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Electrospun biopolymer blends of poly(lactic acid) and poly(hydroxybutyrate) reinforced with biochar derived from kitchen waste

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Abstract: Biodegradable composites reinforced with natural fillers are exciting alternatives to expensive biodegradable polymers. This study aimed to investigate the effect of kitchen waste–derived biochar on the morphological, chemical, thermal, and mechanical properties of electrospun fibrous mats from a blend of biodegradable polymers poly(lactic acid) and poly(hydroxybutyrate). The electrospun neat PLA/PHB mats and mats with 5, 10, 15, 20, and 30 wt.% content of kitchen waste-derived biochar were produced. The techniques of scanning electron microscopy, Fourier transform infrared spectrometry analysis, thermogravimetric analysis, and different scanning calorimetry and tensile tests were used for the fundamental characterization of the produced electrospun mats. The results indicate that adding biochar to PLA/PHB does not significantly affect the properties of electrospun materials. This may be advantageous for packaging, filtration, or agriculture applications.

Keywords: biochar, biodegradable composites, electrospinning, material characterization, environmentally friendly polymers.

1. Introduction

The increasing global concern for environmental sustainability has driven the demand for natural, biodegradable, and biocompatible materials. Biopolymers, derived from renewable sources, offer an alternative to traditional petroleum-based plastics [1]. Among these, poly(lactic acid) (PLA) and poly(hydroxybutyrate) (PHB) have emerged as two of the most promising biopolymers due to their excellent biodegradability and biocompatibility. Blending PLA and PHB offers several advantages. The combination of PLA´s ductility and PHB´s crystallinity can enhance mechanical properties; the synergistic effect of the two polymers can improve the thermal stability of the blend. Adjusting the blend ratio allows the material's properties to be fine-tuned to meet specific applications [2, 3].

Electrospinning is a versatile technique that enables the fabrication of nanofibers with exceptional properties, such as high surface area, porosity, and tunable mechanical properties [4]. By combining electrospinning with biodegradable biopolymers like PLA and PHB, innovative materials with potential applications in various fields, including tissue engineering, filtration, drug delivery, and packaging, can be created [5-7]. However, the global market for biodegradable polymers is still young and struggling with problems resulting from the high prices of its products and the limited infrastructure enabling its organic recycling. The material advantages of applying this type of polymer are undeniable but expensive; therefore, price reduction opportunities may be crucial. An enjoyable alternative in this field is the use of fillers, which, together with the polymer, form a composite; this leads to a reduction in price and modifies the material's properties [8].

Biochar, a carbon-rich material derived from biomass pyrolysis, offers a promising solution as a filler due to its unique properties, such as high surface area, porosity, and excellent thermal and electrical conductivity. Kitchen waste, such as vegetable scraps, fruit peels, and coffee grounds, is an excellent source of biomass for biochar production. By converting such organic waste into biochar, landfill waste can be reduced, and a valuable resource for sustainable industry can be created.

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Another benefit of using kitchen waste is the reduction of greenhouse gases in landfills produced when kitchen waste decomposes [9-12].

The current work aims to design fibrous composites with kitchen waste-derived biochar as filler for a sustainable way to manage kitchen waste. In the study, the electrospun mats PLA/PHB and fibrous composites PLA/PHB with different biochar content were prepared. The biochar used in this study was prepared from the peelings of root vegetable and potatoes contained in the of kitchen waste. The original combination of biodegradable polymers blend PLA/PHB, the kitchen waste derived biochar as filler of fibrous composites and electrospinning technology brings novelty to the study, as the authors have no knowledge of a similar combination of materials and their study from the literature. Due to this specific combination of materials it was necessary to determine the properties of these prepared fibrous composites and on the basic characteristics. The morphology of electrospun mats, chemical changes, and thermal and mechanical properties were investigated by SEM, FTIR, and RAMAN, TGA/DSC, and Instron, respectively. The effects of biochar as fillers on the mechanical, morphological, and thermal properties of electrospun poly(lactic acid) and poly(hydroxybutyrate) blends were characterized.

