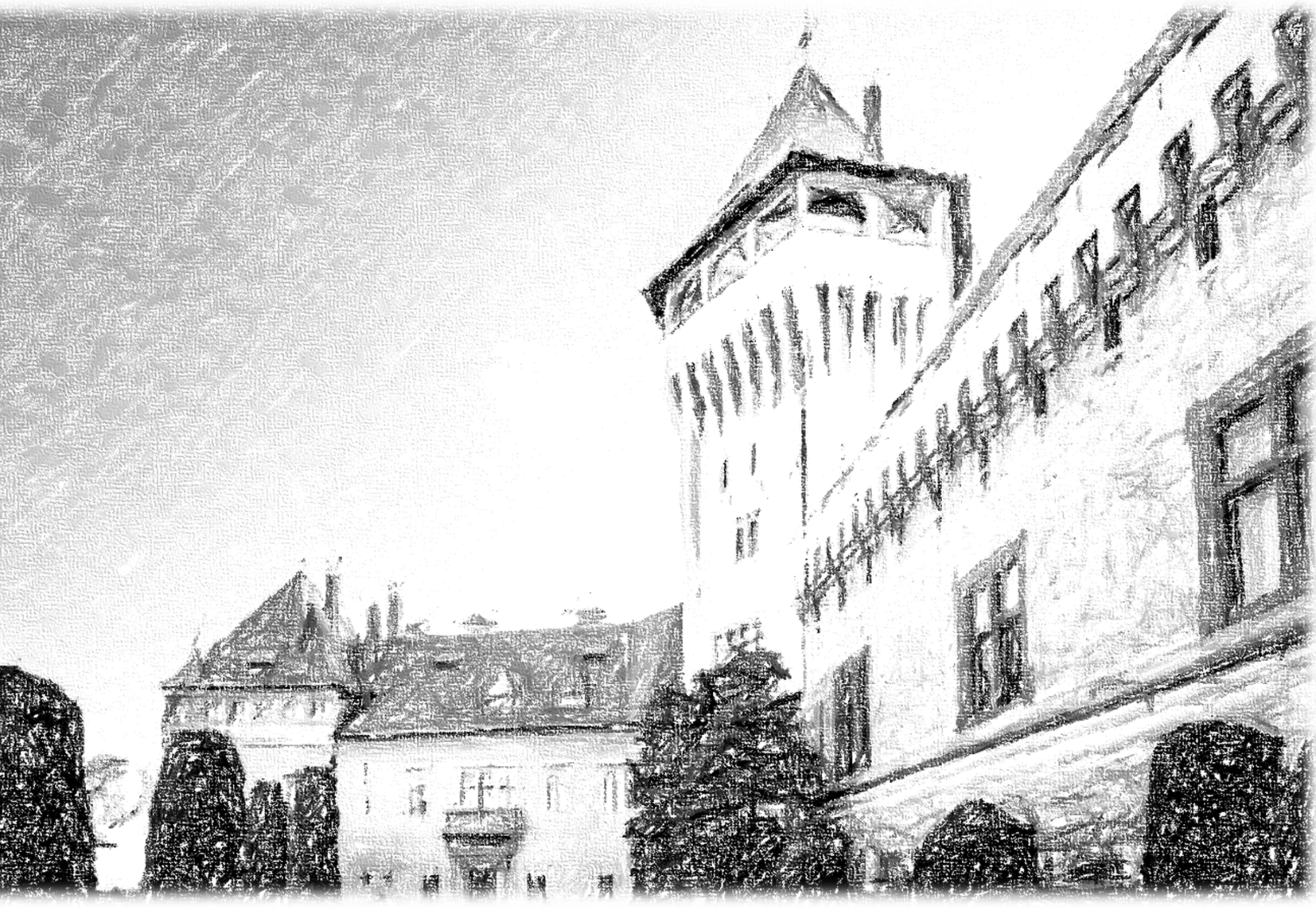
Slovak University of Technology, Faculty of Chemical and Food Technology
Polymer Institute, Slovak Academy of Sciences

FOREWORD

In 2005 the first International Conference on Polymeric Materials in Automotive was organized in Bratislava starting new tradition of events reflected steeply rising importance of automotive industry in Slovakia. That is related to the presence of dominant investors in Slovakia, namely Volkswagen, PSA, Kia and Jaguar Land Rover and including a number of other companies – suppliers of plastics and rubber parts being a significant part of them – building up new facilities in the country. The appreciated feature of the PMA series consists in a fact that, although targeted to polymeric materials used in automotive industry, the scope of the conference is kept highly scientific. Thus, new ideas have been presented, many of these being far away from industrial application, still contributing significantly to a progress in the area.

Similar to the first International Conference on Polymeric Materials in Automotive PMA 2007, PMA 2009, PMA 2011, PMA 2013, PMA 2015, PMA 2017, PMA 2020 and PMA 2023 the upcoming International Conference on Polymeric Materials in Automotive PMA 2025 is targeted on various aspects related to plastics and rubber in the automotive industry, with the aim to present and discuss innovative approaches towards new polymer products increasingly having a decisive influence on the design and appearance of new generation of cars. Almost over 1000 participants from 27 countries attended the PMA conferences which were ranked as successful and interesting.

International scientific conference on rubber, Slovak Rubber Conference, used to be organized every year by the Rubber Research Institute of Matador Púchov. From 2005 this traditional event is held as a part of the International Conference on Polymeric Materials in Automotive and this will be the case also for PMA 2025 organized together with the 26th Slovak Rubber Conference in Smolenice Castle.



List of LECTURES

List of POSTERS

CONFERENCE PROCEEDINGS

RECYCLED PLASTICS IN THE AUTOMOTIVE INDUSTRY

Libor DOBEŠ

Moravskoslezský automobilový klastr, z.s., Technologická 375/3, 708 00 Ostrava-Pustkovec, Czech Republic
email: l.dobes@autoklastr.cz

The automotive/mobility ecosystem is one of the 14 key ecosystems in the European Union. The development and production of automobiles and their accessories are characterized e.g. by a high degree of consumption of precious metallic materials and polymeric materials with specific physical, mechanical and other properties, which are required by the Union regulations and the specific demands of the car manufacturers/OEMs. When talking about the ecological sustainability of the production and usage of automotive products, the circular use of materials is getting the top visibility and attention. The demand for ecological sustainability in the automotive sector is linked to a significant focus on using the lighter and stronger materials that are recyclable. In the case of products made from polymeric materials, the parameters of the use of recyclate in new products are defined by the final customers and the adherence to the rules is strictly monitored. The original polymer granulate has the quality parameters set by its manufacturer, and yet there is variability in the parameters of individual batches of material. This variability increases even further when recycled polymers are used in a mixture with the virgin material. It is a challenge for the technological research and development in companies, requiring cooperation with the scientific/research centres, design development, quality management and purchasing depts. (securing suppliers of certified polymer regranulates) so that the final product always meets the requirements of the end customer. The production process must meet the requirements for traceability and continuity, must be stable, high-quality, and economical. We also provide some relevant practical examples and references to the normative base in presentation.

References (info)

1. Mobility Transition Pathway (europa.eu)
2. Škoda Auto Sustainability (skoda-auto.com)
3. Udržitelnost - Škoda Storyboard (skoda-storyboard.com)
4. Reporting | Volkswagen Group (volkswagen-group.com)
5. Materials Management - Supply Chain Management | AIAG
6. daimler-environmental-check-mb-cla.pdf (mercedes-benz.com)

PERSONALIZED AND INTELLIGENT TRAVEL EXPERIENCE IN SMART CABIN

Ivan KEBISEK, Engineering DIRECTOR, Yanfeng International Automotive Technology

Lecture Abstract:

- Yanfeng Cross-Business Integrated and Innovative Solutions

Yanfeng Cross-Business Integrated and Innovative Solutions

Core Product line

Automotive Interiors
Automotive Seating
Passive Safety
Cockpit Electronics
Door Module & Exteriors

INTEGRATED INNOVATED

Seat x Safety
Interior x Safety
Smart Steering Wheel
Door System
...

ECU
Smart Cabin Controller
Display
Smart Switch
Lighting
Smart Surface / Deco
Motor
Mechanism

Platform Parts

- Mega Trends & Automotive Interiors

Mega Trends

Worldwide Wellness Wave
- Consumers are prioritizing mental health and work-life balance
- For young generation, health concerns are led by mental health & stress

Uncertain Future
- Affordability crisis, concern over future of jobs as technology grows
- Increase in alternative mobility solutions; remarketing in sustainability

Retired & Ready to Spend
- Longer life expectancies are increasing global elderly population
- High-income baby boomer and Silent Generation consumers are a sizable cohort

Growth & Impact of Technology
- AI could create 69 million new jobs globally by 2030
- 5G is expected to add $1.7 trillion to U.S. GDP, create 4 million jobs
- Digital communication methods will increase stress among consumers

Youthful Inheritance
- Millennials & Gen X are poised to inherit a big portion of global wealth
- They are prioritizing well-being with a trend towards seeking comfort and serenity in daily life and purchases

Climate Impact & Battle
- Ascending levels of greenhouse gases (CO2), boost in WW temperatures
- >2000 businesses aiming for net zero carbon emissions, move to EV

- Automotive Inspirations From China
  - New and atmospheric interior design
  - “Caring for family” & “Personal space”
  - Refined Detail
  - Sense of ritual
  - Traditional aesthetics and cultural identity
  - “Guochao” China Chic
- Automotive Cabin Innovations inspired by Sustainability

- Experience in Motion = Yanfeng Smart Cabin Demonstrators

YANFENG "EXPERIENCE IN MOTION" SHOW CARS

Yanfeng

2013 2015 2017 2019 2020 2022 2024 2025

Concept Focus	Urban Mobility Autonomous 3.0 China Luxury	Autonomous 4.0 Car Sharing Health & Wellness	Quality of Life Car / Ride Sharing Owned Mobility	User-Centric Integrated Production-Ready	Digital + Luxury Sustainable Premium Intelligent integration	Fluid Space Gen Z focus highly interactive moving experience
Show Car	ID16	XiM18	XiM20	XiM21/s	XiM23	XiM25

- XiM25 – Latest Smart cabin demonstrator, focused on future consumers - gen Z

RECENT PROGRESS ON FLAME-RETARDANT POLYMERS IN AUTOMOTIVE: FROM FUNDAMENTAL TO APPLICATION

De-Yi WANG

IMDEA Materials Institute, C/Eric Kandel, 2, 28906, Getafe, Madrid, Spain
email: deyi.wang@imdea.org

In recent years, polymer materials have been increasingly applied in the automotive industry and have become key materials for achieving vehicle lightweighting, energy conservation, and functional integration. Currently, polymers account for approximately 10% to 15% of a vehicle's total weight[1], and in some lightweight models, this figure exceeds 20%. These materials are primarily used in interior components (such as dashboards, door panels, and seats), exterior body parts (such as bumpers and fenders), and functional structural components (such as fuel tanks and intake manifolds).

Europe automotive polymer composites market dominated the global automotive polymer composites market with a revenue share of 37.1% in 2023. The European automotive industry is a significant sector, accounting for a substantial share of global production[2].

Commonly used polymers include polypropylene (PP), polyurethane (PU), polyamide (nylon), polycarbonate (PC), polyvinyl chloride (PVC), and acrylonitrile–butadiene–styrene (ABS). However, the widespread use of such flammable materials has significantly increased the risk of fire hazards. Therefore, efficient and environmentally friendly flame-retardant modification of polymer materials has become a critical focus in the development of automotive materials.

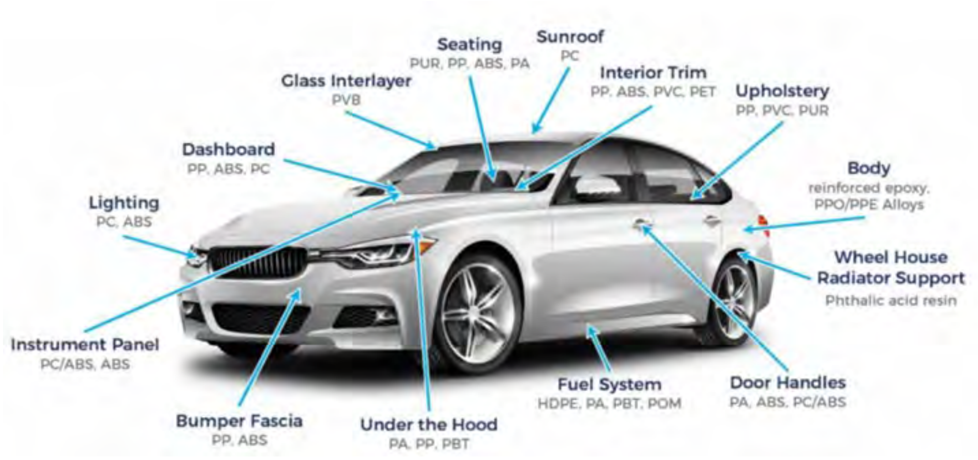


Figure.1. Polymers in vehicles1

In these situations, my group has been focusing on the flame-retardant studies of varied polymers for more than 15 years, including both thermoplastics and thermosets, and/or composites. In this lecture, the latest research and findings will be presented and discussed, such as flame-retardant PP[3], Polyamide, Composites, etc.

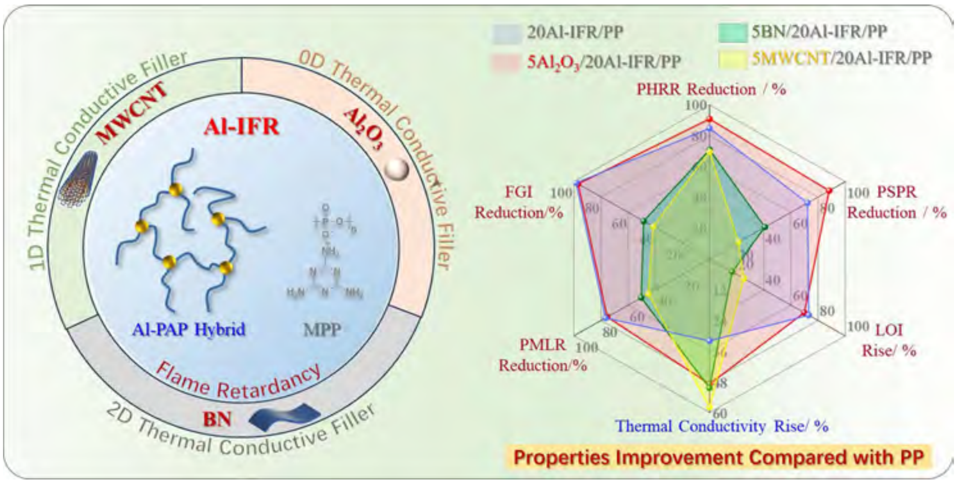


Figure.2. Novel flame retardant method to PP[4]

In addition, coating technology is undoubtedly the optimal solution for metal-based materials, such as aluminum and its alloy. In the field of batteries, aluminum is frequently used for the fabrication of integral pack battery enclosures and laminated pouches for soft-package batteries (SPB). Owing to the high-temperature meltable characteristics and high thermal conductivity of aluminum, it is extremely susceptible to the risk of thermal runaway and explosion of batteries under fire conditions[5,6]. Therefore, the development of high-performance fire-resistant coatings is essential for high safety batteries, in particular to the rapid growth markets of electric vehicles.

The development of high-efficient flame-retardant coatings is crucial for enhancing the fire safety of materials, especially in the high-performance polymer and batteries fields. Traditional intumescent coatings, while effective in providing a rapid response, often struggle to endure prolonged exposure to high-intensity fires. On the other hand, ceramicizable coatings, known for their stability, typically fail to offer immediate protection due to the time required for ceramic formation. As a result, the applicability of these two conventional coatings is significantly constrained. Inspired by differential response behaviors, we designed an innovative bi-layered coating that contains organic and nano-inorganic components, enabling both rapid response and extended protection. With a thickness of only 320 microns, this novel bi-coating can withstand fire temperatures of up to 1400 °C for at least 900 seconds. Consequently, the coating high-effectively prevented burn-through in aluminum plates for batteries enclosure and glass fabric reinforced epoxy resin, which otherwise were burned through in 135 and 173 seconds, respectively.

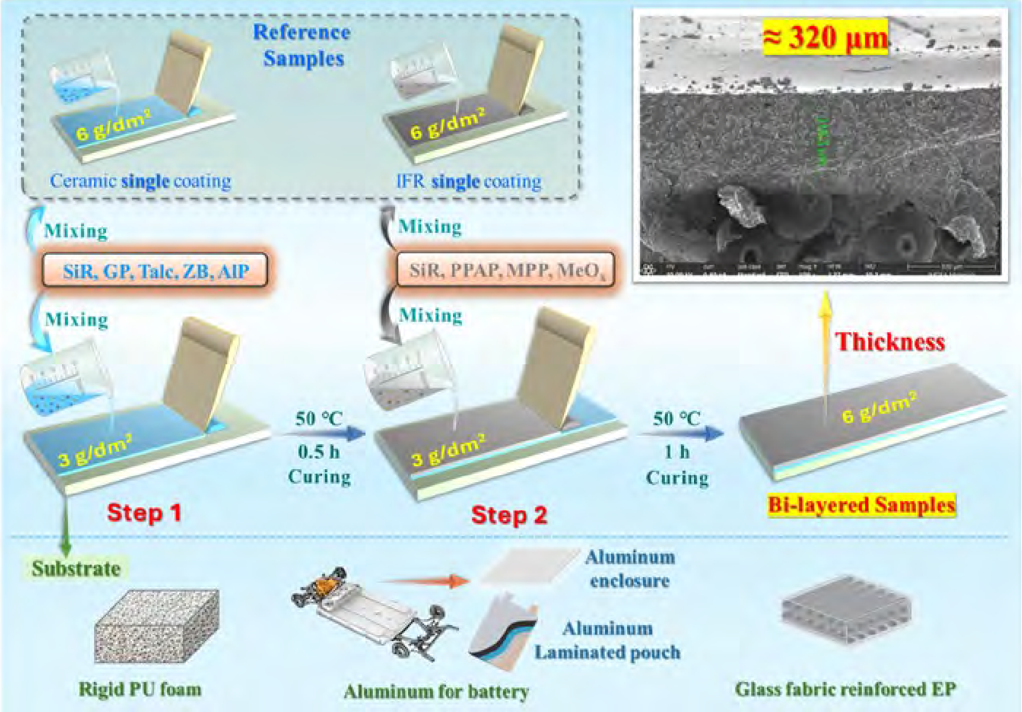


Figure.3. Designation of bi-layered strategy7

Meanwhile, the bi-layered coating suppressed the formation and decomposition of solid interface layer in lithium soft-package batteries, leading to prolonged electrochemical stability and fire safety. Additionally, the bi-layered coating with a fast response endowed rigid polyurethane foam with a self-extinguishing capability, preventing ignition even under direct exposure to strong fire of 1400 °C. Cone calorimeter tests further demonstrated that the coating significantly reduced the peak heat release rate and delayed both heat release and weight loss for polymes. This bi-layered structure successfully integrates the rapid response of the intumescent layer with the ultra-high thermal stability and low thermal conductivity of the ceramic layer. Shortly, our work offers new insights into the design and development of thin, high-performance, and multi-application flame-retardant coatings[7].

Acknowledgement

We acknowledge the financial support of this work by BIOFIRESAFE (PID2020-117274RB-I00) funded by MINECO, Spain.

References

1. https://www.cargroup.org/wp-content/uploads/2019/10/Technology-Roadmap_Materials-and-Manufacturing.pdf?utm_source
2. www.grandviewresearch.com
3. Tang W., Qian L.J., Prolongo S.G., Wang D.Y. Polym Degrad Stab. 2023, 208, 110265.
4. Tang W., Qian L.J., Prolongo S.G., Qiu Y., Wang D.Y. Chem Eng J. 2024, 495(1),153162.
5. Wang Q.S., Ping P., Zhao X.J., Chu G.Q., Sun J.H., Chen C.H. J Power Sour 2012, 208, 210.
6. Zhang Q.K., Zhang X.Q., H. Yuan, Huang J.Q. Small, 2021, 1, 2100058.
7. Tang W., Chen Q., Li J.X., Ao X., Liu Y.H., Qian L.J., Prolongo S.G., Qiu Y., Wang D.Y. Nano-Micro Letters. 2025, 17,231.

PLASMA MEETS POLYMERS: ADVANCED SURFACE ENGINEERING OF PLASTICS

Tomáš HOMOLA*, **Jakub KELAR, Richard KRUMPOLEC, Mirko ČERNÁK and Dušan KOVÁČIK**

Department of Plasma Physics and Technology, CEPLANT - R&D Centre for Plasma and Nanotechnology Surface Modifications, Faculty of Science, Masaryk University, Kotlářská 267/2, 602 00 Brno, Czech Republic
email: tomas.homola@mail.muni.cz

In recent years, plasma surface engineering has emerged as a powerful approach for modifying polymer surfaces, enabling novel functionalities in fields ranging from microfluidics and packaging to biomedical applications. This work presents the capabilities of Diffuse Coplanar Surface Barrier Discharge (DCSBD) — a unique atmospheric pressure plasma technology—as an effective tool for the surface treatment of diverse polymer substrates, including PET, PC, PMMA, PES, PEN, and PS. DCSBD (**Fig. 1**) is introduced here as a high-power density, filament-free, and industrially scalable plasma source capable of precise surface activation without altering the bulk properties of treated materials[1-3].

DCSBD operates in ambient air, eliminating the need for costly vacuum systems and working gases. Its planar geometry and high power density (up to 100 W.cm^{-3}) enable rapid, uniform treatment over large areas and compatibility with roll-to-roll systems, making it ideal for continuous industrial applications[4-7]. During plasma exposure, DCSBD introduces oxygen-containing polar functional groups (e.g., –OH, C=O, O–C=O) onto the polymer surface. This occurs via interaction with energetic species such as electrons, radicals, and UV photons, leading to significant enhancement of surface wettability and adhesion properties[7,8].

The primary mechanism of plasma polymer modification through DCSBD includes both functionalization and mild etching. Functionalization enhances surface energy by grafting oxygen-rich groups, while etching contributes to increased surface roughness. In some cases, plasma-induced degradation leads to the formation of low molecular weight oxidized materials (LMWOM), which are water-soluble and removable, further modifying surface topography[7].

Across various polymer types, DCSBD demonstrated significant improvements in surface properties. For example:

- PS and PES foils exhibited a reduction in water contact angle (WCA) from 83° and 76° to 26° and 32°, respectively, after just 0.5 s of DCSBD treatment.
- PC surfaces saw their WCA drop from 81.5° to 37.9°, accompanied by an increase in total surface energy from 44.3 to ~59 mJ·m^{-2} within seconds.
- PMMA surfaces showed durable hydrophilization and incorporation of polar groups across air, nitrogen, and oxygen environments, with stable wettability over 3–5 days.
- PET and PEN films also achieved rapid activation, with PET WCA reduced from 78.4° to 40.1°, and PEN from 79° to 20° depending on treatment conditions.

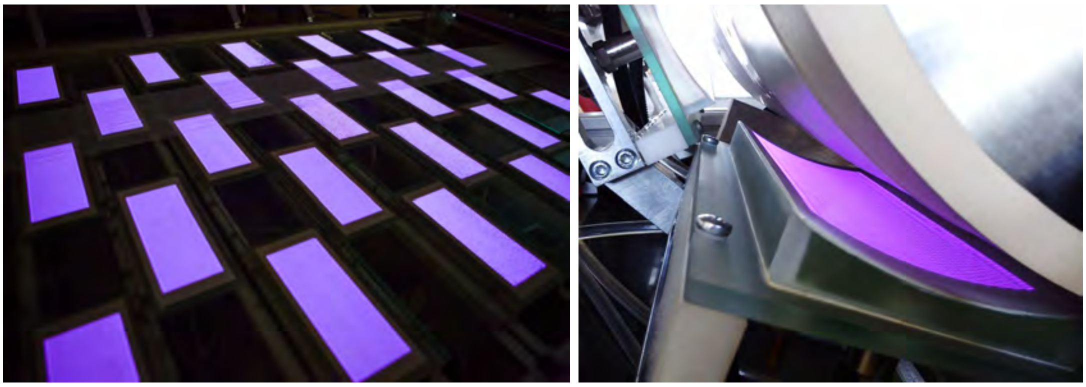


Figure 1. (left) Array of 25 flat DCSBD plasma panels (8 × 20 cm) used in roll-to-roll (R2R) treatment of nonwoven fabric; (right) Concavely curved DCSBD plasma unit designed for the R2R treatment of polymer foil.

These treatments are confirmed by X-ray photoelectron spectroscopy (XPS), which consistently shows elevated oxygen content and the formation of polar groups post-treatment, and by atomic force microscopy (AFM), which evidences nanometric roughening without significant damage.

In conclusion, DCSBD plasma technology represents a versatile, high-performance approach to the surface functionalization of polymeric materials. Its unique characteristics — ambient air operation, high power density, and compatibility with roll-to-roll systems — make it not only suitable for laboratory-scale research but exceptionally well-aligned with the demands of industrial manufacturing. More broadly, plasma technologies are becoming essential tools in the polymer industry. They enable environmentally friendly, solvent-free modification of polymer surfaces without compromising bulk properties. This is particularly crucial in applications requiring enhanced adhesion, wettability, or biocompatibility — such as in food packaging, automotive components, microfluidic diagnostics, membrane engineering, and biomedical devices. Plasma treatment can tailor the interfacial chemistry of inert, low-energy polymer surfaces, turning them into highly functional substrates suitable for coating, printing, or bonding.

Among available plasma systems, DCSBD stands out for its scalability and gentle yet effective interaction with sensitive polymer materials. The treatment is fast (typically < 10 s), uniform, and adaptable to various polymer chemistries and morphologies. It enables controlled introduction of polar functionalities, surface nanostructuring, and removal of weak boundary layers or contaminants — all in a dry, reagent-free process.

Given the growing industrial demand for clean, rapid, and adaptable surface engineering methods, plasma-based technologies — and particularly DCSBD — are poised to become a cornerstone of next-generation polymer processing and functionalization.

Acknowledgement

This work was supported by the projects LM2023039 funded by the Ministry of Education, Youth and Sports of the Czech Republic.

References

1. T. Homola, J. Matoušek, M. Kormunda, L.Y.L. Wu, M. Černák, Plasma treatment of glass surfaces using diffuse coplanar surface barrier discharge in ambient air, Plasma Chemistry and Plasma Processing 33 (2013) 881–894. https://doi.org/10.1007/s11090-013-9467-3.
2. T. Homola, L.Y.L. Wu, M. Černák, Atmospheric Plasma Surface Activation of Poly(Ethylene Terephthalate) Film for Roll-To-Roll Application of Transparent Conductive Coating, J Adhes 90 (2014) 296–309. https://doi.org/10.1080/00218464.2013.794110.
3. T. Homola, J. Kelar, M. Černák, D. Kováčik, Large-area open air plasma sources for roll-to-roll manufacture: High-power density surface plasma generated by diffuse coplanar surface barrier discharge, Vakuum in Forschung Und Praxis 34 (2022) 21–25. https://doi.org/10.1002/vipr.202200785.
4. J. Kelar, M. Shekargoftar, R. Krumpolec, T. Homola, Activation of polycarbonate (PC) surfaces by atmospheric pressure plasma in ambient air, Polym Test 67 (2018) 428–434.
5. M. Shekargoftar, J. Kelar, R. Krumpolec, J. Jurmanová, T. Homola, A Comparison of the Effects of Ambient Air Plasma Generated by Volume and by Coplanar DBDs on the Surfaces of PP / Al / PET Laminated Foil, IEEE Transactions on Plasma Science 46 (2018) 3653–3661. https://doi.org/10.1109/TPS.2018.2861085.
6. T. Homola, J. Matoušek, B. Hergelová, M. Kormunda, L.Y.L. Wu, M. Černák, Activation of poly(ethylene terephthalate) surfaces by atmospheric pressure plasma, Polym Degrad Stab 97 (2012) 886–892. https://doi.org/10.1016/j.polymdegradstab.2012.08.001.
7. J. Vida, M. Ilčíková, R. Přibyl, T. Homola, Rapid Atmospheric Pressure Ambient Air Plasma Functionalization of Poly(styrene) and Poly(ethersulfone) Foils, Plasma Chemistry and Plasma Processing 41 (2021) 841–854. https://doi.org/10.1007/s11090-021-10155-w.
8. T. Homola, J. Matoušek, B. Hergelová, M. Kormunda, L.Y.L.L.Y.L. Wu, M. Černák, Activation of poly(methyl methacrylate) surfaces by atmospheric pressure plasma, Polym Degrad Stab 97 (2012) 886–892. https://doi.org/10.1016/j.polymdegradstab.2012.08.001.

POSSIBILITIES OF USING LOW-TEMPERATURE PLASMA IN THE TREATMENT OF HERITAGE OBJECTS MADE OF PLASTICS

Radko TIŇO*, **Dominika SMATANOVÁ and Katarína VIZÁROVÁ**

Slovak University of Technology, Faculty of Chemical and Food Technology, Radlinského 9, 812 37 Bratislava, Slovakia
e-mail: address:radko.tino@stuba.sk

Plastics have become an integral part of art – the first documented use of plastics as an art material dates back to the pioneering work of Naum Gaba in the early 20th century. However, today's modern collections face conservation challenges related to the wide range of synthetic polymers used in them. These include various arts and crafts items such as furniture parts, jewelry, walking stick headbands and spectacle frames. Plastics have most often been used to imitate more expensive materials of natural origin such as tortoise shells, amber, ivory and coral. The issue of conserving plastics in art collections has been ignored until recently, as plastics have long been considered durable materials due to widespread misconceptions. However, it has eventually become clear that plastic objects are not very durable and are easily affected by environmental influences. However, the long-term behavior of these materials was not sufficiently known when they were first introduced to the market. Due to the intrinsic instability of the materials and suboptimal preparation, many works of art made from plastics tend to deteriorate at an alarming rate. This has been demonstrated by the observation of the rapid degradation of some of the earliest plastics, such as cellulose nitrates and acetates, polyvinyl chloride (PVC) and the first polyurethanes (PUR), which were identified as problematic materials in art collections. The increased awareness of the need for systematic conservation of such materials has led to the emergence of a new field of research aimed at filling the gaps in knowledge, practices and methodologies for the conservation of plastics in contemporary art collections[1-6].

In recent decades, there have been specific works of art that are characterized by the presence of synthetic materials. In the case of many artifacts, the author has reached for the entire range of materials with different chemical composition, structure and aging process. In the collections of modern and contemporary art of the Slovak National Gallery (SNG) and in other galleries of the Slovak Republic, more than 9,000 objects containing plastics have been registered in the CEDVU Database since 1960. The above quantitative expression confirms the need to address the protection of this group of artistic artifacts of the cultural heritage of the Slovak Republic. In addition, representatives of Slovak contemporary art, such as Jozef Jankovič (Fig. 1), Stano Filko and others, are internationally recognized authorities and the lack of knowledge necessary for the protection and conservation of their works will lead to irreparable losses.

Since plastic has accompanied everyday life since the beginning of the 20th century and contemporary art since the 1960s, polymeric materials are present in almost all art collections around the world, and museums and galleries own increasingly large collections of such objects. But every new discovery has its weaknesses, and plastics are no exception, their main disadvantage being their permanence, or rather their instability. At the same time, these current materials are still less studied and the mechanisms of their chemical decomposition have not yet been fully elucidated[7-9].

Since the end of the 20th century, several museums around the world have begun conducting material surveys aimed at identifying and quantifying plastics in their collections. Surveys have provided information on the presence of plastics, the degree and type of their damage[10-15]. Among all, four plastics are considered more susceptible to degradation: cellulose nitrate, cellulose acetate, plasticized PVC and polyurethane foam.

In recent years, the need for detailed research and understanding of the aging mechanisms of plastics has become increasingly apparent. The degradation of synthetic and semi-synthetic polymers is caused primarily by their chemical structure and reactivity, as well as by the influence of external factors (UV radiation, temperature, relative humidity, pollutants)[16-22]. The severity of individual degradation factors is relative and depends on the type of polymer, additives and processing conditions. The categorization of the causes of damage and degradation of plastics is given as the action of physical, chemical and biological factors:

- damage due to previous handling, changes in appearance and mechanical properties due to climatic conditions during storage and migration of additives in plastics;
- reactions of plastics with oxygen, ozone, water, metals and reactions initiated by radiation, especially visible light or heat. Furthermore, the use of chemical additives (in the past) unstable during aging. These factors provide sufficient energy and a suitable environment to break selected chemical bonds present in polymers and additives. In general, degradation leads to a decrease in molecular weight and the formation of new chemical structures;
- the conservation literature states that synthetic polymers are not susceptible to biological degradation. However, residues of oil, proteins and cellulose in contact with polymers can support the growth of bacteria and fungi [21-22]. Some synthetic polymers can be attacked by microorganisms. Acrylic polymers are subject to biodegradation[23]. The results indicate that fungi grow readily on poly(vinyl acetate) and less readily on acrylic polymers. Fungal attack has also been observed on a cellulose nitrate object from the 1960s [24].

Damage is also caused by improper handling and inappropriate conservation interventions.

Current conservation strategies prefer the prevention and elimination of impacts that could contribute to the degradation and depreciation of the artifact. Plastic as part of an artefact is stored in a repository for most of its existence, rather than on display. Therefore, a good conservation strategy should be based on appropriate storage conditions. However, there are no international standards for the storage of plastics in repositories, and museums and galleries therefore apply those used for works of art on paper and other fragile organic materials: a stable relative humidity maintained at around 50%, a temperature of 18°C - 20°C, light levels that are often zero and a maximum of 50–100 lux, and the complete elimination of ultraviolet radiation. Macroclimates are therefore a compromise between the average requirements for all materials in a given location and the available resources. Although microclimates tailored to each type of plastic would be optimal, the cost often precludes them. This problem is partly solved by the use of absorbers, which partially slow down the degradation of plastics. The most commonly used are oxygen absorbers, activated carbon, silica gel and zeolites.

The conservation of plastics today is inherently less safe than that of other materials encountered as artifacts. The process of conserving plastics requires an understanding of the composition of the plastic and an understanding of the possible methods of plastic restoration and their limitations, as well as a full understanding of preventive care for the object after conservation intervention. It is also important to note that the technologies for their conservation are relatively new and have not yet been sufficiently tested. For most traditional materials from cultural heritage artifacts, such as stone, wood, bone, ceramics, glass, metal, oil paints and paper, the technologies used to treat them have been developed for a long time. However, plastics have recently appeared on the scene and their wide range and preparation methods complicate the methods of their conservation. Conservation methods focus on removing dirt and surface damage, i.e. manifestations of mechanical degradation such as various fractures, scratches, deformations and erosion of surfaces. These lead to reduced functionality, aesthetic changes and even complete deterioration of plastics. In addition, in places of mechanical degradation, there may be increased trapping of moisture, dust and other contaminants, which can subsequently lead to chemical degradation.

Among the conservation interventions, cleaning processes are among the most important. Cleaning ensures the removal of mechanical, chemical and biological impurities. In terms of effect (stabilization or degradation), interventions that act on plastics with a physical and/or chemical effect have the greatest impact. In this context, it is necessary to focus attention mainly on mechanical cleaning operations, which, if applied incorrectly, can disrupt (partially or completely) the degraded surface of the plastic. The process of cleaning plastics is carried out in practice using solvents or modern technologies. Cleaning can still be done using water or solvents, but solvents carry a risk of physical damage to object[8]. The Nanorestart project (http://www.nanorestart.eu/) was dedicated to the use of the latest knowledge in the field of nanotechnology for new cleaning and conservation procedures for objects of contemporary art.

Sterilization is no less important. In Slovakia, ethylene oxide sterilization is used as a sterilization method in collection institutions. However, great care must be taken with plastics, as undesirable interactions may occur between the sterilized plastic-containing object and the sterilization medium (EtO). Ethylene oxide gas (EtO) is often used to sterilize materials that are otherwise too sensitive to sterilization by heat or radiation. Many plastics fall into this category, and EtO sterilization is often used for single-use medical devices made of plastic or for devices that are used for a short period of time. However, in this case, it is assumed that these devices are not intended to have a long service life that could be affected by reaction with EtO. After sterilization, some EtO may remain adsorbed on the surface of the object or dissolved in its structural materials. These residues will dissipate over time at a rate that depends on the type of material, its porosity, the size of the object, the degree of ventilation, and other factors. During sterilization, some of the EtO may react with water vapor to form ethylene glycol (EG) or with chlorinated compounds to form ethylene chlorohydrin (ECH), which may also remain on the surface or in the structure of the material. The effect of EtO was investigated in this context on a PEG material containing tyrosine-derived polycarbonates. It was found that the conditions of the EtO sterilization cycle, even those considered "mild", damage fragile structures [42], which in this context can also be expected in degraded polymers that are part of modern heritage objects.

