



Article

Sheep Wool Biochar-Enhanced HDPE Composites

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Abstract

Animal-based biomass is gaining increasing attention in composites technology as a sustainable alternative to conventional fillers, offering a green pathway in the generation of composites exhibiting improved performance via waste valorization. In the present study, carbonized sheep wool was incorporated into high-density polyethylene (HDPE) in various weight ratios up to 10% wt. to fabricate composite specimens. The resulting composites were evaluated through Dynamic Mechanical Analysis (DMA), while their morphology and chemical structure were investigated by Scanning Electron Microscopy (SEM) combined with Energy Dispersive X-ray Spectroscopy (SEM-EDS) and Fourier Transform Infrared Spectroscopy (FTIR), respectively. FTIR analysis revealed the presence of residual keratin-derived oxygen- and nitrogen-containing functional groups, indicating the retention of chemically active surface functionalities upon low-temperature carbonization. This evidence is further corroborated through qualitative (SEM-EDS) elemental mapping of the pristine surfaces of sheep wool fibers and the pyrolyzed biochar product. DMA experimental data demonstrated that sheep wool-derived biochar (SWB) can effectively reinforce HDPE, resulting in stiffness enhancement while reducing viscous dissipation, thereby highlighting its potential as a sustainable, eco-friendly filler and a viable pathway for circular valorization of animal biomass waste.

Keywords: HDPE; animal-derived biochar; thermoplastic composites; sheep wool; Dynamic Mechanical Analysis

1. Introduction

The increasing global demand for materials has led to the rapid accumulation of waste and escalating environment pollution, with plastic waste being a major contributor. Production of plastics has soared from millions to hundreds of millions of tons per year, yet only a small fraction is recycled, while the majority ends up discarded in landfills at the end of their life cycle [1–3]. Much of this waste originates from fossil-derived polymers such as high-density polyethylene (HDPE), polypropylene (PP) and polyethylene terephthalate (PET), which are highly persistent and contribute significantly to pollution [4,5]. Among those, HDPE is one of the most widely used thermoplastics due to its excellent mechanical strength, chemical resistance and low cost, making it prevalent in packaging, containers, etc. [6]. However, the large-scale use and recyclability of HDPE present significant environmental challenges, contributing to long-term pollution [7,8].

Nowadays, the incorporation of natural fibers and bio-based fillers within polymer matrices has gained growing attention as a sustainable strategy to improve materials



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while reducing environmental impact. Specifically, these renewable fillers, derived from plants, animals or minerals, can offer notable advantages, including reduced greenhouse-gas emissions, lowering carbon footprints and minimization of waste [9,10]. Moreover, their low density, design flexibility and biodegradability further strengthen their appeal to composite fabrication [11–13]. However, natural fillers also present some challenges, including thermal instability and moisture sensitivity, which limit their co-processing within high-temperature thermoplastic matrices [14]. To overcome these limitations, an emerging approach involves converting biomass-derived natural fillers into biochar, a carbon-rich, thermally stable material obtained through pyrolysis. Biochar has emerged as a promising alternative to conventional carbon-based nanofillers, which, despite their utilization as additives to enhance the thermomechanical performance of polymers, are neither environmentally friendly nor economically viable [15–17]. In contrast, biochar is a renewable, carbon-rich material generated through pyrolysis of biomass at elevated temperatures, a process that simultaneously valorizes and reduces landfill waste [18,19]. Although the production of biochar requires an energy-intensive pyrolysis process, its overall environmental impact can be offset through the valorization of biomass waste streams and the generation of a stable carbon-rich material [20]. Rather than being disposed of through landfilling or incineration, agricultural and animal-derived residues can be converted into value-added fillers, supporting circular economy principles and reducing dependence on fossil-derived reinforcing materials [21,22]. Numerous studies report that the incorporation of biochar into polymer matrices can lead to polymer composites exhibiting superior properties compared to their pristine polymer counterparts, offering comparable or even enhanced mechanical performance compared to those reinforced with expensive carbon-based nanofillers [23,24].

In this study, sheep wool was selected due to its high abundance (production of 2.3–3.6 kg of wool per sheep per year), as regular shearing is necessary for the animal's health [25,26]. In general, sheep wool has been commonly utilized in the textile industry due to its physicochemical properties, including high elasticity, moisture absorption capacity, thermal insulation and flame resistance [27]. However, its elastic modulus, which is around 1–4 GPa and is similar to that of plastic fibers [28], makes it a great candidate as a reinforcement in composite materials for a variety of applications including construction and agriculture, among others [28–30].

Currently, researchers have been investigating the potential of sheep wool fibers as fillers in cement and mortars, as well as in polymer composites. For example, Alyousef et al. [31] fabricated concrete composites filled with sheep wool fibers, and although their addition led to a small decrease in the compressive strength, the fibers improved the tensile and flexural strength values, highlighting its potential for the production and industrialization of eco-friendly composite concrete. In another study, Demircan et al. [32] investigated a combination of slaked lime, pumice powder and sheep wool fibers. Their findings revealed that the addition of the latter generally improved flexural strength and reduced shrinkage, although excessive fiber content caused agglomeration and reduced performance. In polymer systems, Chowdhury et al. [33] developed PP hybrid composites using water hyacinth and sheep wool fibers as natural reinforcements, aiming to develop environmentally friendly materials with enhanced properties. The composites were prepared with varying fiber loadings of 5, 10 and 15 wt.%, and their thermomechanical, structural and biodegradability characteristics were thoroughly investigated. These studies demonstrate that sheep wool has primarily been investigated in its raw fibrous form, where it has shown promising multifunctional properties across different material systems.