2. Materials

Commercial poly(lactic acid) pellets of PLA 4042 D with a density of 1.25 g.cm^{-3} were purchased from Resinex (Bratislava, Slovakia). Poly(3-hydroxybutyrate) PHB Biomer® powder with a density of 1.20 g.cm^{-3} was supplied by Biomer (Krailling, Germany). The acetyl tributyl citrate (ATBC) 98 % was used as a plasticizer and was purchased from Sigma Aldrich (Saint Luis, MO, USA). 1,1,1,3,3,3-hexafluoro-isopropanol (HFIP, >99.0% purity) was purchased from TCI Tokyo Kasei (Tokyo Japan); dichloromethane p.a. (DCM, 99.8% purity) from Lach-Ner (Bratislava, Slovakia).

3. Methods

The PLA/PHB/ATBC blend (85:15:15) was prepared by melt mixing in Plasti-Corder Brabender at 175 °C at 40 rpm for 10 minutes. The molar mass of the PLA/PHB/ATBC has been estimated on Mw = 74.5 kg.mol^{-1}. Then, PLA/PHB/ATBC solution was prepared in concentrations of 10 % (w/v) in the blend of 1,1,1,3,3,3-hexafluoro-isopropanol (HFIP) and dichloromethane (DCM). The solution was stirred intensively up to wholly dissolved. 60 mL of the solution was prepared.

The kitchen waste, consisting primarily of dried peelings from potatoes and root vegetables such as carrots, celery, and kohlrabi, was used to prepare biochar. The kitchen waste was stabilized in a conventional laboratory oven (Memmert GmbH, Germany). The temperature increased every 24 h, from 50 °C to 250 °C in this oven. Thus, stabilized waste was carbonized at 800 °C in a tube electric resistance furnace (Clasic.cz, Řevnice, Czech Republic) under a vacuum of ~10 Pa, with a heating rate of 2 °C·min^{-1}, and a dwell time of 30 min in the targeted temperature. The cooling was carried out in the oven until room temperature was reached.

The PLA/PHB/ATBC solution was divided into six parts of 10mL each, and biochar was added to five of them to reach concentrations of 5, 10, 15, 20, and 30 wt.%. The samples were thoroughly homogenized to obtain solutions with finely distributed biochar particles with a size D50 of about 50 µm, and 90 % of the powder was smaller than 100 µm. The procedure of carbonization was adopted from [13]. Then, the electrospun mats without and with the different percentages of biochar were prepared by electrospinning under ambient temperature (23 ± 1 °C, H = 57 %) in a horizontal spinning configuration with a flat–end needle with a 21 G inner diameter. The working distance was 12 cm. The applied voltage was between 15 kV, and the voltage was driven by a high–voltage power supply (Spellman SL-150W, Germany). A syringe pump fed the solutions with the feeding rate of 0.5 mL.h^{-1}. The electrospun mats were formed on collectors covered by aluminum foil.

