Research on Engineering Structures & Materials
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Research Group

Research Article

Characterizing polylactic acid/basalt fiber composite: Synthesis, characterization, and mechanical property evaluation

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Article Info

Article History:

Received 12 May 2024
Accepted 31 Oct 2024

Keywords:

Polymers;
Basalt fiber;
Polylactic acid;
Maleic anhydride;
Injection molding

Abstract

This investigation focused on enhancing the mechanical properties of polylactic acid (PLA) through the incorporation of basalt fibers and a maleic anhydride coupling agent. Basalt fibers, renowned for their high tensile strength and modulus, were selected as a potential reinforcement to augment PLA's mechanical performance. To optimize interfacial bonding between the hydrophilic PLA matrix and hydrophobic basalt fibers, maleic anhydride was employed as a coupling agent. The results indicate that the inclusion of 3 wt.% maleic anhydride and 10-20 wt.% basalt fibers significantly improved the composite's tensile strength by 40% and flexural strength by 51%. These findings underscore the potential of basalt fiber-reinforced PLA as a high-performance, sustainable material for diverse applications.

1. Introduction

In recent years, there has been a notable surge in the quest for environmentally friendly and sustainable alternatives to conventional petroleum-based composites. Polylactic acid (PLA), a biodegradable and renewable thermoplastic polymer, has garnered significant attention due to its advantageous mechanical properties and minimal carbon footprint. Researchers are actively exploring ways to enhance PLA's mechanical performance by incorporating various types of fibers.

The studies on PLA/basalt fiber highlights the potential of basalt fiber reinforced composites as sustainable alternatives to synthetic fiber-based composites. While natural fibers offer environmental benefits, their mechanical properties often limit their applications. By combining basalt fibers with PLA, researchers have developed composites with promising tensile and flexural strengths. This work explores various fabrication techniques and mechanical characterization studies on basalt/PLA composites, underscoring their potential as eco-friendly replacements for traditional composites [1]. Previous research also highlighted the challenge of poor interfacial adhesion between basalt fiber and epoxy resin, limiting the widespread application of this promising green material. In a recent study, Wu et al. [2] addressed this issue by developing a novel surface modification technique involving citric acid modified β-cyclodextrin and polyethyleneimine. Their approach resulted in a substantial 76.9% increase in interfacial adhesion strength compared to unmodified fibers. The authors attributed this enhancement to the unique structure of β-cyclodextrin, which, in combination with PEI, created a multifunctional interphase that improved stress transfer, energy dissipation, and crack deflection. This research contributes significantly to the development of high-performance, environmentally friendly basalt fiber composites and offers a potential pathway for industrial-scale production due to the simplicity and sustainability of the modification process. Xue et al. [3] developed ternary PLA composites

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DOI: http://dx.doi.org/10.17515/resm2024.279me0512rs

reinforced with short basalt fiber (SBF), incorporating PBAT and nano-silica. These composites exhibited substantial enhancements in tensile modulus (103%) and crystallinity (185.5%) compared to pure PLA, without compromising structural integrity. The inclusion of PBAT, nano-silica, and SBF also improved thermal stability, expanding the potential of PLA for high-temperature applications.

The recyclability of PLA and PLA-based natural fiber-reinforced composites (NFRCs) through conical twin screw extrusion was studied by Finnerty et al. [4]. PLA exhibited discoloration and an increased glass transition temperature by 15°C after six recycling cycles. While the impact strength of both BF and HNT-reinforced NFRCs remained relatively stable, tensile properties showed variations. BF-reinforced composites experienced a significant 30% decrease in tensile strength after three recycling cycles due to fiber length reduction and poor interfacial adhesion. In contrast, HNT-reinforced composites maintained tensile strength within 50-60MPa for up to three cycles but declined by 12% thereafter. The study highlights the potential of recycling PLA-based composites while identifying challenges related to fiber length, interfacial adhesion, and PLA degradation. Further research is needed to optimize recycling parameters and improve composite performance. Cao et al. [5] investigated the enhancement of PLA mechanical properties through carbon fiber (CF) reinforcement. The addition of CF significantly improved the thermal stability and crystallinity of PLA, as evidenced by TGA and DSC analysis. The melt flow rate decreased from 27.6 g/10 min to 12.1 g/10 min with CF incorporation. Notably, tensile strength increased by 42.49% to 91.58 MPa in PLA/CF composites compared to pure PLA. XRD analysis indicated a refined crystalline structure in PLA/CF. SEM observations revealed a transition from brittle to ductile fracture behavior with CF reinforcement. These findings demonstrate the potential of CF as an effective reinforcement for enhancing the overall performance of FFF-produced PLA materials.