However, sheep wool can also be valorized through the thermochemical conversion process, called pyrolysis, to produce biochar, offering an alternative pathway for waste

utilization. In recent years, several studies have investigated the incorporation of plant-derived biochar as sustainable fillers in thermoplastic composites. For instance, Das et al. [34] investigated wood-derived biochar as a reinforcement in PP composites and reported improvements in stiffness, thermal stability and flame resistance. Similarly, Pudelko et al. [35] incorporated wood waste-derived biochar and sewage sludge biochar into polylactic acid (PLA) composites and observed improved rigidity, thermal stability and dimensional stability while also highlighting the potential of waste-derived fillers in biodegradable polymers. Additionally, recent review studies by Bartoli et al. [16] and Aboughaly et al. [24] highlighted the growing use of biochar derived from rice husks, bamboo residues, nutshells and other agricultural biomass in polymer systems, where improvements in thermomechanical performance and dimensional stability have been reported. Despite this growing field of research, these studies remain almost exclusively limited to plant-based feedstocks, while the use of animal-derived biochar, particularly sheep wool-derived biochar (SWB), remains largely unexplored.

To the best of our knowledge, the incorporation of sheep wool-derived biochar as a reinforcing filler in HDPE composites has not been previously reported. While numerous studies have investigated plant-derived biochar as additives in HDPE matrices and wool fibers as inclusions in polymer composites, the utilization of sheep wool biochar as a sustainable filler for thermoplastic composite applications remains largely unexplored. Specifically, compared to conventional plant-derived biochar, sheep wool-derived biochar possesses a distinct chemical composition originating from its keratin-rich structure, which contains significant amounts of organic elements such as nitrogen, oxygen and sulfur [36]. The presence of nitrogen-containing functional groups during pyrolysis can introduce additional active surface sites and modify the surface chemistry of the resulting biochar, which may promote stronger interfacial interactions with polymer matrices compared to conventional plant-derived biochar inclusions [37,38]. Furthermore, keratin-derived biochar has been reported to exhibit heteroatom-enriched structures without the need for post-synthesis chemical doping, providing a distinct advantage over other biomass-derived biochar that require additional modification steps to achieve comparable functionality [39]. In addition, biochar that is produced from animal-based feedstocks generally exhibits different chemical composition and mineral contents compared to plant-derived biochar, which can influence thermal stability and functional performance in composite applications [40].

Therefore, in the present study, SWB obtained through low-temperature pyrolysis was incorporated into HDPE to fabricate uniform composite specimens. This low-temperature approach further reduces the energy input and associated emissions of the conversion process, thus lowering the environmental footprint while still yielding a stable, carbon-rich filler suitable for composite applications.

The resulting composite specimens were subsequently evaluated through Fourier Transform Infrared Spectroscopy (FTIR), electron microscopy and elemental mapping (SEM-EDS) and thermomechanical characterization, investigating chemical structure, morphology, elemental distribution, filler dispersion and the resulting effects on the thermomechanical performance of the developed composites. This work demonstrates the potential of transforming under-utilized biomass waste into value-added polymer composites and highlights SWB as a promising renewable filler for the development of sustainable material systems.

2. Materials and Methods

2.1. Raw Materials

High-density polyethylene (HDPE), which was kindly provided in the form of pellets by Elysee Irrigation (Cyprus), was used as the polymer matrix. Sheep wool (SW) was obtained from sheep breeding conducted at a local farm (Nicosia, Cyprus).

2.2. Material Preparation and Lab-Scale Pyrolysis

Initially, the as-obtained SW was thoroughly cleaned by washing using natural soap and then left to air dry in the fume hood for 48 h. Figure 1 provides photographs of the SW prior to and after cleaning. The cleaning/drying steps were followed by heating using a 21,100 Tube Furnace (Thermolyne) under a nitrogen atmosphere at $10\text{ }^{\circ}\text{C min}^{-1}$ up to a constant temperature step of 30 min. Three different temperatures were tested, i.e., $650\text{ }^{\circ}\text{C}$, $450\text{ }^{\circ}\text{C}$ and $250\text{ }^{\circ}\text{C}$.



Figure 1. Photographs of the sheep wool fibers prior to (as received) and after washing.

2.3. Fabrication of HDPE/SW Biochar Composites

For the fabrication of the composite plates, neat HDPE pellets were added into the rollers of a twin-roll milling machine while gradually adding the grinded biochar material. The twin milling process was performed at Colorants for Plastics, D. Souris & Co SA, Aspropirgos, Greece, and information on the experimental protocol followed can be found in a previous publication by our group [41]. Following blending, the composites were compression molded into flat plates with dimensions of $60 \times 30 \times 2\text{ mm}^3$, intended for Dynamic Mechanical Analysis (DMA). More precisely, the following processing conditions were employed: milling temperature: $160\text{ }^{\circ}\text{C}$; milling time: 4 min (in average); roll speed: 9.6 rpm; compression molding temperature: $190\text{ }^{\circ}\text{C}$; compression time in press: 60 s; cooling time in press: 23 s and pressure: 100 bar.

A representative illustration of the fabrication process employed is shown in Figure 2. Four composite formulations were produced with contents varying from 2.5% to 10% wt., along with the control sample (0% wt. biochar filler) (Table 1), using the SW biochar, pyrolyzed at $250\text{ }^{\circ}\text{C}$. While the density of HDPE is known from the literature (0.96 g/cm^3) [42], the density of the SWB was determined experimentally using a manual hydraulic press (25 Ton-GS25011, Specac Ltd., Orpington, UK) equipped with a 10 mm diameter cylindrical steel die. More precisely, the biochar powder was compacted into a dense, uniform disc under a pressure of 25 tons at room temperature for 15 min. The resulting specimen was characterized by measuring its mass and geometric dimensions (diameter and thickness) using a high-precision micrometer, allowing for density calculation via Equation (1), where d = density, m = mass and V = volume. The experimental density of the SWB was determined to be 1.080 g/cm^3 .