The morphology of electrospun mats was observed by scanning electron microscopy (SEM) using a JSM Jeol 6610 microscope (Jeol, Japan) at an accelerated voltage of 15 kV. Before observing, the samples were sputtered with a thin layer of gold by the sputter coater Balzers SCD 040 (Balzers Union Limited, Lichtenstein). The average diameters of the samples in the mats were measured using ImageJ software. RAMAN spectroscopy was used to characterize the biochar. The DXR Raman Microscope (Thermo Electron Scientific Instruments LLC, Madison, WI USA) was used. The measurements were performed at room temperature in the range 50 – 3500 cm^{-1}. The Raman spectra were excited using the 532 nm laser, with the maximal laser power of 10 mW. The peak position was calibrated with a polystyrene standard. Power 0.5 - 5 mW was used to examine the samples. Fourier-transform infrared (FTIR) spectra were measured with spectrometer iS50 (Thermo Fisher Scientific, USA) equipped with DTGS detector and Omnic 9.0 software. The spectra were collected in the middle region from 4,000 to 400 cm^{-1} at a resolution of 4 cm^{-1}; the number of scans was 128. Diamond ATR was applied for measurement in solid/liquid state. Thermal analysis was carried out by the combined analyzer Netzsch STA 449 F1 Jupiter. Twenty milligrams of the samples in the Al_2O_3 crucible pans with a diameter of 6 mm were heated from 20 to 500 °C at a 10 °C.min^{-1} heating rate under an argon atmosphere. The weight loss of the samples was recorded as a function of temperature with the help of the Netzsch Proteus software. The tensile tests of the electrospun samples were performed using a Dynamometer Instron 4301 (Instron Corporation, Norwood, MA, USA) following standard ASTM D638. Five testing strips for each formulation were cut from the electrospun mats with the dimensions of the tested strip area of 15 × 50 mm with a thickness of approximately 0.1 mm. The initial length of the tested strips was 120 mm because of better handling, and the gripping distance was 50 mm. A 1 mm.min^{-1} testing rate was applied until 0.5 % deformation was reached, and then the rates were increased to 20 mm.min^{-1}. Average values of the tensile strength, elongation at break, and Young's modulus were determined from the stress-strain curves.

4. Results and Discussions

PLA/PHB/ATBC solutions with 5, 10, 15, 20, and 30 wt.% biochar content were electrospun in the conditions mentioned above in 2. Methods. The color of the electrospun mats reflected the biochar content, as shown in ▶**Figure 1**. The higher the biochar content, the darker the mats. Such electrospun mats were prepared for subsequent characterization, and their morphology, thermal, and mechanical properties were evaluated.

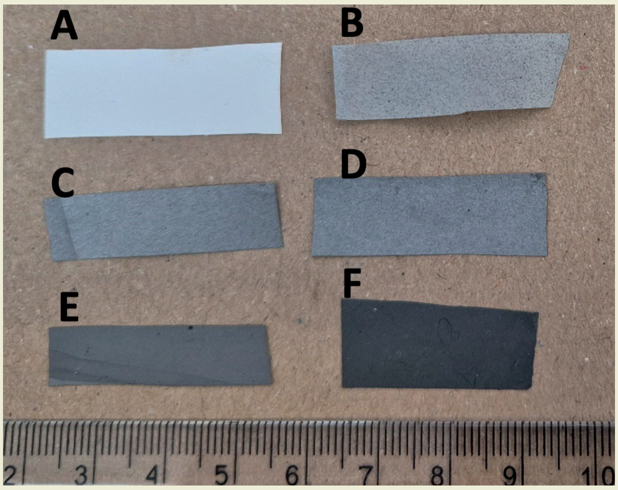

Figure 1. Evidence of color change of the electrospun mats with increasing biochar content in the solution. A – neat PLA/PHB (reference, sample without biochar), B – PLA/PHB + 5% biochar, C - PLA/PHB + 10% biochar, D - PLA/PHB + 15% biochar, E - PLA/PHB + 20% biochar, and F - PLA/PHB + 30% biochar.

4.1. Scanning electron microscopy

▶**Figure 2** shows the scanning electron microscopy images of the investigated samples. The investigation was carried out to check the fibrous structure and determine the fiber diameter depending on the increasing contents of biochar in the polymer solution used for electrospinning.

▶**Figure 2A** presents the morphology of neat PLA/PHB fibers, demonstrating an average diameter of 460 ± 380 nm. While incorporating biochar did not significantly impact the average fiber diameter, it notably reduced the standard deviation (SD) from 380 nm (▶**Figure 2A**) to 190 nm (▶**Figure 2F**). This decrease suggests enhanced fiber uniformity, potentially attributed to increased solution conductivity during electrospinning by adding the higher content of biochar.