Polylactic acid, a biodegradable polymer, has gained significant attention as a sustainable alternative to petroleum-based composites. To enhance PLA's mechanical performance, researchers have explored the incorporation of reinforcing fibers, including basalt fibers. Previous studies have primarily focused on improving interfacial adhesion, exploring diverse processing techniques, and incorporating additional components to optimize composite properties. While advancements have been made in terms of mechanical properties, thermal stability, and corrosion resistance, challenges related to interfacial adhesion, recyclability, and the impact of processing conditions on composite properties persist [6], [7].

This study aims to expand upon the existing body of research by comprehensively investigating the synthesis, properties, and mechanical behavior of PLA/basalt fiber composites. Previous studies have explored the potential of basalt fibers as reinforcement for PLA, but this research delves deeper into understanding the factors influencing the overall performance of these composites. By building upon successful processing techniques used for other polymer-based composites, we seek to optimize the integration of basalt fibers into the PLA matrix and assess the resulting mechanical properties. This research article focuses on the use of maleic anhydride (MAH) as a compatibilizer to improve the interfacial adhesion between basalt fibers and PLA in composites. While compatibilizer-free basalt/PLA composites may have been considered, the study's primary goal is to investigate the effectiveness of MAH in enhancing the mechanical properties and performance of these composites. Without a compatibilizer, the incompatibility between basalt fibers and PLA could have led to poor interfacial bonding, phase separation, and reduced mechanical strength. By using MAH, the study aims to address these potential issues and optimize the properties of the basalt/PLA composites.

2. Materials

Compostable bio-based PLA, known as luminy LX175, is purchased from Banka Bioloo Limited, located in Hyderabad. Basalt chopped fiber is procured from Nickunj Eximp Entp P Ltd, Mumbai. The nominal diameter and length of the basalt fiber are determined to be 13 micrometers and 6 millimeters, respectively. The properties of basalt fiber and PLA are presented in Table 1.

Table 1. Properties of Basalt fibre and PLA

Properties	Basalt fiber	Polylactic acid
Density, g/cm^3	2.65	1.24
Nominal length, mm	6	-
Nominal Diameter, µm	13	-
Tensile Strength, MPa	2365	45
Elasticity modulus, GPa	93.4	93.4
% Elongation at Break	2.9	5
Melting Point, ^{0}C	1500	150

3. Methods

3.1 Sample Preparation

Basalt fibers, constituting 10% and 20% by weight, are combined with a 3% by weight maleic anhydride coupling agent and PLA pellets. This mixture undergoes processing in a twin-screw extruder equipped with a 40 mm diameter, co-rotating screw operating at 80 revolutions per minute as shown in Figure 1. Extrusion temperatures range from 140°C to 170°C. The interlocked screws generate shear pressures that facilitate thorough mixing and dispersion of the fibers within the PLA matrix. The coupling agent improves the interfacial adhesion between the PLA matrix and basalt fibers.

