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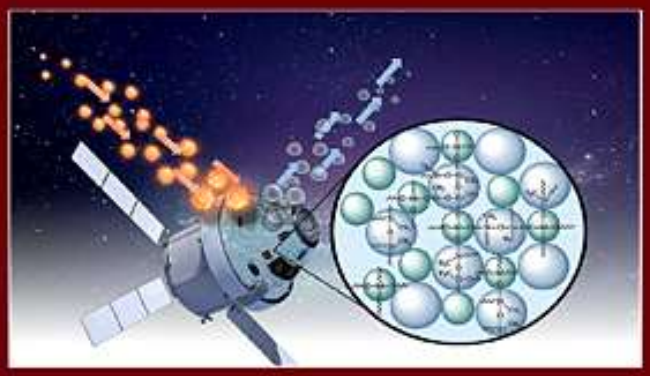

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2. Regular Papers

1. select article Development of an O-ring from NR/EPDM filled silica/CB hybrid filler for use in a solid oxide fuel cell testing system

Research articleAbstract only

Development of an O-ring from NR/EPDM filled silica/CB hybrid filler for use in a solid oxide fuel cell testing system

Sophos Chea, Montri Luengchavanon, Ekasit Anancharoenwong, Kua-anan Techato, ... Sutida Marthosa

Article 106568

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- Abstract

Abstract

This research presents the effects of oxygen pressure and ambient temperatures on the crack behavior of O-rings from a semi-EV of NR/EPDM rubber with silica/CB filler, exposed to the inlet flow and outflow oxygen pressure in a Solid Oxide Fuel Cell (SOFC) environment. Blends of NR/EPDM were prepared with various ratios of silica/CB filler at 00/60, 10/50, 20/40, 30/30, 40/20, 50/10, and 60/00 phr. The fabricated O-ring complied with the standard for O-rings (TIS 2728-2559), with a minimum hardness of 65–75 Shore A, minimum tensile strength of 9 MPa, minimum elongation at break of 200%, and a minimum 100% modulus of 2.7 MPa. The mechanical properties of the compounds were tested, and the appropriate compound was

chosen to make the O-rings to test in SOFC. The crack morphology of the fabricated O-rings was investigated and compared with a commercial O-ring after testing in the SOFC. As a result, the compound with silica/CB of 40:20 ratio provided the optimum mechanical properties, and passed the criteria standard of TIS 2728-2559. The mechanical properties of the prepared and commercial O-rings were similar (*P*-value of commercial with 60/00 = 0.273, 50/10 = 0.273, 40/20 = 0.144, 30/30 = 0.465, 20/40 = 0.465, 10/50 = 1.000 and 00/60 = 0.273; all > 0.05) and and both could still be continued to be used in SOFC despite some inner cracks after 24 h. The price of the prepared O-ring is cheaper than the commercial O-rings due to the low price of NR used in its formulation. Therefore, a prepared O-ring can be used in a SOFC, or other applications due to their mechanical properties and their reasonable price.

2. ☐ select article The sulfur reversion process in natural rubber in terms of crosslink density and crosslink density distribution

Research articleAbstract only

The sulfur reversion process in natural rubber in terms of crosslink density and crosslink density distribution

D. Bornstein, R.J. Pazur

Article 106524

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- Abstract

Abstract

Unfilled natural rubber compounds composed of conventional (CV), semi-efficient (SEV), efficient (EV) and sulfur donor (SD) vulcanization systems were heat aged to promote sulfur reversion. Rheometry, hardness, strain-strain characteristics including Mooney-Rivlin analysis, equilibrium solvent swell and

Double Quantum (DQ) Nuclear Magnetic Resonance (NMR) were used to monitor crosslink density changes. A loss of crosslink density was observed by rheometry, C_1, equilibrium swelling and by DQ NMR as a function of cure extent. No chain scission reactions were operating in the time/temperature conditions used. All crosslink distributions were unimodal and the network homogeneity followed the order of EV > SD > SEV > CV. The crosslink distribution narrowed during the curing process for the CV and SEV systems. Non-oxidative maturation reactions were advantageous in promoting a more random distribution of crosslinks in the polymer matrix.

3. select article Morphological characteristics and properties of TPS/PLA/cassava pulp biocomposites

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Morphological characteristics and properties of TPS/PLA/cassava pulp biocomposites

Phatcharin Jullanun, Rangrong Yoksan

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Abstract

The effect of cassava pulp (CP) on morphological, tensile, and thermal properties of a thermoplastic cassava starch (TPS)/poly (lactic acid) (PLA) blend was investigated. TPS/PLA/CP biocomposites were manufactured by melt extrusion and then converted into specimens using an injection molding. The weight fraction of PLA to TPS/CP was fixed at 40:60, whereas the final CP concentration in the composites was varied in the range of 4.4–22.1 wt%. CP could act as a reinforcement for TPS/PLA blend to enhance its tensile strength up to 354% and Young's

modulus up to 722% when 22.1 wt% of CP was loaded and a nucleating agent for PLA as confirmed from the reduced T_{cc}. In addition, TPS/PLA/CP composites showed a discrete phase structure (i.e., droplets in matrix) when CP with lower concentration (i.e., 4.4 wt%, 8.8 wt%, and 13.3 wt%) was incorporated and a bicontinuous phase structure (i.e., co-continuous) when higher concentration of CP (i.e., 17.7 wt% and 22.1 wt%) was employed. The results suggest that TPS/PLA/CP biocomposites have potential to be used in the manufacturing of injection-molded articles, particularly when biodegradability and renewability of the material are required.

4. ☐ select article Robust flower-like ZnO assembled β-PVDF/BT hybrid nanocomposite: Excellent energy harvester

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Robust flower-like ZnO assembled β-PVDF/BT hybrid nanocomposite: Excellent energy harvester

Gitanjali H. Tabhane, Sushama M. Giripunje

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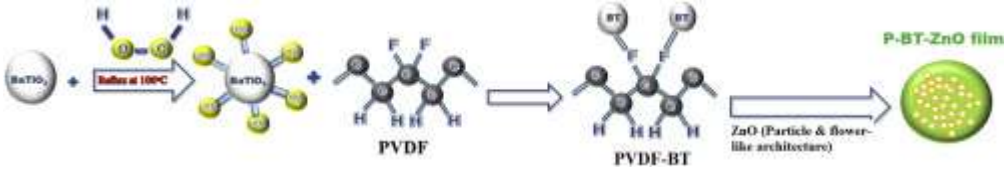

5. ☐ select article Assessment of thermally aged XLPE insulation material under extreme operating temperatures

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Assessment of thermally aged XLPE insulation material under extreme operating temperatures

Hongkun Lv, Tianhao Lu, Lingqi Xiong, Xianggao Zheng, ... Zhuo Li

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Abstract

The degradation of cross-linked polyethylene (XLPE) insulation material is critical for the safe operation of cables. While there are extensive studies on the thermo-oxidative ageing of XLPE at or below 130 °C, the maximum overload conductor operating temperature, the short circuit temperature can rise above 150 °C, and the ageing patterns at these situations have not been fully understood. Here, chemical, crystalline structure, cross-linking density as well as the mechanical properties were characterized. The results demonstrate the importance of antioxidants in the high temperature situation. Before antioxidants are completely consumed, most of the mechanical and physicochemical properties remain stable. While antioxidant is depleted, the thermo-oxidative degradation is detrimental to the structure and properties of the insulation. The activation energy is around 160 kJ/mol for the thermo-oxidative ageing, from which the lifetime allowed before serious destruction of the insulation at different shortage or overload temperatures can be calculated.

6. select article Alkali treatment of lignocellulosic fibers extracted from sugarcane bagasse: Composition, structure, properties

Research articleOpen access

Alkali treatment of lignocellulosic fibers extracted from sugarcane bagasse: Composition, structure, properties

András Bartos, Juliana Anggono, Ágnes Elvira Farkas, Dávid Kun, ... Béla Pukánszky

Article 106549

Article preview

- Abstract

Abstract

Lignocellulosic fibers extracted from sugarcane bagasse were treated with NaOH solutions of different concentration (0-40 wt%) to study the effect of alkali treatment on the composition, structure and properties of the fibers. Composition was determined by the van Soest method, structure was characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM), while mechanical properties by tensile testing. Hemicellulose and lignin content decrease, while cellulose content goes through a maximum as a function of alkali concentration. Crystallinity changes only slightly and microfibril angle (MFA) remains constant thus structural effects and especially MFA are not the primary reasons for changing properties. The Young's modulus of the fibers shows a slight maximum at around 2-4 wt% NaOH content, while tensile strength goes through a much more pronounced one at around 5-8 wt%. Direct correlation between structure and mechanical properties was not found indicating that composition is more important in the determination of properties than structure. Regression analysis proved that the combination of several compositional variables determines mechanical properties in a non-linear manner. The improvement in fiber properties was explained with the dissolution of weak amorphous fractions and the relative increase of cellulose content.