While ▶**Figure 2B-2F** shows the complete integration of biochar particles into the fiber structure, it is evident that the distribution of these particles within the mats is uneven. This uneven distribution may impact the overall properties of the material, especially the mechanical ones.

4.2. FTIR and RAMAN spectroscopy

The Raman spectra in ▶**Figure 3A** confirmed the formation of the stable C–C bonds, indicating D- and G-bands formation.

These two prominent bands are observed at 1576 and 1355 cm^{-1}. The peak at 1576 cm^{-1} (G-band) is attributed to the vibration of sp^2 hybridized carbon atoms in a 2D hexagonal lattice, the peak at 1355 cm^{-1} (D-band) is associated with the vibrations of carbon atoms with dangling bonds inplane terminations of the disordered graphite from the defects and disorders of structures in carbon materials [14]. The D-band intensity ratio (148,076) to G-band (134,147) peaks depend on the type of graphitic materials and reflect the graphitization degree; in this case, the ratio of intensities (ID/IG) is 1,104.

FTIR analysis was carried out to determine the composites' chemical structure change. The FTIR spectra of the neat polymer blend and composite are presented in ▶**Figure 3B**. The spectra were typical for PLA/PHB. The 753 and 871 cm^{-1} peaks indicate that plasticized PLA possesses a semicrystalline morphology.

The three absorption bands centered at 970 cm^{-1}, 868 cm^{-1}, and 1450 cm^{-1} correspond to the motions of the plasticizer's C-O and C-C stretching vibrations and the $-CH_3$ stretching vibration of PLA, PHB, and citrate ester molecules and reduced as their contents increased. The FTIR spectra of plasticized PLA/PHB indicate molecular interactions between PLA/PHB and citrate esters. The interaction between PLA and citrate esters may be attributed to the possible hydrogen bonding between the C=O group and the small number of terminal hydroxyl groups in the PLA main chain. The band supports this claim at 1757 cm^{-1} and shows peaks between 2941 cm^{-1} and 2996 cm^{-1}, corresponding to hydroxyl group stretching. The peaks at about 1100 – 1380 cm^{-1} correspond to the C-O-C, C-O stretching, and CH_3 wagging of PHB [14].

No difference in the infrared pattern was revealed when the biochar was incorporated into the composite composition. The reason may be that biochar is almost completely integrated into the structure of PLA/PHB polymer fibers, supported by SEM images. Still, biochar has similar groups to PLA or PHB (▶**Figure 3A**, FTIR of biochar), and the bands overlap, or there are no interactions between the biochar and polymer.

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4.3. Thermal analysis

TGA analysis was used to determine the thermal stability of the neat PLA/PHB and composites PLA/PHB-biochar with various biochar contents. The neat PLA/PHB TGA shown in ▶**Figures 4A** and **4B** has multiple mass loss steps corresponding to the blend's single materials. The temperature of the maximum decomposition peak, around 272 °C, was assigned to PHB, and the second one was around 350 °C for PLA. These values are in agreement with the results reported in [15]. ▶**Figures 4A** and **4B** also show that the increasing content of biochar slightly increases the decomposition temperature of PHB from 272 to 282 °C. Still, the maximum decomposition temperature of PLA decreased from 352 to 330 °C. This situation can result from increased biochar and better heat transfer, which for PLA increases with increased orientation and crystallization of the molecular chains and stronger molecular chain interactions [7].

DSC analysis was used to follow changes caused by cold crystallization characterized by temperature (T_{CC}) and enthalpy (ΔH_{CC}) and the melting process represented by melting temperature (T_m) and enthalpy (ΔH_m). As previously described [16], T_{CC} and T_m of neat PLA are at 60.3 °C and 150 °C, respectively. Likewise, the neat PHB shows T_{CC} and Tm at 90 °C and 180 °C, respectively. The PLA/PHB blend containing plasticizer was exhibited; therefore, only one T_{CC} was observed, indicating good system miscibility. In addition, two melting peaks at