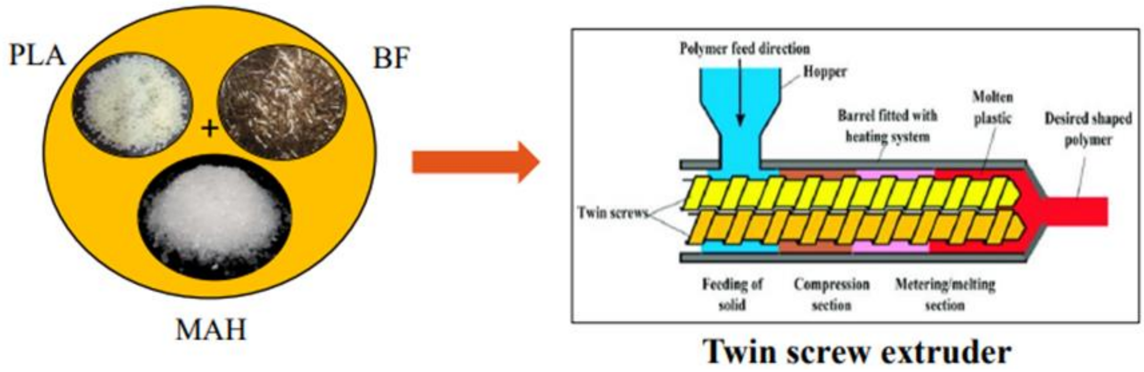


Fig. 1. Composite sample preparation

The resulting composite strands, containing 10% and 20% basalt fibers, are transformed into pellets through palletization and subsequently annealed at 100°C for one hour. These pellets are then processed using an NP100/200 injection molding machine featuring a 40 mm screw diameter and an L/D ratio of 19.6. The machine's heated barrel melts the pellets, which are then molded into desired shapes and cooled to solidify. Table 2 presents the primary injection molding parameters.

Table 2. Injection Molding Parameters

Parameters	Value
Clamping tonnage, ton	60
Injection speed, MPa	50
Injection pressure, bar	80
Refilling speed, rpm	50
Back pressure, bar	4
Shot weight, grams	50
Temperature profile along the barrel (from hopper to nozzle), ^{0}C	140-145-150-155

3.2 Mechanical Testing

Flexural properties, including strength and modulus, were evaluated according to ASTM D790 via a three-point bending test. A crosshead speed of 2 mm/min was maintained during testing. The flexural modulus was calculated from the resulting load-displacement data. The experimental set up is as shown in Figure 2.

Fig. 2. Flexural testing

Tensile properties were determined according to ASTM D638 using a universal testing machine. A constant crosshead speed of 1.5 mm/min was applied, and load-displacement data was collected. Elastic modulus was calculated from the initial linear region of the resulting stress-strain curve. The experimental set up is as shown in Figure 3.

Fig. 3. Tensile testing

Hardness was assessed using the Yuzuki Rubber Hardness Test. A steel ball or diamond cone indented the material's surface, and the indentation depth was converted to a Rockwell hardness number. The experimental set up is as shown in Figure 4.

Fig. 4. Hardness testing test rig

3.3 Scanning Electron Microscopy

SEM was used to evaluate the basalt fiber-reinforced PLA composites' fracture surfaces. The composites were cryogenically fractured, then coated with a conductive substance to prepare the samples. The microstructure of the entire composite as well as the fiber-matrix adhesion and fiber dispersion were revealed by SEM imaging.

4. Results and Discussions

4.1 Mechanical Properties

The influence of basalt fiber reinforcement on the mechanical properties of PLA composites was investigated. Maleic anhydride (MAH) was incorporated as a coupling agent to enhance interfacial adhesion between the basalt fibers and the PLA matrix. The mechanical properties of the composites containing 10 and 20 weight percent basalt fibers were assessed through tensile, flexural, and hardness tests. Table 3 presents the tensile strength (σ_1), Young's modulus (E_1), flexural strength (σ_2), flexural modulus (E_2), and hardness values obtained from these tests.

Table 3: Mechanical Properties of Basalt fiber/Polylactic acid composite

	Tensile			Flexural		Hardness
Materials	σ_1 [MPa]	E_1 [MPa]	ε [%]	σ_2 [MPa]	E_2 [MPa]	HRC Value
PLA	46.369	1825	5.169	84.987	3576	70
PLA/10wt.% basalt fiber	50.224	1978	4.89	89.768	4923	72
PLA/20wt.% basalt fiber	66.025	2905	3.224	127.685	5206	74

The tensile strength, flexural strength, and hardness of PLA composites increased with increasing basalt fiber content. Figure 5 illustrates the comparison of these properties for PLA and its composites. The addition of 20 wt.% basalt fibers resulted in enhancements of 24%, 29%, and 2.7% for tensile strength, flexural strength, and hardness, respectively. These findings underscore the positive influence of basalt fiber reinforcement on the mechanical properties of the PLA matrix. Hardness, a key indicator of a material's resistance to indentation, was evaluated for the basalt fiber reinforced PLA composite using a Yuzuki Rubber Hardness Tester with the Shore D scale. This measurement provides valuable insights into the composite's overall mechanical properties.