One of the methods potentially suitable for cleaning and sterilizing plastics is the application of low-temperature plasma. NTP plasma technologies are currently the most effective alternative to EtO for low-temperature sterilization of thermosensitive materials. Materials and devices that cannot withstand high temperatures and humidity are often sterilized with a hydrogen peroxide gas plasma (generating essentially oxygen reactive species). This method is compatible with most materials and applications. Cold plasma is a partially ionized gas containing ions, electrons, UV and VUV photons, and neutral reactive species such as radicals and excited molecules. In practice, NTP plasma is already commonly used to modify the surface properties of several types of plastics (e.g. activation or modification of plastic surfaces before printing). Low-temperature plasma (NTP) generating reactive oxygen and nitrogen species (RNOS) has proven to be effective in terms of biological decontamination of heritage objects made of natural polymers [25-26]. In general, this method is suitable and effective for disinfection/decontamination of solid surfaces, which can be documented by a large amount of relevant literature. In relation to the decontamination of plastics, low-temperature pulsed corona plasma has been used to sterilize PET bottles[27]. In this context, however, it is necessary to investigate the degradation effects of reactive particles on aged plastics found in collection objects. Eliminating the degradation effects of NTP is a challenge that requires attention before deploying this technology among the conservation methods intended for plastics.

„Plasma cleaning"in general is the removal of impurities and contaminants from surfaces through the use of an energetic plasma created from gaseous species. Gases such as argon and oxygen, as well as mixtures such as air and hydrogen/nitrogen, are used. Plasma is created by using high-frequency voltages (typically kHz to >MHz) to ionize the low-pressure gas (typically around 1/1000 atmospheric pressure), although atmospheric pressure plasmas are now also common[28]. Plasma cleaner exposes the surface to a gas plasma discharge, gently and thoroughly scrubbing the surface. Plasma cleaning can remove non-visible oil films, microscopic rust, dust or other contaminants that typically form on surfaces as a result of handling, exposure or previous manufacturing or cleaning processes; additionally, plasma cleaning does not leave a surface residue. A plasma cleaner can treat the wide variety of materials - including plastics, natural materials, metals, and ceramics - as well as objects and materials with complex surface geometries. By producing high-frequency electric discharges, plasma generates ionized gas that can modify the surface properties of the material it is in contact with. Plasma treatment is a versatile and powerful technique commonly used in many industries for materials such as plastics, textiles, glass, and metals. Plasma is also used for the sterilization of a number of products in the food and pharmaceutical industries.

In the plasma discharge can often be found activated reactive species that include atoms, molecules, ions, electrons, free radicals, metastables, and photons in the short wave ultraviolet (vacuum UV, or VUV for short) range. This 'plasma soup', which incidentally is around room temperature, then interacts with any surface material placed into the plasma. The energy of plasma electrons and ions is sufficient to ionize neutral atoms, break molecules apart to form reactive radical species, generate excited states in atoms or molecules, and locally heat the surface. Depending on the process gases and parameters, plasmas are capable of both mechanical work, through the ablative effect of the kinetic transfer of electrons and ions with the surface, and chemical work, through the interaction of reactive radical species with the surface. Plasma processing is not one process but a “field of opportunities” that can be used to modify a surface through several mechanisms: ablation, activation, deposition, cross-linking and grafting. For cleaning purposes, however, are utilizable ablation, activation, crosslinking and sterilization.

Sterilization is an act or process, physical or chemical that destroys or eliminates all forms of life, especially microorganisms. Plasma sterilization operates differently because of its specific active agents, which are ultraviolet (UV) photons and radicals (atoms or assembly of atoms with unpaired electrons, therefore chemically reactive, e.g., O and OH, respectively)[29]. An advantage of the plasma method is the possibility, under appropriate conditions, of achieving such a process at relatively low temperatures (≤50 °C), preserving the integrity of polymer-based instruments, which cannot be subjected to autoclaves and ovens[29-30]. It has been shown that many pathogens can be destroyed by employing non-equilibrium plasma discharges[31] and the nature of plasma interactions with micro-organisms or biomolecules has been extensively studied in the past few years. However, decontamination of organic surfaces like paper, leather, parchment, wood or plastics is quite complicated because materials of these objects are in principle sensitive to both radiation and active particles formed by plasma.

Ablation is the removal of the surface by evaporation of surface material. Plasma ablation involves the mechanical removal of surface contaminants by energetic electron and ion bombardment. Surface contamination layers (e.g. cutting oils, skin oils, mold releases, etc.) are typically comprised of weak C-H bonds. Ablation affects only the contaminant layers and the outermost molecular layers of the substrate material. Argon is often used for ablation due to its high ablation efficiency and no chemical reactivity with the surface material.

Activation acts as substituting atoms in the polymer molecule with chemical groups from plasma. Plasma surface activation involves the creation of surface chemical functional groups using plasma gases - such as oxygen, hydrogen, nitrogen and ammonia - which dissociate and react with the surface. Such activation alters the chemical activity and characteristics of the surface, such as wetting and adhesion, yielding greatly enhanced adhesive strength and permanency.

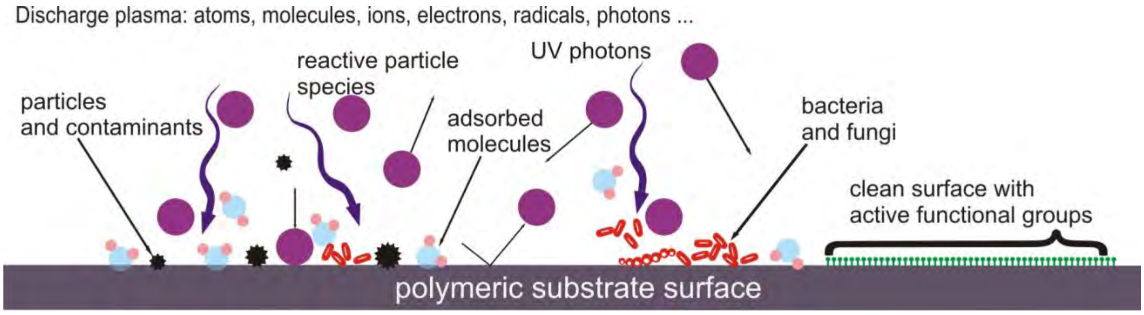


Fig. 1 Schematic description of actions in plasma discharge during the cleaning processing of polymeric materials

Process conditions play an important role in the effect of plasma treatment. Parameters like amount of supplied energy ($W.cm^{-2}$), frequency (Hz), modification time (min.), type of atmosphere (air, N2, O2, CO2, Ar, etc.), gas flow, humidity, pressure and ultimately the type of substrate treated affect the final result of the plasma treatment. Among the endpoints they are shown as the most important the amount of energy supplied, type of substrate, type of atmosphere, and the duration of exposure of the material to plasma.

In the case of atmospheric plasma, which is the preferred choice, it is necessary to narrow down the possible effects due to the higher pressure compared to vacuum or low-pressure plasmas. Under atmospheric conditions, the effects related to the generation and action of reactive RNOS species will dominate. In contrast, the effects related to UV and VUV photons will be minor, since these particles interact already in the plasma discharge and will modify the surface of the treated substrate only to a limited extent,

In our study, we exposed PVC-P (40 % w/w) samples of historic formula (plasticizer dioctyl phthalate, heat stabilizer) to ADRE plasma treatment. The aim of this study was to determine the degradation effects of LTP and its suitability for further sterilization. As plasma gas, air with a flowrate of 6 l/min and relative humidity of 60-70 % was used. Twenty samples of PVC-P were exposed to ADRE plasma at different conditions according to Design of Experiment. Studied variable parameters were time of exposition, energy and frequency. While border values of energy and frequency were chosen as limit values that was possible to set up, the exposure time (1 – 15 minutes) was selected according to previous experiment with ADRE plasma. In those experiment[32], exposure time 10 minutes was sufficient for deactivation of filamentous fungi on paper material. For purpose of studying degradative effect of ADRE LTP on PVC-P exposure time was selected as broader range (1 - 15 minutes). After plasma treatment, there were no visual changes on PVC-P surface, which was confirmed by optical microscopy. When studying morphological changes with SEM, it was observed that pristine PVC-P surface is not homogenous and there are already microcracks presented. ATR-FTIR analysis did not confirm significant degradation by dehydrochlorination, although there seems to be decreased intensity and area of peak characteristic for C=O band from stabilizers. Values obtained for all plasma-treated samples are lower than for pristine, and although they are not significant, they are outside of margin error range. It was also possible to observe the formation of new OH bands (although again not significant) and there were noticeable changes of bands characteristic for heat stabilizers, suggesting that stabilizing reaction occurred. Raman spectroscopy was applied for determination of formation of polyene sequences, but when Raman spectra for pristine and plasma-treated PVC-P were compared, no formation of new peaks was confirmed. All these data were statistically evaluated, however there was found no significance of any of examined parameters. Although obtained results are promising for LTP application in protection of plastic materials, further experiments and evaluations are needed, including study of mechanical properties, thermal and light accelerated ageing and exposition of aged samples to LTP and re-exposition of already treated samples to fully characterize suitability of ADRE plasma for PVC-P treatment in cultural heritage.

Sterilization of softened PVC of historical recipe using ADRE plasma

Procedure:

The samples were prepared on a hot press - during cooling they were cut into 5x5 cm dimensions

and placed in sterile PS dishes of 1, 2 and 3 pcs. Subsequently, random samples were selected from the contamination monitoring of the samples prepared in this way. The selected samples were imprinted into PS Petri dishes with sterile MAE agar. The medium with the imprint was allowed to incubate for about 5 days. For the accuracy of the next experiment, we decided to sterilize the samples with a 70% ethanol solution additionally: the samples were placed in the solution for 15 min and rinsed with sterile deionized water and dried in a sterile glass Petri dish with sterile filter paper to collect water. Such samples were left to dry for 24 h.

Inoculation of samples

PS Petri dishes with MAE agar, on which Aspergillus niger was cultivated, were filled with 1 ml of sterile water without surfactants. Conidia of the filamentous fungus were released using a plastic

"hockey stick." The suspension was pipetted onto a flannel. This procedure was repeated once more and the entire suspension was mixed in a vortex.

The subsequent dark-colored suspension was pipetted into a syringe with a filter with non-woven fabric to separate the rest of the agar and mycelia. The resulting suspension was standardized: the concentration of spores was calculated using a Burker grid. The concentration was 6.7 x 10 6 k/ml. The viability of the spores of the suspension was also monitored - the concentration of the so-called colony-forming units was determined. The suspension was also diluted to a 6.7 x 10 5 cfu/ml concentration. Such a diluted suspension was subsequently pipetted 100 µl onto the surface of the PVC-P samples in an approximately 4x4 drop arrangement, so that the number was 6.7x10 4 conidia.

Such samples, 18 pieces, placed in sterile PS dishes of 3 pieces each, were left to dry overnight (they had to be put into a drying oven at t=35°C, since they did not dry overnight in closed PVC-P dishes). They were then exposed to low-temperature plasma in triplicates.

Samples were always exposed to plasma in 3 pieces at a time. At the beginning, the plasma chamber was separately plasma treated to sterilize the surface. Then, the samples were transferred to the chamber using sterile tweezers (in flame), and were also removed using sterile tweezers into new sterile PS dishes for transfer to the microbiology laboratory.

These plasma-exposed samples were then transferred to a small sterile PS Petri dish with 1 ml of sterile water solution with Tween 80 to wash away spores from the PVC surface and were shaken in these small dishes for 1 h.

After completion of shaking, 100 µl of concentrated solution (10 0) was pipetted and transferred to Petri dishes with MAE agar and spread over the surface with a stick, another 100 µl was diluted to concentrations of 10 -1 and 10 -2 µl, which were also applied to Petri dishes (total number 78).

After approximately 5 days, colony-forming units were counted and a devitalization curve was constructed from the calculated average.

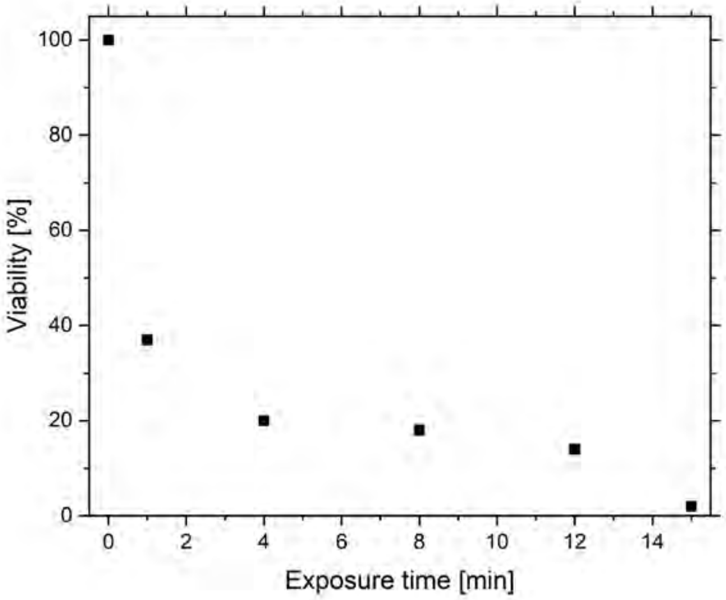


Fig.2 Devitalization curve of the effectiveness of low-temperature ADRE plasma when monitoring the exposure time for changes in the number of colony-forming units of the microorganism *Aspergillus nigger*.

As is evident, the number of viable colony-forming units decreases with the extension of the exposure of the microorganism to NTP. After 15 minutes of exposure, at a power of 0.67 J/s and a plasma source frequency of 1250 Hz, the number of viable colony-forming units decreased to 0. At the same time, ADRE plasma did not show any degradation effects on the surface or structure of the tested PVC-P under the given conditions.

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References

1. Curran, K., & Strlič, M. (2015). Polymers and volatiles: Using VOC analysis for the conservation of plastic and rubber objects. Studies in Conservation, 60(1), 1–14. https://doi.org/10.1179/2047058413Y.0000000125
2. Elsässer, C., Mayr, V., Montag, P., Angelin, E. M., Hilbig, H., Grosse, C. U., & Pamplona, M. (2023). Mock-Ups in Plastic Conservation Research: Processing and Aging of 3D Celluloid Specimens Simulating Historical Objects. Polymers, 15(4). https://doi.org/10.3390/polym15040852
3. Klisińska-Kopacz, A., Krupska-Wolas, P., Obarzanowski, M., Kłosowska-Klechowska, A., & del Hoyo Meléndez, J. (2022). Spectroscopic Characterization of Sensitive Museum Plastic-Based Objects BT - The Future of Heritage Science and Technologies (R. Furferi, R. Giorgi, K. Seymour, & A. Pelagotti, Eds.; pp. 107–118). Springer International Publishing.
4. La Nasa, J., Biale, G., Ferriani, B., Colombini, M. P., & Modugno, F. (2018). A pyrolysis approach for characterizing and assessing degradation of polyurethane foam in cultural heritage objects. Journal of Analytical and Applied Pyrolysis, 134, 562–572. https://doi.org/10.1016/j.jaap.2018.08.004
5. Picollo, M., Bartolozzi, G., Cucci, C., Galeotti, M., Marchiafava, V., & Pizzo, B. (2014). Comparative study of fourier transform infrared spectroscopy in transmission, attenuated total reflection, and total reflection modes for the analysis of plastics in the cultural heritage field. Applied Spectroscopy, 68(4), 389–397. https://doi.org/10.1366/13-07199
6. Shashoua, Y. (2016). Mesocycles in conserving plastics. Studies in Conservation, 61(sup2), 208–213. https://doi.org/10.1080/00393630.2016.1168074
7. Summary of POPART objectives, PopArt, dostupné online (november, 2020): http://popart-highlights.mnhn.fr/introduction/summary-of-popart-objectives/index.html
8. E. Then, V. Oakley: A survey of plastic objects at the V&A Museum. V&A Conser. J. 06, 11–14 (1993).
9. Y. Shashoua, C. Ward: Plastics: modern resins with ageing problems, in Proceedings of SSCR 2nd Resins Conference, Resins-Ancient and Modern, ed. by Wright M.M., J.H. (Scottish Society for Conservation and Restoration, Townsend Edinburgh, 33–37 (1995).

10. F. Waenting: Plastics in Art, A study from the conservation point of view, Michael Imhof Verlag, Petersberg 2008, ISBN 978-3-86568-405-9.
11. The Ghetty Conservation Institute. Conservation of plastics, Conservation perspectives, The GCI newsletter, vol.29, no. 1 (2014).
12. J. Rychlý, L. Rychlá: Stabilita plastov v múzeálnych artefaktoch, možnosti diagnostiky súčasného stavu, Konferencia CSTI 2018 – Premosťovanie disciplín a druhov dedičstva – efektívna ochrana dedičstva v 21.storočí, Conservation Science, Technology and Industrym FHCPT STU, SNM.
13. Damage atlas. Atlas of case studies presenting typical damages, PopArt dostupné online (október, 2020): http://popart-highlights.mnhn.fr/wp-content/uploads/3Collection survey/5_Damage_atlas /Damage_atlas.pdf.
14. G. Barabant: Degradation associated with some plastics found during surveys of three French collections, PopArt, dostupné online (október 2020): http://popart-highlights.mnhn.fr/wp-content/uploads/3_Collection_survey/3_Degradation_associated_with_some_plastics/3_4_DegradationAssociatedWithSomePlastics.pdf.
15. Care and identification of objects made from plastic, National park service, Conserv O Gram, No. 8/4, september 2010.
16. N.S. Allen, M. Edge: Fundamentals of Polymer Degradation and Stabilization (Springer, New York, 1992). (Ed. by S. Halim Hamid).
17. S. Halim Hamid: Handbook of Polymer Degradation, 2nd edn. (CRC Press, Boca Raton, 2000).
18. H.W. Moeller (ed.): Progress in Polymer Degradation and Stability Research (Nova Science Publishers, Hauppauge, 2008).
19. W. Schnabel: Polymer degradation: principles and practical applications. New York: MacMillan; 1982.
20. N. Grassie, G. Scott:m Polymer degradation and stabilisation. Cambridge: Cambridge University Press; 1988.
21. I. C. McNeill: Fundamental aspects of polymer degradation. In: Postprints of Polymers in Conservation Manchester 17–19 July 1991 (N. S. Allen, M. Edge and C. V. Horie, eds.), 14–31, Royal Society of Chemistry (1992).
22. S. Rivers , N. Umney: Conservation of furniture . Butterworth-Heinemann (2003).
23. F. Cappitelli, E. Zanardini, C. Sorlini: The biodegradation of synthetic resins used in conservation . Macromolecular Bioscience , 4 (2004), 399 – 406.
24. M. Silva: Investigating cellulose nitrate degradation caused by fungal attack. In: Postprints of The Future of the Twentieth Century–collecting, interpreting and conserving modern materials. Second Annual Conference Arts and Humanities Research Board Winchester 26–28 July 2005 (P. Garside, ed.), 72–76. Archetype Publications (2006). London.
25. K. Vizárová, B. Kaliňáková, R. Tiňo, I. Vajová, K. Čížová: Microbial decontamination of lignocellulosic materials with low-temperature atmospheric plasma ,Journal of Cultural Heritage
    Available online 5 November 2020, in press. DOI: 10.1016/j.culher.2020.09.016.
26. P. T. Jacobs, L. Szu-Min: Gas-Plasma Sterilization. ACS Symposium Series, Vol. 620 (1996). ISBN13: 9780841233775e. ISBN: 9780841215573. DOI: 10.1021/bk-1996-0620.ch017.
27. Masaoka et al.: Plasma Sterilization of PET Bottles by Pulsed Corona Discharge at Atmospheric Pressure. Biocontrol Science, Vol.12, No.2 (2007), 59-63.
28. Shun'ko, E.V., Belkin, V.V., 2007. Cleaning properties of atomic oxygen excited to metastable state 2s22p4(1SO), J.Appl.Phys.102:083304-1-14. Doi:10.1063/1.2794857
29. Moisan, M, et al. 2002. Plasma sterilization. Methods and mechanisms. Pure Appl. Chem. 2002, 74, 3, 349-358.
30. Moisan, M., Barbeau, J., Moreau, S., Pelletier, J.,Tabrizian, M., Yahia, L'H. 2001b. Int. J. Pharm. 226, 1–21
31. Rossi, F, Kylián, O., 2012. Sterilization and decontamination of surfaces by plasma discharges. Woodhead Publishing series in Biomaterials, p.117-150. doi: 10.1533/9780857096265.117
32. Tiňo R., Vizárová, K. & Krčm F (2019). Plasma surface cleaning of cultural heritage objects, in: Lazzara, G. & Fakhrullin, R. (eds.) Nanotechnologies and Nanomaterials for Diagnostic, Conservation and Restoration of Cultural Heritage, Elsevier, Amsterdam, pp. 239- 251.

HYDROPLASMA - EFFICIENT AND SUSTAINABLE CLEANING SOLUTION FOR THE HIGHEST CLEANLINESS REQUIREMENTS

Petr TICHÝ

Plasmatreat Austria GmbH, Pražská třída 901/145, 500 04 Hradec Králové, Czech Republic
email: petr.tichy@plasmatreat.com

HydroPlasma, developed by Plasmatreat, is an innovative surface treatment technology that combines the company's established Openair-Plasma® process with the chemical reactivity of ionized water. This environmentally sustainable method enables highly effective removal of both organic and inorganic contaminants—such as oils, salts, and residues—without the use of solvents or aggressive chemicals.

The process involves the direct injection of water into a plasma jet, generating a highly reactive cleaning medium. Applied through a precision nozzle and followed by an integrated Air-Knife system for immediate drying, HydroPlasma ensures a residue-free surface that is ready for subsequent processing.

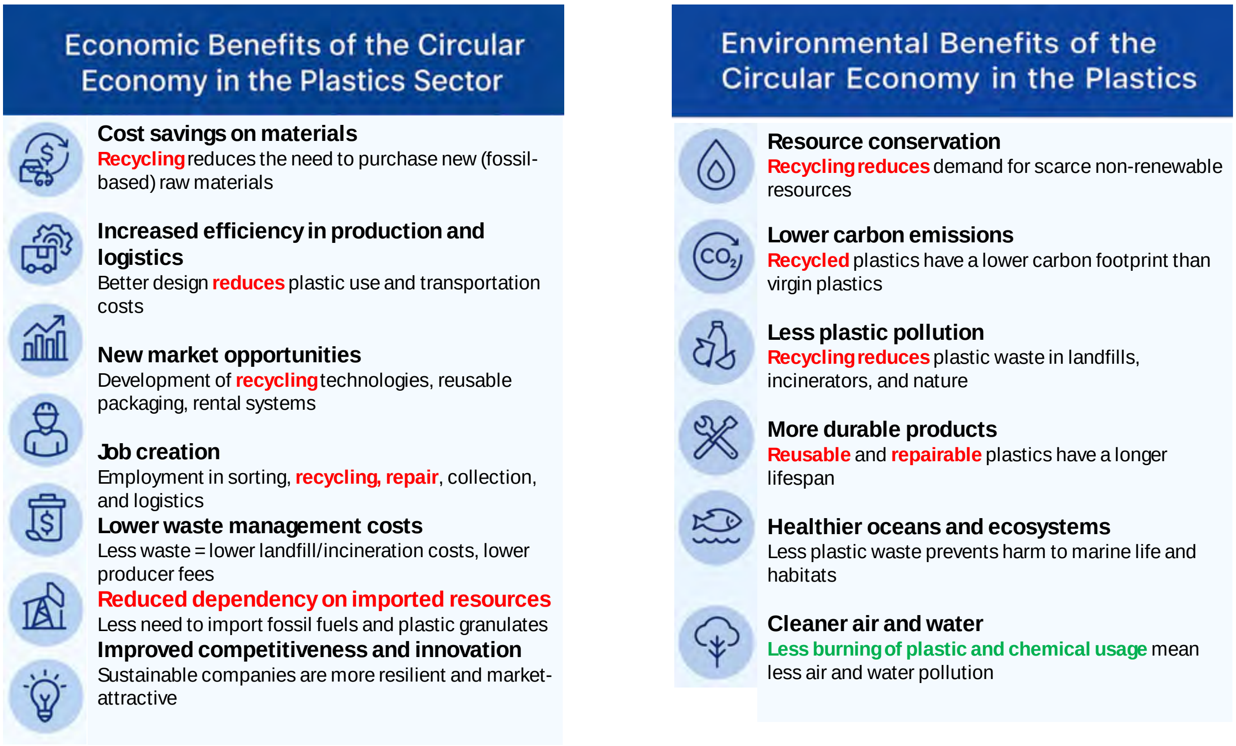
Designed for high-demand sectors such as automotive, electronics, aerospace, and medical technology, HydroPlasma is easily integrated into existing production environments. Its ability to clean sensitive materials without thermal damage makes it a highly versatile and forward-looking alternative to traditional wet-chemical cleaning methods.

PLASTICS AND BIOPLASTICS IN CIRCULAR ECONOMY

Pavel ALEXY*, **Zuzana KRAMÁROVÁ, Zuzana VANOVČANOVÁ, Ida VAŠKOVÁ, Roderik PLAVEC, Katarína TOMANOVÁ, Michal ĎURFINA, Sára VACH AGÓCSOVÁ, Mária MIKOLAJOVÁ, Mária FOGAŠOVÁ and Jozef FERANC**

Slovak University of Technology, Faculty of Chemical and Food Technology, Institute of Natural and Synthetic Polymers, dept. Plastics, Rubber and Fibers, Radlinského 9, 81 37 BratislavaAddress
email: pavol.alexy@stuba.sk

Plastics, as modern materials for the production of a wide range of products, also offer ecological benefits compared to traditional materials. However, in recent years, the negative impact of plastic waste on the environment has been increasingly emphasized, especially because plastics, as they were developed, are highly resistant to biological degradation. If they are not separated from the waste stream, they enter the environment and remain there for hundreds of years, taking up space in natural ecosystems. In addition, the vast majority of current plastics are produced from crude oil as a non-renewable fossil resource. With the rapid growth of plastic production, it is necessary to address both the impact of plastic waste on the ecology and with regard to raw materials and the economy of industrial production. As part of the transition from a linear to a circular economy, the European Union issued a fundamental strategic document in 2018 dedicated to the position of plastics in the circular economy, clearly defining in the introduction the economic and ecological advantages of plastics as such in the conditions of a circular economy (Fig. 1).



Economic Benefits of the Circular Economy in the Plastics Sector

Cost savings on materials
Recycling reduces the need to purchase new (fossil-based) raw materials

Increased efficiency in production and logistics
Better design reduces plastic use and transportation costs

New market opportunities
Development of recycling technologies, reusable packaging, rental systems

Job creation
Employment in sorting, recycling, repair, collection, and logistics

Lower waste management costs
Less waste = lower landfill/incineration costs, lower producer fees

Reduced dependency on imported resources
Less need to import fossil fuels and plastic granulates

Improved competitiveness and innovation
Sustainable companies are more resilient and market-attractive

Environmental Benefits of the Circular Economy in the Plastics

Resource conservation
Recycling reduces demand for scarce non-renewable resources

Lower carbon emissions
Recycled plastics have a lower carbon footprint than virgin plastics

Less plastic pollution
Recycling reduces plastic waste in landfills, incinerators, and nature

More durable products
Reusable and repairable plastics have a longer lifespan

Healthier oceans and ecosystems
Less plastic waste prevents harm to marine life and habitats

Cleaner air and water
Less burning of plastic and chemical usage mean less air and water pollution

Fig. 1. Basic economic and ecological benefits of plastics, defined by the Strategy for Plastics in a Circular Economy

As can be seen from the document and Fig. 1, the main emphasis is placed on recycling plastics. The recycling rate is currently at about 30% of the total plastic production of around 400 million tons per year (2024). If we look at the structure of the representation of individual plastics in annual production (Fig. 2), it can be seen that the most produced are polyolefins, followed by PVC, with these two classes of plastics accounting for almost 60% of the total plastic production.

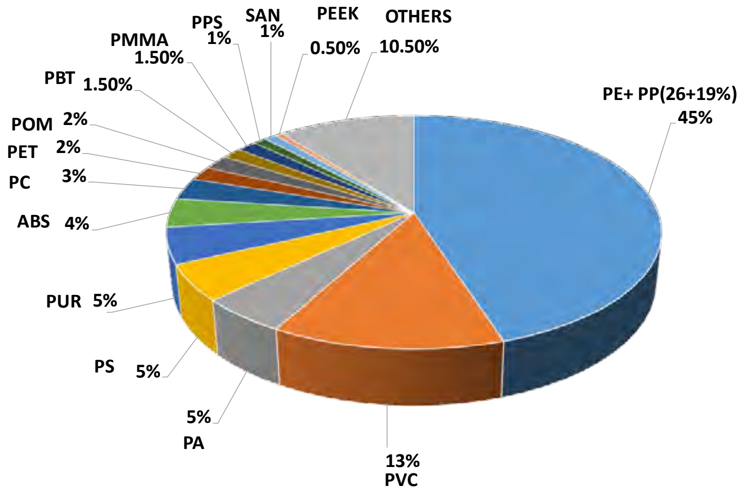


Fig. 2. Representation of individual plastics in total world production

On the other hand, when we look at recycling, PET is the most recycled plastics, despite the fact that its annual production is only 2% of the total volume of plastics, while out of the total 30% of recycled plastics, up to 16.5% is PET, followed by 12.06% of polyolefins, calculated purely on recycled material, PET represents 55% of recycled materials, while polyolefins 40.2% (Fig. 3).

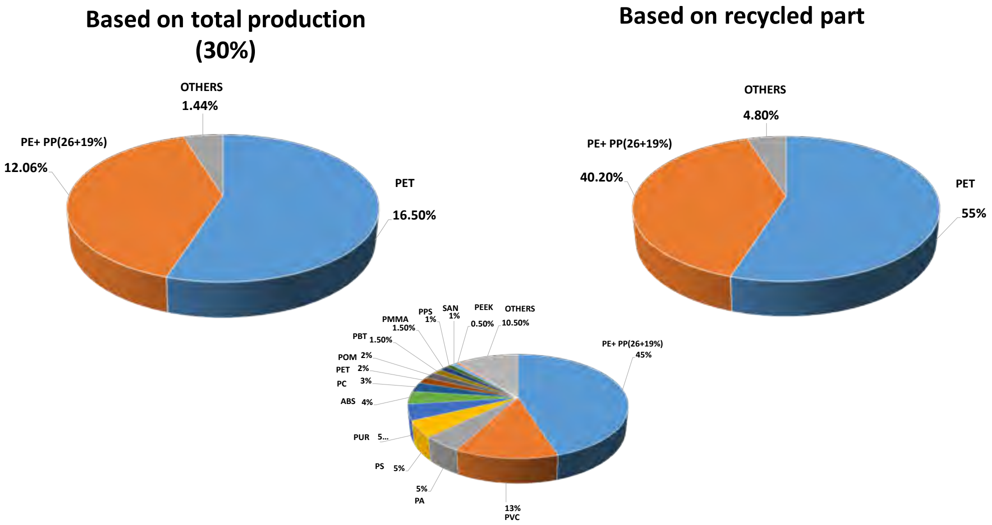


Fig. 3. The share of individual plastics in the recycle in absolute percentages from 30% of recycled plastics (top left), or in relative percentages calculated as 100% of the recycle (top right) compared to the production of plastics (bottom center).

The most plastic waste is generated in the packaging industry (Fig. 4), up to 42.9%, while if we look at the composition of plastics in packaging waste, the most represented, up to 40% are polyolefins, PET only 11% (Fig. 5)

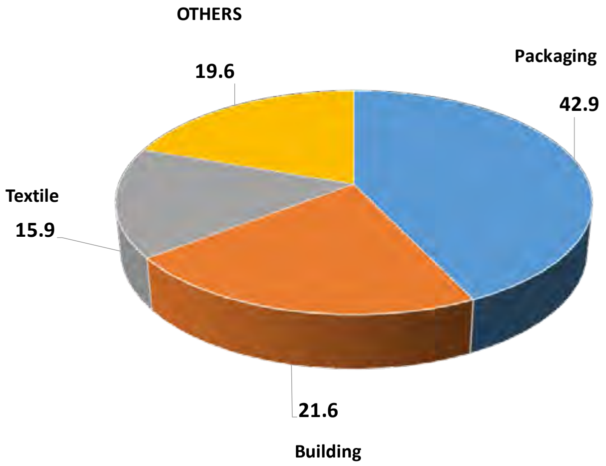


Fig. 4. Representation of individual segments in plastic waste

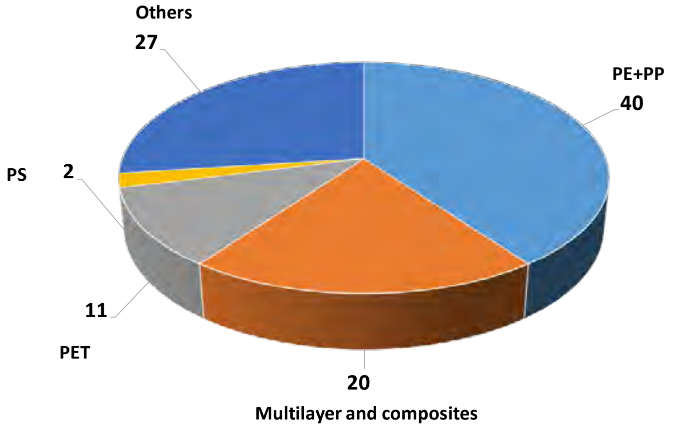


Fig. 5. Composition of plastic packaging waste

The above statistics clearly show that the focus of plastic recycling by type is not set optimally, since those plastics that are represented in the highest proportion of plastic waste (polyolefins) are recycled significantly less than plastics that are represented in the waste significantly less (e.g. PET). Based on these statistics and analyses, it is necessary to take measures to increase the efficiency of polyolefin recycling if the ecological impact of plastic waste on the environment should be significantly reduced.

Another problem with plastics in the circular economy is the fact that the strongly preferred recycling does not ensure complete circularity, which does not lead to complete renewal of resources and thus does not eliminate the exhaustibility of fossil raw materials and does not eliminate the negative impact of plastic waste on the environment, but only slows them down, as is evident from Fig. 6. Recycling (material and chemical) returns the material to the production process at the stage of plastic synthesis or processing but does not return plastic waste directly to the raw material source. After multiple recycling, non-recyclable waste is still generated, which can be utilized by energy recovery, which however produces fossil carbon dioxide and thus causes an ecological issue in form of global warming effect. It is therefore clear from what has already been stated that the currently preferred model of the circular economy is to slow down the degradation of the environment and slow down the extraction of finite, exhaustible sources of raw materials and therefore does not represent a final solution.

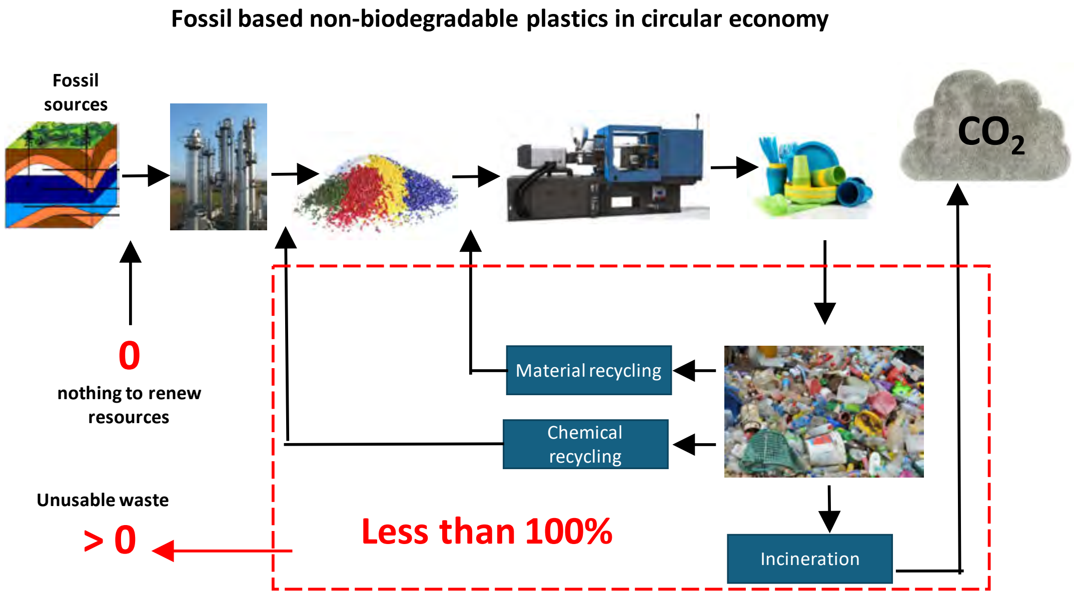


Fig. 6. The life cycle of plastics in a circular economy

A promising solution is the application of so-called bioplastics, of which there are 5 basic types on the market, as can be seen from Fig. 7.

