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Production, Characterization Physical, Chemical, and Structural Analysis of Biochar Fines for Bio-Reinforcement in Composite Materials

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Abstract: Several polymeric compounds are obtained from synthetic organic solids containing petrochemical derivatives. Biochar fines are considered waste and an alternative bio-reinforcement in composite materials, potentially serving as a possible substitute for non-renewable polymers based on petrochemical derivatives. In this context, the present study focuses on analyzing the properties of biochar fines obtained from the pyrolysis of *Eucalyptus* sp. biomass, aiming to support the use of this waste in the fabrication of a composite using biochar as a bio-reinforcement. The biochar was produced through pyrolysis in a muffle furnace at a final temperature of 800 °C, with a heating rate of 5 °C min^{-1} and a residence time of 60 min. The characterization of the obtained fines involved proximate analysis, Brunauer–Emmett–Teller (BET) analysis, scanning electron microscopy (SEM), Fourier-Transform Infrared Spectroscopy (FTIR), thermogravimetric analysis (TGA), and X-ray diffraction (XRD). The results show that the material has a high fixed carbon content, high density, and good thermal resistance, making it stand out for use in composites.

Keywords: HDPE; solid waste; forest biomass; biomaterials; natural reinforcement; pyrolysis

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1. Introduction

The incorporation of industrial waste into plastic composites has proven to be an effective strategy for improving the mechanical and thermal properties of these materials, in addition to reducing their environmental impact. This approach, explored in various studies [1,2], opens up new possibilities for the development of more sustainable materials with diverse applications. In this study, the properties of *Eucalyptus* sp. biochar were investigated as a bio-reinforcement in high-density polyethylene (HDPE) composites, aiming to develop more sustainable materials with enhanced mechanical and thermal properties. Studies conducted by Asyraf et al. (2023) have identified that lignocellulosic biomass fibers can provide significantly higher mechanical performance for several applications. Lignocellulosic biomass composites represent a promising alternative to replace conventional composites, especially in secondary structural applications that require low and medium impact resistance properties [3].

Brazil holds a leading position globally in charcoal production, with 7 million tons produced in 2022 [4]. Biochar is a highly friable material, produced in large quantities through the thermochemical conversion of plant biomass [5]. However, due to its high friability defined as the tendency of a solid material to fragment or disintegrate under friction, impact, or pressure, it generates a significant number of fines, which can account for up to 25% of the total produced [6]. The improper disposal of these residues poses

a major challenge for industries, leading to additional operational costs related to final disposal and the potential for fines due to non-compliance with environmental regulations. Moreover, improper disposal can contaminate soil and water resources, causing harm to the environment and human health.

Charcoal, the main product of pyrolysis, emerges as a promising alternative to partially replace conventional reinforcements such as glass fibers, carbon fibers, natural fibers, and mineral particles in polymer matrices due to its relatively low cost and high availability. Its high thermal stability makes it suitable for melt processing [7]. Additionally, its carbon-based, porous, and hydrophobic structure allows for more significant interaction with polymer matrices [8].

The literature corroborates that the porosity of coal facilitates the interpenetration of polymeric resins, establishing a more intimate interface between the reinforcement and the matrix, which significantly enhances the mechanical strength of the composite [9,10]. However, the penetration of the polymer into the coal's pores depends on the viscosity of the polymeric resin and the pore size, as well as the dimensions of the fine coal particles used as filler [10,11].

Polyethylene (PE), the most widely used polymer in the world, while recognized for its versatility and durability, presents significant challenges, such as its reliance on fossil resources and resistance to environmental degradation. The search for sustainable alternatives that maintain technical performance while reducing environmental impact is essential to mitigate these effects [12,13].

The production of biochar must meet specific chemical and physical properties for its effective application [14]. These characteristics need to be accurately understood and quantified in order to guide production decisions and optimize its performance as a bio-filler in polymer composites. In this context, it is essential to deepen the understanding of biochar's behavior in response to carbonization variables and how its properties correlate, influencing the performance of the resulting composites. To achieve this, the use of relevant analyses, such as thermogravimetry (TGA), X-ray diffraction (XRD), and X-ray diffractograms, is crucial, providing support for greater control over composite production.

