



Development and mechanical characterization of eggshell bio-filler reinforced bamboo fiber composites

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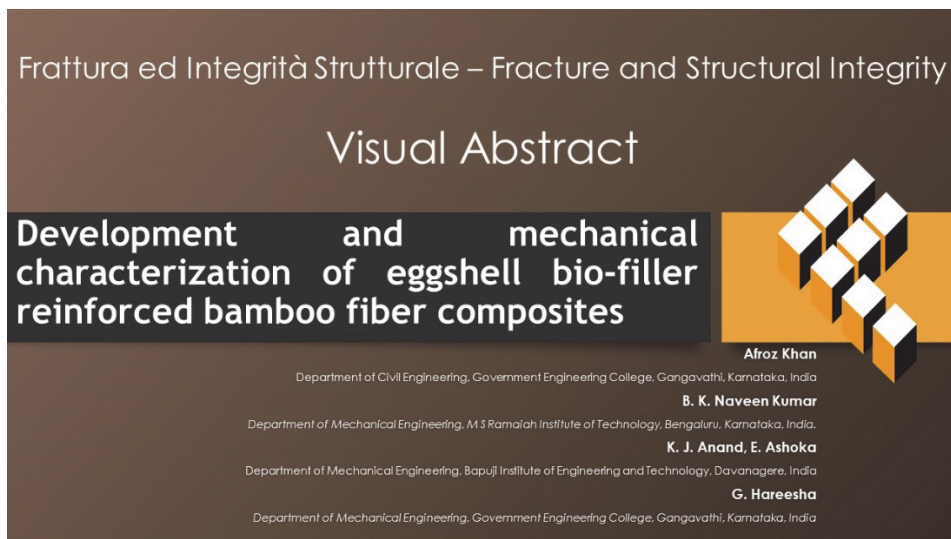
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INTRODUCTION

Sustainability has become a key focus in engineering driven by the need to reduce environmental impact, conserve resources, and create resilient solutions. In materials science sustainable engineering involves developing materials that address environmental challenges such as renewability and recyclability, and promote efficient resource use that is vital for creating a greener built environment in engineering [1]. For instance, sustainable engineering involves designing composite materials that are biodegradable, recyclable, or derived from renewable resources, without compromising performance. Integrating natural fibers into composites enables engineers to enhance sustainability in product design and manufacturing, aligning with environmental goals while meeting engineering demands and promoting a circular economy. These composites utilize fibers derived from renewable resources, such as hemp, jute, flax, and bamboo combined with a polymer matrix to create sustainable composites that are lightweight, strong, and biodegradable [2]. Increasingly, they are being used as sustainable alternatives to conventional synthetic materials in various engineering fields owing to their low cost, availability, and renewable characteristics where they provide comparable mechanical properties while reducing dependency on non-renewable resources. Thus, the development of natural fiber composites represents a significant advancement in sustainable engineering, allowing for the development of materials that are not only high-performance but also environmentally friendly. Achieving a balance between performance and biodegradability is crucial for extending their application across various sectors, highlighting their potential as sustainable materials. Consequently, natural fiber composites have garnered significant scholarly interest over the past decade, leading to notable growth in research and development in this field. While natural fibers offer many benefits, they also display certain drawbacks stemming from their inherent characteristics, including moisture absorption and inadequate compatibility between the fibers and the matrix. Considerable research efforts have been devoted to addressing these challenges, to enhance their functionalities, and broaden their range of applications. Research studies demonstrated that incorporating filler as secondary reinforcement in matrix is a promising technique that enhances composite properties [3]. A growing trend in this area is usage of waste/residual materials, along with by-products from industrial activities, as components of the composite. The incorporation of waste materials as fillers has gained significant importance, and extensive studies have explored utilizing effectively the various industrial and agricultural wastes in developing hybrid natural fiber composites [4]. This strategy not only tackles the issues related to natural fibers but also offers an innovative solution for waste utilization and addresses the problems associated with traditional approach of waste management, such as landfilling. Experimental investigations on feasibility of incorporating different industrial wastes such as red mud, fly ash, and alumina have been reported and their outcome demonstrated satisfactory results [5] [6]. Banana fiber-reinforced polyester composites hybrid composites developed by adding red mud filler exhibited enhanced mechanical, and damping properties. Results revealed that properties are notably affected by filler particle size and filler wt%, and composite with 8 wt% filler exhibiting maximum strength [7]. In another study influence of alumina addition on the mechanical and vibration performance of epoxy-based coir, banana, and sisal hybrid composites analysed [8]. It was reported that including nano alumina improved mechanical properties of these composites. In sisal fiber/bio-epoxy composites inclusion of fly ash fillers enhanced the mechanical properties, significantly improving flexural strength, tensile strength, and impact strength [9].