7. select article Bionic antibacterial modification of IOL through SI-RAFT polymerization of P(TOEAC-co-MPC) brushes to prevent PCO and endophthalmitis

Research articleAbstract only

Bionic antibacterial modification of IOL through SI-RAFT polymerization of P(TOEAC-*co*-MPC) brushes to prevent PCO and endophthalmitis

Hengrui Zhang, Xuemei Zhang, Zheng Kuang, Yingying Jin, ... Qinxiang Zheng

Article 106553

Article preview

- Abstract

Abstract

Various complications such as posterior capsule opacification (PCO) and endophthalmitis occur after intraocular lenses (IOLs) implantation in cataract surgery. It is urgent to construct antifouling and antibacterial IOLs to lower the incidence of PCO and endophthalmitis. Bionic zwitterionic polymer such as 2-methacryloyloxyethyl phosphorylcholine (MPC) shows excellent performance in resisting nonspecific proteins and bacterial adhesion. In this work, a novel bionic brushes coating containing MPC and N, N, N-trimethyl-2-((4-(2-(4-nonylphenoxy) ethoxy)-4-oxobut-2-enoyl) oxy) ethan-1-aminium chloride (TOEAC), a quaternary ammonium monomer, was prepared onto IOLs using reversible addition–fragmentation chain transfer (RAFT) polymerization method. The P(TOEAC-*co*-MPC) brushes exhibited excellent antifouling efficiency against bovine serum albumin, *Staphylococcus aureus*, and human lens epithelial cells. In addition, the P(TOEAC-*co*-MPC) brushes showed excellent antibacterial and antibiofilm abilities and good biocompatibility. An *in vivo* study confirmed that the P(TOEAC-*co*-MPC) brushes effectively prevented PCO and

endophthalmitis. Consequently, the P(TOEAC-*co*-MPC) bionic brushes are promising for IOLs surface modification to resist postoperative complications for long-term implantation.

8. ☐ select article In situ thermo-optical studies of polymer:fullerene blend films

Research articleAbstract only

***In situ* thermo-optical studies of polymer:fullerene blend films**

Bożena Jarząbek, Paweł Nitschke, Barbara Hajduk, Marian Domański, Henryk Bednarski

Article 106573

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- Abstract

Abstract

Optical properties of a blend thin film (1:1 wt) of poly(3-hexylthiophene) (P3HT) and [6,6]-phenyl-C61-butyric acid methyl ester (PCBM) exposed to a stepwise heating and cooling, have been reported and compared with the properties of pure PCBM and P3HT films. The UV–Vis(*T*) absorption measurements were performed *in situ,* during annealing and cooling runs, at the precisely defined temperatures, in a range of 20–210 °C. It was demonstrated that this new method allows to observe the changes of absorption coefficient spectra and absorption edge parameters: the energy gap (E_G) and the Urbach energy (E_U), connected with the length of conjugation and structural disorder of thin film, respectively. Several stages, during annealing/cooling runs, were distinguished for the P3HT:PCBM blend film and related to the following processes, as an increase of P3HT crystallinity in the blend, the orderly stacking of polymer chains, thermally induced structural defects and the phase separation, caused by an aggregation of PCBM in the polymer

matrix. These changes were also observed on the P3HT:PCBM film surface, by means to the microscopic studies.

9. select article Insight from adsorption properties of Xylidyl Blue embedded hydrogel for effective removal of uranyl: Experimental and theoretical approaches

Research articleAbstract only

Insight from adsorption properties of Xylidyl Blue embedded hydrogel for effective removal of uranyl: Experimental and theoretical approaches

Zeynep Mine Şenol, Selçuk Şimşek, Halil İbrahim Ulusoy, Ayyaz Mahmood, Savaş Kaya

Article 106566

Article preview

- Abstract

Abstract

Applications of a hybrid material consisting of polyacrylamide (PAA) and Xylidyl Blue (XB) for the removal of uranyl ions from aqueous solutions has been investigated with all details. Adsorption experiments were performed at batch mode and constant temperature. Experimental parameters affecting adsorption process such as pH, initial uranyl concentration, time and temperature were studied on the removal of the uranyl ions. The isotherms assays were carried out with synthetic solutions and adsorption data were evaluated by using Langmuir, Freundlich and Dubinin–Radushkevich (D–R) isotherm models. Morphological and chemical characterizations of new synthesized material were investigated by UV-VIS-NIR spectroscopy and SEM/EDX techniques and pH_{pzc} experiments. The results of the kinetic experiments are consistent with pseudo-second-order models and intra-particle diffusion models with a slightly better fit to the latter. Equilibrium was achieved

within 3 h. The value of rate constant for adsorption process was calculated as 1.055 mol^{-1} kg min^{-1} at 318 K. The calculated thermodynamic parameters ($\Delta G°$, $\Delta H°$ and $\Delta S°$) indicated that the adsorption of uranyl ions onto XB@PAA was feasible, spontaneous and endothermic nature under the studied temperature. The developed material has also a potential as a sensor because its color turn from pink to red by adsorption of uranyl ions.

10. ☐ select article Evaluation of mechanical, thermal and morphological properties of PLA films plasticized with maleic acid and its propyl ester derivatives

Research articleAbstract only

Evaluation of mechanical, thermal and morphological properties of PLA films plasticized with maleic acid and its propyl ester derivatives

Adalberto Enumo, Idejan P. Gross, Rodrigo H. Saatkamp, Alfredo T.N. Pires, Alexandre L. Parize

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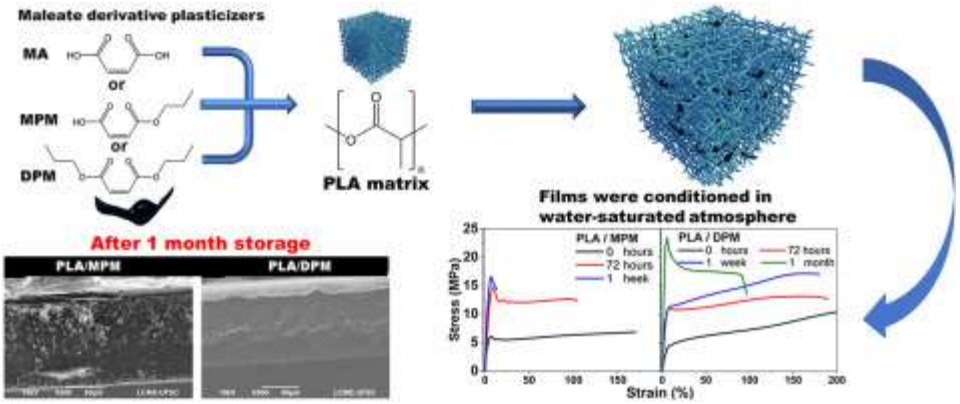

11. select article Mechanical evaluation of polymeric filaments and their corresponding 3D printed samples

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Mechanical evaluation of polymeric filaments and their corresponding 3D printed samples

A.M. Oviedo, A.H. Puente, C. Bernal, E. Pérez

Article 106561

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- Abstract

Abstract

3D printing technologies permits to produce functional parts with complex geometries, optimized topologies or enhanced internal structures. The relationship between mechanical performance and manufacturing parameters should be exhaustively analyzed to warrant the long term success of printed products. In this work, the mechanical performance of filaments based on acrylonitrile butadiene styrene (ABS), polylactic acid (PLA) and polylactic acid/polyhydroxyalkanoate (PLA/PHA) was investigated and also compared with their corresponding 3D printed samples. In general, the specimen dimensional deviations were found to be within the tolerances defined by the standard testing protocols. Density values revealed a high level of filament fusion promoting a nearly solid internal structure. The filaments exhibited improved tensile performance with respect to their corresponding printed samples. Tensile and bending performance looked quite independent of the raster angle. Izod impact behavior was increased, for ABS systems printed with the ±45° raster orientation. Quasi-static fracture tests displayed improved crack initiation resistance with the 0°/90° raster angle. The crack propagation observed for the ±45° specimens, through the bonding of the inter-layers, suggests weak entanglements.