$$d = \frac{m}{V} \quad (1)$$

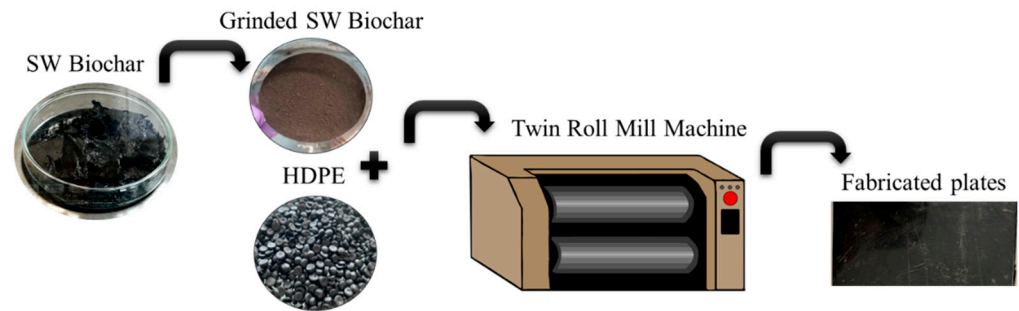


Figure 2. Fabrication of HDPE/SW biochar composite plates using the twin-roll milling process.

Table 1. DMA results for HDPE/SWB composite samples at 38 °C.

Sample	Storage Modulus (MPa)	SD (MPa)	Tanδ	SD
Control (HDPE)	888.86	19.33	0.13	0.004
SWB (2.5%)/HDPE	921.03	42.40	0.13	0.002
SWB (5%)/HDPE	985.20	39.06	0.12	0.003
SWB (7.5%)/HDPE	987.77	34.24	0.12	0.002
SWB (10%)/HDPE	991.25	13.93	0.12	0.002

2.4. Morphological Characterization

The morphological characteristics of the SW fillers before and after pyrolysis were determined by Scanning Electron Microscopy (SEM) (Vega TS5136LS-Tescan, Brno, Czech Republic). Prior to their inspection, all samples were gold-sputtered (sputtering system K575X Turbo Sputter Coater-Emitech, Quorum Technologies Ltd., West-Sussex, UK). The average fiber diameter as well as fiber diameter distribution (where applicable) were calculated from the obtained SEM micrographs via ImageJ Launcher open-source software program, through at least 50 measurements.

2.5. Fourier Transform Infrared Spectroscopy (FTIR)

The chemical structure of the SWB was investigated using a Shimadzu spectrometer 8900. Prior to analysis, the biochar powder was finely ground and mixed with spectroscopic-grade potassium bromide (KBr), followed by pellet formation using a hydraulic press, at a mass ratio of 10:1. FTIR spectra were recorded in the wavenumber range of 4000–400 cm^{-1} at room temperature and were further used to identify the functional groups present on the surface of the biochar and to evaluate the chemical changes induced by the pyrolysis process.

2.6. Energy Dispersive X-Ray Spectroscopy

Energy dispersive spectroscopy (EDS) mapping, along with Scanning Electron Microscopy (SEM) imaging (Apreo ChemiSEM system, Thermo Fisher Scientific), was also carried out to investigate the elemental composition of the pristine surface (no conductive coating) of sheep wool fibers and their pyrolyzed biochar product. This was carried out using a combination of Everhart–Thornley secondary electron detector (ETD) and annular segmented in-lens detector T1 (secondary and backscattering electron detection capability) with an UltraDry Energy Dispersive X-ray Spectroscopy (EDS) detector, respectively, at an electron acceleration voltage of 5 kV (Schottky field emission gun electron source) and 0.4 nA of probe current. As such, topological, morphological and compositional information acquisition from electron-beam sensitive organic matter was enabled under high vacuum. Due to the nature of the organic matter under investigation and the presence of

elements with atomic numbers below $Z = 8$ (besides S), no quantitative elemental analysis has been performed.

2.7. Thermomechanical Characterization

The thermomechanical behavior of neat HDPE and HDPE/SW biochar composites was evaluated by performing Dynamic Mechanical Analysis (DMA) using a Lacerta Dynamic Mechanical Analyzer. DMA temperature scan experiments were performed in single-cantilever bending mode at a constant oscillation frequency of 1 Hz, a heating rate of $4\text{ }^{\circ}\text{C min}^{-1}$ and a displacement amplitude of 0.05 mm. The temperature dependence of the Storage Modulus (E') and loss factor ($\tan \delta$) was examined over the range of 25–140 $^{\circ}\text{C}$. All measurements were initiated at approximately 25 $^{\circ}\text{C}$, while the upper temperature limit was selected based on the melting behavior of the polymer matrix. In accordance with ASTM D7028-07, six test specimens were prepared for each formulation. The specimens were machined to dimensions of 25 mm $\times$ 8 mm $\times$ 2 mm (length $\times$ width $\times$ thickness) using a custom-designed cutting die and a manual hydraulic bench press (Model MP-3, Cogentech; maximum load: 3 tons), as illustrated in Figure 3.

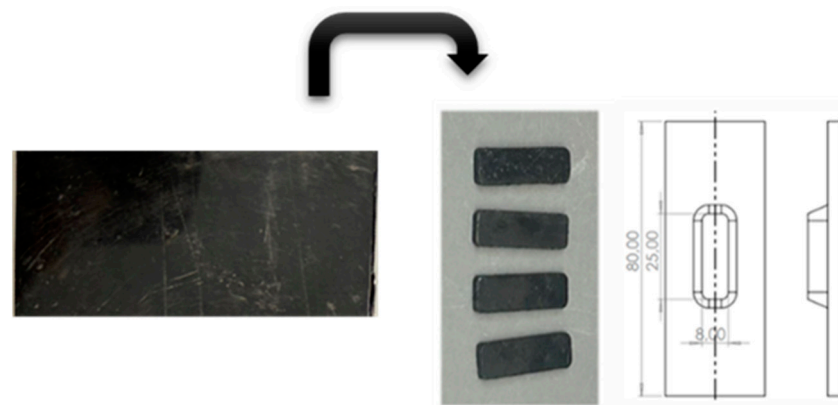


Figure 3. Test specimens prepared for DMA analysis and dimensions of the prepared cutting die.