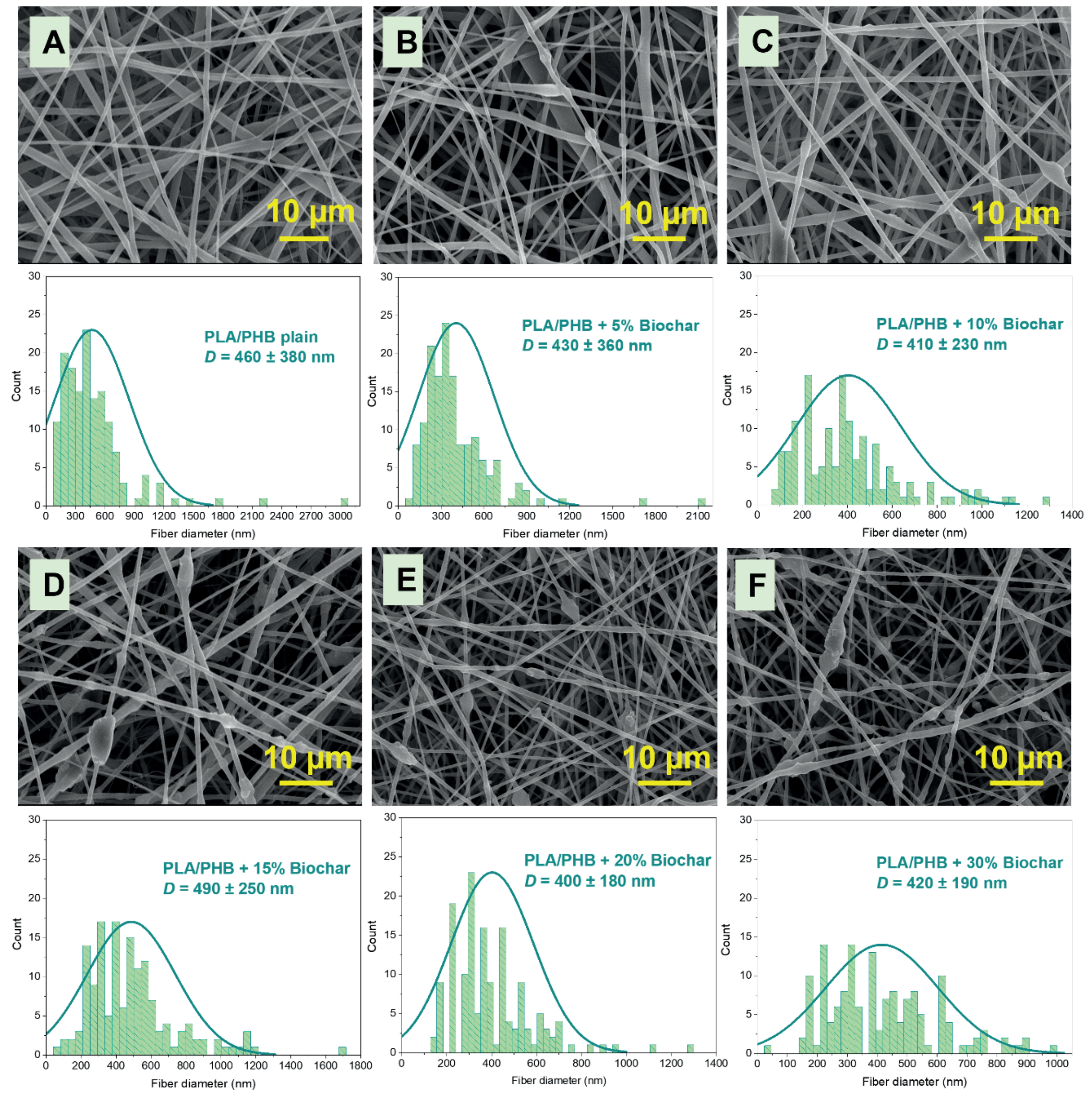

Figure 2. SEM images and histograms of fiber diameters. The image A – neat PLA/PHB (reference sample, without biochar), B – PLA/PHB + 5% biochar, C - PLA/PHB + 10% biochar, D - PLA/PHB + 15% biochar, E - PLA/PHB + 20% biochar, and F - PLA/PHB + 30% biochar. The images were taken at x2000 magnification. Below each image is a corresponding histogram of fiber diameters.