In this context, recent studies [1,2] have explored the valorization of these fines as bio-reinforcement in the development of polymer composites, thereby expanding their industrial applications. Accordingly, the present study aimed to evaluate the properties of biochar derived from commercial *Eucalyptus* spp. wood, with an emphasis on the potential valorization of charcoal residues and the development of new materials.

2. Materials and Methods

2.1. Production of Biochar Fines

For charcoal production, wood disk residues of *Eucalyptus* sp. from different longitudinal positions (BRA) along the trunk (base, 25%, 50%, 75%, and top) were used approximately 1–2 cm in length. The samples underwent a two-stage grinding process: First, the material was coarsely ground in a Tigre A4 hammer mill (Moinhos Tigre, São Paulo, Brazil). Subsequently, to obtain a fine and homogeneous particle size, the material underwent fine grinding in a Willy TE-650 knife mill (Tecnal, Piracicaba, Brazil) to achieve a particle size of approximately 50 mesh. The samples were then oven-dried at 103 ± 2 °C for 20 h prior to pyrolysis. The reduction in biomass particle size facilitated heat transfer during carbonization, resulting in a significant increase in the yield of biochar fines.

Pyrolysis was performed in a fixed-bed reactor, a straightforward, reliable, and widely established technology for processing solid feedstock particles with uniform size [15,16]. In each experiment, 250 g of feedstock was placed in a sealed metal reactor, 8 cm in diameter and 15 cm in length, placed inside a muffle furnace. The experiments were carried out in a

low-oxygen atmosphere, without gas input, with an initial temperature of 30 °C and a final furnace temperature of 800 °C, at a heating rate of 5 °C/min and a residence time at a final temperature of 120 min. A gas condenser cooled with water and ice was coupled to the muffle furnace. These procedures followed the descriptions provided by Delatorre et al. [5].

The dried material was then pyrolyzed in a muffle furnace, reaching a final temperature of 800 °C, with a heating rate of 5 °C min^{-1} and a residence time at the final temperature of 60 min. These procedures followed the descriptions provided by Delatorre et al.

The preparation sequence of the residue, detailed in Figure 1, followed the protocol proposed by Delatorre et al. [5]. The raw residue was subjected to a size reduction process using a hammer mill followed by a knife mill. The resulting particulate material was then subjected to pyrolysis, ultimately yielding biochar fines.



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

Figure 1. Sequential stages of *Eucalyptus* sp. biomass fragmentation up to the production of fines. (**a**) Initial biomass collected, exhibiting heterogeneous particle size distribution; (**b**) biomass after initial fragmentation in the hammer mill, showing a more uniform particle size with smaller particles; (**c**) end biomass after grinding in the knife mill, achieving a particle size of 0.053 mm; (**d**) fines obtained after the pyrolysis process.

To ensure a homogeneous distribution of the carbon fines in the polymer matrix, pilot tests were performed to identify the ideal particle size for the matrix used in this research. To this end, the particles underwent a classification process, selecting those that passed through the 40-mesh sieve (0.425 mm), and were retained in the 60-mesh sieve (0.250 mm). The choice of particles with this size aimed to minimize the formation of agglomerates and ensure a uniform distribution of the reinforcement in the matrix, promoting better interaction between the phases and, consequently, improving the properties of the composite material.

2.2. Characterization of Biochar Fines

The determination of the gravimetric yield in biochar (*GY*) was carried out based on the initial mass of the wood sawdust and the mass of the biochar, using Equation (1).

$$GY(\%) = \frac{Mcv}{Mm} * 100 \quad (1)$$

where *Mcv* = dry mass of biochar; *Mm* = dry mass of wood.

The apparent density was determined following the guidelines of the NBR 11941 standard [17] by immersing biochar pieces in water. Subsequently, the samples were placed in an oven at 103 ± 2 °C and, after drying, removed from the oven and weighed using an analytical balance. The apparent density was calculated with five repetitions based on the ratio of the dry mass of the sample. This was estimated by dividing the mass of the biochar sample by its volume, as per Equation (2).

$$AD = \frac{M}{V} \quad (2)$$

where *M* = co-pyrolytic coal mass; *V* = volume.