3. Results and Discussion

3.1. Morphological Characterization

Figure 4 presents the morphological characteristics of the SW fibers, obtained after washing, highlighting their fibrous structure, surface roughness and overall uniformity [43,44]. The fiber diameter distribution is quantitatively illustrated in the same figure (right image).

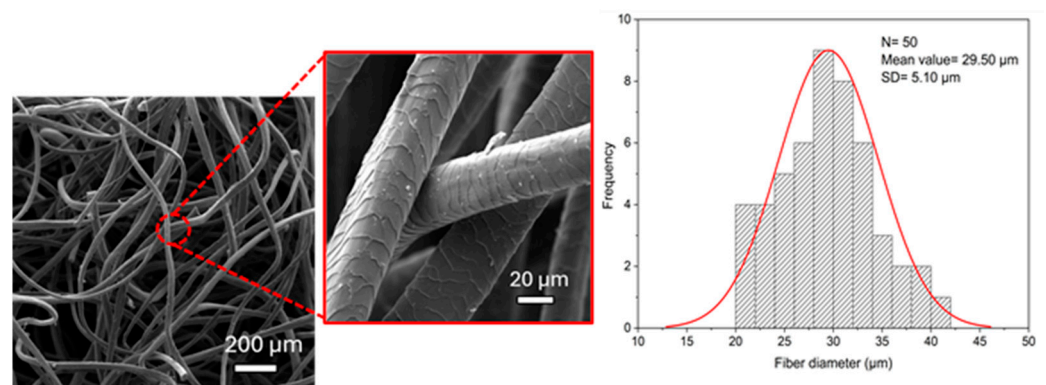


Figure 4. SEM images of sheep wool fibers (after washing/drying) and corresponding diameter distribution.

In Figure 5, characteristic SEM images of SW biochar (SWB) obtained after pyrolysis at various temperatures are provided. SEM analysis revealed that the native fibrous morphology of sheep wool was substantially altered after pyrolysis, with the most pronounced structural collapse observed in the case of the biochar produced at 250 °C (where the material exhibited a flake-like morphology), while partial preservation of the fibrous structure was still evident at 450 and 650 °C. This behavior can be attributed to the thermal degradation and volatilization of keratin components, which induce shrinkage, surface collapse and fragmentation of the original fibrous architecture during carbonization. Similar observations have been previously reported for other bio-based fibers [45]. Despite the more pronounced morphological changes observed at 250 °C, this SWB was selected to be used as a filler in the preparation of HDPE composites because lower-temperature pyrolysis offers important economic and energy consumption advantages compared to higher-temperature treatment, thus significantly reducing the fabrication costs. In addition, biochar produced under low-temperature conditions generally exhibit higher char yields and retain more surface functional groups [46], which may contribute positively to interfacial interactions with the polymer matrix. In the work of R. Kumar and co-workers [47], it was demonstrated that bamboo-derived biochar obtained at lower pyrolysis temperature (i.e., 300–400 °C) led to improved strength properties of the PP-based composites, whereas the strength properties remained unchanged upon pyrolysis at higher temperatures (i.e., between 600 and 900 °C).

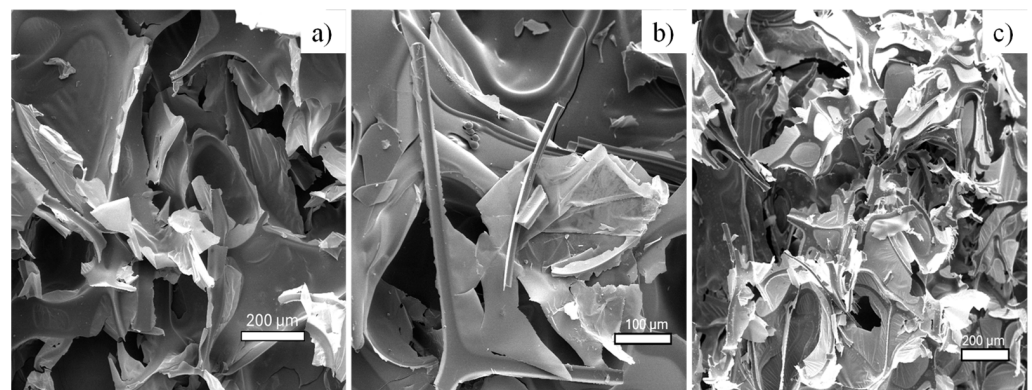


Figure 5. SEM images of sheep wool biochar (SWB) (after washing/drying) pyrolyzed at 250 °C (a), 450 °C (b) and 650 °C (c).

Figure 6 illustrates the surface morphology of the composite SWB/HDPE plates fabricated using the twin-roll milling process. Although macroscopically the composite plates exhibit a uniform appearance and no differences are observed between specimens of different formulations, as seen in SEM images, all samples are characterized by surface defects that seem to become more pronounced upon increasing the biochar content. The SEM images provided in Figure 6i–iv verify the successful inclusion of SWB within the polymer matrix.