12. select article Probing the magnetoelectric response and energy efficiency in Fe₃O₄/epoxy nanocomposites

Research articleAbstract only

Probing the magnetoelectric response and energy efficiency in Fe_3O_4/epoxy nanocomposites

A. Sanida, S.G. Stavropoulos, Th. Speliotis, G.C. Psarras

Article 106560

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- Abstract

Abstract

Fe_3O_4/epoxy nanocomposites were manufactured and studied. Structural, morphological, thermomechanical and dielectric characterization was conducted via X-Ray Diffraction, Scanning Electron Microscopy, Differential Scanning Calorimetry, Dynamic Mechanical Analysis, and Broadband Dielectric Spectroscopy. Nanocomposites' magnetic behaviour was obtained by a Superconducting Quantum Interference Device and their ability to store/harvest energy was investigated by DC charge/discharge tests. Data imply that the incorporation of the nanoparticles has an augmenting influence on the thermomechanical, dielectric and magnetic properties of the systems. Dielectric relaxations recorded in all systems are attributed to interfacial polarization, glass to rubber transition of the polymer matrix, and re-orientation of polar side groups of the polymer chain. Magnetic measurements confirmed the ferrimagnetic nature of the nanocomposites, while induced magnetic properties enhance with filler content. Stored and harvested energies increase with the applied DC field and the coefficient of energy efficiency increases in general with filler content.

13. select article Durability study of flexible bonded joints: The effect of sustained loads in mode I fracture tests

Research articleAbstract only

Durability study of flexible bonded joints: The effect of sustained loads in mode I fracture tests

J. Manterola, J. Zurbitu, J. Renart, A. Turon, I. Urresti

Article 106570

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- Abstract

Abstract

Adhesives in bonded structures are exposed both to external loads and environmental conditions; durability studies are currently needed to assess their service lifetime. Conditioning strategies differ in considering external load conditions (such as stressed or not stressed) for the durability analysis of double cantilever beam (DCB) bonded joints. Different test procedures such as ASTM D3762 (wedge testing) or ISO-25217 (DCB testing) exist to characterise the evolution of the fracture strength and toughness found in bonded joints. These methods depend on crack-length measurements, however, and achieving an accurate visual determination may be difficult due to the large fracture process zones (FPZs) that develop in the adhesive layer, especially in flexible or degraded bonded joints. To compensate, crack-length-independent data-reduction methods such as the compliance-based beam method (CBBM) or the J-integral method can be used, but experimental research is lacking on the suitability of these methods in ageing tests. A lack of consensus also exists in testing methodologies to evaluate the durability of bonded joints, especially when examining flexible bonded joints. The present work evaluates the influence of damage on fracture toughness within flexible bonded joints

exposed to service conditions. Wedge tests and DCB tests are conducted using DCB specimens bonded with a flexible structural adhesive, proving that the degradation of flexible bonded joints exposed to environmental conditions is significantly accelerated when external loads act on them. The findings show that crack length estimation is affected due to environmental effects and thus, that crack-length-dependent test methods are not applicable in ageing tests.

14. select article Detection of liner surface defects in solid rocket motors using multilayer perceptron neural networks

Research articleAbstract only

Detection of liner surface defects in solid rocket motors using multilayer perceptron neural networks

Luiz Felipe Simões Hoffmann, Francisco Carlos Parquet Bizarria, José Walter Parquet Bizarria

Article 106559

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- Abstract

Abstract

Debonding problems along the propellant/liner/insulation interface are a critical point to the integrity and one of the major causes of structural failures of solid rocket motors. Current solutions are typically restricted to methods for assessing the integrity of the rocket motors structure and visually inspecting their components. In this context, this paper presents an improved algorithm to detect liner surface defects that may compromise the bonding between the solid propellant and the insulation. The use of Local Binary Patterns (LBP) provides a structural and statistical approach to texture analysis of liner sample images. Along with color information extraction, these

two methods allow the representation of image pixels by feature vectors that are further processed by a Multilayer Perceptron (MLP) neural network classifier. The MLP neural network analyzes liner sample images and classifies each pixel into one of three classes: *non-defect*, *foreign object*, and *defect*. Several tests were executed varying different parameters to find the optimal MLP configuration, and as a result, the best classification accuracy of 99.08%, 90.66%, and 99.48% was achieved for the corresponding classes. Moreover, the defect size estimate showed that the MLP classifier correctly identified defects less than 1 mm long, with a relatively small number of training examples. Positive results indicate that the algorithm can identify liner surface defects with a performance similar to human inspectors and has the potential to assist or even automate the liner inspection process of solid rocket motors.

15. select article Marker peptide combination for source identification of gelatins obtained from Equidae hides by LC–MS/MS detection

Research articleAbstract only

Marker peptide combination for source identification of gelatins obtained from Equidae hides by LC–MS/MS detection

Shangwei Guo, Guiya Deng, Xiaobo Duan, Xiangshan Zhou, Yaqin Huang

Article 106576

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- Abstract
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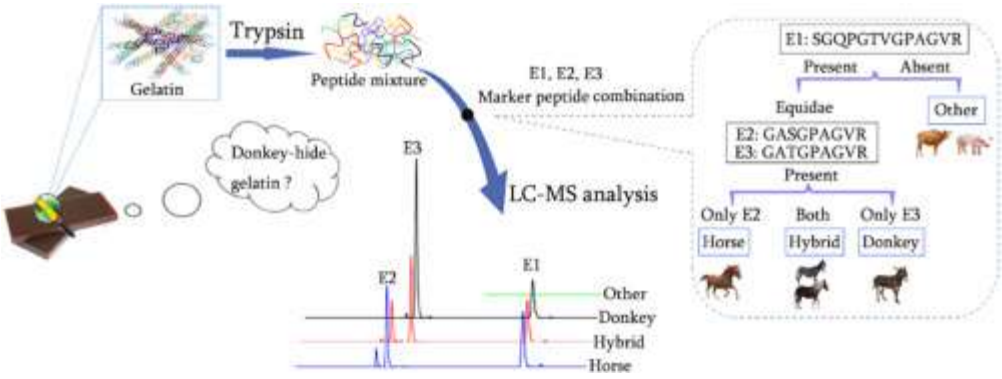

16. ☐ select article Mechanical, acoustical and flammability properties of SBR and SBR-PU foam layered structure

Research articleFull text access

Mechanical, acoustical and flammability properties of SBR and SBR-PU foam layered structure

A. Abdel-Hakim, Tarek M. El-Basheer, Aksam Abdelkhalik

Article 106536

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- Abstract

Abstract

In this work a layer structure from styrene butadiene rubber (SBR) composites and PU foam with improved flame retardancy property and high sound absorption coefficient at frequency range (200–500 Hz). Different types of flame retardants; iron (acrylic-co-acrylamide) as metal chelate (MC), magnesium hydroxide (MOH) and sodium tripolyphosphate (STP) were blended with SBR. The type and loading level of flame retardant had a great effect on filler dispersion and consequently on mechanical properties of SBR. MOH exhibited the best dispersion as indicated from scanning electron microscope (SEM), and SBR/MOH samples had almost the highest crosslink

density (16.04*10^{-5} g^{-1} mol) and the best mechanical properties where the tensile strength was improved by 32.7% at 40 phr MOH. Horizontal burning rate of SBR composites indicated that MC and MOH reduced the rate of burning of SBR at all loading levels. TGA data presented that the addition of flame retardants to SBR increased the maximum decomposition temperature in all composites. A double and triple layer structures of SBR composite and PU foam was designed. The effect of 2.5 cm air cavity on the sound absorption coefficient of SBR-PU foam layered structure was studied. The presence of air cavity behind the layered structure improved the sound absorption in the range of (200–500 Hz) better than the existence of it between the layers. The triple-layer structure gave higher sound absorption coefficient at lower frequencies than that obtained with the double-layer structure where it reached to ≥0.98 at 315 Hz.

17. select article Additive manufacturing of continuous carbon fiber reinforced poly-ether-ether-ketone with ultrahigh mechanical properties

Short communicationAbstract only

Additive manufacturing of continuous carbon fiber reinforced poly-ether-ether-ketone with ultrahigh mechanical properties

Baoning Chang, Xuemu Li, Pedram Parandoush, Shilun Ruan, ... Dong Lin

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- Abstract

Abstract

Continuous carbon fiber reinforced poly-ether-ether-ketone (CCF/PEEK) composites have attracted significant interests in mission-critical applications for their exceptional mechanical

properties and high thermal resistance. In this study, we additively manufactured CCF/PEEK laminates by the Laser-assisted Laminated Object Manufacturing technique, which was recently reported by the authors. The effects of laser power and consolidation speed on the flexural strength of the CCF/PEEK composites were studied to obtain the optimal process parameters. Hot press postprocessing was performed to further improve the mechanical properties of the composites. Various fiber alignment laminates were prepared, and the flexural and tensile properties were characterized. The hot press postprocessing 3D printed unidirectional CCF/PEEK composites exhibited ultrahigh flexural modulus and strength of 125.7 GPa and 1901.1 MPa, respectively. In addition, the tensile modulus and strength of the composites reached 133.1 GPa and 1513.8 MPa. The results showed that the fabricated CCF/PEEK exhibited superior mechanical performance compare to fused filament fabrication (FFF) printed carbon fiber reinforced thermoplastics (CFRTP).