The determination of the true density of the biochar sample was conducted in accordance with the NBR 9165 standard [18]. A pycnometer was used to analyze the

powdered sample, which had a particle size of 60 mesh (0.250 mm) and was previously dried. Thus, the mass of the system (pycnometer + water + coal) was determined using the equation below:

$$DV = \frac{M_s}{(M_s - (M_w - M_{w'}))} \quad (3)$$

where DV = true density (kg/m^3); Ms = mass of the dry material (kg); Mw = mass of the pycnometer + water + material (kg); Mw' = mass of the pycnometer with water (kg).

For the determination of immediate analysis, the fixed carbon content, volatile materials, and ash (on a dry basis) were determined following the procedures outlined in ASTM D1762-84 [19]. A microprocessor-controlled muffle furnace model Q318M24 (Quimis, Diadema, Brazil) was used.

To understand the mass variation in the biochar, thermogravimetric analysis (TGA) was conducted using LABSYS EVO SETARAM (LabWrench, Midland, ON, Canada) equipment with a heating rate of 10 °C min^{-1} and a particle size of the biochar of 60 mesh (0.250 mm). Thermograms were recorded from ambient temperature (25 °C) in a nitrogen gas atmosphere with a flow rate of 1.8 bar up to 600 °C.

The observation of the morphology of coal fines obtained from pyrolysis was conducted using scanning electron microscopy (SEM). Micrographs were obtained with a JEOL JSM 840A equipment (LabWrench, Midland, ON, Canada), operating with an electron beam at an acceleration voltage of 10 kV and a current of 6×10^{-11} A.

Brunauer–Emmett–Teller (BET) analysis was also conducted to characterize and apply the surface area and porosity properties of the fine biochar. For this analysis, the samples underwent a thermal pre-treatment in a muffle furnace at 200 °C for 2 h. The analysis was performed using the JWGB MESO 112 equipment (JWGB Sci & Tech Co., Ltd, Beijing, China, with nitrogen gas for adsorption, at a temperature of 200 °C.

To characterize the crystalline structure of the biochar, X-ray diffraction (XRD) analysis was performed. X-ray diffractograms were recorded from samples with a particle size of 60 mesh using a Rigaku MiniFlex600-C diffractometer (Rigaku, Tokyo, Japan). The diffraction angle 2θ ranged from 10° to 80° in increments of 0.02°, with a scan rate of 0.5 °C min^{-1}.

3. Results

The gravimetric yields of the biochar obtained from the carbonization of the wood are presented in Table 1. The average yield was 28.27%. The average value observed in this study was higher than that reported by Delatorre et al. [20], who used a carbonization system similar to the one in this work and achieved a gravimetric yield of biochar in the range of 24.82% at a temperature of 800 °C in their study on biochar fines obtained from the pyrolysis of *Eucalyptus* sp. wood. Thus, the biochar yields obtained in this study are consistent with the values reported in the literature, where a minimum yield of 30% is commonly observed [21].

Table 1. Average values of gravimetric yield in charcoal (*GCY*) from *Eucalyptus* sp. wood.

Pyrolysis	*GCV* (%)
1	29.20
2	27.35
Mean	28.27
CVe	4.63

It is noteworthy that the achievement of higher biochar yields is a relevant factor for the efficient production of carbonaceous composites, as it ensures a larger volume

of material to compose the matrix. Therefore, the chemical and physical properties of the biochar play a crucial role in the synergy between the components of the composites, significantly influencing the final properties of the composite material [21,22].

The gravimetric yield, although a relevant parameter, does not prove to be decisive for the applicability of biochar in composites. The optimization of the carbonization process, aimed at obtaining high-quality biochar, can compensate for a lower gravimetric yield, as the quality of the biochar is directly related to the properties of the final composite material.

Table 2 presents the means and descriptive analysis of the apparent and true densities of the produced biochar fines. The average values found were 306.4 and 1193.9 kg/m^3 for the apparent and true densities, respectively. The apparent density of biochar has a significant influence on its mechanical properties. Materials with higher density tend to exhibit greater mechanical strength. This correlation can be attributed to the structural reorganization of the biomass during the carbonization process, resulting in a more graphitic structure, which imparts greater stiffness and strength to the material [23].

Table 2. Average values of apparent and true density of biochar fines.

Repetitions	Apparent Density (kg/m^3)	True Density (kg/m^3)
1	324	1278.3
2	300	998.1
3	296	1323.5
4	308	1194.8
5	304	1174.4
Mean	306.4	1193.82
Standard deviation	10.807	125.138
Coefficient of variation	3.527	10.482

The density results indicate that the produced biochar exhibits high potential as a bio-reinforcement in polymer matrices. The higher density of the reinforcement suggests potential for increased stiffness and strength in the composite material, thus expanding its applications in areas requiring high structural performance, as evidenced by Delatorre et al. [11].