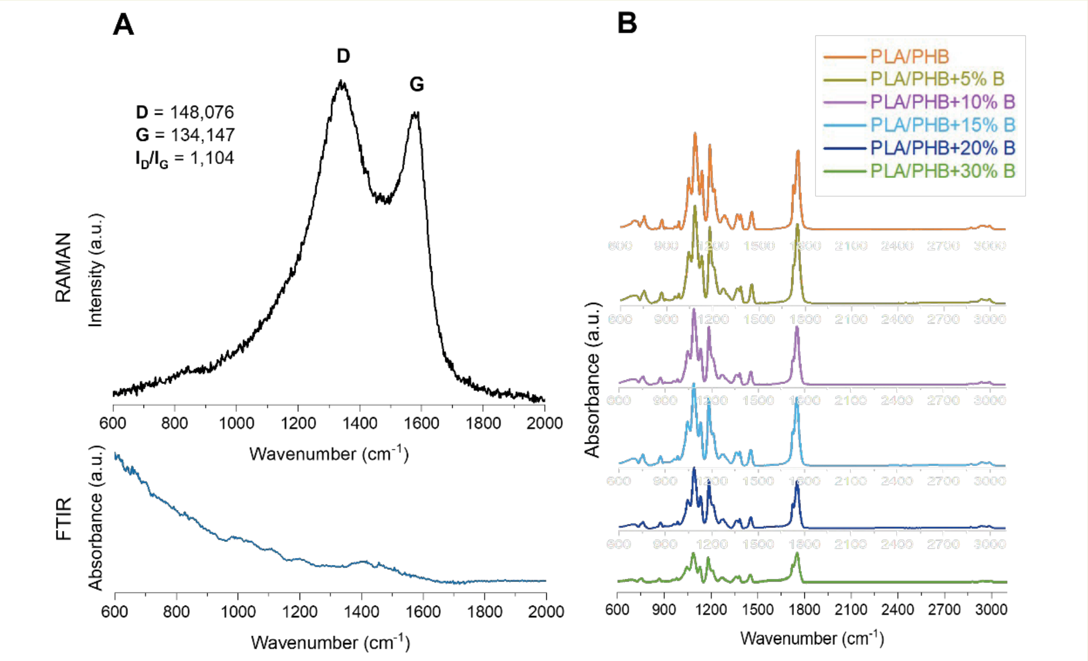

Figure 3. RAMAN and FTIR spectra of biochar (A) and PLA/PHB-biochar composites (B).

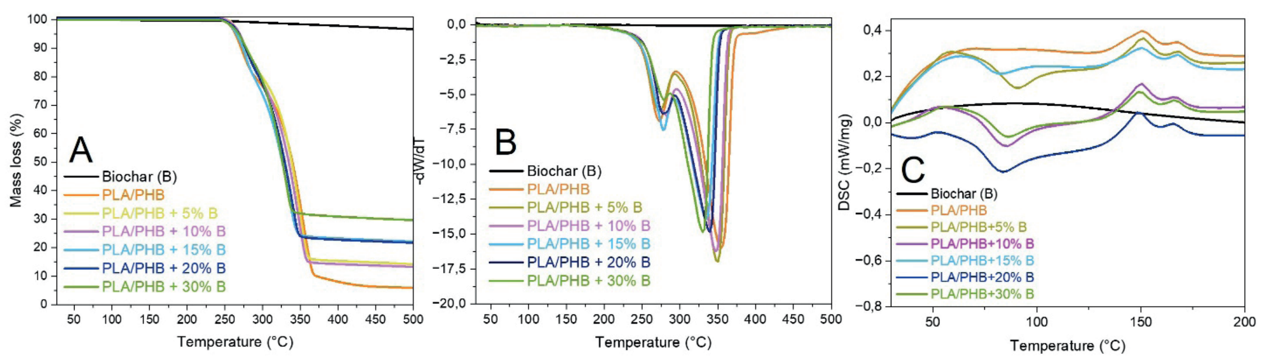

Figure 4. TGA (A), DTGA (B), and DSC (C) spectra of biochar, neat PLA/PHB, and PLA/PHB-biochar fibrous composites.