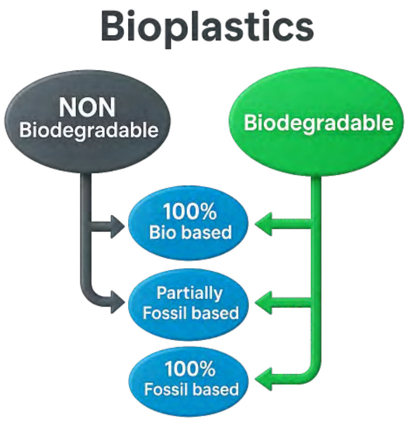


Fig. 7. Basic types of bioplastics

Of the above types of bioplastics, biodegradable bioplastics from 100% renewable raw materials are the most advantageous from the point of view of ecology and also the renewability of raw materials. Their life cycle is shown in Fig. 8.

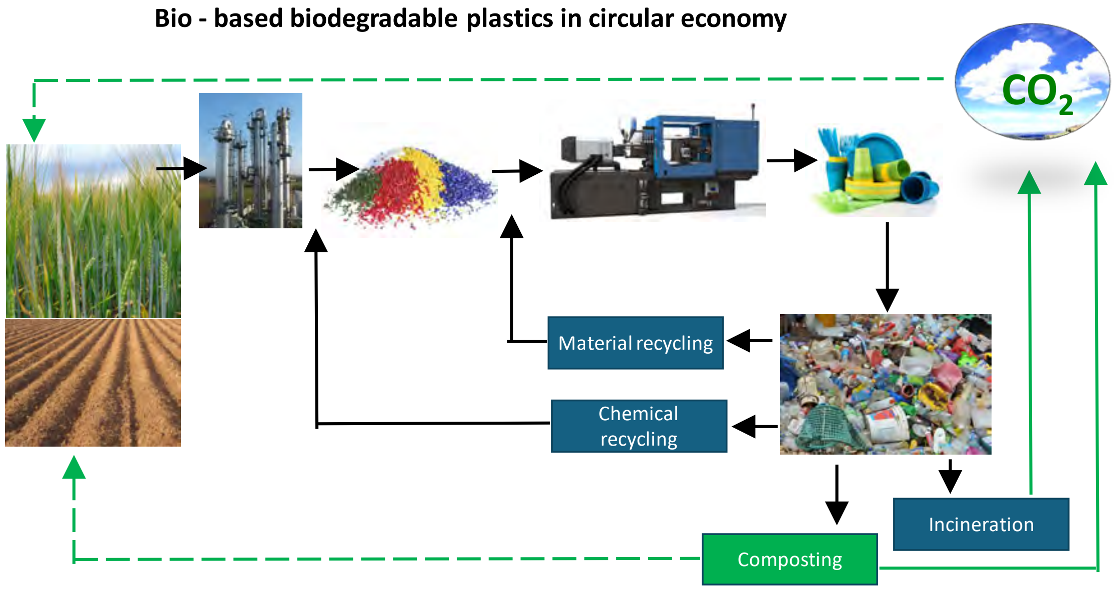


Fig. 8. Life cycle of biodegradable bio-based bioplastics in a circular economy

Bioplastics are currently the fastest growing segment in the field of plastics production, with global bioplastic production almost doubling since 2013. PLA is the most widely used, followed by bioPE and bioPP. The advantages and disadvantages of individual types of bioplastics, as well as Europe's position in this segment relative to the world, will be discussed in more detail at the conference during a lecture

Acknowledgement

Work was supported by grants: APVV-20-0256 Obalové systémy na báze biodegradovateľných polymérov z obnoviteľných zdrojov, APVV-20-0193 Materiálová recyklácia environmentálne prijateľných polymérnych materiálov získaných z obnoviteľných zdrojov, APVV-23-0221Termoplastické elastomérne zmesi na báze obnoviteľných a plne biodegradovateľných polymérov.

PLAN-C | THE PROJECT FOR MOVING PLASTICS AND MACHINE INDUSTRY TOWARDS CIRCULARITY | AT A GLANCE

Iris REINGRUBER

Business Upper Austria – OÖ Wirtschaftsagentur GmbH, Hafenstraße 47 – 51, 4020 Linz, Austria
email: iris.reingruber@biz-up.at

Moving **PL**astics and M**A**chine I**N**dustry towards **C**ircularity (**Plan-C**): Plastics are a versatile material omnipresent in modern life but not sufficiently recycled after first use. Goals of EU Circular Economy Action Plan i.e. 50% recycling rate for plastic packaging by 2025 pose the Danube Regions **-** from the East to the West **-** for significant challenges due to deficits in waste management systems and lack of industrial processing knowledge for the recirculation of secondary plastics materials.

14 project partners from Austria, Bosnia and Herzegovina, Czech Republic, Germany, Hungary, Moldova, Romania, Serbia, and Slovakia aim to boost the transformation along of the plastics value chain in the Danube Region towards circularity through the transnational cooperation of plastics processors and producers with the machine industry. Achieving this requires close collaboration to change mindsets, behaviors and business paradigms.

The goal is to enhance the competitiveness of key actors. By using technology transfer and a design thinking process, representatives of the plastics industry co-create circular plastic prototypes and those of the machine industry develop circular solutions for machine lifecycle phases. After each thematic strand has drafted a transnational strategy towards circularity it’s time to create joint scenarios of the future plastics value chain with the view of plastics processors **and** the view of machine producers.

Overall, at least 140 enterprises from the plastics and machine industries in the Danube Region are collaborating to create a vision for a transformed plastics value chain focused on circularity. This effort demonstrates the benefits of reusing and recycling plastics, as well as maintaining, redistributing, refurbishing, remanufacturing, and recycling machines.

Acknowledgement

Plan-C is an Interreg Danube Region Programme project co-funded by the European Union.

CIRCULAR CHEMISTRY AS THE KEY TO A CIRCULAR ECONOMY: TRANSFORMING PET WASTE INTO SUSTAINABLE MATERIALS

Andra-Cristina ENACHE*, Petrisor SAMOILA and Valeria HARABAGIU

"Petru Poni" Institute of Macromolecular Chemistry, 41A Grigore Ghica Voda Alley, Iasi 700487, Romania
email: enache.andra@icmpp.ro

Plastic pollution has become an urgent global challenge, with plastic production rising from 2 million metric tons in 1950 and projected to reach nearly 600 million metric tons by 2050. The packaging sector accounts for nearly 44% of global plastic production (as shown in Figure 1), with polyethylene terephthalate (PET) being a key material[1,2]. However, a significant portion of PET waste, particularly bottles, ends up in landfills or oceans, posing severe environmental threats. This highlights the pressing need for sustainable recycling solutions, especially within the framework of a circular economy.

The research explores the potential of glycolysis as a chemical recycling method for PET, which can be beneficial in the automotive industry. PET is commonly used in automotive components, including textiles, composites, and polyurethane foams. While mechanical recycling faces limitations such as material degradation and contamination, glycolysis offers a promising alternative by depolymerizing PET into reusable monomers like BHET, ensuring high material purity and the preservation of mechanical properties.

The focus is on heterogeneous catalytic glycolysis, noted for its quick reaction time (20-60 min), high monomer yields (>90%), cost-effectiveness, easy catalyst recovery, and improved durability. The use of efficient oxide-based catalysts for PET glycolysis is highlighted as a promising solution for large-scale industrial use. This makes glycolysis a viable solution for integrating recycled PET into automotive materials production without compromising performance.

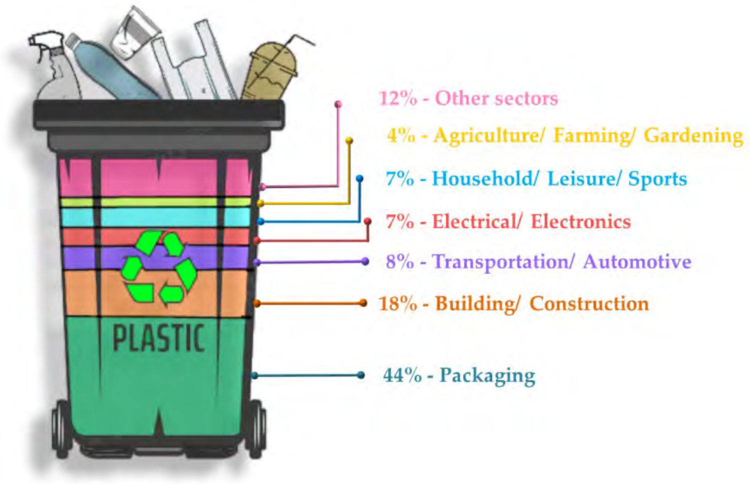


Figure 1. Distribution of the global plastics use in 2021 by sector of application[1,2].

Acknowledgement

The project Moving PLastics and mAchine iNdustry towards Circularity—acronym Plan-C (ID: DRP0200194), supported by the Interreg Danube Region Programme co-funded by the European Union is kindly acknowledged.

References

1. Enache, A.-C., Grecu, I., & Samoila, P.: Materials, *17(12)*, 2991 (2024).
2. Plastic Europe, Plastics—The Fast Facts. 2022. Available online: https://plasticseurope.org/knowledge-hub/plastics-the-facts-2022/ (accessed on 3 April 2023).

OPTIMIZING FILAMENT PRODUCTION FOR FFF TECHNOLOGY: PRESENTATION OF CURRENT RESULTS IN THE AREA OF CIRCULAR ECONOMY SUPPORT

Jakub KAŠČAK

Technical University of Košice, Faculty of Manufacturing Technologies with a seat in Prešov, Bayerova 1, 08001 Prešov, Slovakia
email: jakub.kascak@tuke.sk

The aim of this presentation is to introduce the initial stage of research focused on the optimization of filament production for Fused Filament Fabrication (FFF) technology, with an emphasis on the principles of the circular economy in line with the European Union’s legislative framework. Aligned with the EU Circular Economy Action Plan, the emphasis is placed on product sustainability, life cycle extension, and the efficient use of material resources. In the context of filaments and products manufactured using FFF technology, the most relevant strategic approaches include Reduce, Recycle, and Redesign.

- The Reduce strategy encompasses research and development aimed at optimizing filament properties to improve processability and adhesion to print beds, while minimizing waste caused by defective prints. It also includes the efficient processing of polymer and composite materials into filament form, ensuring more sustainable use of input materials.

- The Recycle strategy focuses on the potential for repeated filament recycling while maintaining the desired mechanical properties, particularly through the integration of bio-based components such as cellulose fibers, which may contribute to material stabilization during reprocessing.

- The Redesign strategy represents a shift in perspective toward re-evaluating the chemical composition of filaments and the structural design of printed products to improve recyclability and environmental compatibility already at the design stage, in accordance with eco-design principles.

The presentation will outline the initial steps taken in filament manufacturing research and composite fiber testing, including future directions in the development of cellulose-integrated polymer matrices. Special attention will be given to the potential of cellulose-based components to alter internal stress distribution in composite materials and to enable new printed part designs aimed at enhancing both functionality and environmental sustainability.

Acknowledgement

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ANTON PAAR – POLYMER CHARACTERIZATION

Richard LANDL

Anton Paar Slovakia, Hattalova 12 831 03 Bratislava - mestská časť Nové Mesto
email: Richard.landl@anton-paar.com

Anton Paar, a global leader in precision measurement and material characterization, now integrates the renowned expertise of Brabender Instruments to offer a uniquely comprehensive suite of solutions for the polymer and rubber industry. This powerful combination brings together Anton Paar's advanced technologies in rheology, viscosity, density, spectroscopy, particle characterization and XRD with Brabender's trusted systems for moisture content, extrusion, mixing, and process simulation. From lab-scale compounding and torque rheometry to real-time viscosity monitoring and mechanical testing, Anton Paar supports every stage of the polymer lifecycle—from raw material analysis and formulation development to quality control and production optimization. By uniting cutting-edge instrumentation with deep industry knowledge, Anton Paar empowers polymer researchers and manufacturers to accelerate innovation, enhance product performance, and meet the evolving demands of modern materials science and quality control.

1. Scheme 1. Mixers and extruders.

CHARACTERIZATION OF POLYMERS USING VARIOUS METHODS OF THERMAL ANALYSIS

Ekkehard FUEGLEIN

NETZSCH-Geraetebau GmbH, Wittelsbacherstrasse 42, 95100 Selb, Germany
email: ekkehard.fueglein@netzsch.com

The methods of thermal analysis are standardized techniques for the investigation of physical and chemical property changes of a variety of materials as a function of time and/or temperature. The applications range from organic samples like fats, oils and proteins to pharmaceutical active ingredients and additives and also from the wide field of polymers such as thermoplastics, elastomers and reactive systems to inorganic materials like glasses, metals and ceramics. In addition to standards for terminology in the field of thermal analysis[1], there are probably a few hundred national and international standards related to thermal analysis describing the setup of instrumentation as well as all relevant influencing factors for evaluation and interpretation of the data obtained.

This contribution will focus on the application of thermal analysis methods in the field of polymers. Examples for the use of techniques such as TG, DSC, DMA, DEA, TG-FT-IR, MMC and others (figure 1) are presented concentrating on the aspect of solving analytical challenges and answering questions related to inspection of incoming goods, quality control or research.

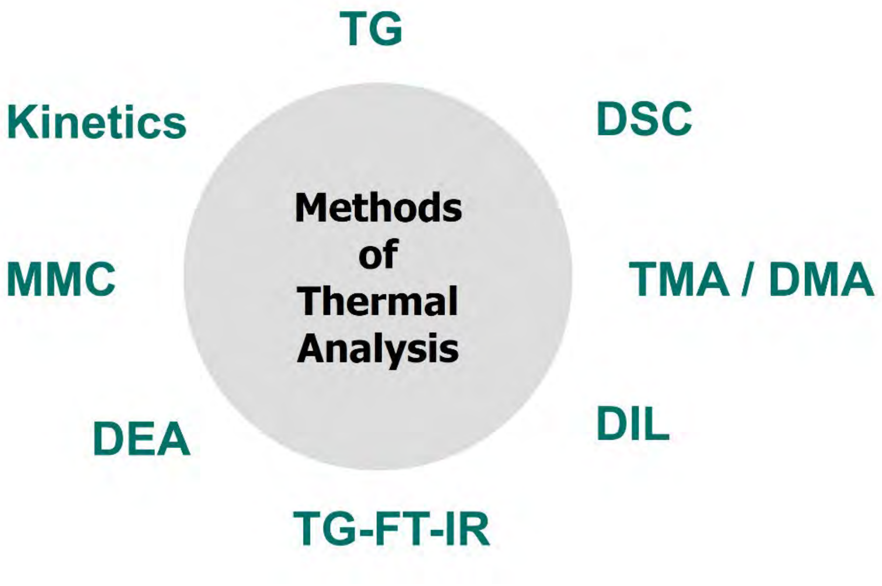

Figure 1. Methods of thermal analysis

The properties of polymers are often improved by using additives, or fillers. A quick and reliable possibility to determine the quantity of additives, the polymer itself and the filler content is thermogravimetry (TG). By heating the sample, not only the temperature of additive evaporation, polymer pyrolysis and filler decomposition can be detected and also the quantification of the detected mass changes give a fast insight into the materials composition.

Via the determination of the melting behavior of polymers by means of differential scanning calorimetry (DSC), it is possible to find impurities, or the ratio of materials used for a polymer blend.

An important material property is the glass transition temperature of amorphous or semi-crystalline materials. Methods such as DIL, TMA, DMA or even DSC can be applied to determine the range of transition from a glassy state into a flexible state. The degree of crystallinity or the specific heat capacity are further properties detectable by means of DSC.

Aging of polymers is usually accompanied by degradation and shortening of the polymer chain length. This, on the other hand, increases the reactivity of polymers at elevated temperatures against oxygen. So-called oxidation-induction time tests (OIT) can serve for classification of the state of aging of polymers.

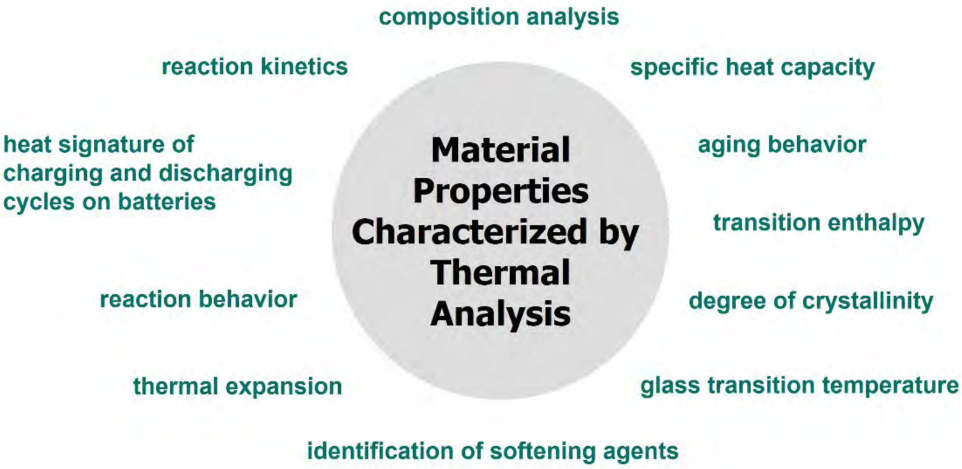

Figure 2. Material properties of polymers determined by thermal analysis methods

Additives such as softening agents are useful and necessary. On the other hand, some of them, phthalates in particular, are known to be cancerogenic and toxic to reproduction. TG-FT-IR is a combination of thermogravimetric analysis and Fourier-transform-infrared spectroscopy (FT-IR). By using this combination, it is not only possible to determine the temperature of gas release and the quantity of gas released, but furthermore to identify the kind of gas released.

Finally, a specialized setup of a calorimetric measurement technique, called multiple-module calorimeter (MMC), is designed to not only characterize materials used in state-of-the-art batteries but also full cells. By controlling a coin cell precisely in temperature, a cycler applies charging and discharging cycles to a battery. The calorimeter detects the heat signature of the battery independently for charging and discharging. The quantification of heat allows for calculating the efficiency of each cycle[2].

References

1. Deutsches Institut für Normung, DIN 51005, 2022-03, “Thermal Analysis – Vocabulary”.
   ASTM-E473, Standard Terminology Relating to Thermal Analysis and Rheology
2. “Isothermal Calorimetry for Investigation of Heat Signatures of Batteries During Charging and Discharging”, E. Füglein, C. Bracht, C. Kallfass, CEEC-TAC7, Brno, Czech Republic, 2023.

DEBONDING OF EV BATTERIES AS AN ENABLER FOR FUTURE BATTERY DESIGNS IN VIEW OF A CIRCULAR ECONOMY

Milan TINÁK* **and Eduard OSWALD**

Henkel Central Eastern Europe GmbH, Erdbergstraße 29, 1030 Wien
email: milan.tinak@henkel.com, eduard.oswald@henkel.com

This presentation introduces the concept of debondable adhesives as a key enabler for future electric vehicle battery designs aligned with circular economy principles. It addresses the challenges of battery disassembly, emphasizing the need to incorporate recyclability, repairability, and second-life applications into battery system designs from the outset. Conventional adhesives often hinder efficient, non-destructive disassembly, complicating compliance with emerging regulatory, environmental, and economic demands. The presentation outlines Henkel’s development of innovative adhesives that can be selectively debonded using various triggers such as heat, ultrasound, or electrical current. It highlights the importance of integrating such technologies early in the battery design process to enhance repairability, extend battery life, and reduce recycling costs. Though small in volume, debondable adhesives play a crucial role in enabling sustainable and cost-efficient EV battery systems.

BIOBASED BIODEGRADABLE BLENDS FOR TISSUE ENGINEERING WITH ENHANCED 3D PRINTABILITY AND MECHANICAL PROPERTIES

Sára VACH AGÓCSOVÁ*, Ivana BÍROVÁ, Ida VAŠKOVÁ, Michal ĎURFINA, Mária MIKOLAJOVÁ, Katarína TOMANOVÁ and Martina ČULENOVÁ[1]

Slovak University of Technology in Bratislava, Faculty of Chemical and Food Technology Radlinského 9, 812 37, Bratislava, Slovakia
[1]Institute of Medical Biology, Genetics and Clinical Genetics, Faculty of Medicine, Comenius University in Bratislava, 811 08 Bratislava, Slovakia
e-mail: sara.agocsova@stuba.sk

Abstract: Tissue engineering is one of the most rapidly advancing scientific disciplines. Significant progress in tissue engineering has been driven by the development of additive manufacturing techniques for the production of scaffolds, enabling a high degree of personalization. The evolution of additive manufacturing opens new possibilities in the medical field, which has led to extensive research on materials suitable for this technology. The development of new materials for medical applications is crucial for the perspective of more effective treatments or addressing diseases previously considered untreatable. This study investigates the potential use of poly (lactic acid) and polyhydroxyalkanoates as biodegradable polyesters in combination with special toughness modifier, as well as their polymer blends in combination with thermoplastic starch (TPS), for application in tissue engineering as scaffold materials. The focus was on optimizing these materials for processing using Fused Deposition Modeling (FDM) technology, as the most accessible method for fabricating patient-specific tissue scaffolds. The study also examines the influence of blend composition and processing parameters on the physical and mechanical properties of the final product.

Introduction

Tissue engineering is an interdisciplinary research field that integrates knowledge from medicine, cellular molecular biology, materials science, and engineering to develop biological substitutes intended to restore, maintain, or improve the function of damaged tissues [1,2]. The development of scaffolds made from bioresorbable biomaterials for tissue engineering applications, including the potential for biological 3D printing, has emerged as a promising solution to the shortage of implants. The future application of 3D bioprinting in medicine represents a method for rapidly and cost-effectively producing implants without defects, with minimal waste, and, importantly, for personalizing them for individual patients. The scaffold provides mechanical and dimensional stability to the developing tissue until it becomes self-supporting. The mechanical properties of the scaffold should ideally match those of the target tissue [3,4].

An ideal scaffold should be prefabricated into a specific shape, while also providing a level of mechanical properties that allow the scaffold to temporarily replace the tissue's function until the tissue undergoes remodeling and sufficient regeneration. Although achieving perfect mechanical properties akin to the target tissue is challenging, the scaffold must be able to withstand handling and mechanical loads encountered in the physiological environment [5, 6]. Given that the most accessible additive manufacturing technology is Fused Deposition Modeling (FDM), efforts were made to optimize the blend properties to make them suitable for FDM processing, including the preparation of filament as a semi-finished product for FDM. This is crucial as, during printing, the 3D filament must be strong and flexible enough to ensure proper feeding, preventing any degradation before entering the printhead, and ensuring a consistent material flow to the 3D printer's head. The high proportion of thermoplastic starch (TPS) in PLA/PHA, however, caused excessive brittleness in the 3D printing filament, which necessitated modifications to the matrix to ensure adequate printability. To address this, the special biobased biodegradable toughness modifier was incorporated into the blends.

Materials and Testing

The basis of the studied polymer matrix consists of three types of polymeric materials: poly(lactic acid) (PLA), polyhydroxyalkanoate (PHA), toughness modifier and thermoplastic starch (TPS).

The polymer granules were prepared using a twin-screw extruder (LabTech) featuring intermeshing and co-rotating screws:

1. Screw diameter: 16 mm
2. L/D ratio: 40
3. Screw geometry: corotating with intermeshing screws
4. Screw configuration: four kneading zones at positions 11-14D, 18-19D, 21-23D, and 26-29D, with one reverse-flow segment positioned at the end of the last kneading zone (positions 30-31D).
5. The temperature profile and screw rotational speed were adjusted according to the composition of the blend.
6. A cooling unit was incorporated after the extrusion device, followed by a granulation unit.

3D filaments were prepared using a single-screw extruder (Plasticorder Brabender) with the following parameters: L/D = 25, D = 19 mm, and a compression ratio of 1:1. The granules were fed into the hopper. The temperature profile of the four heating zones, from the hopper to the nozzle, was 160-170-180-190°C, with a screw rotational speed of 20-25 rpm.

The melt was extruded through a straight nozzle with a circular diameter of 2 mm. Subsequently, the melt was cooled in a cold-water bath. The emerging filament was drawn onto a conveyor belt, with the drawing speed adjusted to ensure a filament diameter of 1.75 to 1.80 mm.

Rheological Properties

The rheological properties of the blends were measured using the RPA 2000 oscillatory rheometer from Alpha Technologies. The samples were placed in the biconical chamber of the rheometer, and the testing program was initiated. After sealing the chamber, the temperature was set to 180°C, and the samples were preheated for 1.5 minutes at an oscillation angle of 6° and an oscillation frequency of 60 CPM. Following the preheating stage, the temperature was adjusted to 160°C, and the flow curve measurement began at the same oscillation frequency of 60 CPM. The oscillation angle was gradually increased from 2° to 50°, resulting in shear rates ranging from 1.34 to 40 s^{-1}. The complex viscosity was recorded using the rheometer software, and the relationships between complex viscosity and shear rate were evaluated for all samples.

The viscosity measurement results, presented as the dependence of complex viscosity at shear rate 15 s^{-1} on the concentration of toughness modifier in the blend, are shown in Fig. 1.

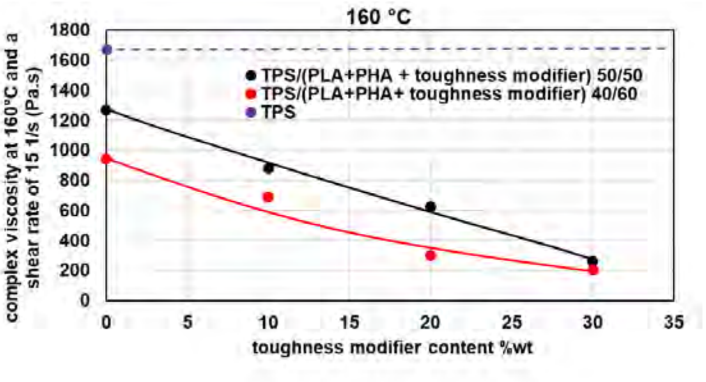

Fig. 1: Dependence of complex viscosity at 160°C and a shear rate of 15 s^{-1} on toughness modifier content in the blend

In Fig 1, the viscosity of the final blend (black and red circles), is significantly lower compared to the TPS (purple circle). With the gradual addition of toughness modifier, there is a significant decrease in viscosity, for both blends with TPS/(PLA+PHA + toughness modifier) ratios of both 50/50 and 40/60, while blend with ratio TPS/ (PLA+PHA + toughness modifier) = 40/60 exhibits lower viscosity.

Mechanical Properties

Tensile tests were conducted using a Zwick-Roell testing machine at a cross head speed of 50 mm/min, in accordance with the STN ISO 527 standard. During the test, the stress-strain curve of the specimen was recorded. From the tensile curve, the yield strength (σy), the tensile strength at break (σb), the Young´s modulus (E) and the elongation at break (εb) were determined.

The mechanical properties of the prepared blends, which were measured in tension mode on the filaments, are shown in Fig. 2.

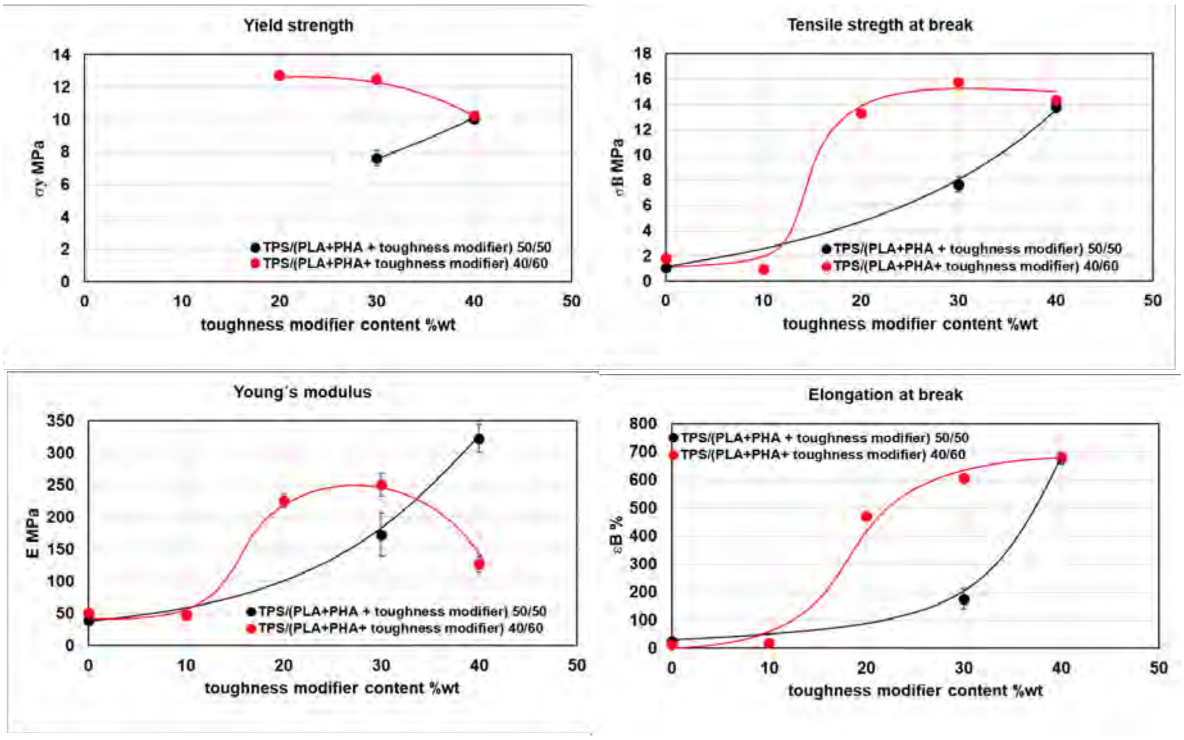

Fig. 2: Dependence of the mechanical properties of filaments on toughness modifier content

Fig 2 shows that the addition of toughness modifier significantly improves the mechanical properties of the filament. However, the effect of toughness modifier is also influenced by the ratio of TPS/ (PLA+PHA + toughness modifier). In both cases, an increase in tensile strength and elongation at break is observed for both ratios, with a considerably more pronounced improvement for the ratio of TPS/ (PLA+PHA + toughness modifier) 40/60. Similarly, the Young's modulus increases, although for the blend with the ratio of TPS/ (PLA+PHA + toughness modifier) 40/60, a transition past the maximum is observed. Improving (increasing) of elongation of break is related to increasing of filament and final scaffold flexibility and it correlates with the fact that blends without toughness modifier or with very low toughness modifier concentration (10 or less %) could not be processed into filaments, or the filaments could not be printed, due to the excessive brittleness of the material.

The influence of toughness modifier on the mechanical properties is regarded very positively, as it allows for better processing of the prepared blends and increases their application value. This includes improved scaffold manipulability, enhanced mechanical properties, and adjustable scaffold flexibility.

Conclusion

The incorporation of toughness modifier significantly enhances both the mechanical properties and processability of the blends intended for 3D printing applications. Blends without toughness modifier or with low concentrations of toughness modifier exhibited inferior processability, primarily due to high brittleness. In contrast, blends containing a lower proportion of thermoplastic starch (TPS) demonstrated improved processability.

The presence of toughness modifier notably improved the tensile strength and elongation at break, while simultaneously reducing the viscosity of the material during processing, which is advantageous for 3D printing. Furthermore, the effect of the TPS/ (PLA+PHA + toughness modifier) ratio was observed, with blends having higher TPS content showing superior processability and flexibility. Blends with a lower TPS content and higher proportions of PLA and PHA + toughness modifier, however, exhibited superior mechanical properties, particularly at TPS/ (PLA+PHA+ toughness modifier) ratio of 40/60.

Rheological measurements, including the analysis of complex viscosity, confirmed that the addition of toughness modifier led to a significant reduction in viscosity, facilitating better processing conditions without compromising the mechanical integrity of the blends. This characteristic is essential for the production of stable and functional scaffolds in tissue engineering applications.

Overall, the addition of toughness modifier is a key factor in improving the processability, flexibility, and functional applicability of 3D-printed scaffolds, while maintaining the necessary mechanical stability for tissue regeneration. This study highlights the potential of PLA, PHA, and TPS-based blends with toughness modifier as promising materials for advanced tissue engineering applications, offering a feasible approach for personalized regenerative therapies.

Acknowledgement

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References

1. A. Nair, S. Nadine, S. Ahadian, J. Mano, N. Tanideh, and F. G. Thankam, ‘Chapter 23 - Translational tissue engineering’, in Tissue Engineering, C. P. Sharma, T. Chandy, V. Thomas, and F. G. Thankam, Eds., Academic Press, 2022, pp. 557–573. doi: https://doi.org/10.1016/B978-0-12-824064-9.00023-X.
2. A. M. Jorgensen and A. Atala, ‘Chapter 19 - Tissue engineering of organ systems’, in Tissue Engineering (Third Edition), J. De Boer, C. A. V. Blitterswijk, J. A. Uquillas, and N. Malik, Eds., Third Edition.Academic Press, 2023, pp. 661–688. doi: https://doi.org/10.1016/B978-0-12-824459-3.00019-6.
3. L. Suamte, A. Tirkey, J. Barman, and P. Jayasekhar Babu, ‘Various manufacturing methods and ideal properties of scaffolds for tissue engineering applications’, Smart Materials in Manufacturing, vol. 1, p. 100011, 2023, doi: https://doi.org/10.1016/j.smmf.2022.100011.
4. N. Sultana Phd Ceng Csci, M. Hassan, and M. Lim, Composite Synthetic Scaffolds for Tissue Engineering and Regenerative Medicine. 2015. doi: 10.1007/978-3-319-09755-8.
5. K. Pluta, D. Malina, and A. Sobczak-Kupiec, ‘KLAUDIA PLUTA, DAGMARA MALINA, AGNIESZKA SOBCZAK-KUPIEC * SCAFFOLDS FOR TISSUE ENGINEERING RUSZTOWANIA KOSTNE DLA INŻYNIERII TKANKOWEJ’, doi: 10.4467/2353737XCT.15.108.4056.
6. S. Amado, P. Morouço, P. Pascoal-Faria, and Nuno Alves, ‘Tailoring Bioengineered Scaffolds for Regenerative Medicine’, in Biomaterials in Regenerative Medicine, L. A. Dobrzański, Ed., Rijeka: IntechOpen, 2017. doi: 10.5772/intechopen.69857.

BIO BASED POLYMER COMPOSITES

Eduard OSWALD*, Jozef FERANC, Michal ĎURFINA, Roderik PLAVEC, Pavol ALEXY, Slávka HLAVÁČIKOVÁ, Katarína TOMANOVÁ, Zuzana VANOVČANOVÁ, Zuzana KRAMÁROVÁ, Vojtech HORVÁTH, Andrej BACO and Laura PALKOVIČOVÁ

Institute of Natural and Synthetic Polymers, Faculty of Chemical and Food Technology, Slovak University of Technology, Radlinského 9, 812 37 Bratislava, Slovak Rep.
email: eduard.oswald@stuba.sk

Our daily life needs depend on plastics, as they are cheap and durable, so they become the most commonly used synthetic chemical products. But from an environmentalist's point of view, major concern related to these plastics is their non-biodegradable nature[1]. Accumulation of synthetic plastics in the environment for several years, if not degraded, causes huge problems. It may result in infertility of soil where crops are not able to grow and make the land infertile. If animals consume it as a food source, then they may suffer with many diseases and can be the cause of their death. Plastics accumulated in sewage causes blockage, which invites different kinds of flies, insects, and parasites for shelter, which further spread diseases. On an average, 10% by weight of municipal waste stream is plastic. Plastic debris causes aesthetic problems, and it also presents hazard to marine habitats including fishing and tourism. Discarded fishing nets result in ghost fishing that may result in loss of commercial fisheries. Many wildlife species have been reported to ingest or become entangled in plastic waste. So satisfactory degradation of plastics is really a challenging aspect. Replacement of synthetic plastics with biodegradable plastics seems to be a good approach to solve pollution issues from our society[2]. Biodegradable polymers represent hot topic in current environment. Here, biodegradable means polymers that are capable of decomposing into simplest molecules like CO_2, CH_4, H_2O, and residual biomass. Variety of naturally occurring and artificially synthesized biodegradable polymers are reported, namely, cellulose, chitin, starch, polyhydroxyalkanoates (PHAs), polylactide, and polycaprolactone. The PHAs are natural biodegradable thermoplastics with properties resembling common synthetic petrochemical–based polymer. Gradual replacement of synthetic plastics with eco-friendly biodegradable polymers will help to reduce plastic waste accumulation in our fragile environment[3]. Polyhydroxyalkanoates has been extensively used for packing film in shopping bags, household utensils, napkins, lady cares, composting bags, coating material of containers, razors, and papers. As medical material, application of PHAs has been widely explored in sutures, bandages, blood vessel patches, biocompatible implants, bone pegs, muscle tendon restoration apparatus, spinal cord bearing frames, nerve catheters, wound dressings, histology restoration, artificial esophagus, and controlled drug release carriers. Recent research demonstrates the application of PHA in nerve rehabilitation and artificial blood vessel and successfully used in skeleton reviving because of its excellent piezoelectricity. Besides the different structures and characteristics, PHA also has the property from rigidity to elasticity, an obligatory requirement for entire plastic field. Continuous advancements in product quality, stability, and manageable production cost make PHA a suitable candidate for industrial applications[4]. Reinforcing fillers are not only added by means to make the material cheaper, means commercially better available. The added value of additives which are used is to increase main parameters like the impact resistance, resulting in a higher values in other mechanical properties like for example elongation at break or tensile strength or yield[5]. Six different types of inorganic and organic fillers compounded to polymer blend of PHA1/PHA2 were observed in current study. Content of filler in polymer blend was 10% vol. The main point of current work was to study effect of different type of inorganic and organic filler on mechanical properties, morphological structure, rheological and thermal properties on prepared compounds.