Apparent relative density values for biochar from Eucalyptus clones and species are frequently found in the literature, ranging from 266 kg/m^3 to 351 kg/m^3 [24,25], validating the methodology used and reinforcing the reliability of the results.

The results obtained regarding the influence of volatile matter content, fixed carbon, and biochar ash on the production of composites are presented in Table 3. The fixed carbon content is one of the primary indicators of the quality of biomass charcoal [26]. However, a comprehensive assessment of biochar properties requires consideration of additional attributes, such as porosity, to optimize its application [14].

Results similar to those found in this study were reported by Dufourny [27], who identified fixed carbon contents ranging from 80.5% to 98.1% in the carbonization of Eucalyptus globulus wood at 800 °C.

The presence of carbonaceous material is crucial for the use of charcoal as a reinforcement in composites. Biochar possesses a stable internal structure, similar to that of graphite, and a reactive peripheral structure due to the presence of various chemical groups (carboxylic, hydroxyl, ester, phenolic, carbonyl, among others) that can bond with organic substances, water, and chemical elements [28]. The graphitic structure present in biochar, formed during pyrolysis, plays a crucial role in the interaction with the polymer matrix. The high surface area and the presence of reactive functional groups on the surface of

the graphite facilitate the formation of chemical and physical bonds with the polymers, potentially resulting in composites with enhanced mechanical and thermal properties.

Table 3. Immediate analysis of produced biochar. FC = fixed carbon (%); VM = volatile matter (%); AS = ash (%).

Repetitions	Volatile Matter Content (%)	Ash Content (%)	Fixed Carbon Content (%)
1	10.66	0.25	89.09
2	10.48	0.05	89.47
3	9.51	0.11	90.38
4	12.93	0.15	86.92
5	10.87	0.61	88.52
Mean	10.89	0.23	88.87
Standard deviation	1.25	0.22	1.28
Coefficient of variation	11.49	95.08	1.44

Regarding ash content, the use of charcoal with high levels of this component is not recommended, as it indicates an elevated presence of minerals in the fuel, which may compromise the quality of the produced material by causing segregation and reducing its properties [26]. Although the ash content is low, a high coefficient of variation was observed, as also noted by Trugilho et al. [29], who highlight the significant variability of this characteristic, which may be related to various environmental and production factors.

Moreover, materials with higher carbon content generally exhibit greater mechanical strength [30]. The reduction in the ash content of the produced biochar indicates an increase in carbon purity, which can enhance the interaction with the polymer matrix and improve the mechanical properties of the composites.

According to Gomes [31], the fixed carbon content has a negative correlation with the volatile matter content and also with the yield of biochar, as obtaining biochar with a high carbon content requires higher pyrolysis temperatures, which results in a lower yield of biochar.

Li et al. [32] investigated the effect of carbon content in coal gangue and polyethylene composites. The results demonstrated a direct correlation between the increase in carbon content and the enhancement of the density, hardness, flexural strength, and elastic modulus of the composites. The microstructural analysis revealed that the carbon content significantly influenced the mechanical properties of the composite.

These findings underscore the potential of coal, with high fixed carbon content, as a reinforcing agent in polymer matrices, providing a new perspective for the development of composite materials with improved mechanical properties.

The microscopy images presented in Figure 2 show the results obtained from the scanning electron microscopy analysis of the produced biochar fines. Scanning electron microscopy (SEM) revealed a porous and heterogeneous microstructure in the particles, as observed in images obtained at magnifications of 1000× and 2000×.

Similar results were reported by Martins et al. [33], who observed a considerable number of pores, particularly mesopores and macropores, in the micrographs of biochar obtained from the biomass of *Eucalyptus* sp. sawdust. This increase in porosity was particularly evident when the pyrolysis temperature was raised from 400 to 500 °C.

The porous microstructure of biochar, characterized by a complex network of interconnected pores and voids, serves as a mold for the infiltration of the polymer matrix during composite processing [34]. Polymers with good fluidity enable deep penetration into the pores, establishing a significant interfacial interface between the phases. This enhanced

interaction contributes to improved adhesion between the matrix and the reinforcement, optimizing the mechanical properties of the composite.