18. select article Simple and immediate quantitative evaluation of dispersive mixing

Short communicationAbstract only

Simple and immediate quantitative evaluation of dispersive mixing

Vivek Pandey, Tianke Chen, Molin Guo, João M. Maia

Article 106587

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- Abstract

Abstract

A dimensionless mixing number is developed to quantify dispersive mixing in a simple and immediate way. This takes into account number-average diameter, volume-average diameter

and interfacial area simultaneously. It is an experimentally determined number, meaning the calculations are performed using microscopy images. This parameter was validated using various polymer blends (droplet morphology) and composite systems and can be used to compare various technologies on the basis of dispersive mixing efficiency.

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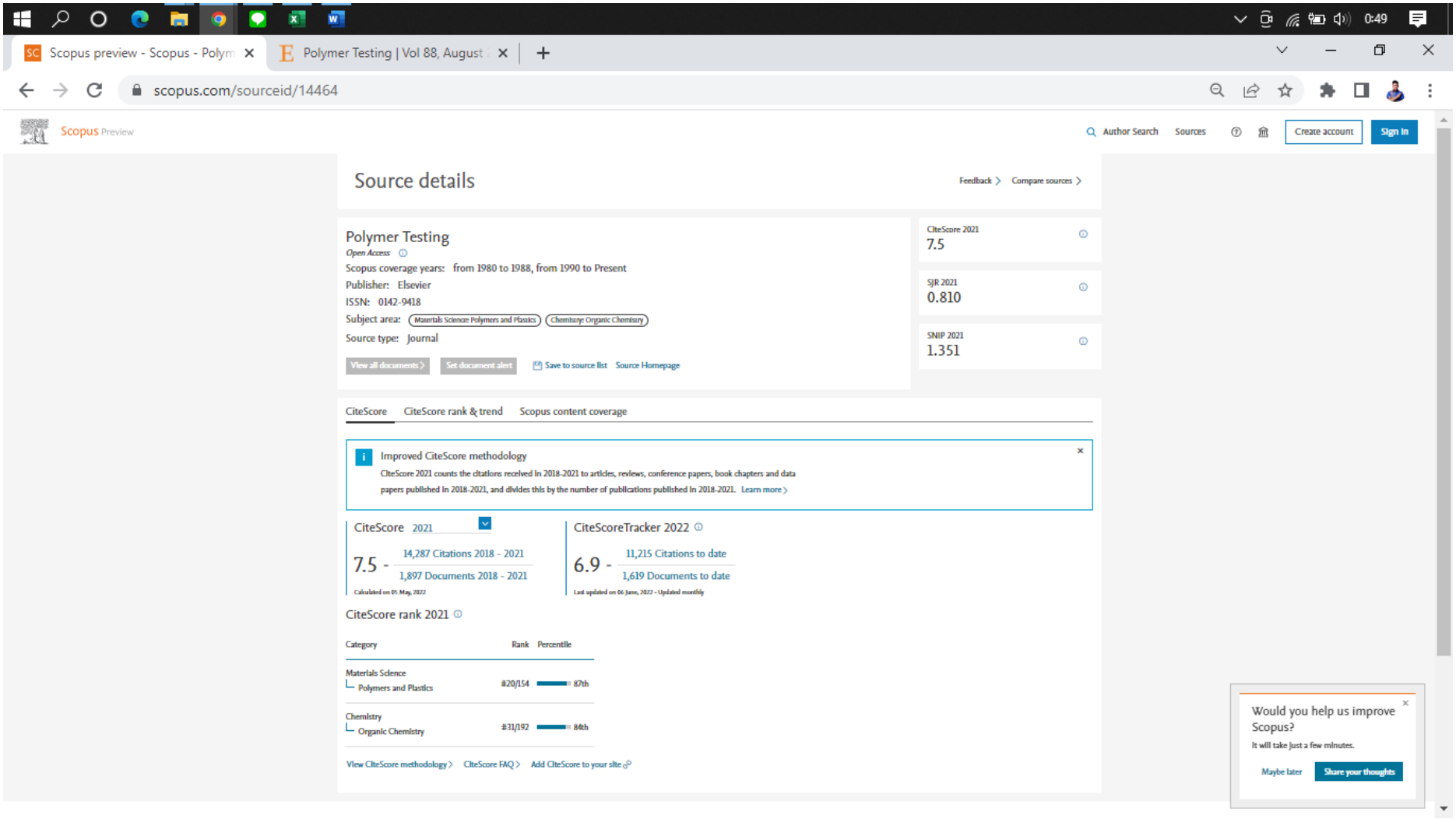
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Alkali treatment of lignocellulosic fibers extracted from sugarcane bagasse: Composition, structure, properties

András Bartos [a,b,*], Juliana Anggono [c], Ágnes Elvira Farkas [a,b], Dávid Kun [a,b], Felycia Edi Soetaredjo [d], János Móczó [a,b], Antoni [e], Hariyati Purwaningsih [f], Béla Pukánszky [a,b]

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ARTICLE INFO

Keywords:
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Crystallinity
Microfibril angle
Mechanical properties

ABSTRACT

Lignocellulosic fibers extracted from sugarcane bagasse were treated with NaOH solutions of different concentration (0-40 wt%) to study the effect of alkali treatment on the composition, structure and properties of the fibers. Composition was determined by the van Soest method, structure was characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM), while mechanical properties by tensile testing. Hemicellulose and lignin content decrease, while cellulose content goes through a maximum as a function of alkali concentration. Crystallinity changes only slightly and microfibril angle (MFA) remains constant thus structural effects and especially MFA are not the primary reasons for changing properties. The Young's modulus of the fibers shows a slight maximum at around 2-4 wt% NaOH content, while tensile strength goes through a much more pronounced one at around 5-8 wt%. Direct correlation between structure and mechanical properties was not found indicating that composition is more important in the determination of properties than structure. Regression analysis proved that the combination of several compositional variables determines mechanical properties in a non-linear manner. The improvement in fiber properties was explained with the dissolution of weak amorphous fractions and the relative increase of cellulose content.

1. Introduction

Because of the continuous search for new materials and the increasing environmental awareness of the industry as well as the public, the interest in materials from renewable resources increases continuously [1]. Biopolymers synthesized from natural raw materials [2,3] as well as starch [2,3], cellulose [3,4] and lignin [5,6] are used in increasing quantities in all areas of life. Plastics are often reinforced with fibers [7–11] in order to increase their stiffness and strength, and traditional fibers are replaced with wood or natural fibers in larger and larger extent. Many products are prepared with natural reinforcements including wood plastic composites (WPC) or various automotive parts [12–14]. However, besides their advantages, natural reinforcements have several drawbacks like sensitivity to heat during processing, limited dimensional stability due to water adsorption, poor adhesion between the matrix polymer and the reinforcement, as well as small transverse strength [15]. In order to overcome these deficiencies, attempts are made to improve properties by surface modification, coupling or the treatment of the fibers [16].

One of the approaches to improve the inherent properties of natural reinforcements and thus those of their composites is the alkali treatment of the fibers. Mercerization is a commercial technology developed a long time ago which consists of the treatment of the fibers with a sodium hydroxide solution of 20–27 wt% under tension [17]. The treatment results in increased strength, improved sheen and easier dyeing. Today, all kinds of alkali treatment are called mercerization [18]. The

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improvement of strength is the main reason and goal of using this approach for the treatment of fibers applied as reinforcements in plastics.

The effect of alkali treatment on the structure and properties of fibers was studied by numerous groups and the increase of stiffness and strength was observed quite often [19–22]. However, the explanation for the improvement of mechanical properties is rather controversial in many cases. The concentration of sodium hydroxide and the time of treatment varies in a very wide range from 0.03 wt% to 40 wt% and from a few minutes to 48 h [19–27]. It is generally accepted that the composition of the fibers changes as the result of the treatment; the amorphous parts are dissolved thus the hemicellulose, lignin and wax content of the fibers decrease, although Taha et al. [19] claimed that lignin content remains constant. The groups agree much less about the reasons of the improvement in strength. Although structural changes are thought to result in these changes in most cases, the opinions about the main factors differ widely. Crystallinity is one of these factors, but it was shown to increase [21,28], decrease [29] or go through a maximum [22,26,30,31] as a function of NaOH concentration and time. Mechanical properties are often plotted against crystallinity but the correlations are rarely convincing. Another factor might be a change in the microfibril angle [18,21,32,33] or crystal modification [32], i.e. the transformation from the cellulose I to the cellulose II form as the result of the treatment [30,34]. Alkali treatment removes waxes and other amorphous components from the surface of the fibers thus modifying surface roughness [28,35–37], which was claimed to improve interfacial adhesion [30]. On the other hand, Gassan and Bledzki [23] think that changing surface quality does not modify composite properties. Unfortunately, the statements are not very often supported with sufficient experimental evidence and the controversies are difficult to resolve.