Similar morphological features have been reported in biochar-reinforced polymer systems, where incomplete filler dispersion often leads to particle clustering and interfacial void formation, particularly at higher filler loadings [48,49]. These defects are frequently associated with processing limitations, filler morphology and the high viscosity of polymer melts, which may hinder the complete breakdown of biochar agglomerates and result in non-uniform filler distribution [50,51]. Although twin-roll milling enabled the successful fabrication of the present composites, its relatively lower shear intensity compared to more advanced melt-compounding techniques, such as twin-screw extrusion, may have contributed to the formation of localized defects. For instance, Shah et al. [52] investigated

rice husk biochar-reinforced recycled HDPE composites fabricated through extrusion processing and reported relatively improved filler integration within the polymer matrix. SEM analysis revealed that HDPE penetrated and filled the porous surface structure of the biochar particles, enhancing interfacial interaction and contributing to improvements in tensile and flexural performance. These findings indicate that processing conditions play a critical role in determining filler dispersion and interfacial interaction within biochar–polymer composites.

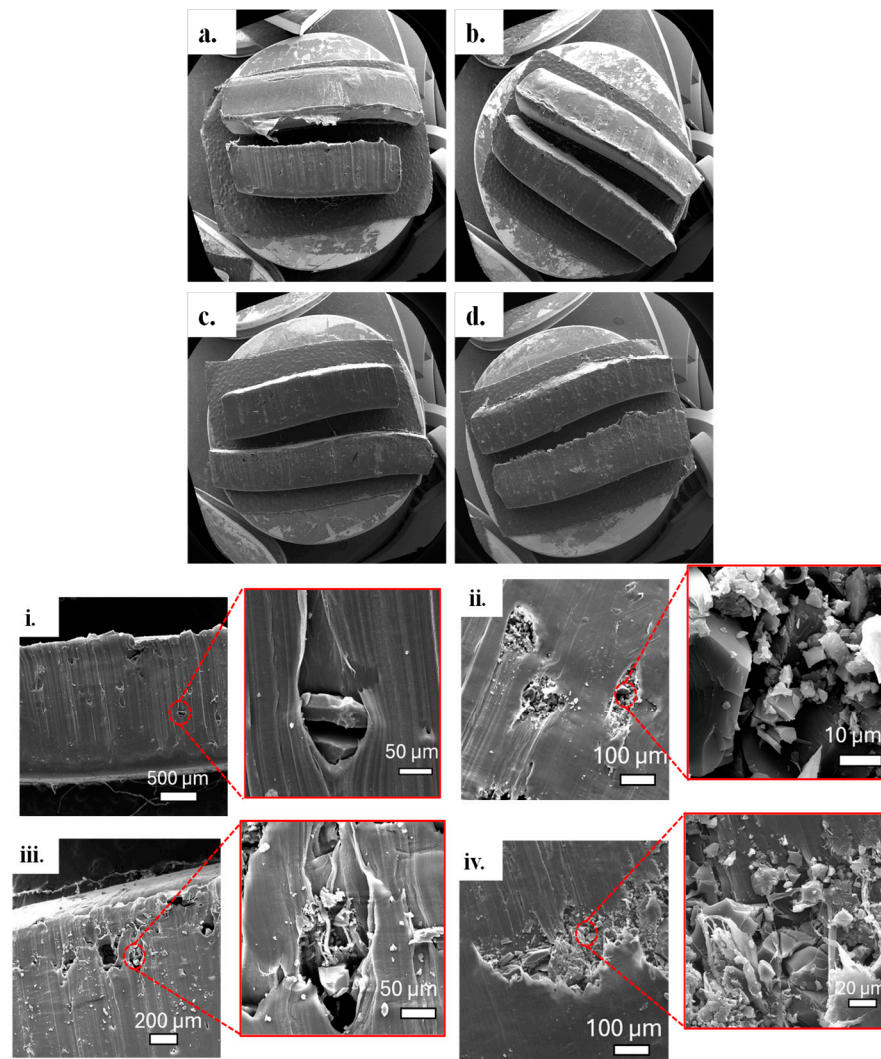


Figure 6. SEM images of SWB (2.5%)/HDPE (a,i); SWB (5%)/HDPE (b,ii); SWB (7.5%)/HDPE (c,iii) and SWB (10%)/HDPE (d,iv).

3.2. Fourier Transform Infrared Spectroscopy (FTIR)

In Figure 7, the FTIR spectrum of the SWB is presented. A broad absorption band centered at approximately 3373 cm^{-1} was observed and can be attributed to N–H stretching vibrations (Amide A), hydroxyl groups and adsorbed moisture. The absorption at 2928 cm^{-1} can be assigned to the C–H stretching vibrations. A pronounced band appearing at 1642 cm^{-1} can be primarily attributed to the Amide I vibration (C=O stretching of peptide bonds), while a weaker feature in the 1540 cm^{-1} region corresponds to Amide II vibrations arising from N–H bending and C–N stretching. These bands are characteristic of wool keratin and indicate that substantial protein-derived structures remain after thermal treatment [53,54]. Additional absorption signals appearing between 1200 and 1000 cm^{-1} can be associated with C–N and C–O chemical bonds originating from keratin [55,56].

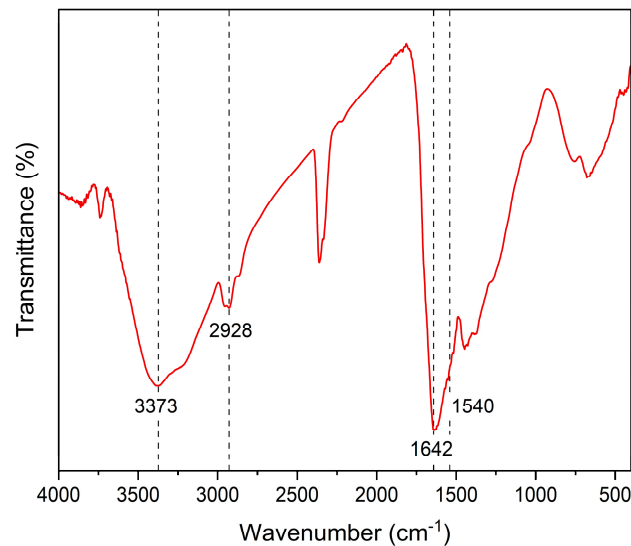


Figure 7. FTIR spectrum of SWB pyrolyzed at 250 °C.