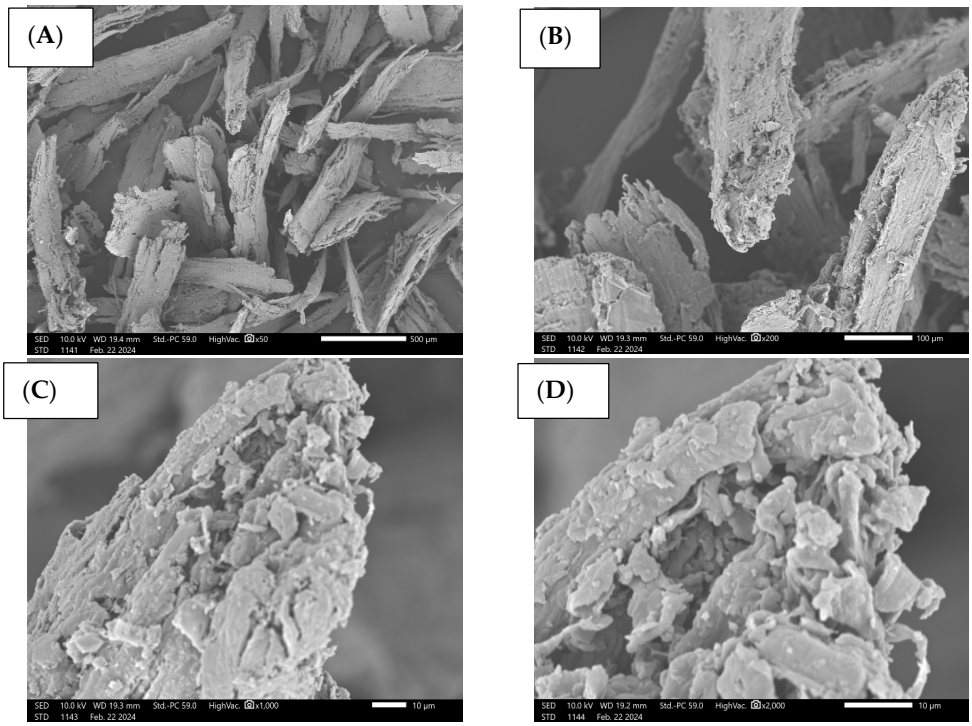


Figure 2. Micrographs of the produced coal fines at magnifications of (**A**) 50×; (**B**) 200×; (**C**) 1000×; and (**D**) 2000×.

The porous microstructure of biochar, characterized by a pore distribution demonstrated by scanning electron microscopy (SEM), can be thoroughly analyzed using Brunauer–Emmett–Teller (BET) analysis. BET analysis reveals the average pore diameter (nm), surface area ($m^2 \cdot g^{-1}$), and total pore volume ($m^3 \cdot g^{-1}$) of the produced charcoal (Table 4).

Table 4. Average values of the mean pore diameter, specific surface area (SSA), and total pore volume of the produced biochar.

Average Pore Diameter (nm)	Surface Area ($m^2 \cdot g^{-1}$)	Total Pore Volume ($m^3 \cdot g^{-1}$)
2.374 ± (0.683)	293.136 ± (36.68)	0.174 ± (0.0254)

The surface area analysis of the biochar yielded a value of 293.136 m^2/g (Table 4). This high surface area is largely attributed to the production conditions, especially the elevated temperatures used during the carbonization process.

Dias Junior et al. [14] have shown that carbonization parameters, including the final temperature, play a pivotal role in the formation and development of biochar's porous structure, directly impacting its surface area. The pore characterization of the biochar fines indicated an average pore diameter of 2.374 nm. According to the International Union of Pure and Applied Chemistry (IUPAC) [35], pores are classified as micropores (diameter < 2 nm), mesopores (diameter between 2 and 50 nm), and macropores (diameter > 50 nm).

Considering the IUPAC classification [35] for porosity, the produced biochar can be categorized as mesoporous. The predominance of mesopores in this material is consistent with the high specific surface area obtained, as the presence of intermediate-sized pores,

such as mesopores, provides a greater contact area for interactions, contributing to the increase in specific surface area.