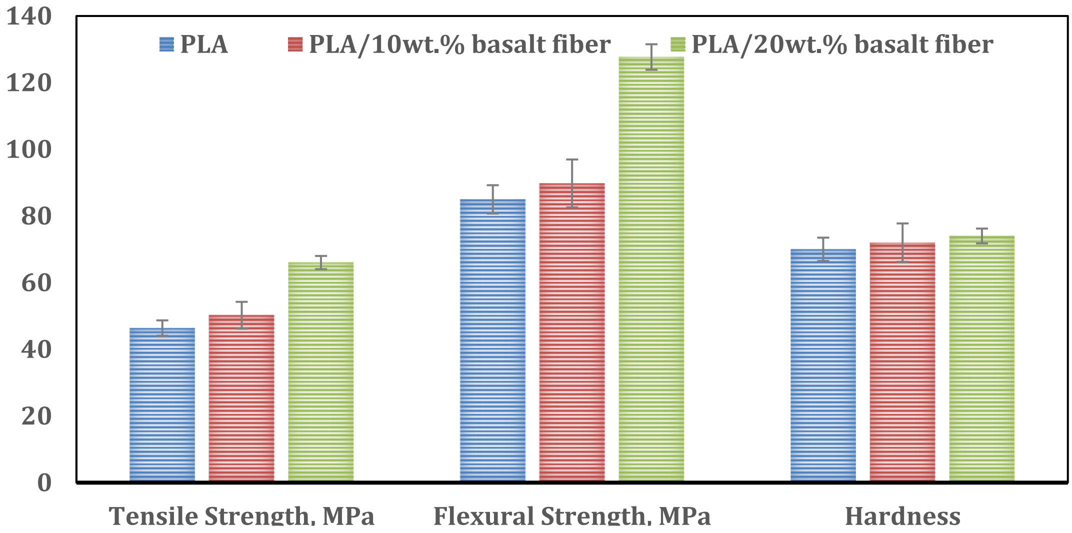
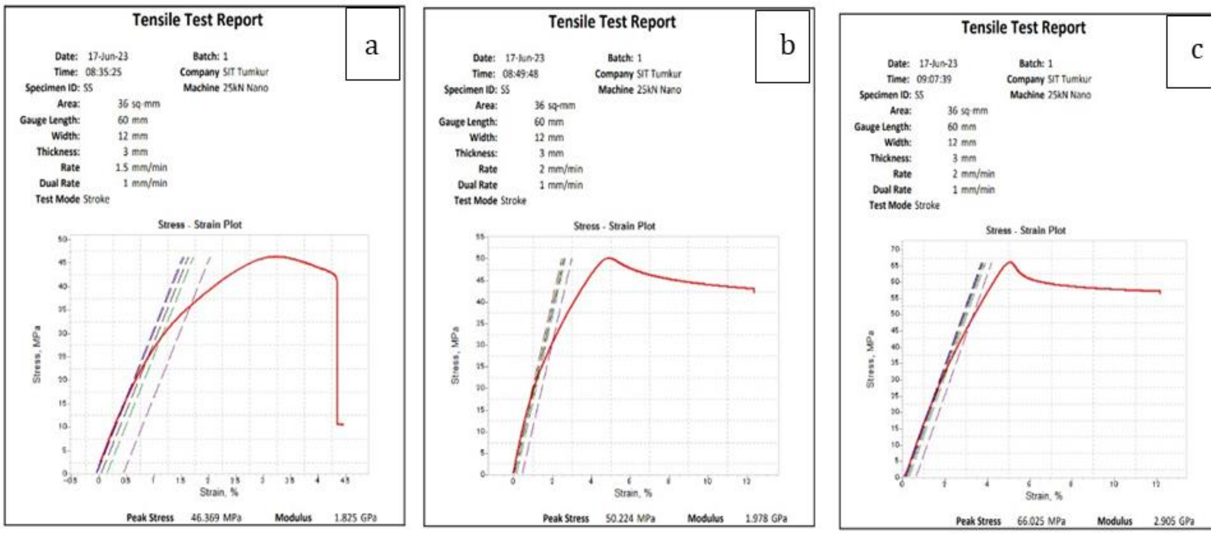