TECHNIQUES AND SUBSTANCES

Substances

Polyhydroxyalkanoates (PHAs) – two types of PHAs were delivered by company Panara a.s., Nitra, Slovakia. PHA1 – polyhydroxyalkanoate with high crystallinity (52%), Tg = 5.1°C, Tm = 166.7°C, in powder form. PHA2 – amorphous polyhydroxyalkanoate with 0% crystallinity, Tg = -14.9°C, Tm not detectable on DSC. All we utilized as biodegradable bio-based blend. On behalf of filler we have utilized Talc MinTalc 97-05C (Ravago Chemicals Czech Republic s.r.o.), Calcium Carbonate Omyacarb 2-VA (Omya s.r.o.), Cellulose Jelucel HM30 (JELU-WERK, Josef Ehrler GmbH & Co. KG), Wood Floor (J. RETTENMAIER & SOHNE GmbH + Co KG), Starch C Gel (BRENNTAG SLOVAKIA s. r. o.) and Bio Piva 100 Kraft Lignin (UPM Biochemicals).

Production of the mixtures

The blends of PHA1/PHA2 with ratio 50/50 and with 10% vol. loading of inorganic and organic filler were prepared by using of twin-screw extruder with screw diameter of 26mm, L/D = 40. The testing films were prepared by attaching the chill-roll line directly to the twin-screw extruder. The melt exiting the nozzle of rectangular cross-section was extruded onto a system of water-cooled rollers and subsequently wound under constant tension onto a winding device.

Production of testing bars

Dog-bone tensile bars for measuring the physical-mechanical properties of the studied filler composites were prepared by injection molding on a hydraulic injection molding machine BOY 60E, in accordance with the tensile testing standard STN ISO 527. The tensile tests were performed using a Zwick Roell tensile-testing device at a crosshead speed of 50 mm/min. Tensile strength at yield (σy), tensile strength at break (σb), and relative elongation at break (εb) were recorded and evaluated.

Assessment of processing stability

RPA 2000 oscillating rheometer with a timed test was used to assess the processing stability of the prepared polymer blends. The conditions, that we have used during the measurement are: preheating period: 1 min, analysis period: 10 min, Oscillating rheometer operative temperature: 180 °C, Oscillation frequency: 50 CPM, Oscillation angle: 60 °. In order to achieve proper assessment for the influence of temperature, causing polymer degradation, resulting with decrease of complex viscosity, we have eliminated the differences in primary viscosity by figuring the complex viscosity to the values of relative viscosity utilising formula: $\eta^*_{rel}(t) = \eta^*(t)/\ \eta^*_0$ (1)

$\eta^*_{rel}(t)$ is relative complex viscosity at time t, $\eta^*(t)$ is complex viscosity at time t and η^*_0 is complex viscosity at start of the test.

Measuring of thermal properties

Thermal properties for all blends were determined using a differential scanning calorimeter (DSC 1, Mettler-Toledo Inc., USA). Key thermal characteristics, including the glass transition temperature (Tg), crystallization temperature (Tc), and melting temperature (Tm), were evaluated from measurements conducted on dog-bone tensile bars. Nitrogen was employed as the inert gas with a flow rate of 50 mL/min. The measurement protocol was as follows:

- Isothermal hold at -70°C for 3 min
- First heating cycle: -70°C to +200°C at a heating rate of 10 K/min
- Isothermal hold at +200°C for 3 min
- Cooling cycle: +200°C to -70°C at a cooling rate of 10 K/min
- Isothermal hold at -70°C for 3 min
- Second heating cycle: -70°C to +200°C at a heating rate of 10 K/min

Scanning Electron Microscopy

The internal structure of the prepared films was examined using a scanning electron microscope (SEM) from JEOL, model JSM-7500F. Fracture surfaces were obtained by breaking the samples after cooling in liquid nitrogen.

RESULTS AND DISCUSSION

Processing stability and rheological properties

Understanding rheological characteristics is essential for the processing of polymer blends into final products. To assess the processing stability of the prepared mixtures, the dependence of complex viscosity on time was measured using an oscillatory rheometer at 180 °C. This test provides valuable insight into the material’s behavior under thermomechanical stress, where degradation typically results in a reduction in molecular weight and, consequently, a decrease in viscosity. To comprehensively evaluate the influence of different fillers, both absolute and relative viscosity values at the fourth minute of testing were plotted for each formulation. It is generally assumed that fillers increase the viscosity of polymer blends; however, this was not consistently observed. Fillers such as talc, starch, cellulose, and wood flour exhibited similar or higher viscosity profiles. Among them, cellulose and talc based composites displayed the highest viscosities - exceeding that of the unfilled matrix. Conversely, blends containing calcium carbonate and lignin-based fillers demonstrated noticeably lower viscosities. In these cases, the decrease in viscosity suggests that the fillers may act as prodegradants, promoting degradation processes within the polymer matrix during thermal and mechanical stress. This degradation is reflected in the relative complex viscosity, with the most significant declines observed in samples containing calcium carbonate and lignin. In conclusion, the extent of degradation in PHAs blends under thermomechanical stress is strongly influenced by the type and chemical nature of the filler. Fillers with prodegradant behavior, such as calcium carbonate and lignin, significantly accelerate viscosity loss, highlighting their impact on the stability of the polymer system during processing.

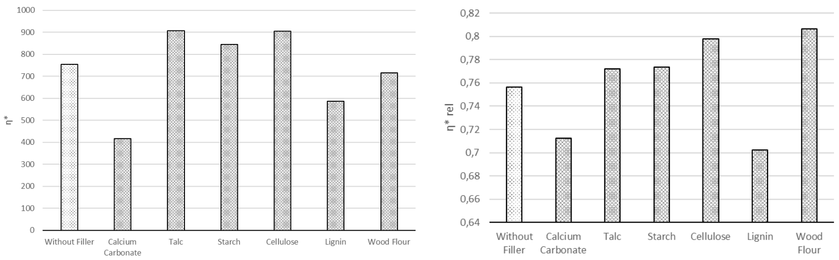


Figure 1. Rheological characteristics measured after 4th min. - viscosity complex and relative

Mechanical properties

As part of evaluating the impact of filler type on the mechanical properties of the prepared samples, we measured relative elongation at break, tensile strength at break, and tensile yield strength. These properties were assessed based on measurements taken one day after samples preparation. In terms of relative elongation at break, the highest values—around 500%—were, as expected, observed in the unfilled material. All filled composites, with the exception of the starch-filled sample, exhibited significantly lower elongation values, ranging between 20% and 50%. Notably, the starch-filled composite demonstrated a markedly higher elongation at break, reaching approximately 150%, making it the most ductile among the filled systems. For tensile strength at break, the unfilled material again exhibited the highest value, approximately 18 MPa. However, the difference compared to filled samples was less pronounced than in the case of elongation. The filled composites showed tensile strength values ranging between 8 and 12 MPa. Lastly, measurements of tensile yield strength revealed minimal variation across all samples, regardless of filler type, with values consistently falling within the range of 12 to 14 MPa. The only exception is talc, where the yield strength values increased by 2 MPa compared to the unfilled blend, reaching a value of 18 MPa.

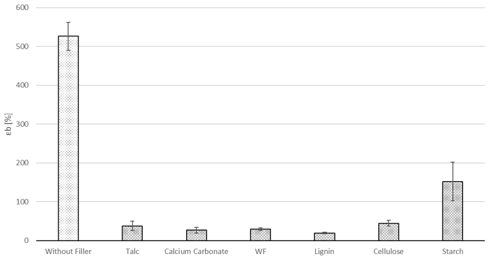


Figure 2. Mechanical properties - Elongation at break and deviation

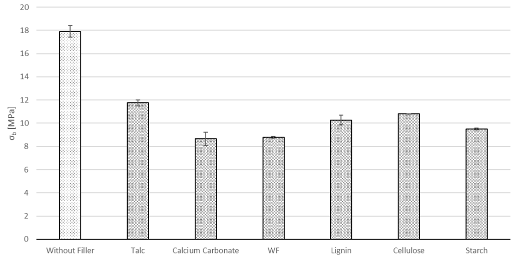


Figure 3. Mechanical properties - Tensile strength at break and deviation

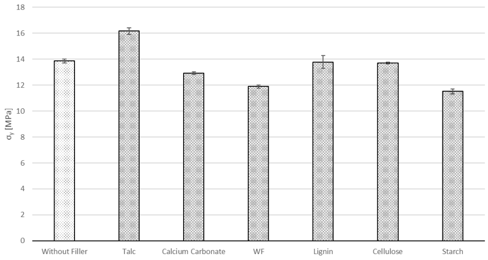


Figure 4. Mechanical properties - Tensile stress at yield and deviation

Thermal properties

Each sample underwent a sequence of thermal analysis involving first heating, cooling, and second heating using differential scanning calorimetry (DSC). The first heating reflects the sample's thermal history, which includes influences from its preparation method, cooling rate during fabrication, and conditions during storage. In contrast, the second heating reveals the material's inherent thermal properties, free from any prior thermal influence. No cold crystallization peaks were observed in the first heating run, indicating that the injection molding process was properly conducted and allowed the formation of a crystalline phase in all samples. The melting temperature of the crystalline phase changed only slightly compared to the unfilled blend, within a range of 1 °C, except for calcium carbonate, where a decrease of 2 °C was recorded. However, during cooling, more significant differences in crystallization temperatures were observed. For the blend with talc, the crystallization temperature increased from 85 °C (unfilled blend) to 101 °C, whereas for the other fillers, a decrease in crystallization temperature was observed — ranging from 4 °C (calcium carbonate) to 35 °C (lignin). The highest crystallization enthalpy was recorded for the talc-containing blend, which, combined with the higher crystallization temperature, confirms its nucleating effect. The addition of other fillers led to a decrease in crystallization enthalpy, with a more pronounced reduction observed for organic fillers. In the case of lignin, the crystallization peak was almost suppressed, and the crystallization enthalpy dropped to only 3 J/g. This phenomenon subsequently manifested during the second heating run in the form of a cold crystallization peak. The second heating run also revealed changes in the glass transition temperature (Tg). The addition of talc caused the most significant decrease in Tg, lowering it to -18.7 °C, which is 2 °C lower than that of the unfilled blend. Conversely, the incorporation of lignin resulted in an increase in Tg to 1 °C. The other tested fillers exhibited glass transition temperatures similar to that of the unfilled blend.

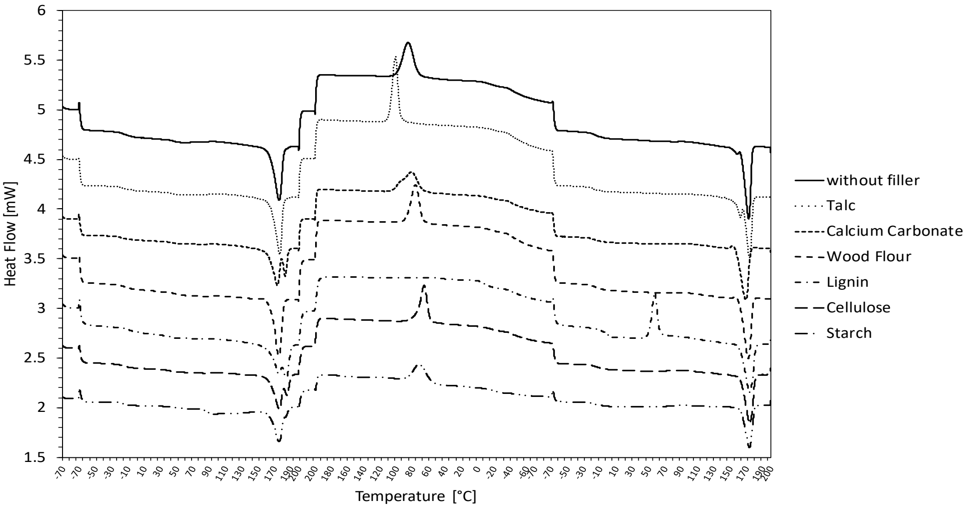


Figure 5. Thermal properties - DSC record for first heating, cooling and second heating

Morphological structure

Mechanical and physical properties we have measured are linked with the morphological form of the manufactured plates. Significant influential role plays the dispersion of the filler composite and the interaction between the filler and the blend. Talc-based fillers with their lamellar structure, plays the definitive element for the ability to delamination in the shear stress measurement. We can see in the pictures a broad particle size distribution for the pure calcium carbonate filler. For all the organic fillers the size of the partials is significantly higher, despite for lignin. Further more, we can see at the morphological forms, that the filler is properly distributed in the polymer matrix thanks to the superb mixing step.

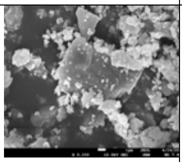
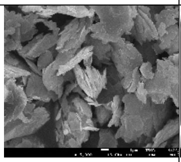
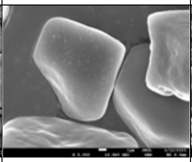
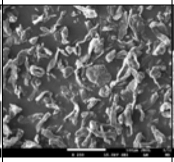
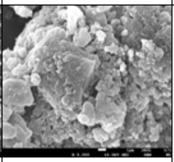
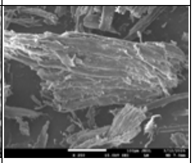
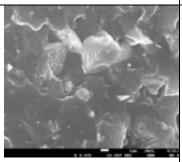
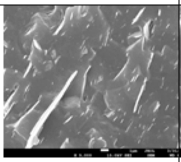
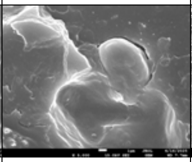
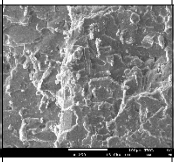
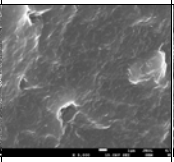
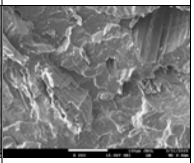
	Calcium Carbonate	Talc	Starch	Cellulose	Lignin	Wood Flour
Filler						
Blend with Filler						

Figure 6. Morphological form of SEM pictures at 5000x magnification for mixtures and with fillers, except for Celulose and Wood Flour filler, with magnification at 250 x

CONCLUSION

We can conclude, that for the rheological parameters, the viscosity changes are dependent on the fillers. For the Cellulose and wood flour blends there is significant increase. On the contrary, the mixtures with composite fillers of calcium carbonate and lignin, are behaving as important prodegradant, leading to viscosity decrease. The addition of fillers resulted in a significant decrease in elongation at break. Tensile strength also decreased. Thermal analysis indicates that talc acts as a nucleating agent in the studied blends. The properties measured: processing stability, thermal, physical-mechanical and rheology are the result of the chemical composition and not only depends on particle size of the composite filler. If we would some modifier, the fitting within the polymer blend and the filler composite may have better results, also in the case of calcium carbonate-based composite fillers, meaning the degradation process can be prevented.

ACKNOWLEDGEMENT

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REFERENCES

1. SHAH, A. A., HASAN, F., HAMEED, A., AHMED, S. *Biological degradation of plastics: A comprehensive review*. Biotechnology Advances 26, 2008. p. 246-265.
2. TOKIWA, A. et al. 2009. Biodegradability of plastics. *International Journal of molecular Sciences*. 2009, vol. 10, ISSN 1422-0067, p. 10.
3. KYRIKOU, I. - BRIASSOULIS, D. 2007. Biodegradation of Agricultural Plastic Films: *Acritical rewiev, J Polymer Environ* 2007, p. 125-150.
4. Sin, L. - Rahmat, A. 2012. Polylactic acid - A volume. In: Plastics Design Library, 4-Chemical Properties of Poly(lactic Acid), Malaysia 2012, ISBN 978-1-4377-4459-0.
5. Liu, X. et al. 2014. Effects of Inorganic Fillers on the Thermal and Mechanical Properties of Poly(lactid acid). In International Journal of Polymer Science. 2014, vol. 2014, Article ID 827028

CURRENT TRENDS AND ISSUES IN TYRE RECYCLING

Jiří BREJCHA [1,2]*, **Jan KRMELA** [1] **and Libor BENEŠ** [2]

[1]Faculty of Mechanical Engineering, J. E. Purkyně University in Ústí nad Labem, Czech Republic
[2] Brejcha Rubber Consulting, Prague, Czech Republic
email: jiribrejcha@seznam.cz

As the volume of road and off-road traffic continues to grow, so does tyre production. But at the same time, the volume of end-of-life tyres is becoming a real environmental problem. Global aspects of tyre market push also producers, policy makers take variety aspects into consideration. The presentation summarises some important aspects, influences and parameters. Include technical points, economic and also environmental and geopolitical tasks.

The presentation will compare benefits and losses of all decision processes, with respect to chemical and physical science implications. The scientific aspect must also be considered in a real industrial environment and shows the need to balance performance and cost points of view.

Important trends in new capacity building respect industrial application point of view and also need of reasonable paybacks to investors, but at the same time accept development of performance characteristics of new tyres and requirements of authorities and homologation, especially in terms of safety, environment and production cost.

The role of standardization as a strong supporting tool is discussed, the difficulties of developing new standards and the complete comitology process are well described.

In the last part of the presentation, new challenges arising from recent legislative acts of the European Commission and the European Parliament are presented. Different approaches are clearly discussed, and possible solutions are identified.

In all cases, the basic objective of material transformation back to the cycle is shown and preferred.

This presentation also identified very important role of science, research and industrial application development for fulfilling future economic figures. Last but not least, a number of questions are left open for further studies and projects.

Together with poster presentation by Anna Smetanová makes a comprehensive set of presentation of activities that are carried out at the Faculty of Mechanical Engineering Jan Evangelista Purkyne University in Usti and Labem in close cooperation with companies and other universities in the Czech and Slovak Republic.

Jiří Brejcha is a consultant in the field of development, production and chemical safety of polymeric materials and an internal PhD student at the Faculty of Mechanical Engineering of the Jan Evangelista Purkyně University in Ústí and Labem. He is a member of the evaluation panel of the E4S European Rubber Journal, worked for 20 years as a materials development manager at Mitas and Trelleborg Wheel Systems and was a member of working groups of the European Tyre and Rubber Association. https://www.linkedin.com/in/ji%C5%99%C3%AD-brejcha-bb662353/

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END-OF-LIFE TIRES AS SECONDARY RAW MATERIALS FOR SUSTAINABLE ENVIRONMENT

Hamed PEIDAYESH[1]*, Ivan CHODÁK[1] and Jozef SOTÁK[2]

[1]Polymer Institute of the Slovak Academy of Sciences, Dúbravská cesta 9, 845 41 Bratislava, Slovakia
[2]Resumo s.r.o., Ladislava Hudeca 15326/2A, 974 01 Banská Bystrica, Slovakia
email: ivan.chodak@savba.sk

Globally, the industry produces over 1.6 billion tires annually and consumers retire around 1 billion waste tires. Current disposal strategies to retiring tires, consisting of incineration, crumb rubber generation, and landfilling, are not effective. Waste tire rubber recovery or regeneration is highly desirable for rubber sustainability and rubber product circular economy in automotive industry. The aim of this contribution was to generate interest in the applications of devulcanized rubber (DvR) prepared from end-of-life tires in the rubber industry, both from an economic and technological point of view.

The company Resumo in Slovakia purchased Recycling Technology from China, a high-performance line for truck tires recycling. First, the tire rubber powder is prepared from the outer tread of truck tire. The rubber powder represents the basic raw material for devulcanization process. The compounding of vulcanizates was done using an internal mixer and a two-roll mill. Afterward, vulcanization of the samples was performed through compression molding. The reproducibility of properties of DvR prepared by the same processes in several batches during approximately two years shows that the differences in the values of mechanical properties of the DvR-based vulcanizates are very reproducible, justifying that the process of DvR production in RESUMO is well mastered. The results indicated that the substitution of virgin rubbers by 20 phr (parts per hundred rubber) of DvR has almost negligible deterioration of ultimate mechanical properties. Moreover, substitution up to 35 phr of virgin rubbers could be considered if marginal decrease of strength would be acceptable. This finding leads not only to savings in material costs but also to significant ecological impact due to the effective recycling of used tires.

Acknowledgement
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THE INFLUENCE OF MATERIAL COMPOSITION ON SELF-HEAT BUILDUP OF RUBBER UNTIL BLOWOUT

Ondřej KRATINA[1]* **and Radek STOČEK[1,2]**

1Centre of Polymer Systems, Tomas Bata University in Zlín, třída Tomáše Bati 5678, 760 01, Zlín, Czech Republic
2 PRL Polymer Research Lab., s.r.o., Nad Ovčírnou 3685, 76001, Zlín, Czech Republic
*email:*okratina@utb.cz

Rubber components such as tires and shock absorbers are frequently subjected to cyclic mechanical loading. Due to the viscoelastic nature of rubber, part of the mechanical energy is inevitably transformed into heat. This internal heat accumulation, exacerbated by rubber's poor thermal conductivity, can lead to dangerously high temperatures that compromise material performance and durability. In extreme cases, a thermal runaway effect causes internal pressure to rise until the rubber catastrophically ruptures, which is an event known as blowout.

This study investigates how the type of elastomer and the concentration of carbon black affect self-heating behavior and the conditions leading to blowout. Rubber compounds were prepared using natural rubber (NR), styrene-butadiene rubber (SBR), and their 50/50 blend, each filled with either 40 or 60 parts per hundred rubber (phr) of N330 carbon black. Specimens were subjected to multi-axial cyclic loading designed to mimic real-world stress conditions. Internal temperature was measured via a built-in sensor, while surface temperature was simultaneously monitored using a non-contact method.

The results reveal a clear link between material composition and the self-heating process. In NR based compounds, heat build-up followed a three-phase progression: an initial rapid temperature rise, a plateau-like middle stage, and a final steep increase culminating in blowout. This staged behavior was consistent at both filler loadings. In contrast, pure SBR compounds did not exhibit the final rapid temperature escalation, and no blowout occurred within the test parameters.

These findings provide critical insights into the thermal response and safety margins of rubber materials under repeated mechanical loading. Understanding and predicting the self-heating threshold based on composition enables better design of rubber products with higher resistance to thermal failure, which is essential for improving operational safety and extending service life.

Acknowledgement

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References

1. De Lima, A., Rade, D., Lacerda, H., & Araújo, C. (2015). An investigation of the self-heating phenomenon in viscoelastic materials subjected to cyclic loadings accounting for prestress. Mechanical Systems and Signal Processing, 58–59, 115–127. https://doi.org/10.1016/j.ymssp.2014.12.006
2. Liu, Y., Chen, W., & Jiang, D. (2022). Review on heat generation of rubber composites. Polymers, 15(1), 2. https://doi.org/10.3390/polym15010002
3. Li, F., Liu, J., Yang, H., Lu, Y., & Zhang, L. (2016). Numerical simulation and experimental verification of heat build-up for rubber compounds. Polymer, 101, 199–207. https://doi.org/10.1016/j.polymer.2016.08.065
4. Luo, W., Huang, Y., Yin, B., Jiang, X., & Hu, X. (2020). Fatigue life assessment of filled rubber by hysteresis induced self-heating temperature. Polymers, 12(4), 846. https://doi.org/10.3390/polym12040846
5. Kratina, O., Stoček, R. (2023), How the rubber compounds of different tire's components heat up? In: Marano et al (eds), Constitutive Models for Rubber XII, 199-204. CRC Press / Balkema, Boca Raton, FL, USA. https://doi.org/10.1201/9781003310266-34.
6. Park, D. M., Hong, W. H., Kim, S. G., & Kim, H. J. (2000). Heat generation of filled rubber vulcanizates and its relationship with vulcanizate network structures. European Polymer Journal, 36(11), 2429-2436. https://doi.org/10.1016/s0014-3057(00)00020-3
7. Kratina, O., Stoček, R., Zádrapa, P., Kipscholl, R., Peter, O. (2023). Kinetics of Self-Heat Build-Up in Carbon Black Filled Natural Rubber Caused by Cyclic Mechanical Loading. In: Advances in Polymer Science. Springer, Berlin, Heidelberg. https://doi.org/10.1007/12_2023_167
8. Kratina, O., Pöschl, M., & Stoček, R. (2024). The effect of apparent density of sulfidic crosslink and their chemical nature on self-heat build-up in carbon black filled natural rubber under cyclic mechanical loading. Polymer Degradation and Stability, 227, 110871. https://doi.org/10.1016/j.polymdegradstab.2024.110871.
9. Gent, A. N. and Hindi, M. J. (1988). Heat build-up and blowout of rubber blocks. Rubber Chemistry and Technology, 61(5), 892-905. https://doi.org/10.5254/1.3536225.

NATURAL-BASED ANTIOXIDANTS FOR RUBBER COMPOUNDS

Drahomír ČADEK*, Nikolai PERMINOV, Alena KADEŘÁBKOVÁ and Antonín KUTA

The University of Chemistry and Technology, Prague, Faculty of Chemical Technology, Department of Polymers, Czech Republic.
email: drahomir.cadek@vscht.cz

Keywords: Natural rubber; natural antioxidant; degradation.

Vulcanized rubber, during long-term storage, slowly changes its properties; this process is called ageing. Usually, this is caused by chemical reactions with oxygen or ozone from the atmosphere. The weakest places in rubber to undergo these reactions are double bonds. To prevent degradation, rubber should be stabilized with antidegradants. Antioxidants protect rubber from the impact of oxygen. Antioxidants can be divided into two major groups based on their mechanism of action: those that interrupt the kinetic chain and hydroperoxide deactivators.

In this work different natural antioxidants were used, like: 2-isopropyl-5-methylphenol (Thymol), 5-isopropyl-2-methylphenol (Carvacrol), DL- α-tocopherol, Cashew nut shell liquid (CNSL), Green tea extract, Lignin and trans-β-farnesene. The chosen antioxidants will then be compared with two antidegradant systems: pure 1,2-dihydro-2,2,4-trimethylquinoline (TMQ) and a typical commercial system containing TMQ + 6PPD. This work deals with the comparison of natural rubber mixtures with various natural antioxidants and mixtures with synthetic antioxidants. The degradation of vulcanizates by different methods was evaluated and the mechanical properties after artificial ageing were investigated.

EFFECT OF THE CARBON BLACK TYPE AND LOAD ON FATIGUE PROPERTIES AND THERMAL EXPANSION OF THE RUBBER

Marek PÖSCHL*, **Radek STOČEK**, **Ondřej KRATINA**, **Martin STĚNIČKA** and **Petr ZÁDRAPA**

Centre of Polymer Systems, Tomas Bata University in Zlín, tř. Tomáše Bati 5678, 76001 Zlín, Czech Republic
email: stoček@utb.cz

This study investigates how different types of carbon black (CB) and mechanical loading conditions influence the thermal expansion and fatigue behavior of styrene-butadiene rubber (SBR)-based compounds. Special attention is given to how static compressive loading impacts thermal expansion, with the aim of identifying early indicators of structural damage within the rubber matrix. Five grades of carbon black N115, N220, N330, N550, and N772 were incorporated into the rubber compounds and compared with an unfilled SBR reference. Fatigue properties were characterized using an Intrinsic Strength Analyzer (ISA), which provided two key parameters: the **intrinsic strength** (T_0), representing the fatigue threshold, and the **ultimate strength** (Tc), corresponding to the critical failure point[1,2,3]. These parameters are essential for predicting the service life and crack resistance of rubber materials. Thermal expansion was evaluated by measuring the **coefficient of thermal expansion (CTE)** in two states: (1) unloaded specimens and (2) specimens subjected to 50% static compressive deformation for 24 hours.

The results reveal a clear load dependency of thermal expansion. In all cases, loaded samples showed higher CTE than their unloaded counterparts. Notably, the unfilled SBR displayed the highest CTE overall. For unloaded samples, CTE increased with carbon black particle size up to N550, then slightly decreased with N772. However, in loaded samples, CTE decreased consistently as CB particle size increased. Fatigue testing revealed that unfilled materials exhibited the poorest crack resistance. Among filled compounds, crack resistance decreased with increasing CB particle size up to N550, then improved with N772. Importantly, a strong inverse relationship between crack resistance and CTE was observed: materials with better fatigue performance exhibited lower thermal expansion, and vice versa.

These findings highlight the significant role of both mechanical loading and CB type on the thermal and mechanical behavior of rubber. The study demonstrates that CTE is not only sensitive to material composition but also a useful indicator of structural integrity under load. This underscores the importance of considering thermal expansion in the design and evaluation of rubber components, especially for applications where mechanical stresses are unavoidable.

Acknowledgement

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References

1. Schieppati, J., Schrittesser, B., Wondracek, A., Robin, S., Holzner, A., & Pinter, G. (2021). Effect of mechanical loading history on fatigue crack growth of non-crystallizing rubber. Engineering Fracture Mechanics, 257, 108010. https://doi.org/10.1016/j.engfracmech.2021.10801
2. Mull, M. A., Othman, A., & Mohammad, L. (2004). Fatigue crack propagation Analysis of Chemically Modified Crumb Rubber–Asphalt mixtures. Journal of Elastomers & Plastics, 37(1), 73–87. https://doi.org/10.1177/0095244305049898
3. Stoček, R., Stěnička, M., & Zádrapa, P. (2020). Future trends in predicting the complex fracture behaviour of rubber materials. Continuum Mechanics and Thermodynamics, 33(2), 291–305. https://doi.org/10.1007/s00161-020-00887-z

RUBBER VALLEY PUCHOV & OTROKOVICE, MATERIAL DEVELOPMENT

Zdeno PLEVÁK* **and** **Juraj BELOBRAD***
email: zdeno.plevak@conti.sk, juraj.belobrad@conti.sk

Pyrolysis Process

Obtaining Recovered Carbon Black (rCB)

- Solid tires from **Continental´s** tire plant in Korbach since September 2023 contain recovered carbon black Pyrum rCB
- By 2030 we aim to have over 40 renewable and recycled materials in our tires

Source:
https://www.continental.com/en/press/press-releases/20230912-solid-tires/

STATE OF PLASTICS LEGISLATION IN THE EU / SMART PRODUCTION CELL

Juraj MAJERSKÝ

Slovenský plastikársky klaster, Novozámocká 117/222, Nitra-Dolné Krškany, Slovak Republic, 949 05
email: juraj.majersky@wittmann-group.sk

The first part of the contribution is a review of the currently used and upcoming EU legislation related to plastics. The first document presented is Directive 2008/98/EC as amended, known as the Waste Framework Directive. It primarily addresses plastics through the waste management hierarchy, which prioritizes waste prevention, reuse, recycling, other recovery (e.g., energy recovery), and disposal. The directive sets binding recycling targets that Member States are required to achieve for various waste streams, including plastics. These targets are regularly updated to increase recycling rates and reduce the amount of waste destined for disposal. The directive gradually restricts landfilling of waste, especially of waste that can be recycled or recovered for energy, which includes plastics. For certain product groups, the directive introduces extended producer responsibility (EPR) (e.g., packaging, batteries, accumulators, vehicles, electronic devices). This system ensures that producers contribute to the costs associated with the collection, recycling, and disposal of their products at the end of their life cycle.

Directive (EU) 2019/904 of the European Parliament and of the Council of 5 June 2019, also known as the Single-Use Plastics Directive, introduces binding reuse targets for packaging for the years 2025, 2030, and 2040. By 2030, at least 40% of transport and consumer packaging and 10% of grouped packaging should be reusable. The rules restrict or ban the use of single-use plastic packaging, such as packaging for pre-packaged fruits and vegetables weighing less than 1.5 kg, food and beverage containers served in restaurants, small single-use cosmetic containers, and very lightweight plastic bags. Packaging must also be designed to use the minimum amount of material necessary to protect the product, thus preventing unnecessary packaging use. New packaging must contain a certain minimum percentage of recycled materials. By 2040, single-use plastic bottles must contain at least 65% recycled content. The presence of substances that could negatively affect recyclability or reusability of packaging, including per- and polyfluoroalkyl substances (PFAS), must be limited.

The Packaging and Packaging Waste Regulation will take effect on 12 August 2026, with Article 67(5) applying from 12 February 2029. According to this regulation, packaging must be at least 70% recyclable, assessed based on design, with gradual inclusion of sorting and recycling possibilities in existing facilities. The regulation introduces recyclability classes: Class A (95% and above), Class B (80% and above), and Class C (70% and above). Specified plastic packaging will be required to contain a mandatory proportion of recycled content. Some packaging types, such as fruit stickers or tea/coffee bags, must be compostable. Individual EU Member States may extend this list, for example, to include lightweight and very lightweight plastic bags. The regulation bans single-use plastic packaging, including multipacks, small portion containers for fruits and vegetables, food and drink packaging consumed directly in food service establishments, small cosmetic and toiletry products in hotels, and plastic bags for loose food items.

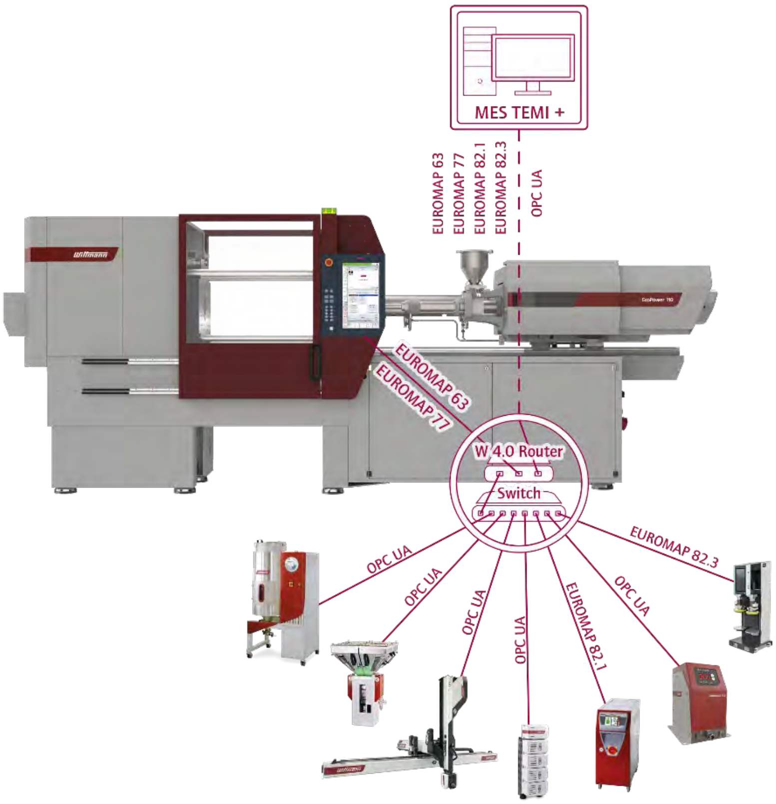
The regulation sets reuse targets for packaging, aiming for 100% reuse of most standard transport packaging by 2030. For packaging of alcoholic and non-alcoholic beverages, at least 10% of such products must be in reusable packaging. Retailers with a sales area over 400 m^2 must allocate 10% of their space for refill stations for both food and non-food products. Customers will have the option to take food and drinks in their own containers or in reusable packaging. The regulation also addresses "empty space" in packaging. In transport packaging, empty space should be limited to 50%, and to the minimum possible in consumer packaging. Empty space also includes material used to fill the packaging, such as air cushions or paper shreds.

The regulation sets targets to reduce the total amount of packaging waste per capita compared to 2018 levels, excluding waste from commercial and industrial packaging. Packaging labeling must be standardized and indicate material composition to facilitate proper waste sorting and increase recycling rates. The regulation introduces the concept of eco-modulated fees, meaning that producer fees will be based on the recyclability class of the packaging and may also consider the content of recycled material. These measures aim to support a circular economy, reduce the environmental impact of packaging, and increase the recycling rate of plastic packaging in the EU.

The Regulation on Preventing Plastic Pellet Loss, aimed at reducing microplastic pollution, is currently under interinstitutional negotiations. Its goal is to limit pellet leakage into the environment — according to estimates, between 52,140 and 184,290 tons of pellets (2,100–7,300 trucks) were lost in the EU in 2019. Current handling practices lead to losses at every stage of the supply chain. The regulation introduces less stringent requirements for small and medium-sized enterprises (with pellet consumption under 1,000 tons annually). Operations handling over 1,000 tons annually must perform an internal assessment each year along with additional measures under a risk assessment plan. This assessment can be integrated into an ISO 45001 audit if such a system is already in place. The regulation will introduce standardized methodologies for measuring pellet leakage. It will also impose monitoring obligations on pellet transporters and facilities that handle pellets.

The most recent document related to plastics is the Green Claims Directive. This directive focuses on B2C communication and is voluntary for businesses — it applies only to those that choose to label their goods/services with environmental claims. Its goal is to prevent greenwashing and protect honest manufacturers from misleading practices. The directive helps prevent abuse of environmental labels and consumer deception. Honest producers who use renewable and biodegradable materials will be protected against dishonest competitors who previously misused such labels for marketing purposes.