The combined analysis of SEM and BET allows us to conclude that the material exhibits a well-developed mesoporous structure. The SEM micrographs clearly demonstrate the presence of intermediate-sized pores, while the BET analysis quantifies the specific surface area associated with these pores.

The porous structure of coal serves as a support for the anchoring of polymer chains, promoting a closer interaction between the phases and, consequently, enhanced mechanical properties. However, the efficiency of this mechanism depends on the compatibility between the pore size of the coal and the viscosity of the polymer resin [10,11,34].

The highly developed porous structure of the studied biochar makes it a promising candidate for application in composites. The large number of pores and the high surface area facilitate excellent interaction with the polymer matrix, optimizing the mechanical properties of the composite material.

X-ray diffraction analysis was conducted to identify the crystalline phases present in the biochar fines and to understand how these phases may contribute to the interaction with the polymer matrix. The XRD pattern exhibits three main peaks: the first between 22° and 23° (2θ), the second between 25° and 26°, and the third between 43° and 44° (2θ) (Figure 3).

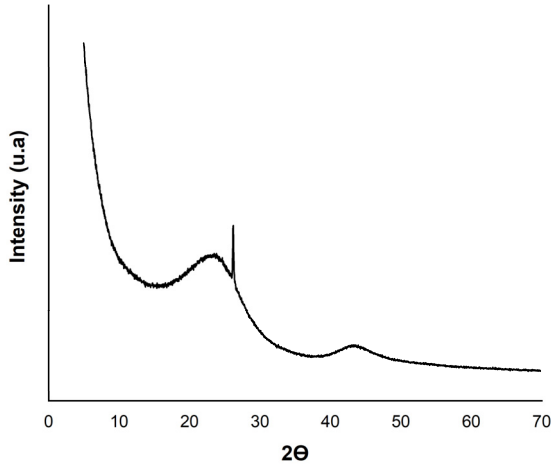


Figure 3. X-ray diffraction patterns of produced biochar.

The detection of peaks around 25° (2θ) suggests the occurrence of crystallization processes in materials that, while exhibiting an increase in structural order, have not yet attained a fully developed crystalline organization [36].

The X-ray diffraction (XRD) analysis, as highlighted by Fernandes et al. [37], reveals that pyrolysis at high temperatures promotes the formation of a predominantly amorphous carbon structure in biochars. The progressive loss of crystallinity in cellulose, evidenced by the broadening of the peak at 23.5° (2θ), along with the emergence of a broad peak between 22° and 26° attributed to carbon (002), supports this assertion. The increase in the intensity of the peak at 43.7° (2θ) starting from 750 °C suggests the formation of more complex and disordered carbonaceous structures.

Li et al. [32] highlighted the fundamental role of carbon content in modulating the properties of polyethylene composites. Variations in carbon content significantly influence the crystallinity of the polymer, with direct implications for the mechanical properties of the composite material. These findings underscore the potential of carbon as an effective reinforcement for the production of composite materials with optimized properties, opening up new avenues for the development of advanced materials across various fields.

4. Conclusions

The results of this study indicate that charcoal produced from *Eucalyptus* sp. biomass at 800 °C exhibits promising characteristics for application as a bio-reinforcement, enabling greater interaction and compatibility with polymer matrices.

- The high fixed carbon content present in the biochar fines significantly contributes to the increased strength and thermal stability of the composites.
- The thermal resistance of the material is a crucial factor that broadens the applicability of composites, making them suitable for high-temperature environments.
- The biodegradable nature of biochar fines contributes to reducing the environmental impact of composites that utilize synthetic reinforcements, as it facilitates their natural decomposition and minimizes waste generation.
- The experimental data support the hypothesis that coal fines can be utilized as bio-reinforcement, contributing to the development of composite materials with enhanced mechanical properties and reduced environmental impact.
- This research opens new perspectives for the utilization of an industrial byproduct, promoting circular economy and sustainability. However, for the full exploration of the potential of biochar as a bio-reinforcement, more in-depth studies are essential regarding the optimization of the pyrolysis process and the characterization of the interfaces between the biochar and the polymeric matrix.

However, to fully exploit the potential of charcoal as a bio-reinforcement, further in-depth studies on optimizing the pyrolysis process and characterizing the interfaces between charcoal and various polymer matrices are essential.

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Conflicts of Interest: The authors declare no conflicts of interest.

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