Nevertheless, it is a fact that under certain conditions the stiffness and strength of natural fibers are improved by alkali treatment [19–22], which results in an increase of composite modulus and/or strength, as a consequence [20,23,32]. Occasionally, an improvement or maximum is observed in the strength of composites when a polymer is reinforced with treated fibers. Van de Weyenberg et al. [25] reinforced epoxy resin with fibers modified by alkali treatment and found considerable increase in the transverse strength of the composites. They explained the improvement with a change in interfacial adhesion, which was contradicted by Gassan and Bledzki [23].

In spite of the contradictions published, the beneficial effect of alkali treatment for fiber characteristics is clear and it often results in better composite properties as well. Considering the contradictions mentioned above, the goal of our study was to treat sugarcane bagasse fibers with sodium hydroxide and then thoroughly characterize their composition, structure and properties including strength and stiffness. Sugarcane bagasse was selected, because it is a cheap, natural raw material, which can be obtained from local sources in Indonesia. Bagasse fiber form a waste and its value added application would be beneficial for the country. In a part of the study, flax fibers were used as reference material in order to extend the validity of our conclusions. An attempt was made to correlate the measured variables and find a plausible explanation for the increase of fiber strength with alkali treatment. The consequence for practice is also mentioned in the final section of the paper.

2. Experimental

2.1. Materials, fiber treatment and sample preparation

The bagasse fibers were obtained directly from the sugar mill. They were washed with ethanol, dried and sieved. The fraction between the sieves of 45 and 20 mesh was separated; the length of the fibers was 4560 ± 1870 μm and their diameter 340 ± 156 μm. Longer individual fibers were separated for mechanical characterization, tensile testing. A solution containing 40 wt% sodium hydroxide was prepared from NaOH flakes (Molar Chemicals, Hungary). The solution was diluted to obtain solutions with 1, 2, 4, 8, 15, 20 and 40 wt% alkali content.

1 g of fiber was weighed into a beaker and 40 ml NaOH solution was poured on it. The treatment lasted for 1 h, when the fibers were separated from the solution and placed into 50 ml acetic acid (Molar Chemicals, Hungary) for 10 min. Subsequently the fibers were washed on a filter with water until the pH of the washing water became neutral. pH was checked by litmus paper. Finally, the fibers were soaked in distilled water and then the pH was checked again. The fibers were dried at 105 °C in an air circulation oven for 48 h. Fibers were prepared in a similar way for tensile testing with the only difference that fibers of 8–12 cm length were separated first and then dried under tension to avoid curling. All treatments were done at ambient temperature.

Fibers had to be milled for X-ray diffraction and FTIR measurements. Two grams of the fibers were placed into a Retsch MM 400 ball mill (Retsch GmbH., Germany) and the fibers were ground for 2.5 min at 30 s^{-1} frequency. Sufficiently small particles were obtained for the preparation of KBr pellets for the recording of the FTIR spectra.

2.2. Characterization, measurements

The chemical composition of the fibers was determined by the van Soest method. The detailed description of the method can be found in the paper of van Soest [38]. According to the method, hemicellulose content is determined after treatment with an acidic detergent solution, the amount of cellulose by treating the fibers with sulfuric acid of 72 wt % concentration and lignin by burning the sample in an oven. Composition was analyzed also by Fourier transform infrared spectroscopy (FTIR). Spectra were recorded on KBr pellets using a Bruker Tensor 27 A (Bruker, Massachusetts, US) apparatus. 2 mg fiber was mixed with 248 mg KBr for the preparation of the samples. Spectra were recorded from 4000 to 400 cm^{-1} at 2 cm^{-1} resolution with 32 scans. Absorbances appearing at 1428 (CH_2 stretching vibration [19,39–41]) and 1372 cm^{-1} (C-H bending vibration [19,24,42]) were assigned to cellulose, those detected at 1730 (C=O stretching vibration [19,39,40,43]), 1249 (C-O stretching vibration [19,40,42,43]) and 1040 (C-H and C-O bending vibration [40,42]) cm^{-1} to hemicellulose and the one observed at 1514 cm^{-1} (C=C aromatic vibration [19,40]) to lignin. The crystalline structure of the fibers was characterized by X-ray diffraction (XRD). The traces were recorded on powder samples using a Philips PW 1830/PW 1050 (Philips, Netherlands) apparatus with CuK_α radiation at 40 kV and 35 mA anode excitation in the 2θ range between 4 and 40° with 0.04° steps. Crystallinity and microfibril angle (MFA) were determined from the reflection of the cellulose detected at 22.8° according to the method described by Yamamoto [44].

The mechanical properties of the fibers were characterized by tensile testing. Fibers were slightly stretched and fixed onto paper frames for the measurements. An Instron 5566 tensile testing machine (Instron, Massachusetts, US) was used for the tests at the gauge length of 20 mm and crosshead speed of 0.5 mm/min. Ten parallel measurements were carried out for each sample. Scanning electron microscopy (SEM) was used for the characterization of the morphology of the neat and treated fibers. The equipment used was a Jeol JSM 6380 LA (Jeol, Japan) apparatus. Before the recording of the images, the samples were sputtered with gold.

3. Results and discussion

The results of the experiments are presented in several sections. First, the effect of chemical treatment on the composition of the fibers is discussed, followed by the presentation of changes in their structure. Properties are analyzed next and then correlations between composition and properties are shown in the last section together with relevance for practice.

3.1. Composition

The analysis of results published in the literature clearly showed that one of the consequences of the alkali treatment of fibers is a change in their chemical composition. The amorphous parts of the fibers, mainly hemicellulose, lignin and waxes, are dissolved in different extents. At larger concentrations of NaOH and at longer times, also cellulose crystals disintegrate and dissolve. The change of chemical composition inevitably leads to the modification of properties as well. Accordingly, the effect of treatment on composition was followed by two methods, chemical analysis and FTIR spectroscopy.

The cellulose and lignin content of the fibers are plotted against the concentration of the treating solution in Fig. 1. The cellulose content is large, exceeds 50-60 wt%, while the amount of lignin is below 20 wt%, as expected for such fibers. In accordance with most literature references, lignin content decreases continuously with increasing NaOH concentration [22–24,31,36,40]. On the other hand, cellulose content shows a maximum. The maximum results from the fact that the dissolution of amorphous components is considerably faster than that of the cellulose crystals, but the latter also degrades at very large alkali concentrations. The observed increase in cellulose content may result in the improvement of mechanical properties as reported many times in the literature [22,31]. Hemicellulose content also decreases with increasing NaOH concentration, while the amount of ash remains constant [21,28,30,36,37,45,46].

As mentioned above, compositional changes were followed also by FTIR spectroscopy. The spectra of the fibers at selected NaOH contents are presented in Fig. 2. The quantitative analysis of peaks assigned to the various components of the fiber yielded the same results as chemical analysis. The inset of Fig. 2 shows the decrease of hemicellulose (1730 cm^{-1}, C=O stretching vibration) and lignin (1514 cm^{-1}, C=C aromatic vibrations) content with increasing NaOH concentration. Both chemical analysis and FTIR spectroscopy confirmed the expectations that the chemical composition of the fibers changes during treatment and the changes must result in modified structure and properties [19,21–23,31].

The chemical composition of the fibers changes because some of the components are dissolved during treatment. The rate of dissolution is different for the various components, but all lead to the decrease of the weight of the sample. Weight loss is plotted against the NaOH concentration of the treating solution in Fig. 3. According to the figure, the decrease of weight is very substantial at large NaOH contents, the fibers start to lose their integrity. Considering the large change in weight, we may conclude that treatments with solutions above 15 wt% NaOH deteriorate the fibers, at least in the time frame (1 h) used in these experiments.

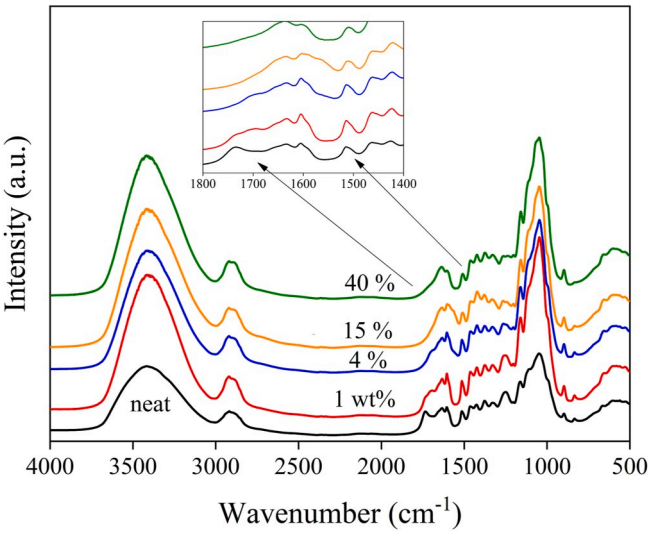

Fig. 2. FTIR spectra of neat bagasse fibers and of those treated with NaOH solutions of various concentrations. The inset shows the decrease of hemicellulose (1730 cm^{-1}) and lignin (1514 cm^{-1}) content with increasing alkali concentration.