150 and 170 °C for PLA and PHB crystals, respectively, were observed during the first heating cycle, how as shown in **▶Figure 4C**.

The loadings of the biochar changed the T_{CC}, which decreased with adding biochar. The values are summarized in **▶Table 2**. The lowest T_{CC} was measured in PLA/PHB+15%B. The changes of T_{m1} and T_{m2} were less significant. However, there was not a clear trend, and the T_{CC}, T_{m1}, and T_{m2} fluctuated with the increasing content of biochar, possibly due to the uneven distribution of biochar in electrospun fibers. Despite that, it can be said that adding biochar does not significantly impact the thermal stability of the polymer blends, which may be advantageous in subsequent applications.

4.4. Mechanical properties

The tensile properties of the polymer blends and composites are significantly affected by compatibilizers and filler particles. The results of mechanical testing

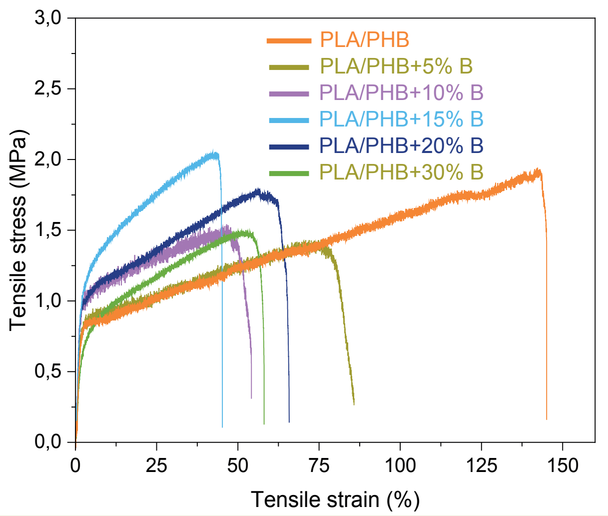

Figure 5. Stress - strain curves of electrospun PLA/PHB and bio-composites PLA/PHB with various biochar content.

Table 1. Summarized results of mechanical testing of plasticized PLA/PHB blend and composites differ in biochar loading.

Description	Tensile stress at tensile strength (MPa)	Tensile strain at break (%)	Young´s modulus (MPa)
PLA/PHB	2.31 ± 0.22	161 ± 22.8	55.7 ± 11.3
PLA/PHB + 5% Biochar	1.37 ± 0.13	101 ± 29.2	64.3 ± 11.9
PLA/PHB+10% Biochar	1.54 ± 0.15	53.8 ± 12.9	81.7 ± 15.7
PLA/PHB+15% Biochar	1.90 ± 0.15	37.8 ± 11.3	79.1 ± 12.3
PLA/PHB+20% Biochar	1.81 ± 0.21	63.2 ± 6.83	77.5 ± 10.7
PLA/PHB+30% Biochar	1.40 ± 0.17	59.7 ± 10.9	39.1 ± 8.56

Table 2. DSC analysis of PLA/PHB and PLA/PHB+B mats.