Fig. 5. Comparison of tensile strength, flexural strength and hardness of PLA composites

The stress–strain curves of the PLA/ basalt fiber composites with different basalt fiber content are shown in Figure 6. For all samples, the curves were linear at low strain, followed by plastic deformation in the region of 1.5% strain. At higher strains, the composites yielded up to a breaking strain of 5% for the PLA. The curves clearly show that the tensile strength of the composites increased markedly with increasing basalt fiber. The elongation at break, indicating a material's ductility, decreased with increasing basalt fiber content in the PLA composites. While neat PLA exhibited an elongation at break of 5.169%, this value reduced to 4.89% and 3.224% for composites containing 10 wt.% and 20 wt.% basalt fibers, respectively. This trend signifies a trade-off between enhanced strength and reduced ductility upon basalt fiber incorporation.



Tensile Test Report

Date: 17-Jun-23
Time: 08:35:25
Specimen ID: SS
Area: 36 sq-mm
Gauge Length: 60 mm
Width: 12 mm
Thickness: 3 mm
Rate 1.5 mm/min
Dual Rate 1 mm/min
Test Mode Stroke
Batch: 1
Company SIT Tumkur
Machine 25kN Nano

Peak Stress 46.369 MPa Modulus 1.825 GPa

Tensile Test Report

Date: 17-Jun-23
Time: 08:49:48
Specimen ID: SS
Area: 36 sq-mm
Gauge Length: 60 mm
Width: 12 mm
Thickness: 3 mm
Rate 2 mm/min
Dual Rate 1 mm/min
Test Mode Stroke
Batch: 1
Company SIT Tumkur
Machine 25kN Nano

Peak Stress 50.224 MPa Modulus 1.978 GPa

Tensile Test Report

Date: 17-Jun-23
Time: 09:07:39
Specimen ID: SS
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Gauge Length: 60 mm
Width: 12 mm
Thickness: 3 mm
Rate 2 mm/min
Dual Rate 1 mm/min
Test Mode Stroke
Batch: 1
Company SIT Tumkur
Machine 25kN Nano

Peak Stress 66.025 MPa Modulus 2.905 GPa

Fig. 6. Stress - Strain curve for PLA (a) PLA (b) PLA/Basalt fiber composite 10 wt. % (c) PLA/Basalt fiber composite 20 wt. %

Figure 7 illustrates the tensile and flexural moduli of the PLA composites. The addition of basalt fibers significantly enhanced the Young's modulus, indicative of increased stiffness and resistance to deformation under tensile load. Pure PLA exhibited Young’s modulus of 1825 MPa. This value increased to 1978 MPa and 2905 MPa for composites containing 10 wt.% and 20 wt.% basalt fibers, respectively, representing improvements of 8.3% and 59.5%.