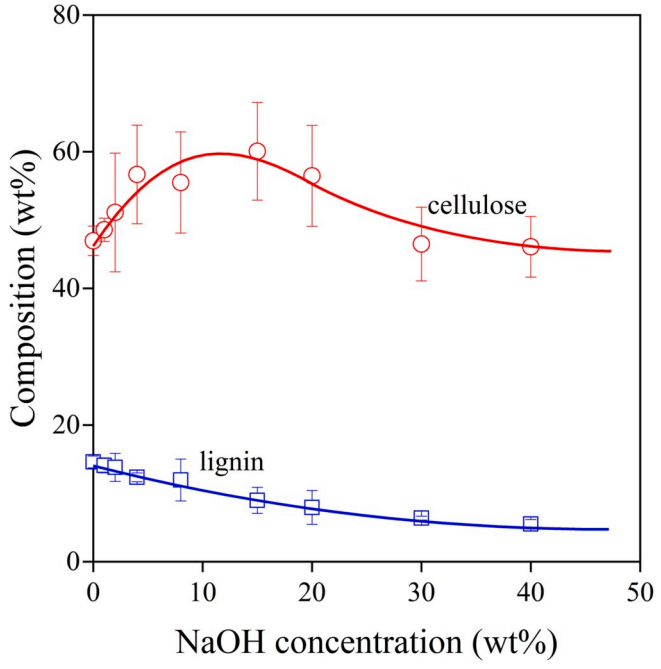

Fig. 1. Effect of the NaOH concentration of the treating solution on the composition of sugarcane bagasse fibers. Treatment time: 1 h. Symbols: (○) cellulose, (□) lignin content.

3.2. Structure

Cellulose has various crystal forms. In plants, it crystallizes in the cellulose I form, which may transform into the cellulose II modification

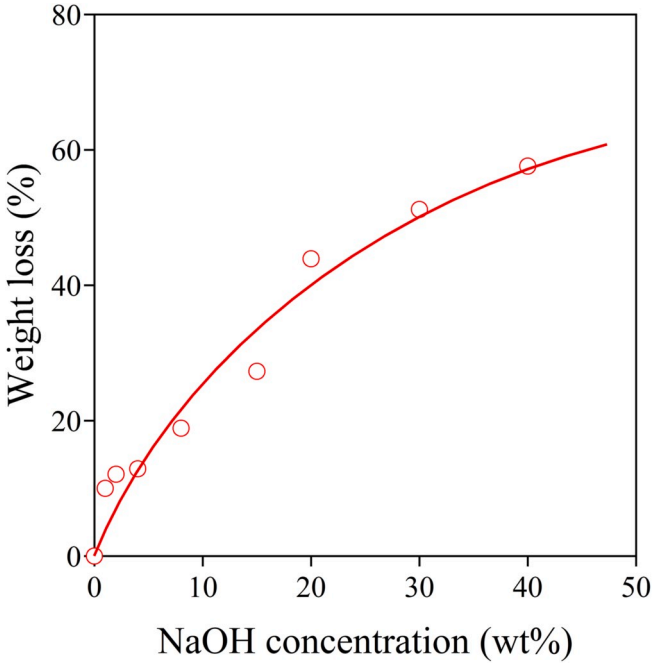

Fig. 3. Weight loss during the alkali treatment of sugarcane bagasse fibers. Treatment time: 1 h.

as the result of alkali treatment [30,34]. Treatment loosens up structure and the sodium ions as well as the water present make possible the transformation. Interesting to note that such transformation was rarely reported during the alkali treatment of fibers used as reinforcements in plastics. Parallel to the change in crystal modification, crystallinity may be also modified during treatment. XRD records obtained on fibers treated with NaOH solutions of different concentration are plotted in Fig. 4. According to the traces, crystal form does not change in our case even at the largest alkali concentration. On the other hand, crystallinity and order change quite considerably as NaOH concentration increases. Cellulose crystals retain their integrity up to 8 wt% NaOH content, but the regularity of the crystals decreases considerably above that concentration. The quantitative analysis of the results shows that crystallinity increases from about 58% to 63% at 5 wt% NaOH content of the solution, but decreases steeply above this alkali concentration down to around 40%, confirming the conclusions drawn by the visual observation of the traces presented in Fig. 4.

The improvement of the mechanical properties of the fibers is often explained with the change of the microfibril angle (MFA) during alkali treatment [32]. Decreasing MFA results in better alignment of the fibrils to the direction of the load and thus larger stiffness and strength [25]. MFA can be determined from XRD records using the approach proposed by Yamamoto [44]. MFA values are plotted against the NaOH content of the treating solution in Fig. 5. MFA did not change at all for the sugarcane bagasse fibers used in this study, and we detected only very slight changes in the case of the flax fibers used as reference. According to these results the change in microfibril angle cannot explain the improvement or changes in the mechanical properties of the fibers, if there is any.

For polymers reinforced with natural fibers, the changes in properties are often explained with the modification of the surface of the fibers. The removal of waxes and increased surface roughness are claimed to improve interfacial adhesion and thus stiffness and strength [28,35–37] of composites reinforced with natural fibers. The surface morphology of the fibers is demonstrated by the SEM micrographs presented in Fig. 6. The surface of the neat fiber is relatively smooth and even (Fig. 6a), but

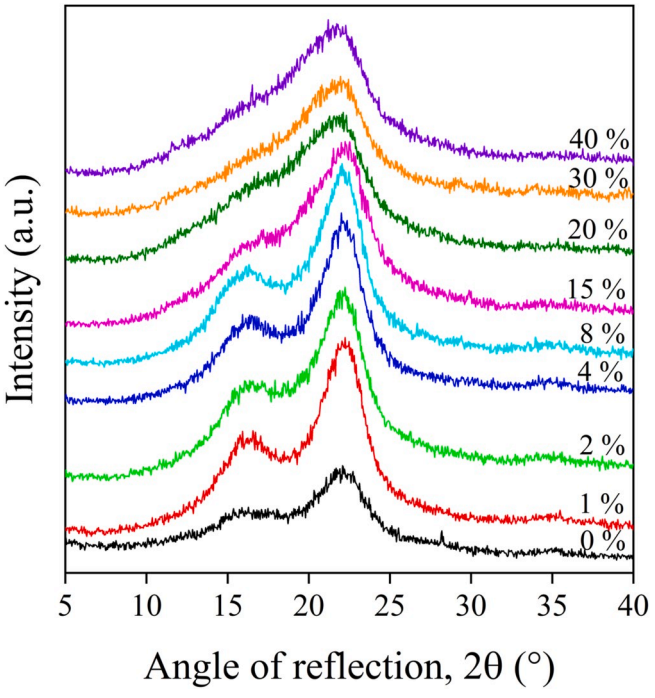

Fig. 4. XRD traces of the neat bagasse fiber and of those treated with alkali solutions of various concentration. Changes in the crystalline structure of the fibers.

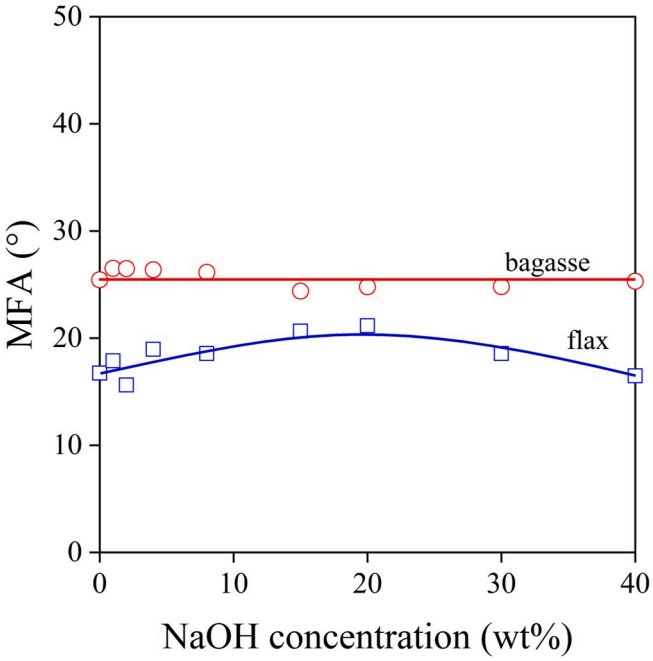

Fig. 5. Independence of the microfibril angle (MFA) of bagasse fibers of the NaOH concentration of the treating solution. Flax is presented as comparison. Symbols: (◯) bagasse, (□) flax.

sharper contours appear already after the treatment with 4 wt% NaOH solution (Fig. 6b). Deep groves (Fig. 6c) and even larger holes (Fig. 6d) can be observed after the treatment with solutions of larger concentrations, and the fibers start to lose their integrity as indicated also by the XRD traces. Several authors claim that changing surface morphology improves interfacial adhesion and fiber or composite properties [24, 46–48], but Gassan and Bledzki [23] question this claim and relate property changes to the shrinkage of the fibers.