The second part of the contribution focuses on a smart manufacturing cell in the plastics industry — specifically in plastic injection molding. Currently, such a system is defined by customer demands for low acquisition and operating costs, a minimal number of defective parts, production using recyclates and regranulates, and the collection and evaluation of production data.



Scheme 1. Injection molding machine with peripheries – diagram of communication via router and communication protocols

The manufacturing cell is divided into several smaller components. The first and most important of these is the injection molding machine. It is primarily expected to meet requirements in terms of low energy consumption, precision, reliability, adequate equipment, and interfaces for connecting peripherals and MES systems. Another component consists of the peripherals — including manipulators (robots), flow meters, temperature control units, dosing devices, drying and conveying systems for granulates, as well as granulators for rejected parts and sprues. These peripherals are also expected to be energy-efficient, reliable, and equipped with interfaces for connection to the injection molding machine and the MES system.

Machine manufacturers have developed numerous systems that simplify production and assist with parameter settings — including adjustments in response to deviations, or modifications aimed at reducing energy consumption. Examples include:

- assistance with machine setup based on Moldflow analysis data
- centralized storage of machine and peripheral programs in one place (within the machine)
- unified data set transmission to the MES system
- Plug & Produce – automatic recognition of connected peripherals and evaluation of their compatibility
- system responses in case of incorrectly used or insufficiently specified peripherals
- automatic parameter input for peripherals when loading a program
- real-time parameter evaluation and online process optimization during the cycle

- automatic process evaluation and selection of optimal parameters for minimal energy consumption (robots, temperature control units, dryers and granulate conveyors, granulators)
- condition monitoring of the machine with communication to technical support

In the near or more distant future, the introduction of new functionalities in plastic processing equipment is expected. The most significant of these is likely to be artificial intelligence. It should enable the following capabilities:

- automatic adjustment of optimal machine and peripheral parameters based on moldflow data, material datasheets, and changes in conditions within the production hall
- process optimization based on feedback from evaluation systems — such as peripheral devices used for assessing the quality of the molded part
- analysis of data from the MES system — including alerts on deviations and potential changes to machine and peripheral settings, prediction of product complaints, and even evaluation and identification of discrepancies based on received complaints, followed by "learning" of the evaluation system to recognize new types of defects

On the hardware side, manufacturers are actively developing and implementing injection units for processing PCR (post-consumer recycled) materials, which would not require complex recycling and regranulation processes. This would significantly reduce the carbon footprint of these materials.

Acknowledgement

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References

1. SLOV-LEX.SK. 2024. Nariadenie Európskeho parlamentu a Rady (EÚ) 2025/40 z 19. decembra 2024 o obaloch a odpade z obalov, o zmene nariadenia (EÚ) 2019/1020 a smernice (EÚ) 2019/904 a o zrušení smernice 94/62/ES (Text s významom pre EHP). [online] Available on the internet: https://www.slov-lex.sk/pravo-eu/56dceb32-04c5-46a2-895b-18626dbab350
2. EUR-LEX.EUROPA.EU, 2019. Smernica Európskeho parlamentu a Rady (EÚ) 2019/904 z 5. júna 2019 o znižovaní vplyvu určitých plastových výrobkov na životné prostredie (Text s významom pre EHP). [online] Available on the internet: https://eur-lex.europa.eu/legal-content/SK/TXT/?uri=CELEX:32019L0904

OPTIMISATION OF QUALITY PLASTIC PARTS

Miroslav KOLLÁR

Simul Plast, Na letisko 28, 05801 Poprad, Slovakia
email: simulplast@simulplast.sk

The basic advantage of injection moulded products is the combination of functional and appearance properties in a single production step. This put high demands on their designers as well as their manufacturers. Despite the existence of a large amount of literature, standards and experiences, there is always a detail that can reduce the mechanical or appearance properties of the finished part.

A great help to discover the causes of the problem is the use of CAE analysis, e.g. with Moldex3D software. The analytical elements of this system make it possible to monitor the behaviour of the product during and after manufacture.

Today's time is marked by AI solutions. Company CoreTech System Co., Ltd. the creator of Moldex3D CAE software has implemented an AI solution as an additional solution to classical DOE method based on the principle of successive iterations of calculations for the process of product defect optimization. AI evaluates the benefit and proposes the optimal solution.

In this item on simple product that has several defects in its design will be presented the effectivity of both methods. Uneven wall thickness 1.5 mm and 2 mm with stepwise connection. The thickness of ribs at the connection to the main wall is higher than the required ratio for sink marks visibility.

The first visible shortcoming is the sprue gate is located in the middle of the thin wall (1.5mm). This wall solidifies earlier than the perimeter walls, which are 2mm thick.

This combination of part and mould design lead to problems with part surface appearance. There are visible sink marks and deformation “Box effect”.

In this work was used classic Taguchi DOE method and new AI method to optimize the injection molding process for defect elimination or minimize. Parameters of both methods were set identical for better comparison of results and effectivity. By AI method is by the software Moldex3D recommended to use optimal 30 iterations. It was tested the precision also by faster optimization where were use only 20 or 10 iterations respectively.

This study has evaluated two different ways of process optimisation. Both methods DOE and AI are reaching the same results but through different approach and time consumption

Both methods offer results view in Parallel Coordinates Plot, where is possible choose the optimal process setting and also the influence of every parameter on controlled quality factor. By DOE is possible by moving points explore other setup alternatives.

Restriction of DOE is in setting parameters values only in specific level from narrow interval. The calculation is going strictly on these levels. Therefore, the results for other parameters values are only calculated.

DOE - Stable, time-tested optimization method. It requires the best possible definition of control parameters as well as the range of their changes. With less than 6 control parameters and max. 3 levels, it is faster than AI optimization in Moldex3D software.

AI optimization - uses a system of intelligent comparison of calculation results with targeted generation of new calculations to optimize the process. Parameters chosen for calculation are chosen from prescribed interval and don’t have only level value as is in DOE method. The time required depends on the number of iterations the user chooses. Unlike DOE, it more easily handles a higher number of control parameters as well as a larger range of their changes. In principle, 20 iterations are sufficient but 30 is the optimal number.

References

1. Beaumont J.P., Runner and Gating Design Handbook, 512 (2020).
2. Gunther E., Design with plastics, 517 (2006).
3. Malloy Robert A., Plastic Part Design for Injection Molding, 549 (2011)

COMPUTATIONAL MODELLING AND HIGH PERFORMANCE COMPUTING DRIVEN INNOVATION IN THE DEVELOPMENT OF POLYMERIC MATERIALS IN THE CONTEXT OF THE EUROPEAN COMPETENCE CENTRE FRAMEWORK.

Lucia MALICKOVA

Slovak National Supercomputing Centre, Bratislava, Slovakia
email: lucia.malickova@slovakianscc.com

Abstract

The increasing complexity of polymeric materials and rubber-based components in the automotive sector requires advanced digital tools to support innovation, efficiency, and sustainability. High Performance Computing (HPC) has emerged as a key enabler of predictive modelling, material testing, and product optimization. This article explores the role of HPC-driven innovation in polymer development through an analytico-synthetic research method based on secondary data analysis within the EuroCC Competence Centre framework. Two Slovak success stories are analysed: optimization of CFD workflows in HPC environments, and AI-driven structural parameter analysis of capsule-based products. Although originally developed for different sectors, these methodologies offer valuable insights and transferable approaches for modelling, processing, and evaluating polymeric materials used in automotive applications. The study underscores the strategic potential of HPC and artificial intelligence as tools supporting cross-sectoral material innovation, aligned with current challenges in circular economy, performance design, and advanced manufacturing processes.

Introduction

The automotive industry is undergoing a fundamental transformation driven by increasing demands for lightweight construction, sustainability, advanced functionality, and electrification. Polymeric and rubber-based materials play a critical role in addressing these challenges, from structural applications in vehicle exteriors to complex components in interiors, sealing systems, and battery housings. As the complexity and performance requirements of these materials grow, traditional experimental approaches are no longer sufficient to ensure rapid development cycles and optimized design. High Performance Computing (HPC) offers an advanced infrastructure for computational modelling, numerical simulation, and data-driven decision-making across the material development pipeline. From predicting flow and curing behaviour in thermoplastic elastomers to simulating mechanical responses under various operational loads, HPC enables a new level of precision and efficiency in designing materials with tailored properties. To make these capabilities accessible to a broader ecosystem of researchers and businesses, the European Union launched the EuroCC initiative, which established a network of National Competence Centres (NCCs) for HPC across Europe. The Slovak National Supercomputing Centre (NSCC) supports industrial and academic stakeholders by facilitating access to supercomputing infrastructure, expertise, and applied methodologies. This article analyses two success stories from the NSCC to illustrate how computational workflows developed in other domains can be effectively transferred to polymer science and automotive applications. By examining the optimization of computational fluid dynamics (CFD) simulations and the application of artificial intelligence (AI) in structural analysis, the study identifies key enablers and replicable techniques that align with the strategic goals of HPC-enabled innovation in polymeric materials.

Methodology

This study adopts an analytic-synthetic approach, grounded in qualitative research methodology and based on secondary analysis of publicly available technical documentation and success stories produced within the European network of EuroCC. Specifically, the paper focuses on two Slovak case studies facilitated by the NSCC. Although the original contexts of these projects—computational fluid dynamics (CFD) optimization and AI-based structural analysis in pharmaceutical applications—do not directly address polymeric materials, their methodologies and digital workflows exhibit a high degree of transferability to the polymer and rubber domains in automotive manufacturing.

The research method comprises the following stages:

Data collection: Technical content was sourced from official NSCC publications, EuroCC Slovakia web documentation, and project deliverables associated with the EuroHPC Joint Undertaking. Priority was given to projects that implemented scalable HPC solutions, machine learning models, and complex simulations with direct implications for materials development and evaluation.

Analytical processing: A systematic breakdown of each success story was performed to identify computational frameworks, simulation workflows, pre- and post-processing techniques, and the integration of AI/ML tools. The emphasis was placed on technological generalizability, automation potential, and scalability of the methods used.

Comparative analysis: Both case studies were evaluated with respect to their methodological structures, innovation mechanisms, and compatibility with typical requirements in polymer processing and material performance testing. Similarities in data handling, simulation objectives, and computational strategies were mapped to draw conceptual analogies with polymeric material research challenges.

Abstraction and synthesis: Insights from both projects were synthesized into a generalized understanding of how HPC and AI can be leveraged to support innovation in polymeric materials. Key transferable concepts—such as accelerated simulation cycles, image-based structural recognition, and parallel workflow orchestration—are discussed in the context of their potential applications in automotive design and polymer testing environments.

The methodological framework enables the integration of interdisciplinary approaches, combining insights from computational sciences, data engineering, and material technology, which reflects the hybrid nature of modern polymeric research and the strategic goals of the EuroCC initiative.

Results and Discussion

The analysis of the selected success stories from the NSCC reveals two distinct yet methodologically aligned applications of high-performance computing (HPC) and artificial intelligence (AI) with direct relevance to polymeric materials development. Both implementations demonstrate core HPC principles such as workflow optimization, parallel computing, and integration of AI/ML techniques—approaches that are readily transferable to research and industrial processes in the automotive polymer sector.

Optimization of CFD Workflows in HPC Environments and its Transferability to Polymer Processing

Computational Fluid Dynamics (CFD) simulations play an essential role in understanding the behavior of complex materials during flow, heating, cooling, and deformation processes. The success story from NSCC demonstrated the migration and optimization of a CFD simulation workflow onto HPC infrastructure, resulting in significantly reduced computational time and improved efficiency across multiple parameters such as meshing complexity, solver stability, and parallel processing performance[1]. While the original project focused on fluid simulations for non-polymeric applications, the parallels with automotive polymer processing are evident, particularly in:

- *Injection molding*, where accurate flow simulation is critical for determining fill patterns, identifying weld lines, evaluating shrinkage and warpage, and predicting potential defects such as voids or sink marks.
- *Extrusion and calandering*, where pressure distribution, shear rates, and temperature profiles affect product consistency, layer adhesion, and dimensional stability.
- *Multimaterial and 2K systems*, which demand simultaneous modelling of materials with different thermal and rheological properties.

HPC provides the computational capacity required to run fine-grained simulations with high temporal and spatial resolution. For instance, solving the Navier-Stokes equations in complex geometries for non-Newtonian fluids typical in polymer melts is computationally expensive but achievable in real-time scenarios only through scalable HPC resources. Moreover, by integrating CFD simulations into an automated HPC workflow, several advantages are gained:

- *Automated parameter studies and design of experiments* (DoE) become feasible, reducing human error and speeding up optimization loops.
- *Parallel simulation execution* allows for simultaneous testing of multiple design configurations or material variants.
- *Post-processing via scripted data analytics* enables fast extraction of critical quality parameters as seen on Figure 1 (e.g., pressure, velocity, viscosity gradients).

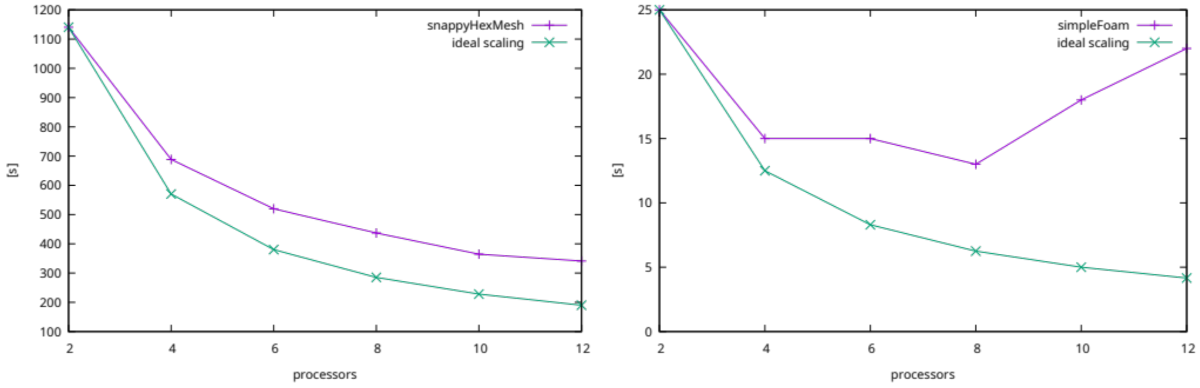

Figure 1. Comparison of computation times and scalability before and after HPC optimization of CFD simulations. The optimized version shows significant improvement in runtime and parallel efficiency. Source: Adapted from NSCC Slovakia, 2023.

In polymer research, these computational advantages enable more effective virtual prototyping, thereby shortening time-to-market while improving performance predictability and process reliability. Particularly in the development of new thermoplastic elastomers or bio-based polymers, such CFD-driven HPC workflows can identify optimal processing windows and avoid costly trial-and-error in pilot lines (Fig. 2).

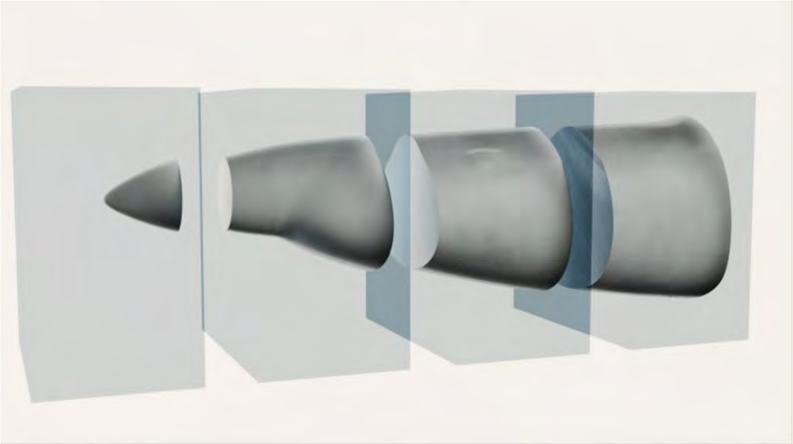

Figure 2. Visual representation of flow geometry reconstructed during HPC-based simulation phases. This type of spatial domain segmentation supports improved solver accuracy and flow behavior prediction. Source: Adapted from NSCC Slovakia, 2023.

In summary, the methodological approach used in this NSCC project has the potential to become a backbone technology for future-oriented polymer processing innovation, especially as sustainability demands precise control over energy input, waste generation, and recyclability of materials.

AI-Based Structural Analysis and its Application in Rubber and Polymeric Testing

The second success story provided by the NSCC demonstrates the application of artificial intelligence (AI) and machine learning (ML) for the structural analysis of pharmaceutical capsules[2]. Although developed for pharmaceutical purposes, the methodology is transferable to the testing of rubber and polymer-based components used in the automotive sector. The process combines optical microscopy, neural network-driven object detection, segmentation techniques, and geometric analysis to provide high-precision measurements of small, complex structures.

The procedure begins with high-resolution input images captured through optical microscopy at magnifications of 2.5x and 4x. These images serve as the base for detecting capsules using trained neural networks, specifically the YOLOv5 architecture[3], trained on annotated datasets similar to COCO128[4], and prepared using the Labeling too[5]. The first stage of the analysis involves identifying and extracting capsules from images Fig. 3.

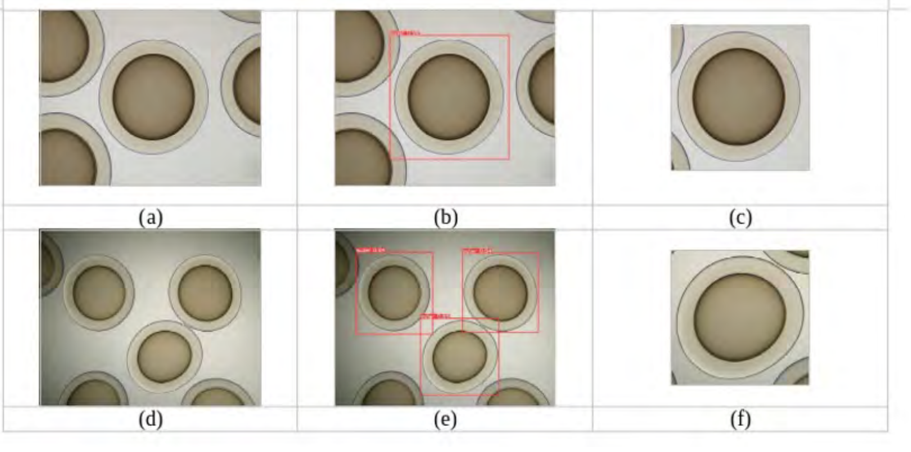

Figure 3. (a) Input image from optical microscopy; (b) detected capsule; (c) magnified region of the detected capsule at 4x; (d) another input image; (e) detected capsule; (f) magnified region at 2.5x. Source: Adapted from NSCC Slovakia, 2023.

Following detection, segmentation is carried out using the U-Net convolutional network[6], which classifies each pixel to separate the capsule's interior and exterior regions. Binary masks are created and enhanced using image pre-processing techniques, such as Retinex correction[7] and binary hole-filling algorithms from the SciPy library[8]. These masks are then analyzed through watershed segmentation and shape measurement methods available in the Scikit-Image package[9,10]. The segmentation workflow is shown in Fig. 4.

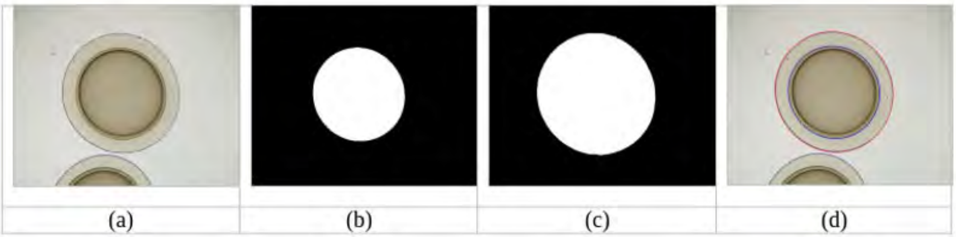
(a) (b) (c) (d)

Figure 4: (a) Input image; (b) inner binary mask; (c) outer binary mask; (d) output image with fitted ellipses derived from binary masks. Source: Adapted from NSCC Slovakia, 2023.

The final stage involves the geometric analysis of fitted ellipses to determine dimensional deviations. The ellipses represent the outer contours of the capsules and are evaluated based on the primary and secondary axes. The results compare U-Net and Retinex-based segmentation approaches. Tolerance thresholds set by the Slovak Academy of Sciences (SAS) are used as reference limits. Differences in axis measurements are shown in Fig. 5, where deviations beyond the red line indicate unacceptable tolerances.

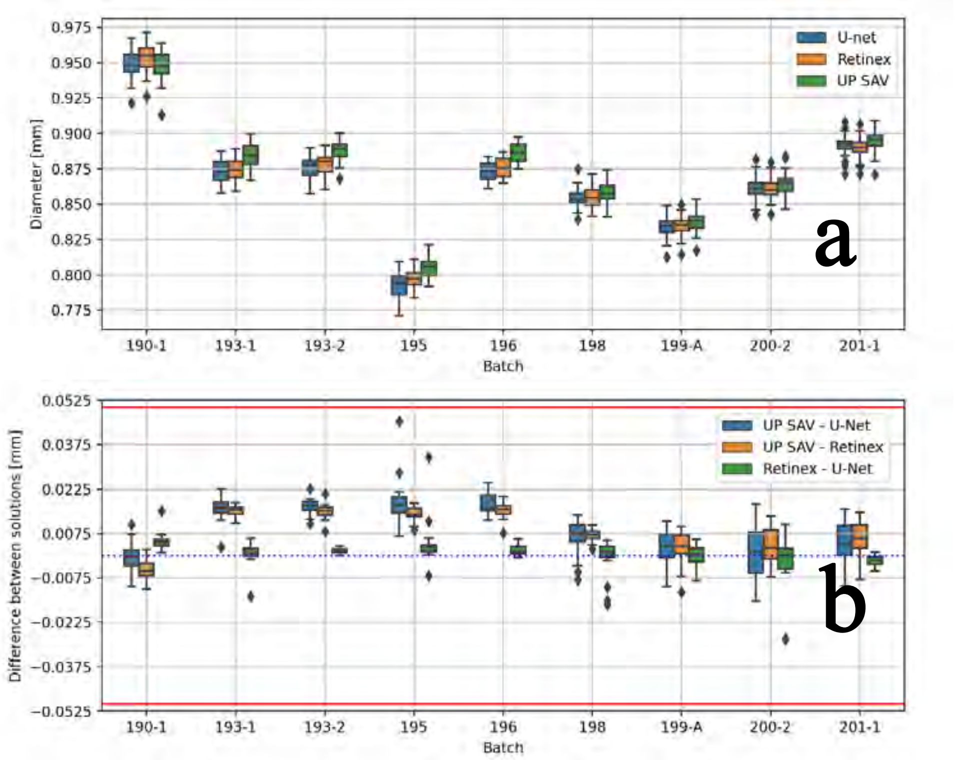

Figure 5. (a) Capsule dimensions based on U-Net and Retinex methods, using average of the major and minor axes; (b) difference in the major axis of ellipses for outer diameter. Red line indicates the maximum acceptable deviation defined by SAS. Images obtained at 4x magnification. Source: Adapted from NSCC Slovakia, 2023.

This workflow demonstrates high reproducibility, objectivity, and scalability—features that can be applied to non-destructive testing of automotive components such as gaskets, elastomeric bushings, and flexible seals. The combination of AI detection, high-resolution image segmentation, and geometric analysis offers a powerful approach for quality control and predictive maintenance in polymer and rubber part manufacturing[11].

Moreover, when supported by HPC resources, the time needed for AI training and image processing is significantly reduced, enabling deployment in industrial environments with high throughput. These technologies allow automotive suppliers to implement automated inspection and traceability protocols in line with Industry 4.0 and smart manufacturing initiatives.

Conclusion

The accelerating transformation of the automotive industry has led to the emergence of advanced requirements for materials used in both structural and functional applications. Polymeric and rubber materials are at the forefront of this shift, enabling lightweighting, improved durability, and better performance in demanding environments. However, the development of such materials demands rapid prototyping, accurate prediction of mechanical behaviour, and robust quality control systems, all of which can be significantly enhanced using high-performance computing (HPC) and artificial intelligence (AI).

This article presented an analytic-synthetic evaluation of two selected success stories from the NSCC, which were contextualized and reinterpreted for their relevance in polymer and rubber materials development for the automotive sector. Both cases showcased how digital innovation, by HPC infrastructure and AI techniques, can support the design, simulation, evaluation, and continuous improvement of polymer-based products in a highly competitive and quality-sensitive industry.

In the first case, CFD simulation workflows were optimized by transferring them to an HPC environment. The resulting increase in computational efficiency, reduction in time-to-solution, and ability to run high-resolution simulations across multiple CPUs demonstrate the value of HPC in modeling processes such as injection molding, extrusion, and thermoforming. These processing technologies are critical in shaping polymeric components with strict performance specifications. When applied to polymer research, these HPC-enabled methods allow for more accurate simulations, more extensive design-of-experiment loops, and improved understanding of material flow and behavior under stress.

In the second case, AI-driven structural analysis originally developed for pharmaceutical capsules was reinterpreted for polymeric component testing. Using optical microscopy, deep learning models (such as YOLOv5 and U-Net), and geometric measurement methods, the approach demonstrated high precision in detecting structural deformations and dimensional anomalies. This methodology holds significant promise for automotive applications involving non-destructive testing (NDT), automated quality control, and predictive maintenance of elastomeric components such as seals, gaskets, and flexible couplings. By leveraging HPC for training complex AI models, the time required for model convergence can be dramatically shortened, enabling industrial deployment at scale.

Both case studies underscore the broader impact of the EuroCC initiative and the role of national competence centers like NSCC in supporting digital transformation across sectors. The approaches described are aligned with the thematic focus of the International Conference on Polymeric Materials in Automotive and the Slovak Rubber Conference, particularly in the areas of computational modelling, prediction, material testing, advanced processing, and sustainability.

Furthermore, the insights gained in this article contribute to the research on the role of HPC in enabling innovation, research, and entrepreneurship in academic and industrial ecosystems. The practical implications of these success stories support the integration of HPC as a strategic tool for polymer material innovation, helping to bridge the gap between research and industrial application, and reinforcing the value of cross-sector collaboration in the digital age.

In conclusion, the convergence of HPC, AI, and materials science presents a compelling opportunity for the automotive polymer industry to accelerate its innovation cycles, enhance product reliability, and meet increasing regulatory and environmental expectations. The integration of these technologies is not a matter of future potential, but a present-day necessity for maintaining global competitiveness and fostering sustainable development.

Acknowledgement

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References

1. EuroCC Slovakia: Prenos a optimalizácia pracovného toku CFD výpočtov v HPC prostredí. https://eurocc.nscc.sk/use-case-prenos-a-optimalizacia-pracovneho-toku-cfd-vypoctov-v-hpc-prostredi/ (2023).
2. EuroCC Slovakia: Meranie štrukturálnych parametrov kapsúl použitím techník umelej inteligencie (AI) a strojového učenia (ML). https://eurocc.nscc.sk/meranie-strukturalnych-parametrov-kapsul-pouzitim-technik-umelej-inteligencie-ai-a-strojoveho-ucenia-ml/ (2023).
3. Ultralytics: YOLOv5 repository. https://github.com/ultralytics/yolov5 (2023).
4. Ultralytics: COCO128 dataset. https://www.kaggle.com/ultralytics/coco128 (2023).
5. Heartex: LabelImg annotation tool. https://github.com/heartexlabs/labelImg (2023).
6. Ronneberger, O., Fischer, P., Brox, T.: U-Net: Convolutional Networks for Biomedical Image Segmentation. https://lmb.informatik.uni-freiburg.de/people/ronneber/u-net/ (2015).
7. Jobson, D.J., Rahman, Z., Woodell, G.A.: A Multiscale Retinex for Bridging the Gap Between Color Images and the Human Observation of Scenes. IEEE Transactions on Image Processing **6**(7), 965–976 (1997).
8. SciPy Documentation: binary_fill_holes. https://docs.scipy.org/doc/scipy/reference/generated/scipy.ndimage.binary_fill_holes.html (2023).
9. scikit-image: Watershed segmentation example. https://scikit-image.org/docs/stable/auto_examples/segmentation/plot_watershed.html (2023).
10. scikit-image: Shape measurement API. https://scikit-image.org/docs/stable/api/skimage.measure.html (2023).
11. Richardson, S. M., et al.: AI-assisted Material Testing and Predictive Modelling. Springer (2021).

COLOUR STABILITY OF PACKAGING MATERIALS

Mária PETKOVÁ[1]*, Marcela HRICOVÁ[1], Jozef RYBA[1], Anna UJHELYIOVÁ[1] and Zita TOMČÍKOVÁ[2]

[1]*Slovak University of Technology in Bratislava, Faculty of Chemical and Food Technology, Department of Plastics, Rubber and Fibres, Radlinského 9, 812 37 Bratislava, Slovakia*
[2]*Research Institute for Man-Made Fibres a.s, Štúrova 2, 05921 Svit, Slovakia*
email: maria.petkova@stuba.sk

This work presents the results of evaluating the colouristic properties of biodegradable packaging films. The films were prepared from Nonoilen, which is a commercial blend of PLA/PHB. Our applied research is focused on coloured masterbatches using natural pigments to prepare films as packaging material for foods. The natural pigments are extracted from plants and food pigments. The dyed Nonoilen films in the mass were prepared from colour masterbatches, using natural pigments such as chlorophyll, caramel, and violets. The properties of the pure and dyed films were investigated before and after accelerated light aging using Q-SUN equipment. The changes in colorimetric properties were recorded using a device spectrometer from TECHKON SpectroDens. Natural pigments showed sufficient colourability after preparation, although the colouration was lost relatively quickly after accelerated light aging. If they were used as food packaging materials, these pigments would be highly safe, besides being biodegradable.

Synthetic plastics have good properties as mechanical stability, low cost, and oxygen barrier, making them suitable for packaging products during storage, transportation, and handling. Nowadays, there is a growing demand for solutions to the ecological situation. As a large amount of synthetic plastic waste is generated, petroleum resources are also limited, and the ability to recycle synthetic polymers is still limited, the demand for biodegradable packaging materials to replace them is increasing [1, 2]. Demand for bioplastics has been increasing in recent years, as we can see in research. Many research studies are devoted to these polymers because they are not only biodegradable plastics, but they are also made from renewable sources [3]. A certain part of the research is devoted to finding possible substitute materials. Preparing mixtures to improve the properties of biodegradable materials to achieve the best mechanical, thermomechanical, and other properties. However, it is necessary to make this material visually interesting for more attractive marketing. Therefore, we are trying to find ecological pigments to preserve the material's biodegradability. There are inorganic and organic materials that meet this requirement[4-5]. But we focused on colouring with natural pigments, so that it is the least harmful material. So that during the storage and protection of food, no contact with chemicals could lead to health risks.

EXPERIMENTAL PART

Materials used:

Polymer: Nonoilen IM 3056-2 (NOil) is an injection moulding grade of a PLA/PHB-based blend (Panara Ltd., Slovakia),

Pigments: Caramel; Chlorophyll; Violet,

Dispergation agent: Licocare RBW 102 Vita (Clariant).

Colour masterbatches and films preparation:

For the preparation of color PLA masterbatches were used by a laboratory line with twin - screw extruder WP ZDSK. Melting was performed on the screw extruder with a diameter of 28 mm, in the temperature range of 180 – 210°C. The extruder string of PLA was cooled and pelletized. The concentration of food pigments in the masterbatches was 3 wt.%, and the dispersing agent was contained in 0.6 wt.%.

In the next step, the pellet of pure NOil and colour masterbatches were dried in a laboratory oven for 3 hours at 60°C. Subsequently, blends of pure NOil and a specified quantity of colour masterbatches with selected pigments were mixed so that we obtained dyed films with 0.1 and 1.5 wt.% pigment in the films. The cast films were made from the prepared masterbatches. The cast films were prepared on a laboratory line of Plasticizers with an extruder D=18 mm and a slot width of 48 mm.

Method used:

Colorimetry deals with the measurement and characterization of colours. Each colour can be expressed in the CIELab in colorimetric trichromatic coordinates (L, a, b) were obtained according to ISO 12647–2:2013. L is the parameter of specific lightness, and it is the position between black and white on the axis of colour space. Parameter a determines colours from green to red, and parameter b characterizes colours from blue to yellow. The total colour difference of two colours is defined based on the colour difference of the sample and the CIE 1976 standard (ab) ΔE. The colour deviation can also characterize the effect of thermal-light aging of the dyed sample. The total color difference ΔE was calculated from the Equation. (1).

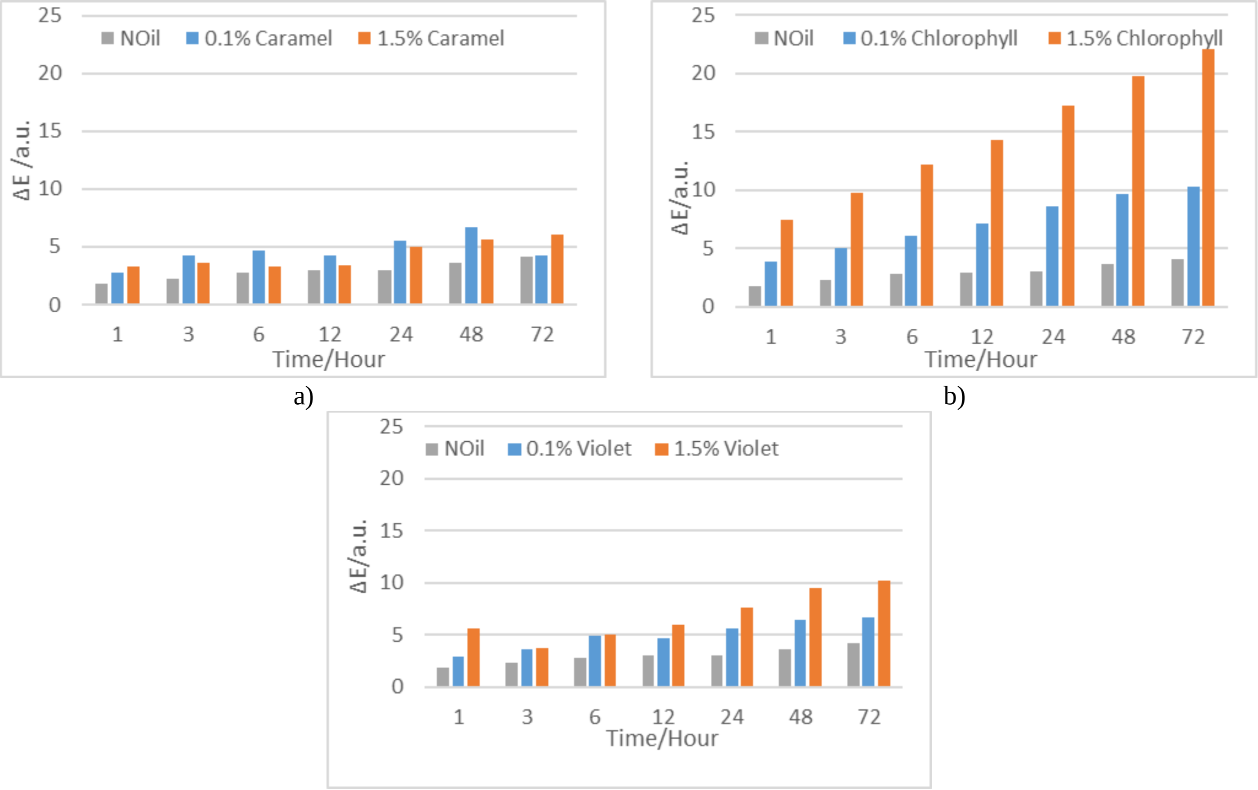
$$\boldsymbol{\Delta E = (\Delta L)^2 + \Delta a^2 + \Delta b^2} \qquad (1)$$

The accelerated light aging was performed with the intensity of radiation 300-800 nm approximately. 494 $W.m^{-2}$ in the Q-SUN Xe-1-S chamber (Q-LAB, Westlake, OH, USA), in which temperature was regulated at 65 °C (black panel). The samples were evaluated before and after aging. 72 hours of accelerated light-heat aging corresponds to approximately 2.3 years of real external aging. The aging period corresponds to continuous exposure of materials to conditions of increased temperature, and intense UV radiation and visible light (midday sunlight). Therefore, the real, total time of use of the materials exceeds this period of calculated laboratory aging.

The coloristic properties of the dyed prepared films were evaluated before and after thermal-light aging using the TECHKON SpectroDens instrument with settings of illumination type D65, standard observer 10°, and absolute white standard. Before thermal-light aging, the parameters L, a, and b were determined. Subsequently, the films were placed in the Q-sun instrument for thermal-light aging. At time intervals of 1 hour and 3, 6, 12, 18, 24, 48, and 72 hours, samples were removed from the chamber, and CIELab coordinates were measured again.