3.3. Energy Dispersive X-Ray Spectroscopy Elemental Mapping

Further support of the FTIR results is provided by qualitative elemental analysis on the surfaces of pristine (no conductive coating) sheep wool fiber and the resulting post-processed biochar. As expected, SEM-EDS analysis on sheep wool fibers confirms the presence of C elements through a pronounced characteristic X-ray peak, distinctive of organic material, while N and S signals are also detected due to the keratin-rich structure (Figure 8). Figure 8a depicts the fibrous nature of the sheep wool sample through secondary electron imaging, with insets b, c, e and f presenting the blended X-ray count maps for C, N, S and O elements of the same overall area. Moreover, inset d presents the corresponding EDS spectrum from an area of approximately $25 \times 25 \mu\text{m}^2$ on the surface of the main fibril, indicating the detection of C, N, S and O characteristic X-ray peaks (K-lines).

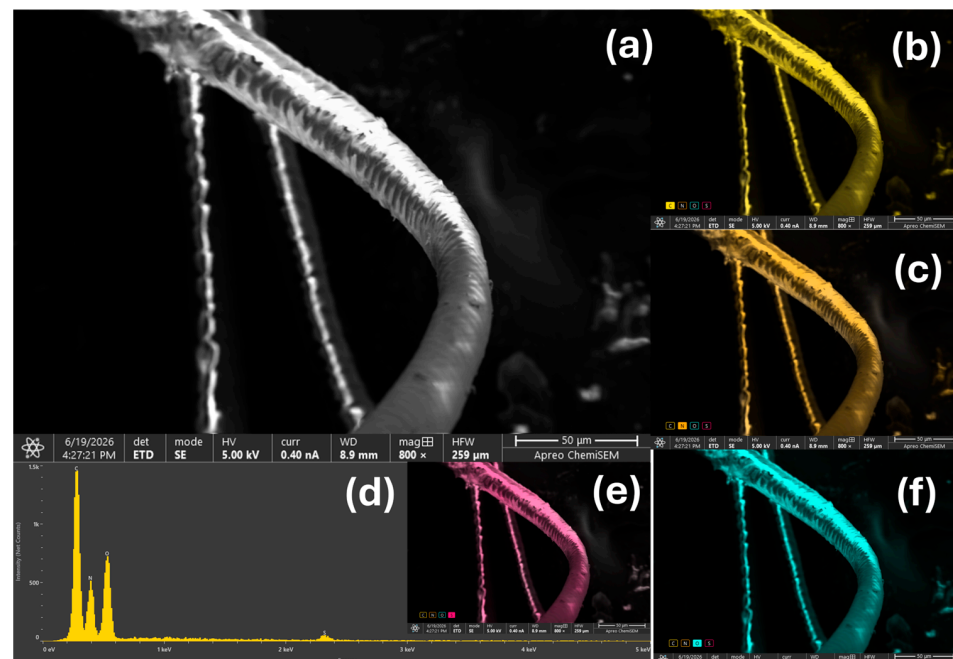


Figure 8. Secondary electron imaging (ETD) of the pristine (no conductive coating) fibrous sheep wool sample (a). Blended X-ray count maps for C, N, S and N elements of the same overall area (insets b,c,e,f). EDS spectrum from an area of approximately $25 \times 25 \mu\text{m}^2$, on the surface of the main fibril (d).

Furthermore, SEM-EDS analysis of sheep wool derived biochar (pyrolyzed at 250 °C) corroborates the evidence suggesting successful carbonization and the retention of N in the structure (Figure 9). On the other hand, S characteristic X-ray signal was substantially diminished in sheep wool-derived biochar, possibly due to volatilization of this element during the pyrolysis. Figure 9a depicts the flake-like nature of the sheep wool sample through composite secondary and backscattered electron imaging, while uniform backscattering signal suggests that flakes are likely comprised of a single homogeneous organic phase. This is also supported in insets b, c and e presenting the uniform distribution of C, N and O elements, depicted in blended X-ray count maps from the same overall area. Inset 1d presents the corresponding EDS spectrum from an area of approximately $25 \times 25 \mu\text{m}^2$ on the surface of the main biochar flake (lower left side of Figure 9a), indicating the detection of C, N and O characteristic X-ray peaks (K-lines). Minute signal counts of S characteristic lines are within the background noise, and as such, S cannot be considered detectable through EDS on these samples given the morphological characteristic biochar and the detection limit of the characterization method.