3.3. Mechanical properties

The effect of alkali treatment on the stiffness of the fibers is presented in Fig. 7a. Flax is used as reference in the figure. The modulus of flax is considerably larger than that of the bagasse fibers. The difference might be caused by the dissimilar microfibril angle, which is around 25° for the bagasse and 18–20° for the flax fibers. In both cases a maximum appears in stiffness at around 3-5 wt% NaOH content of the treating solution. The maximum is more pronounced for flax, but it seems to be present also in the case of bagasse. The location of the maximum on the NaOH concentration axis differs from the one determined for cellulose content, which raises some doubt about the exclusive role of this latter factor in the changes of mechanical properties. On the other hand, the maximum determined in crystallinity is much closer to this value.

The tensile strength of the fibers is plotted against NaOH content in Fig. 7b. The strength of the bagasse fibers is larger than that of the flax fibers indicating that instead of microfibril angle other factors determine fiber strength. We must keep in mind that the fibers were slightly stretched for tensile testing, which influences the measured values, but these latter are acceptable for comparative purposes. A maximum is observed in this case too, it appears at around 5-8 wt% for bagasse and closer to 5 wt% for the flax fibers. The maximum is clear, fiber strength increases considerably as an effect of the treatment, and then it decreases at larger alkali concentrations. The increase in fiber strength justifies the increase observed in the strength of composites prepared with various fibers treated with NaOH [25,47,49]. The decrease in strength, to very small values in the case of flax, at larger NaOH content is in accordance with earlier conclusions drawn from the XRD and SEM

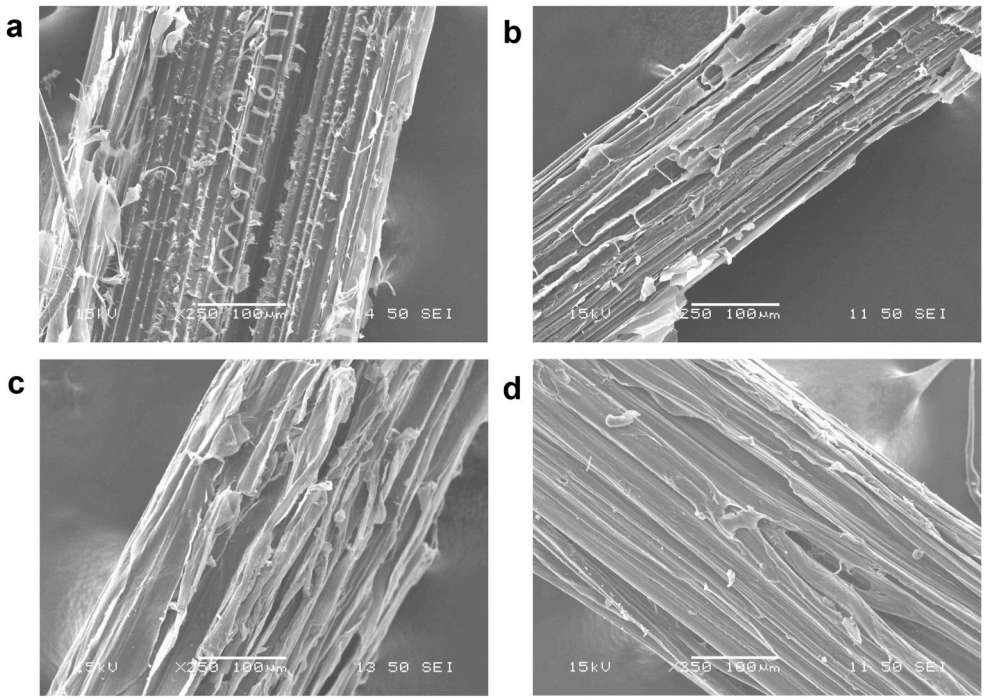

Fig. 6. SEM micrographs recorded on the neat fibers and on fibers treated with NaOH solutions of various concentration; a) neat fiber, b) 4 wt%, c) 8 wt%, d) 30 wt% NaOH content.

studies. We might mention here also the deformability of the fibers and the effect of alkali treatment on them. The elongation-at-break of the bagasse fibers is larger, it is around 5%, while that of the flax fibers is approximately 3% in the average, and it does not change much upon treatment. The dissimilar elongation of the fibers might also influence strength. Although we confirmed the beneficial effect of alkali treatment on the properties of natural fibers and proved that it results from the change of composition and structure, we do not have an unambiguous explanation for the improvement and do not know the determining factor either.

3.4. Correlations

The results presented above indicate clearly that composition and structure determines properties and that alkali treatment modifies both factors. In publications, mechanical properties are very often related to the crystallinity of the fibers [20,28]. The Young's modulus and strength of the fibers are plotted against crystallinity in Fig. 8. Strength apparently increases with increasing crystallinity, while a maximum seems to exist for modulus that is difficult to explain since stiffness should increase continuously with increasing crystallinity. We must call the attention here to the fact, though, that the standard deviation of all data is large and the correlations are rather loose. Crystallinity must influence mechanical properties, but it is not the determining factor, others must play a role as well.

The unambiguous determination of the dominating factor is very difficult, because probably more than one influence properties and their weight might be dissimilar. Moreover, various factors are related to each other, they either change similarly as a function of NaOH concentration or even depend on each other, thus the identification of the dominating one is very difficult or even impossible. Since the correlation between mechanical properties and crystallinity is weak, we decided to analyze the combined effect of various compositional factors on properties. We carried out multiple regression analysis, in which we considered all compositional variables and their interactions. Subsequently we neglected components, which were not significant and arrived to the conclusion that a non-linear model taking into account first-order compositional variables and an interaction term describes property change quite well. The following model:

$$\sigma = -36.7[C] + 39.1[HC] - 340.5[L] + 8.3[C][L] \quad (1)$$

where σ is the tensile strength of the fiber, while $[C]$, $[HC]$ and $[L]$ is its cellulose, hemicellulose and lignin content, respectively, was obtained in the analysis. The model takes into account the fact that the sum of the components must be 100%. The ash content of the fibers was fixed at 2%. The two-dimensional surface plot of the model is presented in Fig. 9. Measured values are also shown in the figure as red symbols together with the corresponding value. Although deviations can be observed between the predicted and measured values, the agreement is reasonable, the determination coefficient, i.e. the goodness of the fit is 0.9110.

Fig. 9 proves that the combination of several compositional variables determine the mechanical properties of the fibers. The overall effect is not linear and the interaction of the variables (see the factor $[C][L]$) further complicates evaluation. In order to see the predictive power and validity of the approach, we plotted calculated fiber strength against the measured values. The correlation is presented in Fig. 10. Considering the uncertainty of the tensile test and the large standard deviation of tensile strength (see Fig. 7b), the correlation is excellent, thus we can state that the changes in composition during alkali treatment unambiguously determine mechanical properties.

Although the correlation shown in Fig. 10 is unambiguous, one might object that the structure of the fibers must determine properties. This might be true, but MFA did not change at all and the relationship between mechanical properties and crystallinity was very weak (Fig. 8). On the other hand, if we consider that the cellulose crystals are much stiffer and stronger than the amorphous components of the fiber, one can easily accept that the dissolution of a part of the amorphous phase results in an improvement of mechanical properties. The maximum in strength was observed at around 5 wt% NaOH content of the treating solution and both hemicellulose and even the lignin content are still considerable even after the treatment. Obviously, the weakest fraction is dissolved during treatment resulting in the improvement of properties. We can expect, as a result, that fibers treated with a NaOH solution of 5