RESULTS AND DISCUSSION

The colorimetric coordinates of three-dimensional space define the relationships between colours. The colour space is commonly evaluated by CIELab for colour evaluation. With the help of coordinates, the colour difference between two colours can be mathematically defined and characterized by the total colour difference. The colour difference is the measurement of agreement or discrepancy between the two evaluated colours.



a)

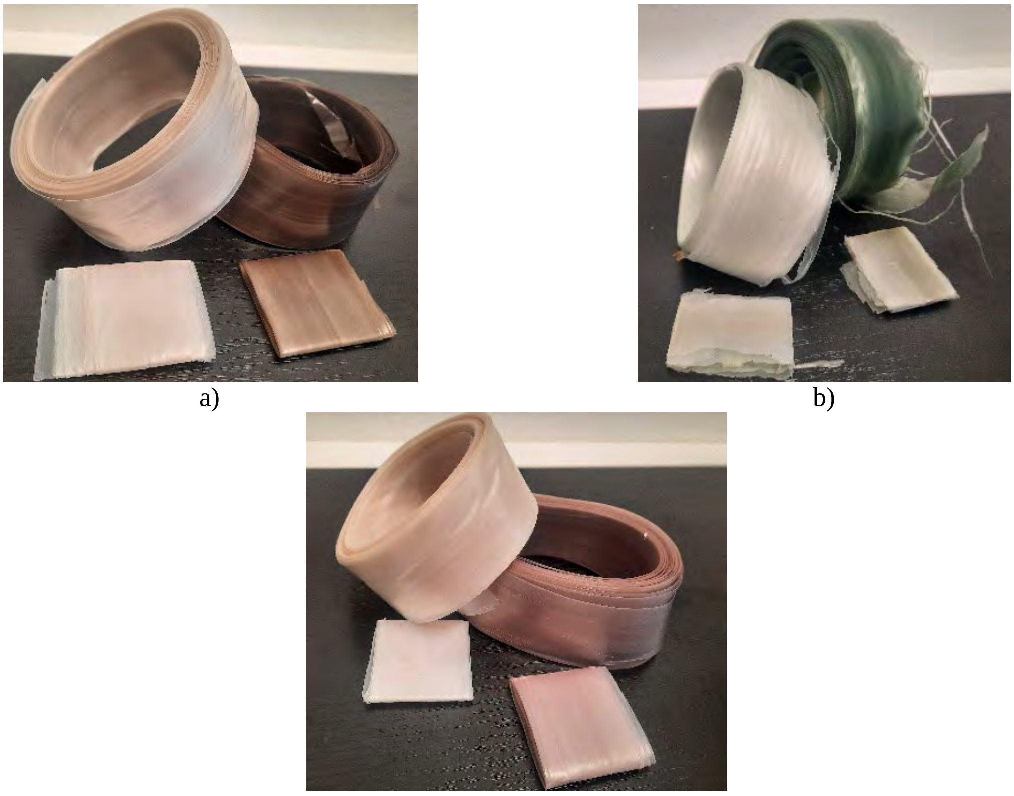
b)

c)

Figure 1. The total color difference of NOil/ color NOil films depending on the aging time for different pigments a) Caramel, b) Chlorophyll, and c) Violet

The evaluation of the total colour difference (Figure 1) from the point of view of the three types of food pigments manifests itself very similar way. It can be shown that there has been an increasing colour change since the onset of aging. A greater increase in the colour difference occurred after 6 hours of aging, and this difference did not significantly increase until the end of the observed aging (72 h). We can observe this process with the pigments Caramel and Violet. With Chlorophyll, we can observe the increase in colour change throughout the aging process with quite significant shifts. Especially with a higher concentration of the dye. We can see the difference even when comparing one pigment at different concentrations of this pigment in the film. The total colour difference reaches higher values at 1.5% concentration. The point is that better dyeing intensity in films was achieved during preparation, but the pigment lost its colour due to aging, which ultimately led to a greater total colour change.

I also declare these claims in Figure 2, where there are photographs of the prepared films and the films subjected to accelerated aging. The changes achieved can be seen with the naked eye. The lighter film has a pigment content of 0.1% by wt., and the darker film contains 1.5% wt. of pigment.



a) b)

c)

Figure 2. Photographs of the films before and after being subjected to accelerated aging for different food pigments and two concentrates of the pigment: a) Caramel, b) Chlorophyll, and c) Violet

CONCLUSION

Food pigments are very safe and harmless to health. Although in this work, we can see that the intensity of colouring achieved during the preparation of the films was not maintained after exposing these films to accelerated aging. In practice, for short-term storage of fresh foods, the short duration of colouring would not be an obstacle.

Acknowledgement

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References

1. Sani M.A., Khezerlou A., Tavassoli M., Abedini A.H., McClements D.J.: Development of sustainable UV-screening food packaging materials: A review of recent advances. In Trends in Food Science & Technology. *145*, 104366 (2024).
2. Ezati P., Khan A., Priyadarshi R., Bhattacharya T., Tammina S.K., Rhim J.-W.: Biopolymer-based UV protection functional films for food packaging. In Food Hydrocolloids. 142, 108771 (2023).
3. Zhang W., Sani M.A., Zhang Z., McClements D.J., Jafari S.M.: High performance biopolymeric packaging films containing zinc oxide nanoparticles for fresh food preservation: A review. In International Journal of Biological Macromolecule. *230*, 123188 (2023).
4. Vizárová K., Reháková M., Kirschnerová S., Peller A., Šimon P., Mikulášik R.: Stability studies of materials applied in the restoration of a baroque oil painting. In Journal of Cultural Heritage. 12 (2), 190-195 (2011).
5. Petková M., Jančovičová. V., Ujhelyiová A., Hricová M.: The coloristic properties of biodegradable fibers. In Fibers. 12, 59 (2024).

MASTERBATCHES FOR BIODEGRADABLE PACKAGING

Marcela HRICOVÁ[1]*, Mária PETKOVÁ[1], Zita TOMČÍKOVÁ[2] and Anna UJHELYIOVÁ[1]

[1]*Department of Plastics, Rubber and Fibres, Slovak University of Technology in Bratislava, Faculty of Chemical and Food Technology, Department of Plastics, Rubber and Fibres, Radlinského 9, 812 37 Bratislava, Slovakia*
[2]*Research Institute for Man-Made Fibres a.s., Štúrova 2, 05921 Svit, Slovakia*
email: marcela.hricova@stuba.sk

In this work, the influence of food pigments on the rheological properties and processability of biodegradable colour masterbatches was evaluated.

Waste, in all its forms, is one of the most pressing environmental issues of our time. As the world's population explodes, the amount of waste in oceans and landfills worldwide continues to rise. There is no one solution to the eco-crisis. It demands a multi-pronged approach, and using biodegradable packaging is one from potential solution. Instead of damaging the environment, biodegradable packaging can become part of a circular economy models, where product life cycles are closed-looped, and waste is minimized. By re-direct biodegradable plastic packaging to compost, we can help to cultivate the soil to support farming and at the same time to eliminate waste. The transition to biodegradable packaging is part of a larger, vital shift towards sustainability that includes reducing waste, conserving resources, and safeguarding our planet for future generations. These materials represent a new philosophy in design that considers the entire lifecycle of a product. The using biodegradable packaging is step towards responsible production and consumption aligning with the health of the planet. The functionality of biodegradable packaging depends on the materials and additives used. The individual components should be non-toxic, allergy-free and mainly biodegradable. This also applies to pigments and dyes used to increase the attractiveness of packaging [1-3].

Colouring of biodegradable polymers requires careful selection of pigments that are in accordance with the European standard EN 13432. This standard defines the characteristics of a "compostable" material that can be processed by organic recovery (composting and anaerobic digestion).

Biodegradable colorants, made from natural vegetable sources, typically cost less and provide glaring colours. In the biodegradable materials sector, polylactic acid (PLA) offering an alternative to petroleum-based plastics. PLA is a bioplastic derived from renewable resources like corn starch or sugarcane. The using of PLA is versatile, from food packaging to biodegradable medical devices.

This article is focused on the evaluation of flow properties of colour masterbatches modified with biodegradable pigments.

EXPERIMENTAL PART

Materials used

Nonoilen IM 3056-2 (NOil), is an injection moulding grade of PLA/PHB based blend (Panara Ltd. Slovakia),
Pigments: *Caramel; Chlorophyll; Blue,*
Dispergation agent: Licocare RBW 102 Vita (Clariant).

Colour masterbatches preparation

The preparation of colour masterbatches was carried out on a melting-kneading double screw WP ZDSK D = 28 mm in the temperature range of 180-210°C, using a stationary dryer and a mixer Drais. The food pigments in quantities of 3 wt.% and dispersing agent with content of 0.6 wt.% were used in the preparation.

Method used

Rheological properties were measured on the rotary rheoviscosimeter PHYSICA MCR 101 with geometry plate/plate with a measuring body diameter of 25 mm in the range of shear speeds from 0.1 s^{-1} to 100 s^{-1} at 190°C.

RESULTS AND DISCUSSION

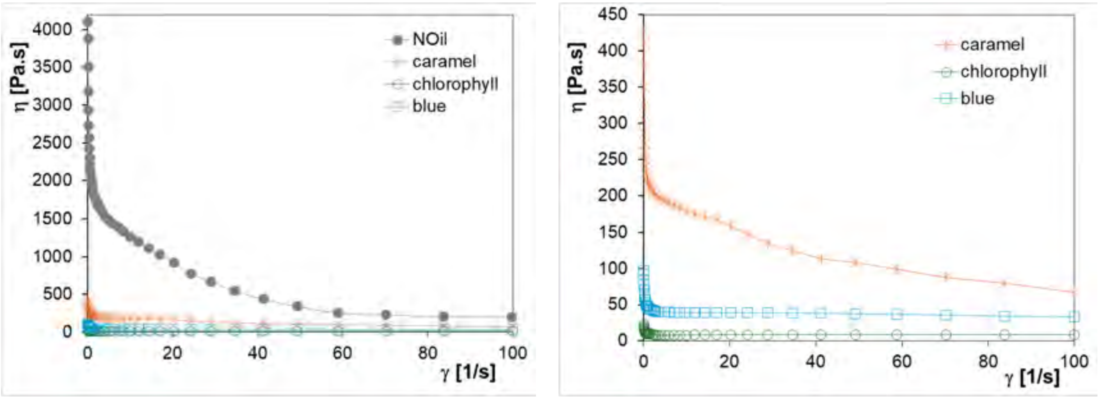
Based on the measurement of rheological properties of prepared colour masterbatches, it is possible to evaluate the processing stability of NOil masterbatches with food pigments.

Measurements of rheological properties were made on a rotary rheoviscometer using a rotary mode with a plate/plate measuring module. Dependences of viscosity and shear stress on shear rate were obtained. The dependences of the rheological values of the prepared colour masterbatches are showed on the Figures 1 and 2.

From the obtained results of rheological measurements, we can conclude that colour masterbatches behave as non-Newtonian pseudoplastic polymer systems. These systems are characterized by a decrease in viscosity with increasing shear rate, as we can see on Figure 1.

The viscosity of the pure *NOil* polymer decreased with increasing shear rate from 4100 Pa.s to 200 Pa.s. The colour masterbatches contain of 3 wt.% of food pigments and 0.6 wt.% of dispersant. All colour masterbatches show a much lower viscosity compared to pure *NOil* (Fig. 1a).

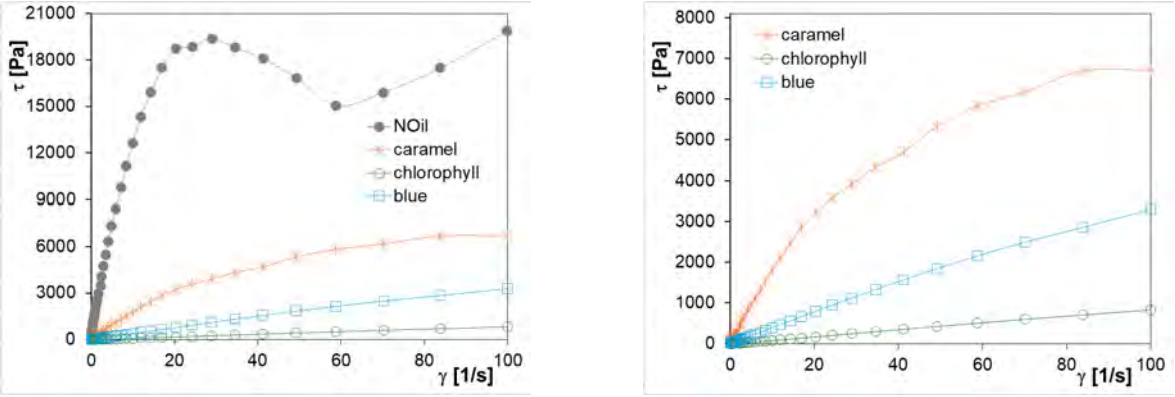
In colour masterbatches with food pigments, increasing shear rate results in a decrease in viscosity for all masterbatches (Fig. 1b).



a) b)

Figure 1. Dependence of viscosity η on shear rate γ for all samples (a) and detailed for pigments (b)

The viscosity of the masterbatch containing *Caramel* pigment decreased from 428 Pa.s to 67 Pa.s. The decrease in viscosity for the other two colour masterbatches was even more pronounced (Fig. 1b). The viscosity of the *NOil*/B*lue* masterbatch at low shear rates was 97 Pa.s and decreased to 32 Pa.s with increasing shear rate. The lowest values were shown by the colour masterbatch containing *Chlorophyll* (Fig. 1b). With increasing shear rate, its viscosity decreased from 22 Pa.s to 8 Pa.s. These viscosity values are already at the limit of the instrument's measurability.



a) b)

Figure 2. Dependence of shear stress τ on shear rate γ for all samples (a) and detailed for pigments (b)

The dependences of shear stress on shear rate showed similar tendencies as the dependences of viscosity on shear rate. The values of shear stress for all colour masterbatches were several times lower than for the pure polymer *NOil* (Fig. 2a). During rheological measurements, the melts of the masterbatches *NOil*/B*lue* and *NOil/Chlorophyll* essentially flowed, what indicated their worsened processability.

Based on the obtained data, we can conclude that not all masterbatches with food pigments can be used for further technological processing. The worst rheological behaviour was found for colour masterbatches with *Blue* and *Chlorophyll* pigments. These pigments can cause processing problems and therefore are not suitable for further use in the preparation of the biodegradable packaging materials.

CONCLUSION

This work was focused on evaluation of the influence of food pigments on the rheological properties and processability of biodegradable colour masterbatches. Based on the obtained data, the results can be formulated into the following conclusions:

1. all food pigments have strong effect on rheological properties of coloured masterbatches,
2. not all food pigments are suitable for the preparation of colour masterbatches,
3. it is necessary to select other food pigments and evaluate their use in colour masterbatches.

Acknowledgement

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References

1. Ivanković A., Zeljko K., Talić S., Martinović Bevanda A., Lasić M.: J. Food Saf. Food Qual. *68*, 26 (2017)
2. Cheng J., Gao R., Zhu Y., Lin Q.: Alexandria Engineering Journal *91*, 70 (2024)

3. Salman S., Mudasir Y., Poonam A.: Current Research in Food Science 4, 503 (2021)

RUBBER COMPOUNDS WITH BIOPOLYMER COMPONENT

Ján KRUŽELÁK*, Andrea KVASNIČÁKOVÁ, Michaela DŽUGANOVÁ and Ivan HUDEC

Faculty of Chemical and Food Technology, Slovak University of Technology, Radlinského 9, Bratislava, 812 37
email: jan.kruzelak@stuba.sk

Abstract

Calcium lignosulfonate as a biopolymer component was incorporated into acrylonitrile-butadiene rubber in the amount – 50 phr. Four low molecular organic molecules – 1,4-butanediol, ethylene glycol, and two glycerols with different purity were used as plasticizers. They were applied in rubber compounds in the amount ranging from 5 to 30 phr. The results revealed that plasticizers softened both the rubber matrix as well as the biopolymer filler. The higher the polarity of the plasticizer, the higher the plasticizing effect on lignosulfonate. The plasticizing effect increased in the following order: 1,4-butanediol < ethylene glycol < glycerols. Softened lignosulfonate formed small soft filler like domains well distributed within the rubber matrix. Good compatibility and adhesion between the rubber and the biopolymer on their interface was observed leading to the enhancement in tensile characteristics of vulcanizates plasticized with ethylene glycol and glycerols.

Introduction

Following cellulose, lignin belongs to the second-most-spread renewable natural polymer in the world. This aromatic biopolymer originates from the biosynthesis of the plants by the radical polymerization of three phenylpropane units: p-hydroxyphenyl (H), syringyl (S) and guaiacyl (G), which are formed from the following lignin precursors: para-coumaryl alcohol, sinapyl alcohol and coniferyl alcohol[1]. Those units are interconnected by a variety of C-C or ether-like C-O-C linkages to form three-dimensional highly branched polymer structures with a variety of functional groups: aliphatic and phenolic hydroxyls, carbonyl, carboxylic and methoxyl groups[2]. Lignin is amorphous polymer, which behaves as a thermoplastic material, having glass transition temperatures Tg that can vary in a broad range depending on the isolation method, plant species, absorbed water, molecular weight and thermal history[3]. It is commonly derived from wood or crops, where it is integral part of the cell and imparts mechanical strength to wood. Thus, it can be considered as a construction material. The biggest producers of lignin are paper mills and the pulp-making industry. Lignin has many interesting properties, including a high availability, eco-friendliness, biodegradability, non-toxicity, antimicrobial and antioxidant behavior, adhesive properties, low price, etc.[4-6]. Those attributes make it different from traditional fillers used in rubber industry, i.e., carbon black or silica. Despite that, only about 2% of worldwide-obtained lignin is commercially used or to produce value-added products. The rest is landfilled or used as cheap burning fuel.

High availability, environmental friendliness, biodegradability, and reinforcing capability make it a suitable candidate as a filler or component for rubber compounds to form novel green composite materials. Through the augmentation of polar groups, enhanced electrostatic attraction, and hydrogen bonding, it can form a conjoined net-work with some polar polymers[7,8]. However, due to the formation of strong intramolecular interactions between the macromolecular chains, its compatibility and adhesion with non-polar polymers is usually weak. Therefore, the incorporation of lignin in its original, unmodified form into polymer compounds usually results in the deterioration in the physical–mechanical and utility properties of the final materials.

The various modification techniques to improve the dispersion of lignin in rubber matrices and compatibility between both components have been used[9-12]. The results have demonstrated very promising prospects and pointed to the high application potential of lignin into rubber compounds. However, a lot of modification procedures are time-consuming and require extra expenses. Sometimes, it is worth using a simple solution to improve the homogeneity and compatibility between the components of rubber blends, mainly for those fabricated on an industrial scale. To improve the adhesion and homogeneity between the rubber and the biopolymer in the current work, four low-molecular-weight polar plasticizers were used, namely 1,4-butanediol, ethylene glycol, and two types of glycerol (with 99% purity and in the form of 86% water solution). Those plasticizers are highly available and cost-effective. They have been chosen due to their polarity, as calcium lignosulfonate and acrylonitrile-butadiene rubber are polar materials. It is well known that low-molecular-weight hydrophilic substances are suitable plasticizers for polar-based rubber formulations. The main aim of using the plasticizers was to improve the dispersion of the biopolymer within the rubber matrix and to enhance the compatibility between both components.

Experimental

Acrylonitrile–butadiene rubber NBR, type SKN 3345 (acrylonitrile content 31–35%), was supplied from Sibur International, Russia. Calcium lignosulfonate (Borrement CA120), provided by Borregaard Deutschland GmbH, Germany, was used as a biopolymer component and was dosed to the rubber compounds in a constant amount - 50 phr. The average molecular weight of lignosulfonate was 24,000 $g.mol^{-1}$. 1,4-butanediol (99%), ethylene glycol (99%), glycerol (99%), and glycerol in the form of 86% water solution (glycerol 86%) were applied into rubber formulations as plasticizers in a concentration scale ranging from 5 to 30 phr. All plasticizers were delivered from Sigma-Aldrich, USA. For cross-linking of rubber compounds, a sulfur vulcanization system was used. The activators, stearic acid and zinc oxide, were supplied from Slovlak, Košeca, Slovak Republic. Accelerators, N-cyclohexyl-2-benzothiazole sulfenamide CBS and sulfur, as curing agents, were provided by Vegum, Dolné Vestenice, Slovak Republic.

The rubber formulations were fabricated by using a Brabender laboratory kneading machine (Brabender GmbH & Co. KG, Duisburg, Germany) in two mixing steps at a temperature of 90 °C and 55 rpm. The curing process was carried out using a Fontijne hydraulic press (Fontijne, Vlaardingen, Holland) following the optimum cure time of each rubber compound. The curing temperature was 170 °C and the pressure was 15 MPa.

The tensile tests were performed according to valid technical standards using a Zwick Roell/Z 2.5 tearing equipment (Zwick GmbH & Co. KG, Ulm, Germany). The cross-head speed was set up to 500 mm.min^{-1} with a gauge length of 25 mm. The dumbbell-shaped specimens (width 6.4 mm, length 80 mm) were cut with a special knife from a 2 mm thick cured rubber plate. The hardness in Shore A was measured by using a durometer.

Results and discussion

From Figure 1, it is shown that in comparison with the reference, the elongation at break first slightly decreased by the incorporation of 5 phr plasticizers. The elongation at break of vulcanizates with 1,4-butanediol fluctuated only in a low experimental value range, almost independently of the content of the plasticizer and was very similar to that of the reference. The elongation at break of the vulcanizates with applied ethylene glycol and both glycerols showed an increasing trend and reached a maximum at 20 phr of plasticizers. The increase in the elongation at break can be attributed to the softening effect of plasticizers on rubber compounds, weakening of intramolecular interactions, and thus increase in rubber chains' elasticity and mobility.

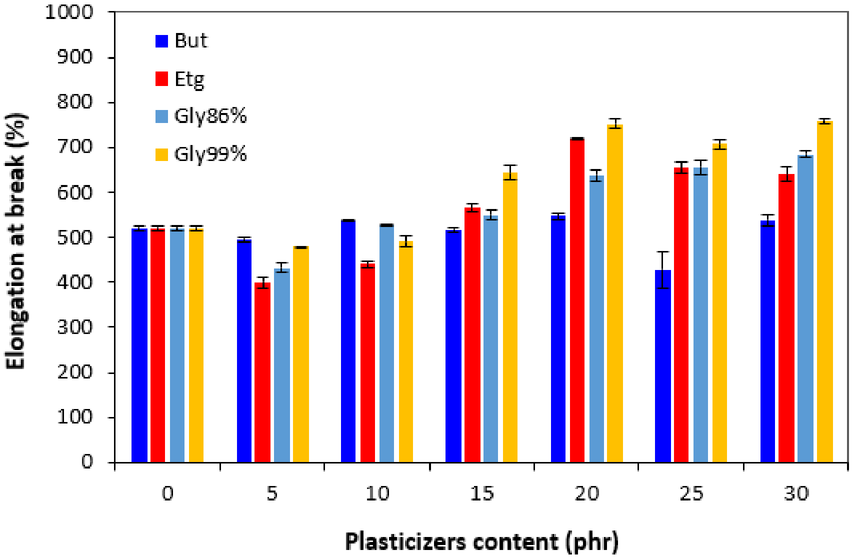

Fig. 1 Influence of plasticizer content on elongation at break of vulcanizates

Similarly, to the elongation at break, the tensile strength first decreased by the application of 5 phr plasticizers (Figure 2). Subsequently, the increase in the tensile strength was recorded (with exclusion of the compounds with 1,4-butanediol). The tensile strength of the compound with a maximum content of glycerol 86% was almost threefold higher when compared to the reference (the tensile strength increased from 3.7 MPa for the reference up to over 10 MPa for the vulcanizate with 30 phr of glycerol). By the application of glycerol 99%, the maximum tensile strength was reached at 20 phr of the plasticizer (again over 10 MPa). Similarly, vulcanizates plasticized with ethylene glycol reached a maximum tensile strength at 20 phr of the plasticizer. When compared to the reference, the application of 20 phr ethylene glycol resulted in an increase in the tensile strength up to over 6 MPa. By contrast, the tensile strength of the vulcanizates plasticized with 1,4-butanediol decreased. As shown in Figure 2, the higher the amount of 1,4-butanediol, the lower the tensile strength. Based upon the obtained results it can be concluded that the presence of plasticizers in rubber formulations (except for 1,4-butanediol) resulted in an increase in the tensile characteristics. However, the enhancement in the tensile strength and elongation at break was observed at higher amounts of plasticizers. It also becomes evident that the highest tensile strength was demonstrated by the vulcanizates plasticized with both glycerols.

The used plasticizers are hydrophilic, low-molecular organic molecules that plasticize both the rubber matrix and calcium lignosulfonate. This leads to a decrease in the viscosity of the rubber compounds. The viscosity of the filler and the rubber became closer, which resulted in better dispersion and distribution of the biopolymer. The improvement in the compatibility and adhesion on the filler–rubber interface was achieved. Lignosulfonate formed smaller and softer domains. The softer domains with a lower hardness and rigidity deform much more easily, when compared to the stiff filler agglomerates, which act as stress concentrators upon deformation strains. This causes a decrease in the mechanical properties of the final rubber materials. On the other hand, small domains with high deformability behave as particles of reinforcing fillers, contributing to the increase in the tensile behavior of rubber systems.

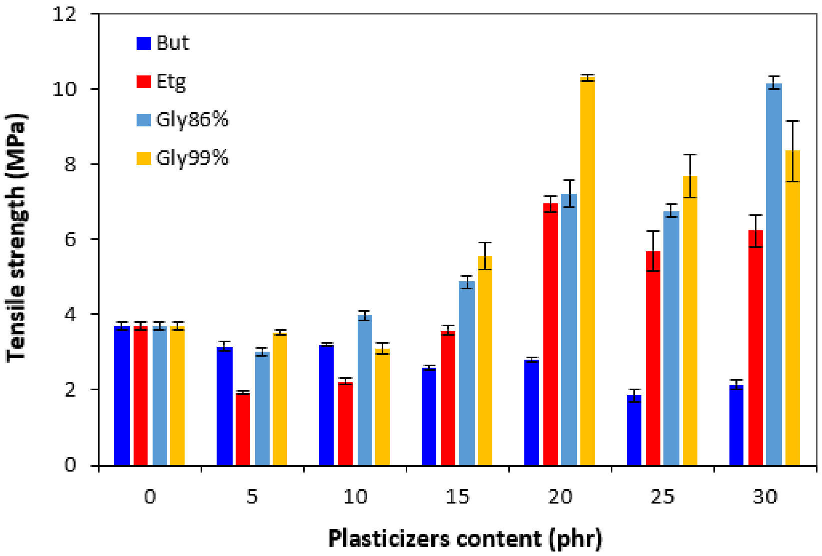

Fig. 2 Influence of plasticizer content on tensile strength of vulcanizates

Conclusion

Calcium lignosulfonate filled rubber compounds were plasticized with polar low-molecular-weight organic substances. The higher the amount of plasticizers, the lower the viscosity. The reduction in internal friction between the chain segments and increase in the chains' elasticity and mobility resulted in the increase in the elongation at break. Simultaneously, plasticizers exhibited a softening effect on lignosulfonate. It can be stated that the higher the polarity of the plasticizer, the higher the softening effect on the biopolymer. The most homogeneous structure was demonstrated by the vulcanizates with applied glycerols having the highest polarity. This suggests that glycerol shows the highest plasticizing effect on lignosulfonate. Softened lignosulfonate formed small, soft filler-like domains well distributed within the rubber matrix. Good compatibility on the filler–rubber interface was observed. The outlined facts are responsible for the enhancement in the tensile characteristics of the corresponding vulcanizates.

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References

1. Kramer CAC. et al.: J Mol Struct. *1251* (2022)
2. Antonino LD. et al.: Molecules. *26* (2021)
3. Laurichesse S. et al.: Prog Polym Sci. *39* (2014)
4. Domenek S. et al.: J Polym Environ. *21* (2013)
5. Verdini F. et al.: Molecules. *27* (2022)
6. Roy K. et al.: J Polym Environ. *28* (2020)
7. Ashraful Alam M. et al.: Int J Biol Macromol. *281* (2024)
8. Kanti Mondal A. et al.: Macromol. *207* (2022)
9. Komisarz K. et al.: Materials *16* (2023)
10. Hait S. et al.: J Compos Sci. *5* (2021)
11. Frigerio P. et al.: Bioresources *9* (2014)
12. Taher MA. et al.: ACS Appl Bio Mater. 6 (2023)

EMI ABSORPTION SHIELDING OF RUBBER COMPOSITES BASED ON FERRITES AND CARBON FILLERS

Ján KRUŽELÁK[1]*, **Andrea KVASNIČÁKOVÁ**[1], **Michaela DŽUGANOVÁ**[1], **Rastislav DOSOUDIL**[2] **and Ivan HUDEC**[1]

[1]*Faculty of Chemical and Food Technology, Slovak University of Technology, Radlinského 9, Bratislava, 812 37*
[2]*Faculty of Electrical Engineering and Information Technology, Slovak University of Technology, Iľkovičová 3, Bratislava 812 19*
email: jan.kruzelak@stuba.sk

Abstract

In this work, magnetic soft ferrites, namely manganese-zinc ferrite, nickel-zinc ferrite and combinations of both fillers were incorporated into acrylonitrile-butadiene rubber to fabricate composite materials. The total content of ferrites was kept constant – 300 phr and the mutual ratio of both fillers was changed. In addition to ferrites, graphite as carbon based filler was incorporated into rubber compounds in constant amount – 25 phr. The work was focused on the investigation of the fillers on absorption shieling performance of the composites, which was investigated within the frequency range 1 GHz – 6 GHz. The achieved results demonstrated that the absorption shielding efficiency increased with increasing proportion of nickel-zinc ferrite, which suggests that nickel-zinc ferrite demonstrated better absorption shielding potential.

Introduction

Electromagnetic interference (EMI) can be defined as a kind of environmental pollution produced by electronic, radiation and electro-communication devices. The rapid progress in modern technology is connected with higher accumulation of EMI in the surrounding. It has been revealed that undesired EMI can lead to the lowering of efficiency of electronic equipment, even to their malfunctions. Not least, negative impact of EMI on human beings' health and ambient has been reported as well[1,2]. Therefore, efficient methods and materials have been developed to reduce EMI in the surrounding and thus to protect the functionality of electronic devices and human beings' health.

In generally, the combination of three shielding mechanisms plays a crucial role in reducing or eliminating of EMI. Those mechanisms are absorption, reflection and multiply reflection of electromagnetic radiation[3,4]. Reflection is basic shielding mechanism that relates to a simple reflection of EMI from the outer surface of the shield. To shield EMI by reflection, the material must have free electric charges (electrons or holes), which interfere with electromagnetic plane wave in the radiation. On the other hand, necessary condition of absorption shielding is the presence of electric or magnetic dipoles in the shield. Multiply reflection takes part as the third shielding mechanism and refers to reflection of EMI from internal surfaces, interfaces and various inhomogeneities in the shielding material. Foam and porous materials exhibit high specific surface, while large phase interfaces have composites filled with different types of fillers. Such materials usually show good ability of multiply reflection[5-7].

Formerly, metal based plates were extensively used as efficient EMI shields due to their unique electrical conductivity. However, heavy processing and manipulation, low flexibility and heaviness limit the utilization of metal based shields. Also, possessing high electrical conductivity, the reflection of EMI is the most common shielding mechanism for metal based shields. The reflected electromagnetic waves can interfere with primary radiation that is emitted from neighboring electronic appliances, causing the so called collateral electromagnetic radiation effect. Composites based on polymer matrices and suitable filler inclusions can be used as alternatives to traditional metal based EMI shields due to their low weight, high flexibility and elasticity, good process-ability, corrosion resistance, tunable properties or low cost. The adjusting of electromagnetic characteristics to polymer matrices can be performed by selection of suitable fillers. The application of fillers into polymer matrices enables not only to tune mechanical and structural properties of the composites, but also provides space for adapting electrical conductivity, permittivity, permeability or thickness to obtain desired EMI shielding performance.

Experimental

Commercially available magnetic soft ferrites, nickel-zinc ferrite NiZn and manganese-zinc ferrite MnZn were supplied from by Epcos Company, Šumperk, Czech Republic. Ferrites have spinel-type structure with particle size distribution ranging from 0.2 to 70 μm for NiZn ferrite and from 0.7 to 50 μm for MnZn ferrite. Graphite powder (99.9%) with particle size lower than 20 μm was provided from Sigma-Aldrich, Saint Louis, Missouri, USA. Acrylonitrile-butadiene rubber (NBR, SKN 3345, acrylonitrile content - 31-35 %) was supplied from Sibur International, Russia. Sulfur vulcanization system consisting of stearic acid and zinc oxide as activators, N-cyclohexyl-2-benzothiazole sulfenamide CBS as accelerator and sulfur as curing agent was used for cross-linking of composites. The additives of vulcanization system were provided by Vegum a.s., Dolné Vestenice, Slovak Republic. The composition of the composites and their designation is presented in Tab. 1.

The rubber compounds were fabricated in two-step mixing process in a laboratory mixer Brabender. In the first step (9 min, 90ºC), the rubber and the filler were compounded. In the second step (4 min, 90ºC), the curing system was introduced.

The curing characteristics of rubber compounds were investigated from the corresponding curing isotherms measured by MDR2000 at 160 °C. The curing process was performed at 160 ºC for the optimum curing time under a pressure of 15 MPa by using a hydraulic press Fontijne. The tensile properties of tested composites were evaluated by using Zwick Roell/Z 2.5 appliance.

High frequency single-layer electromagnetic wave absorption properties (return loss *RL*, matching frequency f_m, bandwidth Δf for *RL* at −10 dB and *RL* at −20 dB, and the minimum of return loss RL_{min}) of composite materials were obtained by calculations of return loss: $RL = 20 \log |(Z_{in} - 1)/(Z_{in} + 1)|$, where $Z_{in} = (\mu/\varepsilon)^{1/2} \tanh[(j\omega \cdot d/c)(\mu \cdot \varepsilon)]$ is the normalized value of input complex impedance of the absorber, *d* is the thickness of the single-layer absorber (backed by a metal sheet), *c* is the velocity of light in vacuum.

Table 1. Composition of composites in phr and their designation

NBR	100	100	100	100	100
ZnO	3	3	3	3	3
stearic acid	2	2	2	2	2
CBS	1.5	1.5	1.5	1.5	1.5
sulfur	1.5	1.5	1.5	1.5	1.5
graphite	25	25	25	25	25
MnZn ferrite	300	200	150	100	0
NiZn ferrite	0	100	150	200	300
designation	Mn300	Mn200 Ni100	Mn150 Ni150	Mn100 Ni200	Ni300

Results and discussion

It was already mentioned that shielding by reflection is undesirable, as reflected radiation still proliferates through the ambience, often causing damages related to secondary electromagnetic radiation effect. Thus, the work is focused on absorption shielding efficiency, which means the ability of composites to absorb electromagnetic energy. The absorbed radiation is then transformed into harmless forms of energy, e.g. to heat. Absorption shielding efficiency of composites was investigated within the frequency range from 1 MHz to 6 GHz and was determined as return loss in decibels unit. Return loss provide information, which amount of incident EMI can be absorbed by the shielding material. It has been reported in scientific works that materials showing return loss at - 10 dB can absorb 90 – 95 % of EMI. The materials reaching return loss at -20 dB can efficiently absorb around 99 % of electromagnetic radiation. The efficiency of absorption shielding depends on the frequency bandwidths. The broader the frequency bandwidths for absorption shielding, the higher the absorption shielding ability of the materials.

The frequency dependencies of return loss for composites filled with ferrites and graphite are graphically illustrated in Fig. 1, while their electromagnetic absorption parameters are summarized in Tab. 2. RL_{min} (dB) represents the minimum value of return loss (maximum absorption shielding performance) at a matching frequency f_m (MHz), Δf (MHz) – is the effective frequency bandwidth for absorption shielding at -10 and -20 dB. From Fig. 1 and Tab. 1 it becomes apparent that the composite filled with graphite and MnZn ferrite (Mn300) exhibited absorption shielding efficiency at the lowest frequencies (matching frequency was 2180 MHz) with absorption maximum -55 dB. This composite manifested the lowest effective frequency bandwidth at -10 and -20 dB (from 1500 MHz to 3200 MHz at -10 dB and from 1950 MHz to 2430 MHz at -20 dB). Based upon the narrowest absorption peak it can be stated that the composite Mn300 is the worst EMI absorber. With increasing amount of nickel-zinc ferrite in magnetic fillers combination, the absorption maxima and absorption shielding efficiency shifted to higher frequencies. Simultaneously, the effective frequency bandwidths at *RL* = -10 and -20 dB become broader, which suggests that the absorption shielding efficiency of composites showed an increasing trend with increasing proportion of nickel-zinc ferrite. The composite based on graphite and NiZn ferrite (Ni300) exhibited the absorption maximum -49 dB at 3820 MHz. The effective frequency bandwidth ranged from 2250 MHz to 6000 MHz at -10 dB (Δf = 3750 MHz) and between 3250 – 4480 MHz at -20 dB (Δf = 1230 MHz). The widest absorption peak points to the fact that this composite is the most effective EMI absorber shield.