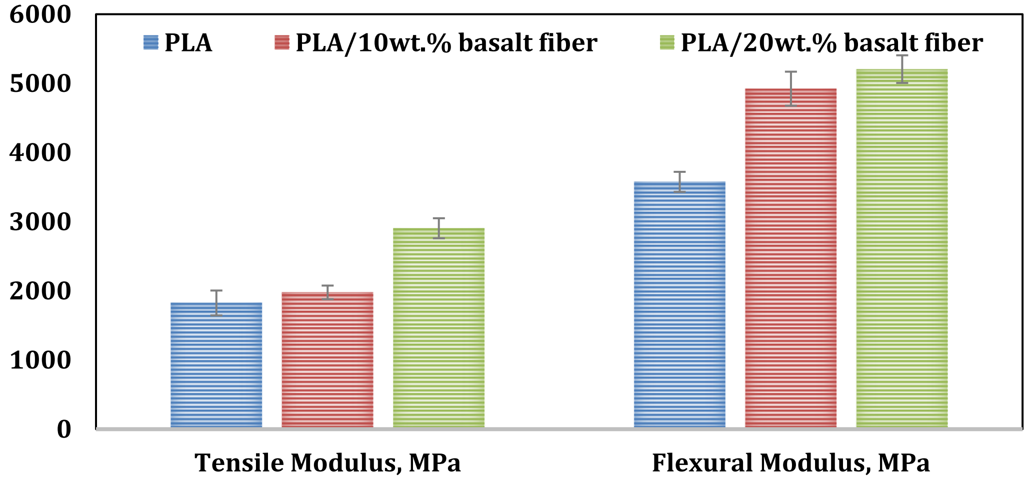

Fig. 7. Comparison of tensile and flexural modulus of PLA composites

Flexural modulus, a measure of a material's stiffness under bending, also increased with basalt fiber addition. While the PLA/10 wt.% composite showed a notable improvement in flexural modulus compared to both pure PLA and the PLA/20 wt.% composite, the latter also exhibited an increase over the neat PLA. The addition of MAH as a compatibilizer to 20 wt.% basalt fiber reinforced PLA composites resulted in improved flexural strength to 127MPa compared to 105MPa as reported by Aslan et al. (2018) [8]. The enhanced tensile strength of the PLA/basalt fiber composites can be attributed to the basalt fiber's stiffness and the presence of immobilized or partially immobilized polymer phases. The following factors contribute to the increase in mechanical properties,

- Physical Interactions: Basalt fibres reinforce PLA by transferring load, with their orientation, length, and volume fraction impacting the composite's properties [1] [9] [10].
- Chemical Interactions: Strong interfacial adhesion between basalt fibres and PLA, enhanced by coupling agents or surface treatments, is crucial for optimal composite performance. Polymer matrix compatibility also affects interaction strength [11], [12], [13], [14]

4.2 Morphological Analysis

The SEM investigation employed a high-resolution scanning electron microscope to analyze the samples. To improve image quality and mitigate charging effects, a thin layer of conductive material was sputter-coated onto the samples. SEM micrographs of the basalt fiber reinforced PLA composites were captured at 100μm and 200μm magnifications. Uniform dispersion of basalt fibers within the PLA matrix was observed, along with strong adhesion at the fiber-matrix interface, indicating the effectiveness of the maleic anhydride coupling agent. No fiber pull-out or surface irregularities were evident on the basalt fibers for both compositions (Figure 8 and 9). At 200μm magnification, individual basalt fibers were more clearly visible. Their random orientation within the composite was confirmed, with minimal voids or gaps observed. This suggests well-bonded interfacial regions.

A well-bonded interfacial region in polymers enhances mechanical strength and adhesion, improves thermal and electrical properties, and reduces defect formation. This results in better overall performance and durability of the material [15]. In the 20wt.% BF/PLA composite (Figure 9), the basalt fibers were evenly distributed throughout the PLA matrix, and strong adhesion was maintained at the fiber-matrix interface. No fiber pull-out or breakage was observed, and the basalt fiber surfaces appeared smooth with well-bonded interfacial regions. Tensile testing revealed stretched fibers within the composite. No grouping or aggregation of basalt fibers was observed, and their random orientation was confirmed. The above morphological observations are found to incline with SEM results reported by Aslan et al. (2018) [8]. Few voids or gaps were present, indicating well-bonded interfacial regions. The coarse surfaces observed in Figures 8 and 9 suggest a brittle fracture mechanism.