Code	T_{cc} [°C]	T_{m1}(PLA) [°C]	T_{m2} (PHB) [°C]	ΔH_{cc} [J.g-1]	ΔH_{m1} [J.g-1]	ΔH_{m2} [J.g-1]
PLA/PHB	103.8	150.1	171.4	-0.1	6.8	0.4
PLA/PHB+5%B	89.8	150.1	168.5	-10.0	7.9	0.8
PLA/PHB+10%B	85.2	149.7	168.3	-7.7	8.6	0.7
PLA/PHB+15%B	79.8	149.2	166.9	-3.1	4.9	1.0
PLA/PHB+20%B	83.4	148.9	168.3	-4.6	6.2	0.7
PLA/PHB+30%B	82.5	148.1	167.2	-3.9	4.4	1.1

of plasticized PLA/PHB blend and composites differ in biochar loading are summarized in ▶**Table 1**. The stress-strain curves for plasticized PLA/PHB blend and PLA/PHB reinforced with 5, 10, 15, 20, and 30 wt.% are shown in ▶**Figure 5**. As is seen, adding 5 wt.% of biochar leads to a significant decline of elongation at break. It reaches half value compared to the unfilled blend due to the unequal distribution of filler particles in the composite volume. In addition, with increasing biochar loading, the reinforcing effect was manifested by tensile strength increase with a simultaneous drop in percentage elongation up to 15 wt.% biochar content. The decrease in tensile strength was also detected at higher biochar loading due to higher interfacial tension within multiphase composites, leading to the deterioration of mechanical properties.

The uneven distribution of biochar may cause values to fluctuate, in this case, also due to the thickness of the mats. Since it is impossible to control the thickness of the sample fully in a single-needle electrospinning device, the high standard deviation of tensile test results. The influence of biochar content on mechanical properties needs a more profound study in the future.

5. Conclusion

The study aims to design fibrous composites with kitchen waste-derived biochar as filler to biodegradable polymers for a sustainable way to manage kitchen waste. Such composites are an exciting alternative to expensive biodegradable polymers and could reduce the price of biodegradable polymers. In the study, the fibrous composites PLA/PHB + biochar were prepared using the electrospinning method and elementary characterization of such composites were carried out. The morphology of electrospun mats was studied by SEM. The fibers with an average diameter of 400 – 500 nm were received, and there was not observed a significant effect of the percentual content of biochar in the solutions. All the biochar particles were capsulated into the structure of the fibers, which was observed in SEM images but also confirmed with spectroscopy; only the polymers left traces in the spectroscopic spectra. The thermal analysis revealed that biochar addition does not significantly impact the thermal stability of the polymer blends. The mechanical properties were most affected as both tensile strength and strain to stress were reduced, which may limit the practical use of these fiber mats. In the future, it is necessary to pay more attention to the compatibility of individual components in the composite, including validating the sustainability of waste utilization for biochar production, or the usability and feasibility of the composite in the individual applications such as the packaging industry, filtration, or as agrotextile.

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None

Research ethics

Not applicable.

Author contributions

Conceptualization: [Alena Opálková Šišková, Naďa Beronská], Methodology: [Alena Opálková Šišková, Tomáš Dvorák, Andrej Opálek, Katarína Mosnáčková, Viera Dujnič], Formal Analysis: [Tomáš Dvorák, Andrej Opálek, Katarína Mosnáčková, Viera Dujnič], Investigation: [Alena Opálková Šišková, Katarína Mosnáčková], Resources: [Alena Opálková Šišková, Naďa Beronská], Data Curation: [Alena Opálková Šišková, Tomáš Dvorák, Naďa Beronská], Writing - Original Draft Preparation: [Alena Opálková Šišková, Katarína Mosnáčková], Writing - Review & Editing: [Alena Opálková Šišková, Katarína Mosnáčková, Naďa Beronská], Visualization: [Tomáš Dvorák, Andrej Opálek, Katarína Mosnáčková, Viera Dujnič], Supervision: [Alena Opálková Šišková, Naďa Beronská], Project Administration: [Alena Opálková Šišková, Naďa Beronská], Funding Acquisition: [Alena Opálková Šišková, Naďa Beronská]

Competing interests

The author(s) declare(s) no conflict of interest.

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Data availability

The raw data can be obtained on request from the corresponding author.

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