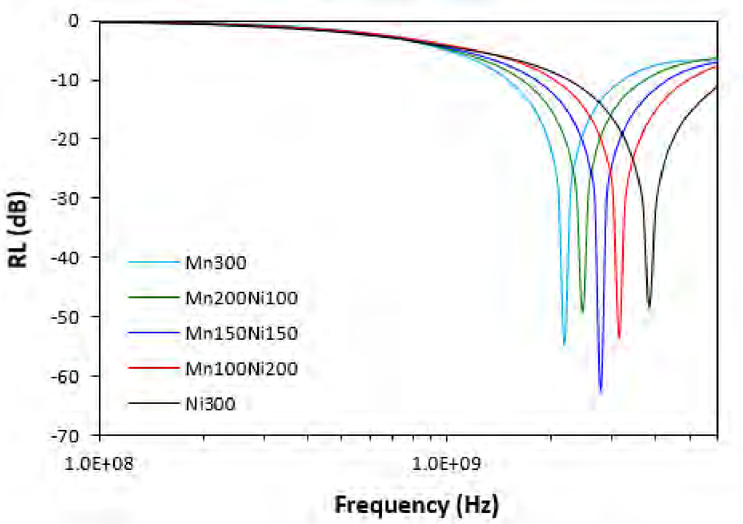

Fig. 1 Frequency dependences of return loss of composites

Table 2. Electromagnetic absorption parameters of composites

sample	RL_{min} (dB)	f_m (MHz)	Δf (MHz) -10 dB	Δf (MHz) -20 dB
Mn300	-55	2180	1700	480
Mn200Ni100	-49	2460	2060	560
Mn150Ni150	-62	2770	2500	690
Mn100Ni200	-54	3130	2870	820
Ni300	-49	3820	3750	1230

The modulus and tensile strength showed a slight increasing tendency with increasing content of nickel-zinc ferrites in magnetic fillers combination, which suggests that NiZn ferrite shows a higher reinforcing effect. Though, it should be noted that the differences were not very significant. Due to the tough crystal structure and relatively wide particles size distribution, ferrites in general are inactive fillers when incorporated into polymer matrices.

Conclusion

The results showed that composites filled with graphite and ferrites exhibited absorption shielding performance in tested frequency range. The higher the ratio of nickel-zinc ferrite, the higher the absorption shielding efficiency, pointing to better absorption shielding potential of NiZn filler. As the best absorption shielding material was proved to be the composite filled with graphite and 300 phr of nickel-zinc ferrite as it exhibited the widest effective frequency bandwidths. Simultaneously, this composite absorbs electromagnetic radiation within the highest frequency ranges.

Acknowledgement

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References

1. Apakuppakul S. et al.: J Arrhythm. *38* (2022)
2. Kaszuba-Zwoińska J. et al.: Przegl Lek. 72 (2015)
3. Raagulan K. et al.: Nanomaterials. *10* (2020)
4. Leão A. et al.: J Polym Environ. *28* (2020)
5. Reshi HA. et al.: J. Appl. Phys. *131* (2022)
6. Huang Y. et al: Materials. *14* (2021)
7. Hwang U. et al: ACS Omega. *7* (2022)

DEVELOPMENT OF INTEGRATED DESIGN SYSTEM FOR AUTOMOTIVE RUBBER COMPONENTS

Chang-su Woo[1]*, Hyun-sung Park[1], Sang-hee Choi[2] and Sang-kyun Kim[3]

[1]Korea Institute of Machinery & Materials, 156 Gajeongbuk-ro, Yuseong-gu, Daejeon, Rep. of Korea, 34103
[2]TSR Co., Ltd, 157 Gongdan-dong, Gumi, Gyeongsangbuk-do, Rep. of Korea, 39267
[3]DRB Co., Ltd, 7 Geumsa-dong, Geumjeong-gu, Busan, Rep. of Korea, 46329
email: cswoo@kimm.re.kr, hspark@kimm.re.kr, shchoi@tsr.co.kr, kim.sang.kyun@drbworld.com

Abstract: Fatigue analysis and useful lifetime evaluation are very important factors to consider in the design procedure to assure the safety and reliability of rubber components. Recently, the design, analysis and evaluation technology were required to achieve the high quality, fidelity, and reliability of rubber products. The objectives of this study are to establish test methods of rubber material and to develop a database of rubber material properties and to evaluate the performance of rubber components and to construct the prediction system of fatigue lifetime. The fatigue lifetime prediction methodology of the rubber component was proposed by incorporating finite element analysis and fatigue damage parameters from the fatigue test. Database of mechanical properties of anti-vibration rubber material, such as compound conditions, mechanical properties, degradation, and fatigue lifetime were developed. Fatigue lifetime of engine rubber mount for automobiles was nearly exactly predicted by the developed method and the validity of the fatigue lifetime estimation method was verified. Using the developed integrated design system of rubber components, the time and expense required to develop rubber components were saved and the reliability of products is expected to be enhanced.

1. Introduction

For products aiming to be first class, the application of rubber components for most machines including vehicles have gradually increased to achieve high quality and reliability through low vibration and noise, and enhanced operation. Therefore, demand for analysis and design technology for rubber components is required[1]. As much attention has been paid to the assurance of the quality and durability of vehicles, the development of technology is required to enhance the durability and reliability of anti-vibration rubber components that are the most important components of vehicles[2]. Until now, however, rubber components have been developed by trial and error as well as experiences due to the special properties of rubber. All respects of performance tests including the design and analysis technology, therefore, are now very vulnerable. In particular, the mechanical data of rubber material can be obtained only by experiment as there are many rubber compounds. Therefore, it is very difficult to develop a database of mechanical properties of rubber component and there are many obstacles to overcome in the fatigue lifetime estimation and evaluation technology due to insufficient manpower and technology[3].

In this study, likewise, a database was developed that includes the mechanical properties of anti-vibration rubber material for vehicles including a compound conditions, mechanical properties, deterioration, and fatigue lifetimes. And then, a fatigue analysis model of rubber components was developed by associating the results of mechanical properties on rubber components with a database. The validity of the developed model was verified with the results of the actual fatigue tests. It is, therefore, expected to save on the time and expenses that are required for developing rubber components and will help to enhance reliability of products.

2 Integrated design system

2.1 System configuration

Standard mechanical properties of rubber material have not yet been established because there are various combination components and content conditions required in the manufacturing processes. Performance improvement through property estimation and securing design technology of rubber component is a key technology that is mandatory for advancing all machines and systems. According to recent advances in large scale and super computer, it is possible to analyze the behaviors of rubber components through nonlinear analysis with finite element methods and to apply systematic and analytical methods to designing rubber components[4]. In this study, the database of the mechanical properties of rubber material including compounding conditions was developed. Design and analysis system (RubDAS; Rubber component Design & Analysis System) was developed by integrating all these items. Integrated design system of rubber component in machine was developed to perform the systematic design and analysis of rubber components. These systems are, as shown in Figure 1(a), composed of: a rubber compound program (RubCOM: Rubber Compound), mechanical property database program (RubPRO: Rubber Property), and a fatigue lifetime estimation program of rubber components (RubFAT: Rubber Fatigue). These systems are integrated design and analysis systems that include all the processes required to design rubber components: rubber compound, the determination of material constants by rubber mechanical property tests, the strength and size design of rubber components, component design through finite element analysis, and the durability estimation of rubber components.

2.2 Rubber compound program

Database of rubber compound and mechanical properties of rubber material have not yet been developed because they are possible through many experiments due to various mechanical properties required by each consumer company and the component of rubber compounds are too vast. Consumer companies of rubber products depend on the results of experiments by rubber part makers rather than selecting rubber products based on clearly standardized mechanical properties. If database of mechanical properties of rubber material is developed, it is expected to achieve standardization and the independent development of prediction software of mechanical properties. After the main factors of rubber compound were selected and the effects of interaction between factors on the mechanical properties of rubber were understood, the characteristic equations that represented various mechanical properties were derived using a rubber compound program (RubCOM) in Figure 1(b). In this study, experiments were carried out by varying the quantity of carbon black, sulfur, vulcanization accelerators, activator ZnO, and stearic acid on the natural rubber polymer base, and then the effects of these materials on the mechanical properties of rubber compound were understood, deriving a relation that could quantitatively predict changes in mechanical properties according to each factor through statistical data analysis. Mechanical properties were obtained by setting five factors (carbon black, sulfur, vulcanization accelerators, ZnO, and stearic acid) to the fifth level to derive the relation. Based on the mechanical properties, the effect of each factor and regression coefficients through statistical analysis were investigated. The validity of models was checked by obtaining characteristic regression equations. The regression equations were better as the value of the R-square drew closer to 1. Experiments were repeatedly conducted to enhance reproducibility and reliability, and their order was arbitrarily determined to exclude the effects of other factors except for main factors. The compound program was developed based on this data from the experiments and characteristic regression equations.

2.3 Database of rubber property

For the finite element analysis of a hyper-elastic model on material such as rubber, it is very important to determine its exact mechanical properties. That is why, static tests, dynamic tests, and friction tests are necessary. Static tests include simple tension, simple compression, and pure shear tests^{5}. If mechanical properties of rubber are obtained with the existing simple tension tests, it is likely to create errors in finite element analysis. Because the mechanical properties of rubber change according to the environment, it is necessary to conduct tests at both high and low temperatures. When the rubber is exposed to high temperature for a long time, it increases in its hardness by aging, and that poses an important problem in material tests and mechanical environments such as the maximum strain and cyclic load needs to be considered in experiments. Database program of rubber mechanical property (RubPRO) is made to obtain the mechanical properties of rubber in various environments before finite element analysis. In these programs, a database of results of static test, dynamic test and friction test according to changes in the mechanical environment as well as at high, low, and warm temperatures were developed. Using the database, the mechanical properties of the necessary materials could be easily obtained when new rubber components are designed. Figure 3(a) shows an initial screen of rubber mechanical property database program that is composed of a database of these: static, dynamic, friction, aging, and fatigue properties. Static properties include a curve fitting program of nonlinear material constants.

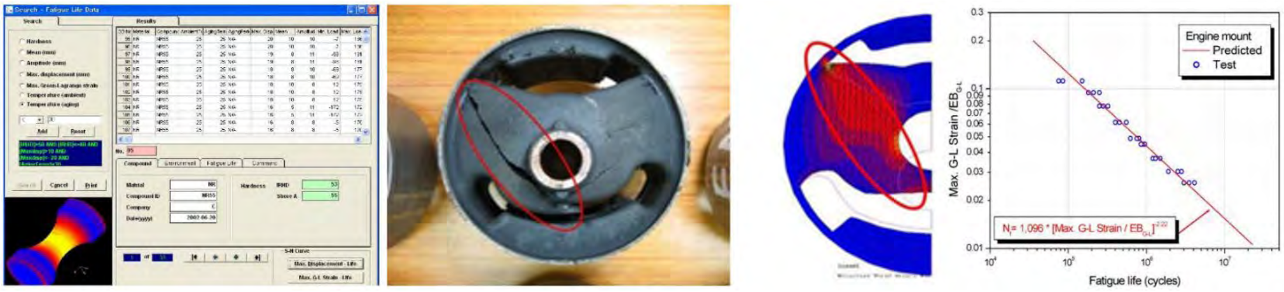
(a) Integrated design system (b) Rubber compound program (c) Database of rubber properties

Figure 1. Integrated design system for rubber components

2.4 Fatigue lifetime prediction

Figure 2(a) shows the procedures to predict the fatigue lifetime of rubber components as follows: (1) using the results of material tests of rubber component to develop, finite element analysis of the component was conducted to the maximum strain in weak points; (2) fatigue tests were carried out for rubber specimens that had the same compound and mechanical properties as the components, and fatigue damage parameter was obtained from the relation of the maximum strain and fatigue lifetime; (3) the fatigue lifetime of rubber components could be predicted and evaluated using the analysis results of rubber component and the fatigue test results of rubber specimens. Fatigue lifetime curves are necessary for the same rubber materials to predict the fatigue lifetimes of rubber components. In this study, fatigue tests were conducted by manufacturing 3-dimensional fatigue specimens^{6} that could reproduce the maximum tension strain by the fatigue load of rubber components as in Figure 2(b). Using the results of fatigue tests in various conditions, the correlation of fatigue lifetime with variables such as amplitude, mean displacement, and maximum tension displacement were obtained. As a result, the fatigue lifetime was similar irrespective of mean displacement and amplitude if the maximum tension displacement was fixed. the fatigue lifetime decreased as maximum tension displacement increased. The fatigue lifetime, therefore, could be represented irrespective of test conditions when the maximum tension displacement was a fatigue damage variable. Using the relation of the maximum tension displacement and the Green-Lagrange strain that was the result of the analysis on specimens, the relation curves of Green-Lagrange strain and fatigue lifetime could be obtained as shown in Figure 2(c). The maximum tension displacement could be well represented with the maximum Green-Lagrange strain and fatigue lifetime was predicted according to the strain[7]. The database of fatigue properties, as shown in Figure2(d), includes this data: hardness, fatigue test conditions, fatigue lifetime, and aging conditions. Users could find the data after setting multiple search conditions by assigning the ranges of this data: hardness, average displacement, amplitude, the maximum displacement, and the maximum Green-Lagrange strain. The fatigue lifetime of anti-vibration rubber component used in the engine mount for automobiles was estimated to verify the validity of the fatigue lifetime prediction method of rubber component proposed in this study. Most fatigue cracks occurred at points where the strain was at a maximum value as shown in Figure 2(e) the results of the analysis were quite consistent with those of tests. Figure 2(f) shows the relation to the fatigue lifetime obtained in fatigue tests on actual rubber components and the predicted lifetime using the fatigue lifetime prediction equations of rubber specimens. Predicted fatigue lifetimes of rubber components for engine mounts as shown in Figure 2 was fairly consistent with the actual lifetime: this result verified the validity of the lifetime estimation method of rubber components proposed in this study.

(a) Procedure to fatigue life prediction (b) 3-dimensional fatigue specimen (c) Strain-fatigue life curves



(d) Database of fatigue properties (e) Fatigue test and finite element analysis (f) Fatigue lifetime prediction

Figure 2. Fatigue lifetime prediction of rubber components

3. Conclusions

The following conclusions were obtained through the development of integrated design systems of rubber components for automobiles.

(1) The development of the fatigue analysis process for rubber component, a fatigue lifetime prediction methodology was proposed by incorporating finite element analysis and fatigue damage parameter.

(2) In various test conditions, fatigue tests on three dimensional specimens were conducted to obtain fatigue lifetime according to rubber material, mean displacement, and changes in amplitude, and then the fatigue lifetime prediction equations were derived with maximum tension displacement and maximum strain as one of the fatigue damage parameters.

(3) Database of mechanical properties of anti-vibration rubber material, such as compound conditions, mechanical properties, degradation, and fatigue lifetime were developed.

(4) Fatigue lifetime of engine rubber mount for automobiles was nearly exactly predicted by the developed method and the validity of the fatigue lifetime estimation method was verified.

(5) Using the developed integrated design system of rubber component, the time and expense required to develop rubber components were saved and the reliability of products is expected to be enhanced.

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References

1. Charlton. D. J., Yang. J., A Review of Methods to Characterize Rubber Elastic behavior for use in Finite Element Analysis, *Rubber Chemistry & Technology* 67, 481-503 (1993).
2. Lake. G.J., Fatigue and Fracture of Elastomers, *Rubber Chemistry & Technology*, 68, 435-460 (1997).
3. Gent. A. N., Engineering with Rubber–How to design rubber components, Oxford University Press, NY, 1992.
4. Kim. W. D. & Kim, D. J., A study on the equi-biaxial tension test of rubber material, *KSAE*, 11, 95-104 (2003).
5. Mullin. L., *Rubber Chemistry & Technology*, 42, 339-362 (1969).
6. Takeuchi. K. & Nagakawa. M., *Int'l Polymer Sci*. 20, 64-69 (1993).
7. Fatemi. & Yang., Cumulative Fatigue Damage and Life Prediction Theories, International J. of fatigue, 20, 9-34 (1998).

LOW-DENSITY POLYETHYLENE COMPOSITES WITH MODIFIED MONTMORILLONITE

Michaela SEDNIČKOVÁ[1]*, Ľuboš JANKOVIČ[2], Zuzana NÓGELLOVÁ[1], Ivan CHODÁK[1] and Juraj KRONEK[1]
[1] Polymer Institute, Slovak Academy of Sciences, Dúbravská cesta 9, 845 41 Bratislava, Slovakia
[2] Institute of Inorganic Chemistry, Slovak Academy of Sciences, Dúbravská cesta 9, 845 36 Bratislava, Slovakia
email: michaela.sednickova@savba.sk

Abstract

Low-density polyethylene composites were prepared by melt intercalation. Intercalate of montmorillonite Kunipia was used as filler with the concentration from 1 to 10 wt%. The mechanical properties of the samples were pointed out to changes of Young´s modulus and strain at break with the rising concentration of filler. Interactions between matrix and filler were analyzed by rheology and X-ray diffraction.

Introduction

Montmorillonites belong to the group of layered silicates. Montmorillonites are hydrophilic in the nature, but they are able to cation-exchange reaction, what make them more hydrophobic. Modified montmorillonites can be used as fillers with hydrophilic polymers for preparation of nanocomposites. The advantage is dispersion of layered silicates to the polymeric matrix. Even a small amount (less than 5 wt%) of modified filler leads to a change in mechanical properties, heating capacity or gas permeability of prepared composites. Some kinds of modified montmorillonites are also commercially available[1,2].

Nanocomposites prepared in a wide range of polymers were tested in the laboratory conditions. Polyamide-based nanocomposites are used in the automotive industry. Nanocomposites based on polypropylene or polyethylene terephthalate are used as packaging material. The polyethylene used as a matrix is also interesting for research. Low-density polyethylene (LDPE) is a thermoplastic polymer, flexible and moisture-resistant. The main advantages are low price and recyclability[3,4].

This work is focused on the preparation and analysis of low-density polyethylene nanocomposites consisting of modified montmorillonite.

Experimental

Low density polyethylene (LDPE) with the trade name BRALEN FB 2-17 supplied by Slovnaft, a.s. was used as the polymer matrix.

Highly purified sodium MMT (>98%) Kunipia-F (Na-KU), a product of Kunimine Industries Co., Ltd., Tokyo, Japan, of relatively high cation exchange capacity (CEC = 119 meq/100 g), was used as a clay mineral.

Basic chemical for the synthesis of polyoxazoline, 2-ethyl-2-oxazoline (TCI Chemicals, Belgium) was dried over potassium hydroxide for 48 h and distilled over calcium hydride under reduced pressure. Acetonitrile (Sigma-Aldrich, Steinheim, Germany) was dried and distilled over calcium hydride. Both chemicals were stored over molecular sieves 4Å (Merck, Darmstadt, Germany) under argon.

The synthesis of polymeric modifiers poly(2-ethyl-2-oxazoline) (PETOX) and statistical copolymer poly(2-ethyl-2-oxazoline-co-ethyleneimine) (PETOX-PEI) is described in cit. 5. In the cit. 5 is also shown the modification of MMT by polyoxazolines. The final filler was labeled PEI-PETOX 100 (short: MMT).

The pure LDPE and composites of LDPE with the concentration 1, 2, 3, 5 and 10 wt% of MMT were prepared in mixing chamber Brabender Plasticorder PLE 331 (Brabender, Germany) at 140°C, 35 rpm and 10 minutes. The composites were pressed to slabs using a laboratory press Fontijne Holland TP 50 (The Netherlands) at 140°C for 2 minutes.

Mechanical properties were measured using the testing machine Instron 3365 (Instron, UK). Seven dog bone specimens were used for each sample and analyzed at a deformation rate of 50 mm/min.

Rheological properties were measured on the rheometer AR 2000 Advanced Rheometer (TA Instruments, USA) with the Peltier and 20 mm steel plate geometry at 140°C with a strain of 0.1% and a frequency range of 0.01-100 Hz.

X-ray diffraction (XRD) patterns were recorded in reflection mode, using a fixed sample stage for flat samples, on the EMPYREAN system (PANalytical B.V, USA), equipped with CuKα ($\lambda\alpha 1$ = 1.54060 Å) radiation and operating at 45 kV and 40 mA. The patterns were scanned in the 2θ range 2.5–10° with scanning steps of 0.026° 2θ and scan step times of 100 s.

Results and Discussion

Mechanical testing is the most common method used to evaluate the mechanical properties of polymer-clay composites. The results of mechanical properties are shown in the Table 1. Young´s Modulus was increased with the rising concentration of MMT filler. Concurrently with increasing concentration of filler, the strain at break was decreased. Pure LDPE had a stress at strength 15.9 ± 0.5 MPa, but surprisingly, the adding of filler reduced the stress at strength to around 10 MPa and this value was almost the same for all filler concentrations.

Table 1. Results of mechanical properties as Young´s modulus, Stress at strength and Strain at break for pure LDPE and LDPE composites with different MMT concentrations.

	Young´s Modulus [MPa]	**Stress at Strength [MPa]**	**Strain at Break [%]**
pure LDPE	203 ± 8	15.9 ± 0.5	632 ± 29
LDPE + 1 wt% MMT	203 ± 8	10.6 ± 0.8	307 ± 107
LDPE + 2 wt% MMT	212 ± 5	10.0 ± 0.2	152 ± 72
LDPE + 3 wt% MMT	221 ± 6	9.9 ± 0.1	128 ± 45
LDPE + 5 wt% MMT	241 ± 9	9.9 ± 0.1	91 ± 11
LDPE + 10 wt% MMT	273 ± 30	10.5 ± 0.4	69 ± 5

Rheological method tells us about the interaction between the matrix and the filler. Figure 1 shows the dependence of storage modulus (G´) on frequency for pure LDPE and LDPE composites. Behavior of storage modulus in dependence on frequency for pure LDPE compare to LDPE composites was almost the same and pointed out to the low interactions at the intercalation level between the polymeric matrix and the modified filler.

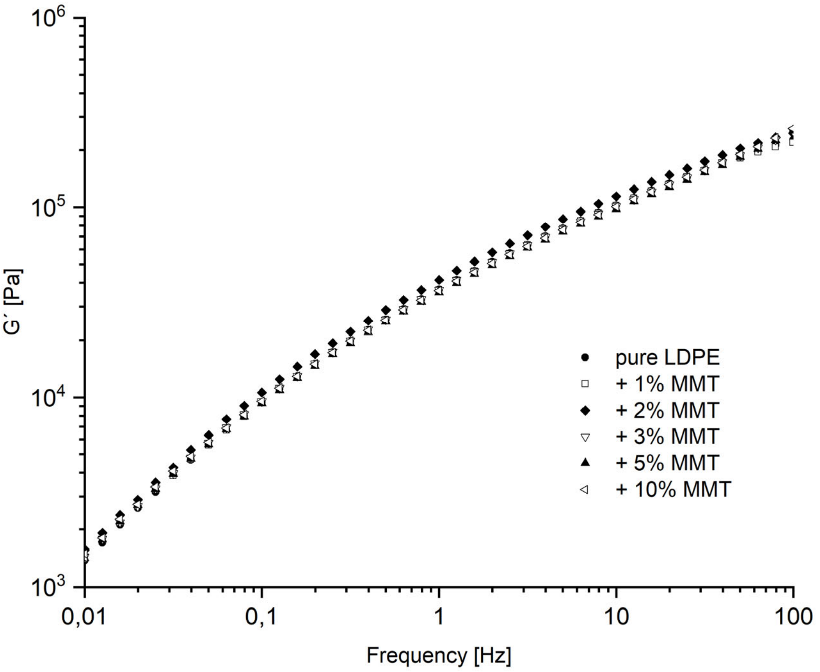

Figure 1. Results of rheological properties. Storage modulus (G´) in dependence on the frequency for pure LDPE and LDPE composites with different MMT concentrations.

X-ray diffraction (XRD) is the method for evaluating the degree of intercalation. The XRD records of pure LDPE and LDPE composites with different concentrations of MMT is shown on the Figure 2. For pure LDPE, no maximum appears in the 2 theta angles region. In the matrix with MMT, a peak was formed around 6.5 in the region of 2 theta angles and increased with the increasing concentration of MMT filler. Unfortunately, no shift of 2 theta angles from 6.5 region to the lower region was observed, which would indicate the intercalation.

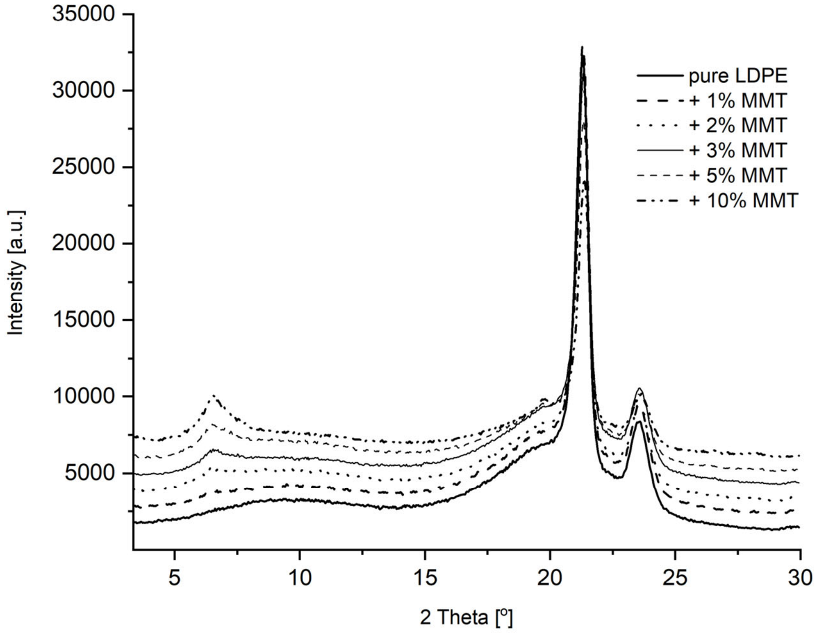

Figure 2. The XRD records of pure LDPE and LDPE composites with different MMT concentrations.

Conclusions

The results indicate low interactions between low-density polyethylene and modified montmorillonite MMT. The rheology and XRD-diffraction indicate that the interactions are not at the level of intercalation or even exfoliation.

Acknowledgement

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References

1. Alexandre M., Dubois P. *Mater. Sci. Eng. 28*, 1-63 (2000).
2. DePaiva L.B., Morales A.R.: Valenzuela Diáz F.R.: *Appl. Clay Sci. 42*, 8-24 (2008).
3. Liptáková T., Alexy P., Gondár E., Khunová V., in: *Polymérne konštrukčné materiály*, EDIS, 2012. ISBN 978-80-554-0505-6.
4. Khunová V., in.: *Odhalené tajomstvá chémie,* Polymérne nanokompozity, VEDA, 2008. ISBN 978-80-224-0957.
5. Boháč P., Nógellová Z., Šlouf M., Kronek J., Jankovič Ľ., Peidayesh H., Madejová J., Chodák I.: Materials 17(16), 4017 (2024). https://doi.org/10.3390/ma17164017.

RECYCLING OF POLYURETHANE – INDUSTRY POINT OF VIEW

Petr MONTAG

BASF spol. s r.o., Radlicka 354/107b, 15800Praha 5, Czech Republic
email: Petr.a.Montag@basf.com

Recycling of polyurethanes is a constantly discussed topic both at the scientific and industrial levels. Many technologies, not only, for the chemical conversion are scientifically described. Primarily then for flexible, semi-rigid, and rigid polyurethane foams back into polyol, or polyol and an intermediate for the production of isocyanate in the form of methylene-diphenyl-amine, from which methylene-diphenyl-diisocyanate can be produced again. A new possibility is the mechanical recycling of polyurethanes. This new technology builds polyol with weak crosslinking suitable for many industries.

Although many technological processes have been developed, recycling is being introduced into industrial scale relatively slowly. There are several factors influencing this slow process. These include so far unclear European legislation, market reluctance to proactively accept recycled materials, and the lack of infrastructure in the form of collection, sorting, logistics.

Despite existing obstacles, projects with recycled polyurethane materials in the automotive industry[1], construction[2], and furniture manufacturing[3] have been successfully initiated.

Some example of scaled up running projects:

Scheme 1. Steering wheels polyurethane foam recycling running project.

Scheme 3. Recycled polyurethane foam in furniture[2].

The company BASF aims to increase the sales of recycled materials from 5 billion euros in 2023 to 10 billion euros by 2030. BASF is open to cooperation at the industrial level.

References:

1. BASF Press release P169/24e. Published on April 15th, 2024.
2. https://www.rampf-group.com/en/news/2022/go-create-something-new-from-old-pur-and-close-the-material-loop-for-pur-rigid-foams/. Available on April 30th, 2025.
3. https://www.vitra.com/en-us/magazine/details/. V-Foam. Available on April 30th, 2025.

POSSIBILITIES FOR DEVULCANIZATION OF RUBBER CRUMB FROM TIRES USING MECHANICAL AND CHEMICAL PROCESSES

Ján HRONKOVIČ[1]*, Jozef PREŤO[1], Ján KRUŽELÁK[2] and Michaela DŽUGANOVÁ[2]

[1] Vipo a.s., Gen. Svobodu 1069/4, 958 01 Partizánske, Slovakia
[2] Slovak University of Technology in Bratislava, Faculty of Chemical and Food Technology, Institute of Polymer Materials, Radlinského 9, 812 37 Bratislava, Slovakia
email: jan.hronkovic@vipo.sk

Abstract

Untreated and treated rubber crumbs by devulcanization were incorporated into SBR based rubber compound in the amount of 25wt.%. The goal of this work was to find a devulcanization process that will require the lowest energy and time consumption. First, based on the output data, the most suitable mechanical and temperature conditions for the processing of rubber crumbs were selected, and then the chemical compositions for devulcanization of the crumbs were tested under these conditions. The experimental work was aimed at the investigation of the effects of treated rubber crumbs on the vulcanization characteristics, physical-mechanical and rheological properties of rubber compounds. Devulcanization was based on thermomechanical, or subsequently mechanical treatment of rubber crumb with a chemical composition in a calender. It was found that the addition of devulcanized rubber crumb has a positive influence on the several observed parameters of rubber compounds compared to untreated rubber crumb.

Introduction

The environmental problems created by waste rubber and discarded tires have become a serious issue. Many attempts have been made to recycle waste rubber for both environmental and economic reasons. The recycling of discarded tires has some very important outcomes including environmental protection, energy savings and provision of raw materials. Therefore, the development of an efficient way to utilize rubber waste is an emergent economic and environmental task faced by the rubber industry worldwide [1]. Rubber recycling is a difficult task because of the vulcanization reaction, which takes place in three-dimensional structures. After undergoing reactions, vulcanized rubber turns into an infusible and insoluble form and cannot be converted into other forms [2]. The crosslinks in the main skeleton of the polymer, which are formed during the vulcanization process, turn the thermoplastic structures into thermosets that are impossible to reshape by heating. Therefore, physical and chemical treatments for waste tires are necessary to destroy the three-dimensional structure [3]. A lot of study has revealed that ground tire rubber (GTR) recycling should target the production of thermoplastic elastomers, and its reuse in the rubber industry [4]. Rubber recycling currently is receiving special attention, because other disposal methods, such as burning for energy generation, are dangerous for the environment. Some new physical and chemical devulcanization techniques for recycling are being developed to recycle used tires [5].

In this work, the chemical composition comprises one or more chemical compounds, which can perform the rubber accelerator function and one or more activators. The compounds, which are used conventionally as rubber accelerators, when used co-jointly with one or more activators capable of initiating proton exchange at temperatures below 70°C must be capable of opening or delinking the vulcanized network of the sulfur-cured elastomeric material to provide sulfur-curable reclaimed elastomeric material. Preferably, the compounds, which may perform the function of rubber accelerator comprise thiocarbamates and thiazoles, in the molar ratio in the range of 1:3 to 1:6. The accelerators are activated by one or more activators, preferably stearic acid and zinc oxide. The presence of a small amount of sulfur was found to aid ultimate vulcanization but is not necessary. Similarly, the presence of diols may help in the dispersion of the powders and perhaps activate the mixture, but this is not essential [6].

Experimental

Truck tire crumb as a waste material from the tread part of the tire, which is generated during the technology of retreading truck tires. First, the mechanical parameters for the calender were chosen, such as a 0.2 mm gap and a friction of the cylinders in a ratio of 1:2. Subsequently, the optimal temperature of the process was selected for further testing, while the values achieved were 65, 95, 105, 150°C. The results obtained from thermomechanical devulcanization showed that the process without additional heat was the most suitable for subsequent chemical devulcanization. This was matched by a rubber crumb temperature of 65°C during processing. The devulcanization of rubber crumbs was performed in the calendar by adding 6 parts of the chemical composition and 100 parts of rubber crumb at temperature below 70°C. A total of four types of composition were tested. The first tested composition (COMP) was unknown, commercially used. The composition AD112S consists of 24% rubber matrix, the accelerators MBT and ZDMC in molar ratio 8:1 and two activators with sulfur and was first prepared in Banbury mixer before being mixed with the crumb. These two compositions were mixed according to procedure A and the other compositions TDT and TD were directly mixed with the crumb according to procedure B as described in Table 1. The composition TDT consists of the MBT, diphenyl disulfide and thio phthalimide in molar ratio 4:1:1. The composition TD consists of MBT and diphenyl disulfide in molar ratio 2:1.

From the results obtained in our previous work it was decided to examine the influence of the rubber crumb as an addition on selected properties of styrene-butadiene rubber compound [7,8]. The compounds consisted of 25wt.% untreated, or devulcanized rubber crumb. The compounds with untreated (UNDC) and no rubber crumb (REF) were prepared to compare. In the first step the rubber compounds were prepared in Banbury mixer at mixing temperature 120°C and rotor speed of 60 rpm. In the second step, the curing additives were added to the calender at a mixing temperature of 60°C.

Table 1) the mixing procedures

Two roll mill mixing procedure A:	time (min)	**Two roll mill mixing procedure B:**	time (min)
1. passing rubber crumb through rollers	10	1. passing rubber crumb through rollers	5
2. adding a devulcanizing additive	5	2. adding a thiazole, diphenyl disulfide, resp. Thio phthalimide	5
		3. adding vulcanizing additives	5

The vulcanization characteristics measurements were carried out in a rheometer MDR 2000E at temperature 160 °C. The vulcanizates sample physical-mechanical properties were determined using Instron 3365 at crosshead speed 500 mm/min according to STN ISO 37. The rheological properties were measured at a dynamic mechanical rheological tester RPA 2000EDR. The surface morphology and microstructure were observed using scanning electron microscope JEOL JSM-7500F.

Results and Discussion

The results of vulcanization characteristics show mainly that the vulcanization rate is increased for blends with chemical devulcanized rubber crumb, as seen in table 2. The rubber crumb in the SBR blends enables to enhance the minimal torque. The ability to crosslinking of rubber compounds, what is expressed by ΔS', is increasing in all chemical modified compounds, but most in the sample AD112S. No vulcanization system was added to the devulcanized crumb, which mainly affected the vulcanization rate and maximum torques, but also the tensile strength and modulus.

Table 2) The values of vulcanization parameters at temperature 160°C and at the measurement time 30 min

	REF	**UNDC**	**DC65**	**DC95**	**DC105**	**DC150**	**COMP**	**AD112S**	**TDT**	**TD**
TS2 (min)	3,81	4,06	3,06	3,95	3,97	3,71	1,75	1,16	2,30	2,03
TC90 (min)	9,12	9,02	7,35	8,76	8,69	8,45	3,86	3,92	5,95	5,47
S'_{Min} (dNm)	1,38	2,91	2,71	2,96	2,95	2,65	2,42	2,31	2,41	2,34
S'_{Max} (dNm)	16,47	12,34	12,50	12,43	12,30	11,45	16,01	17,10	14,66	15,19
ΔS' (dNm)	15,09	9,43	9,79	9,47	9,35	8,80	13,59	14,79	12,25	12,85
Rate(S'/min)	3,93	2,18	2,71	2,31	2,28	2,09	9,51	11,03	4,55	5,37

The influence of chemically devulcanized rubber crumb on rheological properties of tested compounds was evaluated by obtained values of loss angle *tan δ* by measuring the deformation and temperature sweep on the rubber process analyzer as shown in Figure 1. Loss angle *tan δ* is for chemically untreated rubber crumb lower than for devulcanized rubber crumb. These rheological properties indicate a capability to devulcanization process.