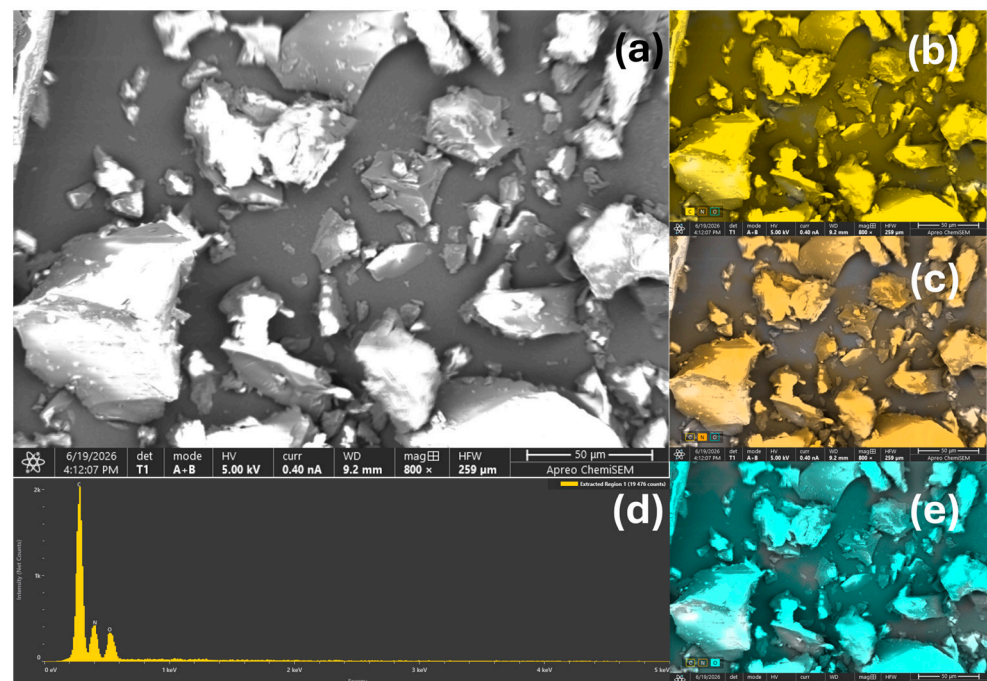


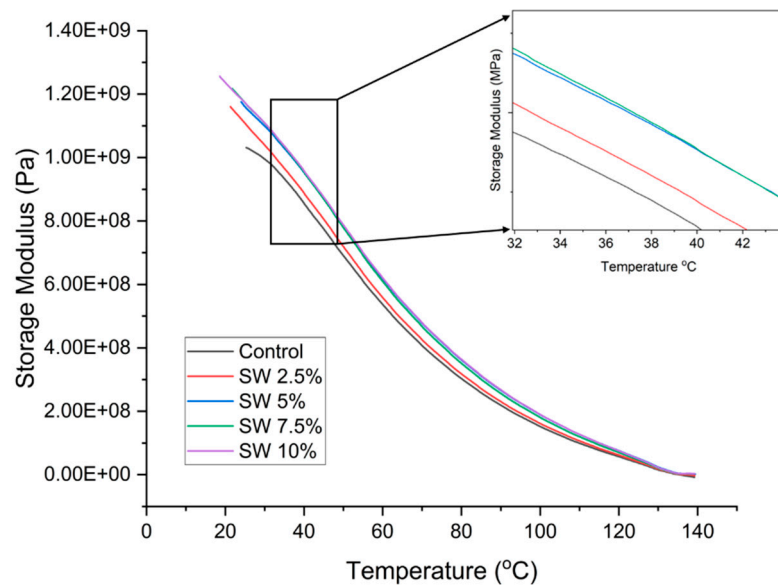
Figure 9. Composite secondary and backscattered electron imaging (T1) of the pristine (no conductive coating) flake-like sheep wool sample (a). Blended X-ray count maps for C, N and O elements of the same overall area (insets b,c,e). EDS spectrum from an area of approximately $25 \times 25 \mu\text{m}^2$, on the surface of the main biochar flake depicted on the bottom left T1 image (d).

3.4. Thermomechanical Performance

DMA revealed a progressive increase in the Storage Modulus with increasing SWB content, indicating an enhancement in the stiffness of the HDPE matrix. Compared to the control sample, the Storage Modulus of the SWB (10%)/HDPE composite exhibited ~12% improvement. Intermediate loadings also exhibited noticeable values, suggesting a consistent filler–matrix interaction across the investigated compositions (Figure 10a, Table 1). In addition to stiffness, the damping behavior, expressed by $\tan\delta$ (defined as the ration of the Loss to the Storage Modulus), was evaluated to assess energy dissipation. The control sample exhibited the highest $\tan\delta$ value, indicating greater molecular mobility to dissipate energy, whereas the composites showed slightly reduced $\tan\delta$ values, with the lowest damping coefficient observed for the SWB (10%)/HDPE sample (Figure 10b,

Table 1). The observed reduction in $\tan\delta$ indicates a shift towards a more elastic response, consistent with the materials' increased stiffness.

(a)



(b)

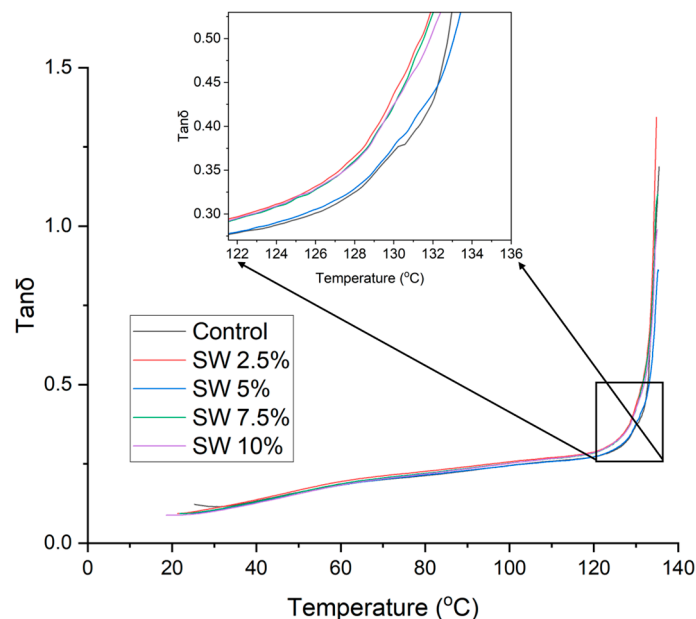


Figure 10. Storage Modulus vs. temperature curves for SWB/HDPE (a) and $\tan\delta$ vs. temperature curves for SWB/HDPE composite samples (b).