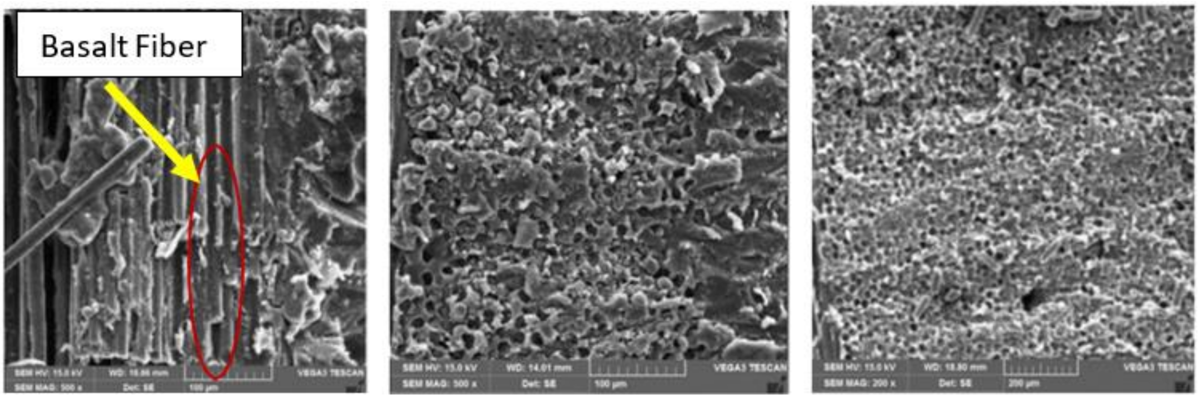

Fig. 8. SEM Observation of 10wt.% BF/PLA

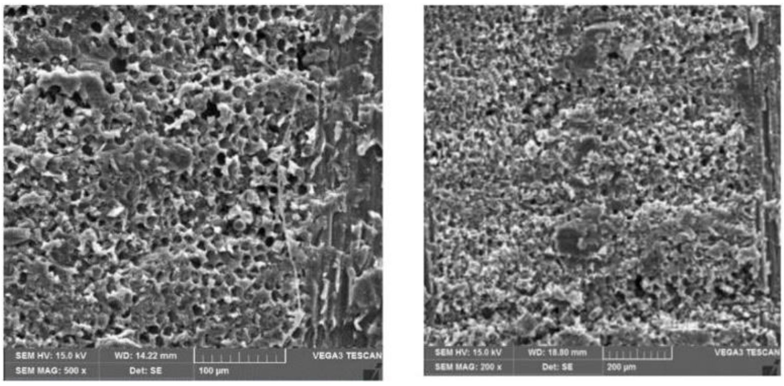

Fig. 9. SEM Observation of 20% Basalt fiber/PLA

5. Conclusion

In summary, the integration of basalt fibers in various proportions has notably bolstered both the tensile and flexural characteristics of PLA composites. Incorporating 10 wt. % basalt fibers yielded an 8.3% upsurge in tensile strength to 50.224 MPa and a 5.6% increase in flexural strength to 89.768 MPa. Elevating the basalt fiber content to 20 wt. % resulted in a significant enhancement of both tensile and flexural strength to 66.025 MPa and 91.797 MPa, respectively. These results underscore the effectiveness of basalt fibers in enhancing the mechanical properties of PLA composites, making them suitable for applications requiring heightened stiffness and strength.

Moreover, the observed enhancements in elastic modulus (E) indicate heightened stiffness and structural integrity of the composites, offering valuable insights for the development of bio-based materials with superior performance characteristics. Specifically, PLA/basalt fiber composites exhibit considerable potential for use in automotive components, where superior strength and rigidity are imperative.

These materials hold promise for the fabrication of structural automotive components such as body panels, interior trims, and reinforcing elements, owing to their enhanced mechanical properties and structural integrity. The increased tensile and flexural strength of these composites contribute to heightened durability and load-bearing capacity, thereby enhancing performance and safety in automotive applications.

Furthermore, the observed improvements in elastic modulus (E) offer benefits in terms of enhanced handling and greater deformation resistance, particularly advantageous for automotive parts. In summary, the study underscores the potential of basalt fiber-reinforced PLA composites in automotive applications, presenting opportunities for the development of lightweight, high-performance components that can contribute to fuel efficiency, weight reduction, and compliance with rigorous safety and performance standards in the automotive industry.

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