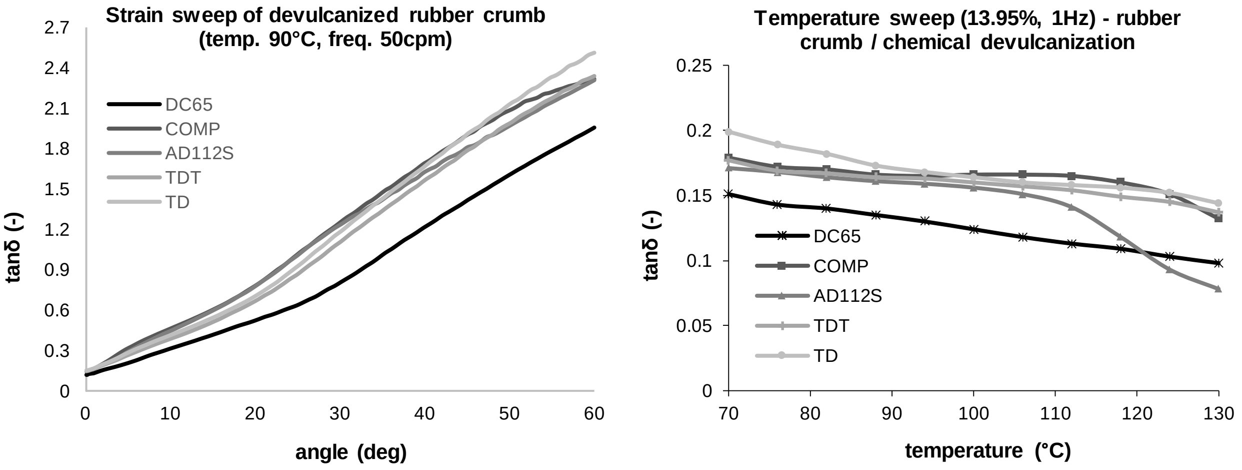


Figure 1) a) Strain and b) Temperature dependencies of loss angle *tan δ* of devulcanized rubber crumb

The selected tensile parameters as tensile strength, elastic modulus M300 and elongation at break of the prepared vulcanizates are shown in Figure 2. The addition of mechanically, resp. thermomechanically prepared rubber crumb to the SBR compound resulted mainly in a decrease of the tensile strength. The values, such as the highest elongation at break, low modulus and hardness were achieved by the sample DC65 containing rubber crumb prepared without using temperature. This indicates the formation of a network with longer sulfur chains and for these reasons it was decided to test this sample using chemical composition.

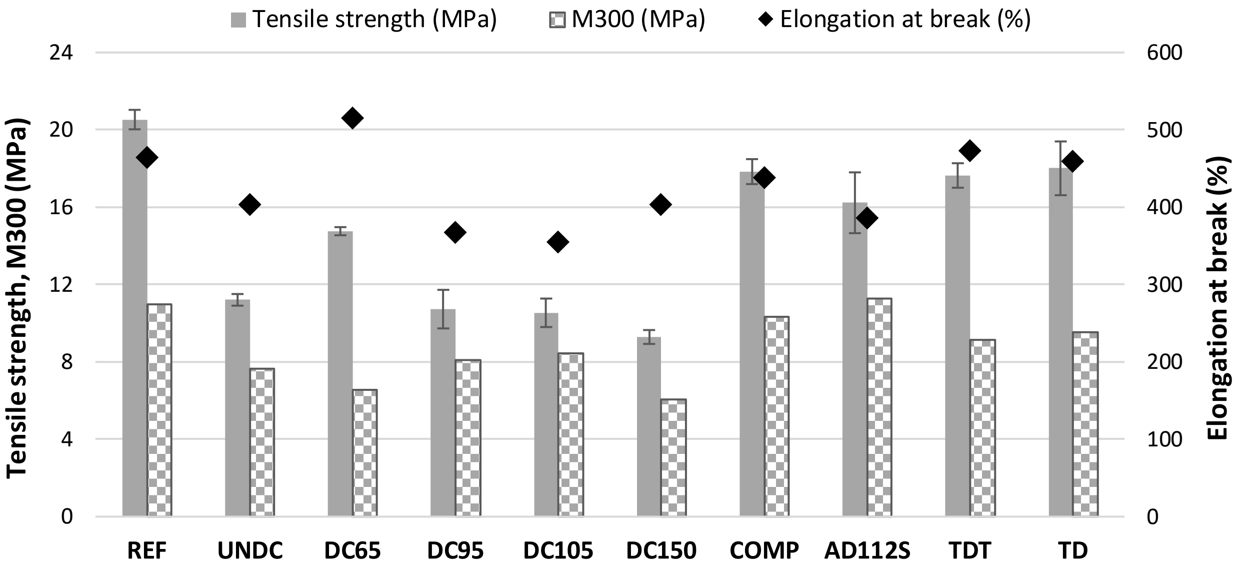
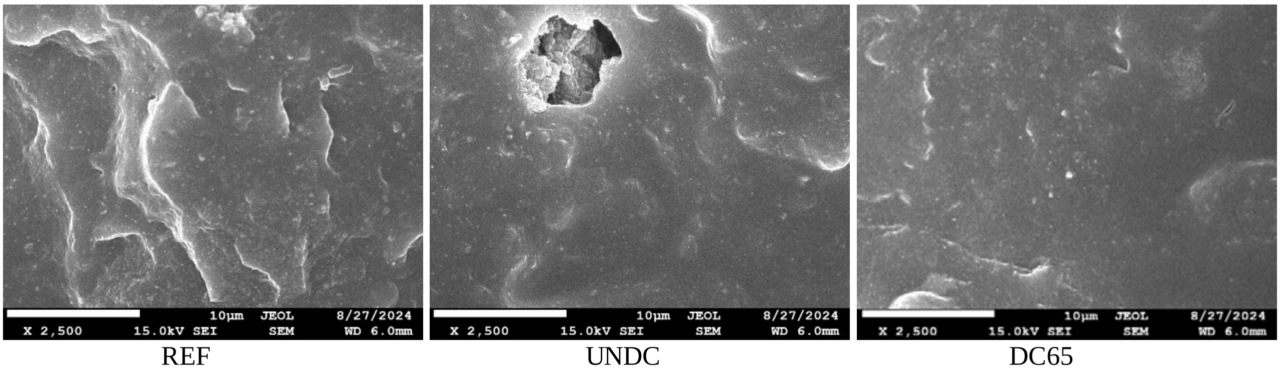


Figure 2) Chosen tensile parameters at standard conditions of rubber compounds

The devulcanization of rubber crumb by adding chemical compositions achieved higher values of tensile strength and modulus, but partially reduced elasticity. Compared to the commercial product (COMP), better tensile properties were recorded for samples with a composition containing diphenyl disulfide (TDT, TD).



REF UNDC DC65

Figure 3) SEM images of SBR compound (REF), SBR compound with untreated rubber crumb (UNDC) and SBR compound with mechanically devulcanized rubber crumb (DC65).

On the surface structure of the SBR compound with untreated rubber crumb (UNDC) were observed several holes by employing Scanning Electron Microscopy as described in Figure 3. Structures disturbed in this way were probably created by weak interactions at the interphase polymer matrix – rubber crumb.

Conclusions

The devulcanization of rubber crumb improves all monitored properties of tested SBR compounds. The rheological properties of investigated rubber crumb determine a certain capability of devulcanization. The addition of mechanically, resp. thermomechanically prepared rubber crumb to the SBR compound resulted mainly in a decrease in the tensile strength, but with adding the chemical composition, better tensile values were achieved. The results obtained from measurements imply that devulcanization of rubber crumb has a significant effect on the process of vulcanization, tensile, rheological and microstructural properties.

Acknowledgement

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References

1. Lamminmaki J, Li S, Hanhi K. *J. Mater. Sci*. 2006, 41, 8301–8307.
2. Rooj S, Basak GC, Maji PK, Bhowmick AK. J. Polym. Environ. 2011, 19, 382-390
3. Scuraccio CH, Waki DA, Silva MLCP. J. Therm. Anal. Calorim. 2007, 87, 893-897
4. Karger-Kocsis J, Meszaros L, Barany TJ. Mater. Sci. 2013, 48, 1–38.
5. Zanchet A, Carli LN, Giovanela M, Crespo JS, Scuracchio CH, Nunes RCR. J.Elast. Plas. 2009, 41, 497-507
6. Pat. US 5,770,632 (1998)
7. Hronkovič J., Preťo J., Trnovec J., Gažovič P., Vereš J., Chodák I.; The rubber crumb processing in rubber compound. In: Book of Abstracts from 14th ISE ‘14, Bratislava 2014, p. 159-161.
8. J. Hronkovič, J. Preťo, J. Vereš, I. Chodák, J. Oravec: The Appropriate Composition to Modify Rubber Crumb and its Application in Rubber Compounds. In: PMA 2015 & SRC 2015, Bratislava 26-28th May 2015, Book of Proceedings, ISBN 978-80-970923-7-5, s. 196-199

LIGNIN AND DEVULCANIZED TIRE CRUMB FOR NEXT-GENERATION RUBBER COMPOSITES

Michaela DŽUGANOVÁ[1]*, Katarína TOMANOVÁ[1], Viera JANČOVIČOVÁ[1], Ján HRONKOVIČ[2] and Ján KRUŽELÁK[1]

[1] Institute of Natural and Synthetic Polymers, Faculty of Chemical and Food Technology, Slovak University of Technology in Bratislava, Radlinského 9, 81237 Bratislava, Slovakia
[2] VIPO a.s., Gen.Svobodu 1069/4, 958 01 Partizánske, Slovakia
email: michaela.dzuganova@stuba.sk

Introduction

Elastomers are a unique class of polymers with diverse applications, from medical devices to the tire industry. With automobiles being an essential part of modern life, the demand for tire rubber has surged in recent decades. Millions of tires are produced annually, eventually becoming waste. Due to their large volume and environmental impact, tire waste poses a significant ecological challenge. Recycling waste tire rubber is crucial for both environmental sustainability and economic efficiency[1–3].

Ground tire rubber (GTR), the primary commercially available source of recycled rubber, varies in quality depending on tire type (e.g., truck, passenger, off-road) and regional conditions, though overall composition remains similar. Typically, GTR consists of 58–60 wt% natural/synthetic rubber (NR, IR, BR, SBR), 27–28 wt% carbon black and silica, 3–4 wt% vulcanization agents, and 8–11 wt% additives such as antioxidants, antiozonants, and softeners[2,4]. The vulcanization systems that are used to crosslink rubber polymers during vulcanization, create very strong chemical bonds and products that do not easily decompose. Vulcanization enhances the physical and mechanical properties of elastomers through cross-linking, but it also complicates recycling. Devulcanization aims to break C–S and S–S bonds (cross-links) while preserving C–C bonds (the polymer backbone) in the rubber network. Although C–C bonds theoretically require more energy to break than C–S and S–S bonds, in practice, both chain scission and cross-link breakage occur during devulcanization. The most effective devulcanization method maximizes cross-link breakage while minimizing chain scission. Regardless of the devulcanization method, some of the main polymer chains are broken during devulcanization and, thus, the properties of the treated material are different compared to the original rubber. The use of devulcanized rubber can reduce the cost of final products. Depending on the desired application, the devulcanized rubber can be reused as obtained or mixed in different amounts with virgin rubber or other polymers. Environmental aspects should be considered, because rubber reclaiming process is required to be low- pollutant and the used components should be non-toxic[4]. This paper describes mechanical devulcanization (or reclaiming) of rubber crumb, the influence of kraft lignin on the devulcanization process and compounding such devulcanized rubber crumb with virgin SBR compound.

Materials

Butadiene-styrene rubber (KER 1502, SynthosGroup, PL) was used as a rubber matrix filled with 50 phr of carbon black (CONTINEX N330, Continental Carbon Company, Belgium). Sulfur vulcanization system consisting of 5 phr of zinc oxide (Slovzink a.s., Košeca, Slovakia), 2 phr of stearic acid (Setuza, Ústí nad Labem, Czech Republic), 1.5 phr of N-tert-butyl-2-benzothiazolesulfenamide (WESTCO TBBS, Western Reserve Chemicals, Akron OH, USA) and 2 phr of sulfur (Siarkopol Tarnobrzeg, Poland) was used. Above mentioned components were used for preparation of virgin SBR compound, which was used as a reference and subsequently mixed with devulcanized rubber crumb and lignin. Devulcanized rubber crumb (RC), obtained as a waste from tread part of truck tires (A.R.S. spol. s.r.o. Prievidza, Slovakia), which is generated during the process of retreading truck tires, was incorporated at 25 wt.%. To further enhance sustainability, an additional 5 wt. % of the virgin compound was substituted with kraft lignin (BioPiva 100, UPM Biochemicals, Finland). Glycerol (purity ≥99%, Sigma-Aldrich, St.Louis, MO, USA) was added as a plasticizer to improve processing and enhance the lignin/rubber interface. Based on previous research, a glycerol content of 20 wt. % relative to the lignin content was used[5].

Experimental

Devulcanization of the RC was carried out by mechanical treatment on a two-roll mill (Buzuluk 400x400, Buzuluk Inc., Komárov, Czech Republic), with roller friction 1:2. The processing time was 15 minutes and the temperature of RC during processing was kept at 65°C (RC 65). One sample (RCL) was processed together with 5 wt.% of lignin, under the same conditions as RC 65. To evaluate the devulcanization process, and unprocessed RC was also included in the experiment.

Subsequently rubber compounds were prepared using a two-step mixing process. The first step was carried out in a semi-industrial kneading machine (Buzuluk Inc., Komárov, Czech Republic) set at 100°C and 60 rpm. Initially, the rubber was masticated for 1 minute, followed by the addition of RC 65 and lignin (or RCL) and glycerol, and mixed for another minute. Additives and half of the carbon black were then introduced and mixed for 2 minutes. Finally, the remaining carbon black and processing oil were added and mixed for an additional 2 minutes. The resulting compound was shaped into strips and homogenized using a two-roll mill. After cooling, the second mixing step was performed on the two-roll mill, where TBBS and sulphur were blended into the compound at 70°C for 8 minutes.

The FTIR spectra were measured with the FTIR spectrophotometer Nicolet iS5 FTIR Spectrometer with iD7 ATR accessory (Thermo Scientific, Waltham, MA, USA), using the ATR adapter with single reflection germanium crystal. The vulcanization characteristics measurements were carried out in a rheometer MDR 2000E at temperature 160 °C. The vulcanizates sample physical-mechanical properties were determined using Zwick Roell/Z 2.5 appliance (Zwick GmbH&Co.KG, Ulm, Germany), according to technical norm ISO STN 37. The vulcanization characteristics were investigated using an oscillatory rheometer (MDR 2000, Alpha Technologies, Akron, OH, USA) according to the STN 62 1416 at a temperature of 160°C. Mechanical properties were measured using a Zwick Roell/Z 2.5 appliance (Zwick GmbH & Co.KG, Ulm, Germany) in accordance with the technical norm ISO STN 37. The cross-link density was calculated using the Flory-Rehner equation modified by Krause [6], based on the equilibrium swelling state. For the calculations, the Flory-Huggins interaction parameters were used, which for SBR in xylene is 0.3908. The surface morphology and microstructure of composites were observed using a scanning electron microscope JEOL JSM-7500F (Jeol Ltd., Tokyo, Japan). The samples were initially frozen in liquid nitrogen, fractured, and then coated with a layer of gold on the fracture surface. The electron source used was a cold cathode ultra-high vacuum (UHV) field emission gun. The acceleration voltage ranged from 0.1 to 30 kV, resulting in a resolution of 1.0 nm at 15 kV and 1.4 nm at 1 kV. Scanning electron microscopy (SEM) images were captured by CCD-Camera EDS (INCA X-ACT, Oxford Instruments, Abingdon, UK).

Results & discussion

During the devulcanization process, crosslink scissions generate a significant number of free radicals, which tend to slowly re-crosslink over time. To address this issue, the radical scavenging activity of lignin is proposed as a potential stabilizing strategy. The antioxidant capability of lignin arises primarily from the homolytic dissociation of its phenolic O–H bonds. This process, characterized by its bond dissociation enthalpy, enables lignin to act as a radical stabilizer by transferring hydrogen atoms to the polymer-based radicals, thereby neutralizing them and inhibiting further crosslinking[7–9].

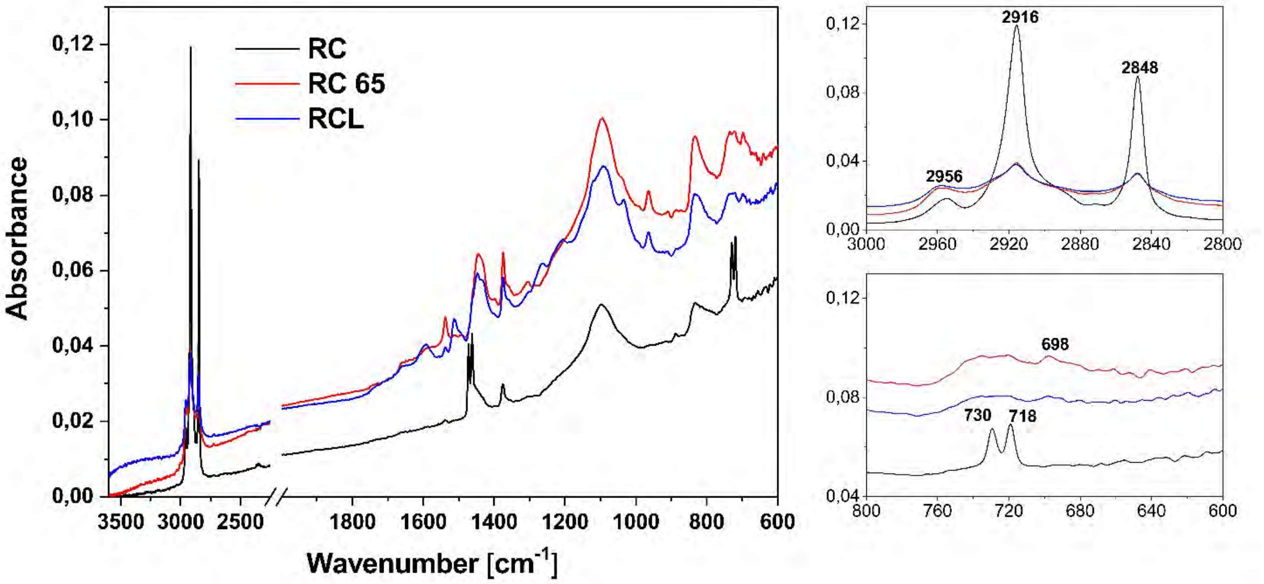

Figure 1: FTIR (ATR Ge) spectrum of reference sample RC and devulcanized rubber crumb RC65 and RCL in MIR region (left) and their sections in the region of the alkane bands (top right) and the polysulfide and sulphide bands (bottom right)

Figure 1presents the FTIR spectra of reclaimed rubber (RC) processed with and without lignin. The analysis focused on changes in the alkane stretching vibration region (3000–2800 cm^{-1}) as well as the 600–800 cm^{-1} region. RCL exhibited a higher A2956:A2916 ratio compared to RC 65. This suggests a lower degree of main elastomer chain scission in the presence of lignin, pointing to its potential stabilizing effect during the devulcanization process. Additionally, the lower A698:A730 ratio indicates higher C-S-C or C-S-H bonds in proportion to R-Sx-R for RCL when compared to RC 65, indicating preferred R-S$_x$-R scission.

The reclaimed rubber samples RC 65 and RCL were subsequently compounded with a virgin SBR blend. The vulcanization characteristics of these blends are presented in Figure 2. Compared to the reference SBR sample, all key parameters show a decrease. The decrease in the value of the maximum rheometric torque may be due to the presence of short rubber chains and crosslink precursors in the RC 65 and RCL. The behaviour of lignin containing samples can be also attributed to the steric hindrance introduced by lignin as well as its polar nature.

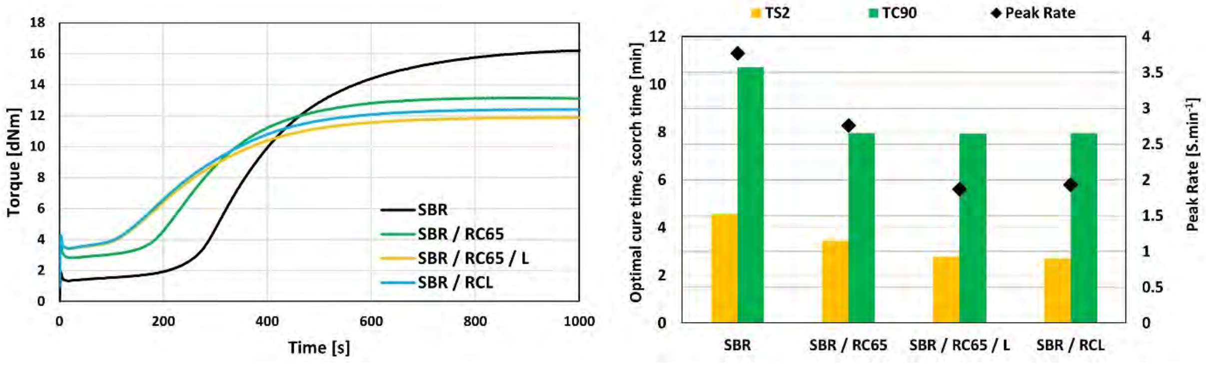

Figure 2: Cure behaviour of tested rubber samples

The mechanical properties of the composites are summarized in Figure 3a. The addition of RC in general reduces tensile strength and moduli compared to reference. Incorporating lignin in the SBR/RC65/L composite further decreases these properties because lignin displaces virgin rubber, reducing the available vulcanizable matrix. In this role, lignin behaves as an inert filler - forming domains that do not participate in cross-linking - consistent with the lower cross-link densities shown in Figure 3b. In contrast, when RC and lignin are pre-processed together on a two-roll mill before incorporation (SBR/RCL), the resulting composite's tensile strength matches that of SBR/RC65, and its 300% modulus is approximately 2 MPa higher than in SBR/RC65. This improvement suggests altered cross-linking behaviour and supports the hypothesis that lignin participates in the devulcanization of RC during processing. The higher cross-link density observed in SBR/RCL indicates an improved ability to undergo revulcanization, possibly due to the preservation of longer polymer chains during the devulcanization treatment.

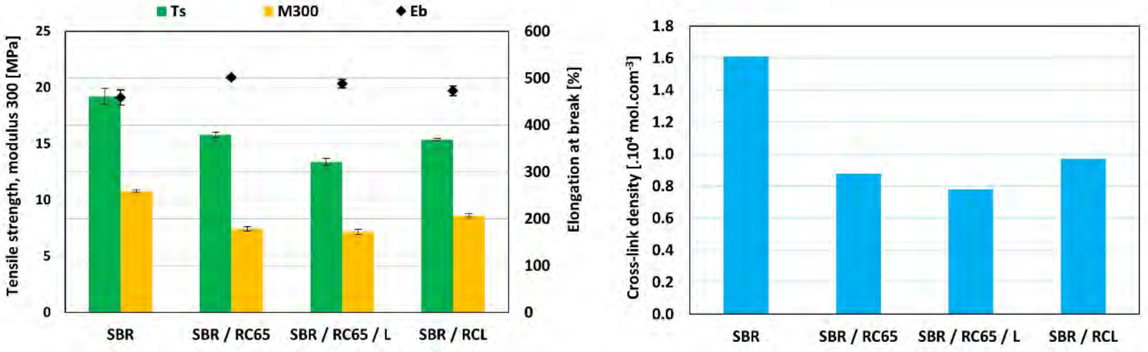

Figure 3: Mechanical properties (a) tensile strength, modulus 300 and elongation at break and (b) cross-link density of studied composites

Figure 4 shows the fracture surfaces of SBR/RC65/L and SBR/RCL samples. While both samples appear structurally similar, the SBR/RCL shows better interphase adhesion between rubber matrix and RCL. This observation further supports earlier claims regarding the beneficial role of lignin in the devulcanization process and its continued influence on the re-vulcanization of next-generation rubber composites and also explains the superior mechanical properties of SBR/RCL.

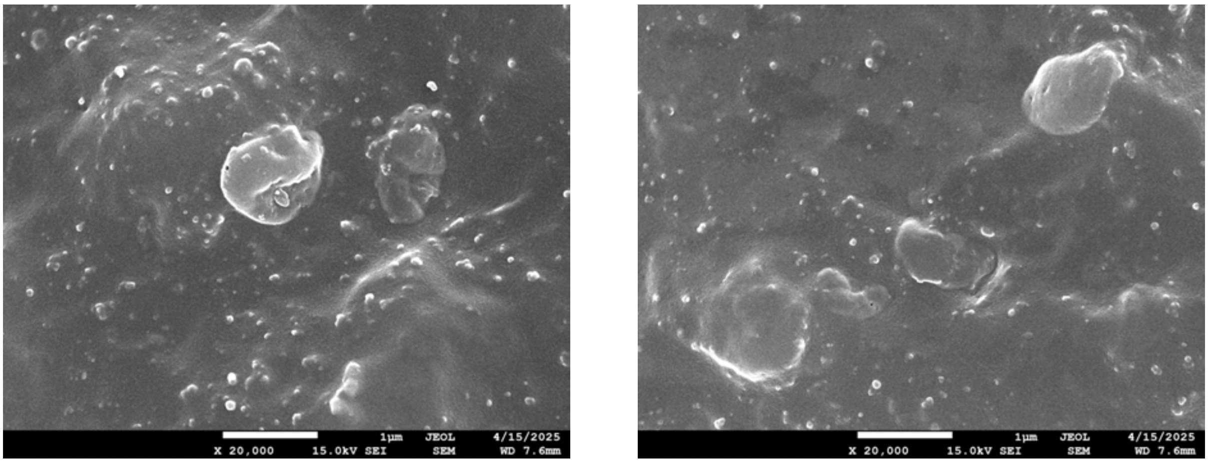

Figure 4:SEM analysis of SBR / RC65 / L (left) and SBR /RCL (right) at 20 000x magnification

Conclusion

This study demonstrated that kraft lignin can play a beneficial role in the devulcanization and reprocessing of rubber crumb obtained as a waste from tread part of truck tires, for use in next-generation rubber composites. Mechanical devulcanization on a two-roll mill, particularly when combined with lignin, resulted in reclaimed rubber with improved properties. FTIR analysis indicated that lignin helps preserve the integrity of the polymer backbone while enhancing selective cross-link scission. When incorporated into a virgin SBR matrix, the pre-treatment of rubber crumb with lignin improved cross-link density and mechanical properties compared to untreated or post-mixed lignin systems. SEM analysis further confirmed better incorporation of reclaimed RCL into the virgin rubber matrix. These findings suggest that kraft lignin not only contributes to the sustainability of rubber recycling but also enhances the performance of the resulting composites, making it a promising additive for circular economy applications in the rubber industry.

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References

1. Shabani M, Jamshidi M. Recycling NR/SBR waste using probe sonication as a new devulcanizing method; study on influencing parameters. *RSC Adv*. 2022;12(40):26264-26276. doi:10.1039/D2RA04726H
2. Seghar S, Asaro L, Rolland-Monnet M, Aït Hocine N. Thermo-mechanical devulcanization and recycling of rubber industry waste. *Resour Conserv Recycl*. 2019;144:180-186. doi:10.1016/j.resconrec.2019.01.047
3. Goevert D. The value of different recycling technologies for waste rubber tires in the circular economy—A review. *Front Sustain*. 2024;4. doi:10.3389/frsus.2023.1282805
4. Wiśniewska P, Wang S, Formela K. Waste tire rubber devulcanization technologies: State-of-the-art, limitations and future perspectives. *Waste Manag*. 2022;150:174-184. doi:10.1016/j.wasman.2022.07.002
5. Kruželák J, Hložeková K, Kvasničáková A, Džuganová M, Chodák I, Hudec I. Application of Plasticizer Glycerol in Lignosulfonate-Filled Rubber Compounds Based on SBR and NBR. *Materials*. 2023;16(2):635. doi:10.3390/ma16020635
6. Kraus G. Swelling of filler-reinforced vulcanizates. *J Appl Polym Sci*. 1963;7(3):861-871. doi:10.1002/app.1963.070070306
7. Guo L, Wang C, Lv D, et al. Rubber reclamation with high bond-breaking selectivity using a low-temperature mechano-chemical devulcanization method. *J Clean Prod*. 2021;279:123266. doi:10.1016/j.jclepro.2020.123266
8. Troncoso-Ortega E, Castaño-Rivera P, Romero R, et al. Organic radicals stabilization in natural rubber: Discerning the influence of thermo-oxidation using chemically modified or unmodified lignin as antioxidant. *J Ind Eng Chem*. 2024;137:435-447. doi:10.1016/j.jiec.2024.03.029
9. Barana D, Ali SD, Salanti A, et al. Influence of Lignin Features on Thermal Stability and Mechanical Properties of Natural Rubber Compounds. *ACS Sustain Chem Eng*. 2016;4(10):5258-5267. doi:10.1021/acssuschemeng.6b00774

DEVELOPMENT OF COMPLEX TESTING METHODOLOGY OF TYRE ABRASION

Anna SMETANOVÁ[1], Jiří BREJCHA[1]*, Zdeněk HRDLIČKA[2] and Libor BENEŠ[1]

[1]Faculty of Mechanical Engineering, J. E. Purkyně University in Ústí nad Labem, Czech Republic
[2]Department of Polymers, University of Chemistry and Technology, Prague, Czech Republic 3rd year of Bc, study programme Engineering Technology
email: jiribrejcha@seznam.cz

With the continual increase in road traffic, tyre abrasion has become a significant environmental and materials science challenge. The particles generated by tyre wear contribute to microplastic pollution, raising concerns about their ecological and health-related impacts. The study of tyre abrasion can be approached from two main perspectives: the environmental implications of wear particle release and the mechanical behavior of rubber compounds under stress. This study focuses on predicting and evaluating the material behavior of rubber compounds with respect to their wear resistance.

Tyre wear results from a complex interaction of mechanical, chemical, and environmental factors. Key contributors include the frictional interaction between the tyre and road surface, the specific composition of the rubber compound, and external conditions such as temperature and humidity. Under laboratory conditions, various test methods are used to replicate and predict the wear performance of tyres. However, a universal standardized testing protocol has yet to be established.

This research investigates how both the methodology of testing and the composition of rubber compounds influence the resulting wear characteristics. A comprehensive experimental approach was adopted, including:

- Dynamic Mechanical Analysis (DMA) to assess the viscoelastic properties of the rubber compounds and their behavior under dynamic loading conditions.
- DIN abrasion testing to quantify material loss under standardized mechanical abrasion.
- Laser Induced Breakdown Spectroscopy (LIBS) for elemental analysis of worn surfaces and detection of possible compositional changes due to abrasion.
- Microscopic analysis, including both optical and scanning electron microscopy (SEM), to investigate surface morphology and wear mechanisms at the microstructural level.

These methods will be used to establish correlations between material composition, test methodology, and wear performance. The results will contribute to a better understanding of rubber compound behavior and may support the development of more durable, environmentally sustainable tyre materials.

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References

1. GENT, A., 1989. A hypothetical mechanism for rubber abrasion. Rubber Chemistry and Technology, 62(4), 750–756. https://doi.org/10.5254/1.3536272&#8203.
2. HAN, D., WANG, K., WANG, C., & HAN, W., 2022. The effect of different dosages of TESPT on metal friction and metal wear in the mixing process. Polymers, 14(12), 2314. https://doi.org/10.3390/polym14122314&#8203.
3. LI, D., 2017. Abrasive wear. In: ASM Handbook. Volume 18: Friction, Lubrication, and Wear Technology. s. 243–251. https://doi.org/10.31399/asm.hb.v18.a0006382&#8203.
4. PULFORD, C., 1985. Failure of rubber by abrasion. Rubber Chemistry and Technology, 58(3), 653–661. https://doi.org/10.5254/1.3536083&#8203.
5. KOMÁREK, J., 2015. Opotřebení běhounových směsí silně namáhaných pneumatik. Diplomová práce. UTB ve Zlíně.
6. MYERS, R. R., 2000. Natural Rubber and Its Use in Modern Tires. Tire Science and Technology, 28(4), 264–278.
7. SHANG, S. et al., 2014. Effects of BR and SBR Blends on Wear Resistance of Rubber Compounds. Polymer Testing, 39, 84–90. https://doi.org/10.1016/j.polymertesting.2014.07.004.
8. AHMAD, N. et al., 2019. Influence of Rubber Matrix on Abrasive Wear Behavior.

EFFECT OF A MULTICOMPONENT HALOGEN-FREE FLAME RETARDANT ON THE PROPERITES OF POLYETHYLENE FOR THE CABLE INDUSTRY

Anna VYKYDALOVÁ[1]*, **Ján ŠIPLA[2], Petr KADLEC[2] and Radek POLANSKÝ[2]**

[1] *Polymer Institute, Slovak Academy of Sciences, Dúbravská cesta 9, 845 41 Bratislava 45, Slovakia*
[2] *Department of Materials and Technology, Faculty of Electrical Engineering, University of West Bohemia, Univerzitní 8, 306 14 Pilsen, Czech Republic*
email: anna.vykydalova@savba.sk

Polymers are widely used in everyday life as well as in specialized applications (packaging, construction or electrical industries, etc.). However, their poor flame retardant properties as well as durability limit some of their applications[1]. In general, polymer combustion is based on a combination of energy feedback from the flame to the polymer surface and the formation of flammable degradation products from polymer gasification[2]. To protect the polymer from fire, it is necessary to reduce the released heat and also to reduce the amount of flammable volatile substances emitted during the combustion of the polymer. Therefore, it is necessary to incorporate the flame retardants (FR) to the polymer matrix, since they are able to reduce flammability of polymer[1]. In general, the addition of flame retardants can also change the thermal and thermooxidative stability of the material. For this reason, it is necessary to study the effect of FR on the thermal and thermooxidative stability and dielectric properties of polymers and try to predict the lifetime of the newly developed materials[3,4].

Polymer composites based on linear low-density polyethylene (LLDPE) with halloysite nanotubes (HNT) offer great promise for developing low-fire hazard cables due to their proven fire-retardant properties. However, incorporating HNT into a non-polar polymer matrix inevitably changes the composites' polarity, homogeneity, and thermal properties. Consequently, this can lead to a degradation of the dielectric and mechanical properties. This work describes such changes for LLDPE/HNT composites that differ in the amount of HNT present in the LLDPE matrix. A series of LLDPE/HNT composites with filling levels of 1, 3, and 7 wt% of HNT and pure LLDPE as the reference material were prepared by extrusion. Dielectric properties such as volume resistivity, dielectric strength, complex relative permittivity, and dissipation factor were determined. Tensile tests and cone calorimetry were also carried out to enhance the characterization of the tested materials regarding dielectric properties and other parameters necessary for using the tested materials as cable insulation. Adding HNT to LLDPE up to 7 wt% has no substantial effect on the mechanical parameters. However, due to its molecular polarity, HNT significantly deteriorates the dielectric parameters at 7 wt% of HNT. The results obtained using the cone calorimeter show that adding HNT reduces the value of the maximum heat release rate curve and the burning time. Burning of composites with HNT does not produce more smoke than burning of reference LLDPE.

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References

1. Jasinski E., Bounor-Legaré V., Taguet A., Beyou E., Polym. Degrad. Stab. 183 (2021) 109407.
2. Dasari A., Yu Z.Z., Cai G.P., Mai Y.W., Prog. Polym. Sci. 38 (2013) 1357–1387.
3. Vykydalová, A., Dubaj, T., Cibulková, Z., Mizerová, G., Zavadil, M., Polym. Degrad. Stab. 158 (2018) 119–123.
4. Polanský R., Kadlec P., Kolská Z., Švorčík V., Appl. Clay Sci. 147 (2017) 19–27.

ELECTRICAL CONDUCTIVITY BEHAVIOR OF RUBBER COMPOSITES WITH VARYING CROSSLINK DENSITY UNDER CYCLIC MECHANICAL DEFORMATION

Hamed PEIDAYESH* **and Zdenko ŠPITALSKÝ**

Polymer Institute of the Slovak Academy of Sciences, Dúbravská cesta 9, 845 41 Bratislava, Slovakia
email: hamed.peidayesh@savba.sk

During the last decade, conductive polymer composites (CPCs) have been considered to be relatively cost-effective materials for many engineering applications, such as sensors, antistatic coatings and films, conducting adhesives, and electromagnetic interference shielding materials. For any possible application, the intimate relationship between the conductivity changes and external stimuli, such as electrical, thermal, mechanical, and chemical stresses, must be understood. From this point of view, mechanical deformation can be expected to be the most important factor affecting the electrical properties of flexible conducting composites. Although the practical application of this knowledge is clear, the mechanism of conductivity changes during mechanical deformation must be understood in detail to get reliable product. Investigating both electrical conductivity and mechanical properties in parallel using online measurements seems to be of particular interest. Moreover, to date, no investigation has been done to evaluate the effect of vulcanization time on the electrical conductivity of styrene-butadiene rubber (SBR) and ethylene-propylene-diene terpolymer (EPDM) rubber composites under mechanical tensions.

Herein, this contribution is focused on understanding the mechanism of conductivity changes during mechanical deformation for conductive rubber composites based on SBR or EPDM vulcanized for various times. Higher tensile strength, Young's modulus, and static conductivity values of the composite were observed with the increase in vulcanization time. The electrical conductivity during five runs of repeated cyclic mechanical deformations for SBR composites increased permanently, although not linearly, whereas EPDM composites showed a slight increase or at least a nearly constant current, indicating healing of minor defects in the conductive pathways or the formation of new conductive pathways. For the SBR composite, the current value starting from the second cycle increased with rising vulcanization time in the SBR composite, while the current values were nearly constant for all cycles in the EPDM composite.

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