The Storage Modulus and $\tan\delta$ for all formulations were recorded at 38 °C, a temperature that lies within the service temperature range of HDPE and provides a representative point for comparing the reinforcing efficiency of the different filler loadings (see Table 1, Figure 11). Additionally, it enabled a direct comparison with our previous publication on HDPE/Luffa Cylindrica-derived biochar composites, where the same analysis methodology was employed [41]. Furthermore, Figure 11 presents box plots for each formulation (at 38 °C), illustrating the data distribution, including error bars, as well as the mean and median values for each concentration.

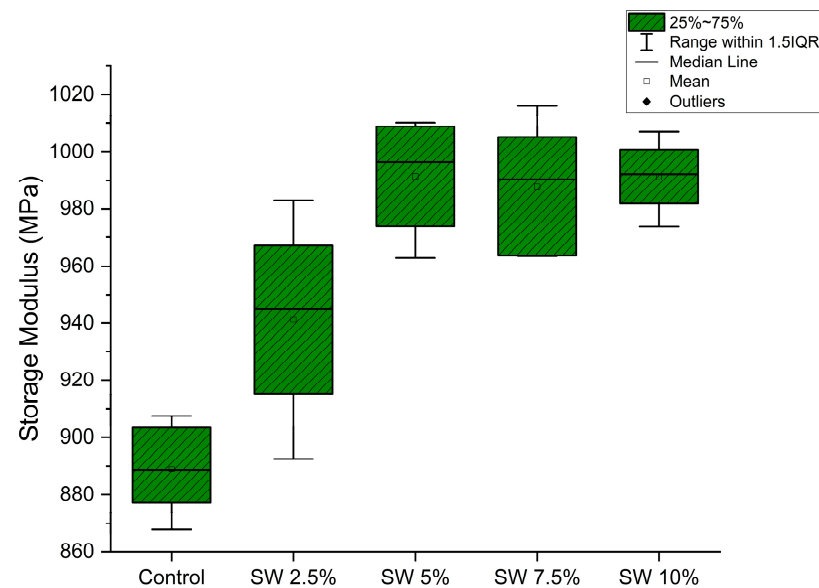


Figure 11. Box plots indicating the Storage Modulus of HDPE/biochar composites recorded at 38 °C.

Despite the morphological defects observed via SEM, the DMA-derived experimental data demonstrated a progressive increase in the Storage Modulus with increasing SWB content, indicating that the rigid carbon structure of the filler was still able to enhance the stiffness of the HDPE matrix (see Figure 11). The observed defects mainly consisted of localized biochar agglomerates and interfacial voids created between the filler inclusions and the polymer matrix, which are indicative of incomplete filler dispersion during processing. Such features may act as stress concentration sites, thus reducing the efficiency of stress transfer and consequently promoting an adverse effect on the long-term durability and mechanical performance of the composites. A similar trend was also observed in our previous study on HDPE/*Luffa Cylindrica*-derived biochar composites, where SEM revealed localized agglomeration, yet thermomechanical performance still improved with increasing filler content [41]. Comparable reinforcement behavior has also been reported in other biochar-reinforced HDPE systems, where the incorporation of carbon-rich fillers increased stiffness and restricted polymer chain mobility despite the presence of filler agglomerates. In one such example, Arrigo et al. [57] reported that biochar particles restrict the mobility of polyethylene macromolecular chains through confinement effects and interactions within the porous biochar structure, indicating the establishment of polymer-filler interactions that hinder chain relaxation. Similarly, Zhang et al. [7] demonstrated that the rigid porous structure of biochar contributed to improved stress transfer and enhanced thermomechanical performance in HDPE composites. Furthermore, the porous morphology of the produced biochar may facilitate partial penetration of the molten HDPE within its cavities during processing, creating a physical interlocking effect at the filler–matrix interface. This mechanism can partially compensate for the detrimental effects of agglomeration by improving stress transfer between the biochar particles and the polymer matrix, thereby contributing to the observed increase in Storage Modulus [35,58]. In the present study, this reinforcing effect may additionally be associated with the keratin-derived carbon structure of SWB, which is rich in aromatic carbon domains and heteroatom-containing functionalities that can further promote rigidity and interfacial interactions within the HDPE matrix.

4. Conclusions

The obtained results demonstrate that SWB can serve as an effective reinforcement filler for HDPE composites, contributing to increased stiffness and reduced viscous dissipa-

tion, as evidenced by the DMA analysis. Although SEM observations revealed localized agglomerates and interfacial voids, the composites still exhibited progressive improvements in the thermomechanical performance with increasing biochar content, indicating that the rigid carbonaceous structure of the filler successfully contributed to matrix reinforcement. FTIR characterization further confirmed the retention of keratin-derived functional groups after pyrolysis at 250 °C, indicating that the resulting biochar possesses chemically active surface functionalities that may contribute to the observed reinforcement effect in the HDPE matrix. This evidence was further supported by qualitative SEM-EDS imaging and elemental mapping. Nevertheless, the present study was focused on the thermomechanical characterization, and therefore, the influence of SWB on other mechanical properties, such as tensile strength, impact resistance, flexural behavior and long-term durability, remains to be investigated. Future studies will focus on optimizing filler dispersion and interfacial adhesion through improved processing strategies, such as twin-screw extrusion, compatibilization approaches or advanced melt-compounding techniques while also incorporating a broader range of mechanical and physicochemical characterization methods. Overall, this study highlights the strong potential of SWB as a sustainable and renewable carbon filler while simultaneously demonstrating an alternative pathway for the circular valorization of biomass waste into high-value polymer composite materials.

Author Contributions: V.T.: Methodology, investigation, validation, formal analysis and writing of original draft. I.P.: Methodology (pyrolysis procedure) and writing—review and editing. P.S.I.: Methodology (SEM-EDS imaging and elemental mapping) and writing—review and editing. T.K.-C.: Conceptualization, supervision and writing—review and editing. All authors have read and agreed to the published version of the manuscript.

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