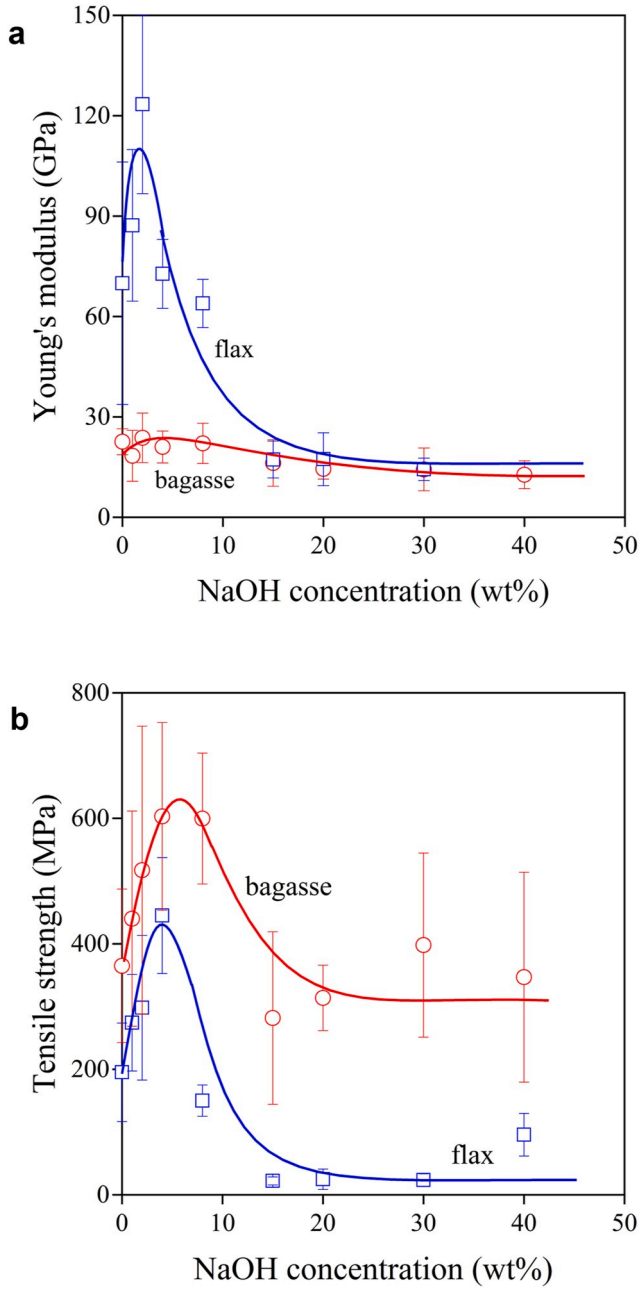

Fig. 7. Effect of the concentration of the NaOH solution used for treatment on the stiffness (a) and tensile strength (b) of sugarcane bagasse fibers. Flax is used as reference. Symbols: (◯) bagasse, (□) flax.

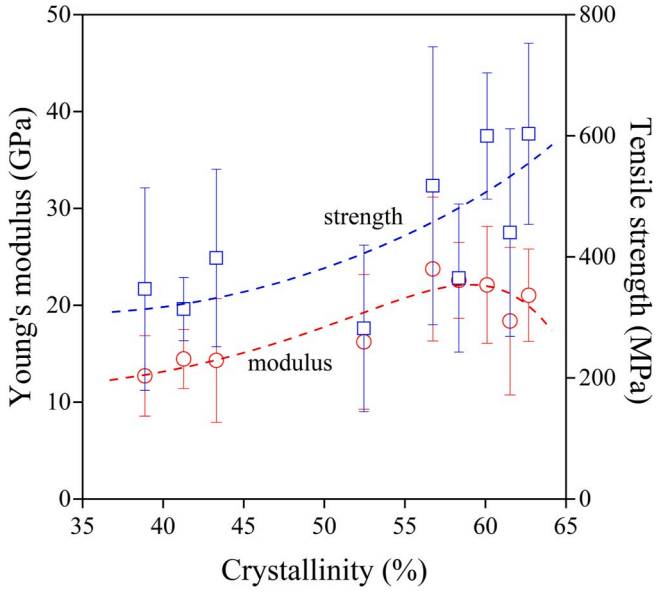

Fig. 8. Weak correlation between the mechanical properties of bagasse fibers and their crystallinity. Symbols: (◯) Young's modulus, (□) tensile strength.

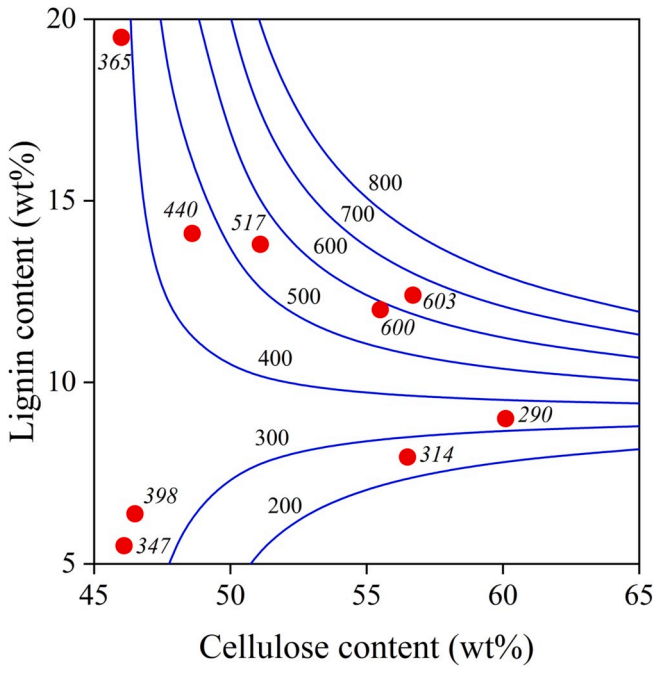

Fig. 9. Simultaneous effect of cellulose and lignin content on the tensile strength of sugarcane bagasse fibers. The solid lines were determined by regression analysis. The red symbols indicate measured values.

wt% concentration will reinforce polymers more than neat fibers and composite properties will improve in accordance with published results [30,43,50].

4. Conclusions

The results on the alkali treatment of sugarcane bagasse fibers proved that the treatment modifies the composition, structure and properties of the fibers. Hemicellulose and lignin content decrease, while cellulose content goes through a maximum as a function of the alkali content of the treating solution. Crystallinity changes only slightly and microfibril angle remains constant with increasing NaOH content thus structural effects and especially MFA are not the primary reasons for changing properties, contrary to many reports published in the literature. The stiffness of the fibers shows a very slight maximum at around 2-4 wt% NaOH content, while strength a much more pronounced one at around 5-8 wt%. The increase in fiber strength is quite considerable. Direct correlation between structure and mechanical properties were not found indicating that changes in composition are more important in the determination of properties than structure. Regression analysis proved that the combination of several compositional variables determines mechanical properties in a non-linear manner. The improvement in fiber properties was explained with the dissolution of weak amorphous fractions and with the increase of cellulose content. The optimum concentration of the treating solution is around 5 wt% NaOH content if the time of treatment is fixed at 1 h. The increase of fiber strength is expected to result in the improvement of composite

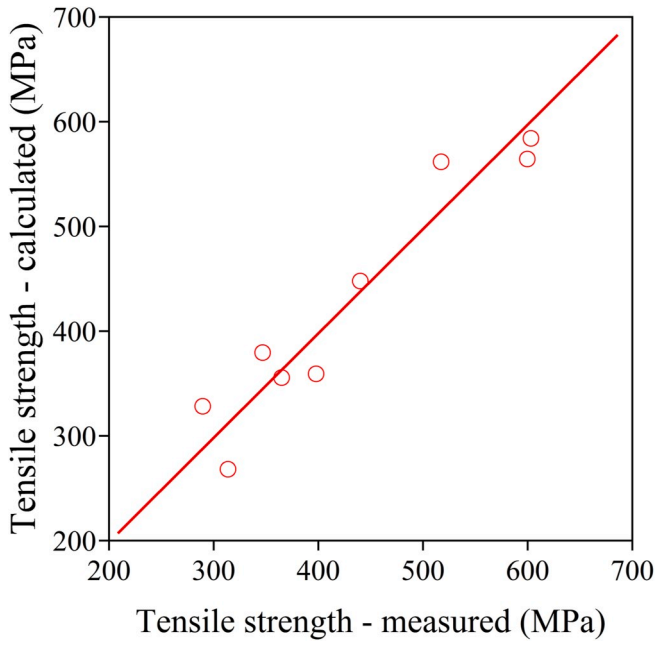

Fig. 10. Correlation between the measured strength of sugarcane bagasse fibers and values calculated by regression analysis from compositional variables using Eq. (1).

properties.

Data availability statement

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

Declaration of competing interest

None.

CRediT authorship contribution statement

András Bartos: Methodology, Investigation, Data curation, Writing - original draft, Writing - review & editing, Visualization. **Juliana Anggono:** Conceptualization, Writing - review & editing. **Ágnes Elvira Farkas:** Investigation. **Dávid Kun:** Formal analysis. **Felycia Edi Soetaredjo:** Investigation. **János Móczó:** Conceptualization, Methodology, Data curation, Writing - original draft, Writing - review & editing. **Antoni:** Investigation, Resources. **Hariyati Purwaningsih:** Investigation, Resources. **Béla Pukánszky:** Conceptualization, Methodology, Writing - original draft, Supervision.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.polymertesting.2020.106549.

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