Studies on fillers derived from agricultural wastes were reported in recent years. Rice husk, bagasse, areca sheath particulates, groundnut, and coconut shell have been utilized to develop composites [10] [11]. It was reported that adding bagasse ash filler in hybrid epoxy composites significantly improved mechanical properties [12]. A comprehensive study on effect of fillers such as sawdust, kolam, and coconut shell powder on the mechanical and wear properties of hybrid sisal, banana, and pineapple fiber-reinforced epoxy composites revealed that these fillers had a positive influence on composite properties, with coconut shell filler giving the best results compared to other compositions [13]. In another study, it was found that adding groundnut filler in luffa fiber composites exhibited superior mechanical properties compared to composites without filler [14].

Calcium carbonate primarily sourced from limestone is the most common filler used in polymer composites. Other than that, CaCO_3 is naturally available as the main content in eggshells and shells of marine organisms such as clams and snails. Recent studies have demonstrated that fillers derived from these biowaste sources can be considered in the development of natural fiber composites because of their availability, low cost, and pollution reduction in terms of landfills [15,16]. Studies revealed that the main content of eggshells was CaCO_3 in the form of calcite (90–96 wt.%), with organic matter 3–4 wt.%, and negligible traces of phosphorus, magnesium, and other elements [17]. In a study eggshell particles with chicken feather was used to develop a hybrid jute-epoxy composite [18]. Results showed that composite with 10% eggshell exhibited the highest hardness, tensile, and impact properties. Eggshell powder in different proportion was incorporated with three different natural fibers coir, sisal, and jute and its influence on mechanical properties evaluated. Results revealed that mechanical properties significantly enhanced in all three composites with addition of eggshell filler and among them coir



fiber with 6% filler showed a highest improvement [19]. In another study composite with banana fiber and eggshell powder fabricated and results showed that the composite with 25 wt% banana fiber and 2.5 wt% eggshell powder exhibited the maximum tensile strength and bending strength [20].

Among natural fibers, bamboo fibers stand out due to their attractive features. The fast-growing, renewable, sustainable, and biodegradable nature of bamboo combined with its efficient strength properties makes it an attractive and environmentally friendly choice as reinforcement in composite [21]. Considerable investigational works have been reported on hybridizing bamboo composites by adding a wide variety of macro/nano-sized particulate fillers and outcome of these studies showed that bamboo-composite properties were significantly influenced by filler inclusion. Recently several studies on the inclusion of fillers from different types of wastes were reported by various authors. Industrial waste red mud in different wt% was incorporated in bamboo epoxy composite and it was reported that up to 10 wt.% red mud inclusion enhanced mechanical properties [22]. Similar observations were made in another study where cement by pass dust filler inclusion improved mechanical properties of bamboo-epoxy composites with highest tensile and flexural strength achieved for 10 wt% filler and further addition up to 20 wt% resulted in strength decline [23]. When bamboo epoxy composite is hybridized with cenosphere filler in different wt%, it resulted in improvement of mechanical properties, but the improvement observed was depends upon the amount of cenosphere. Highest strength properties for composites were achieved for 33 wt % bamboo fibre and 3 wt % cenosphere [24]. A recent study on incorporating bio filler derived from waste clamshells revealed that bamboo composites with 6 wt% filler exhibited the highest tensile and flexural properties, while clamshell filler addition resulted in a decrease in impact strength [25]. The findings from these studies demonstrated that these waste fillers positively influenced the characteristics of bamboo composites where mechanical properties were improved. However, higher filler content had a detrimental effect.

A review of studies on the influence of fillers on bamboo fiber composites revealed that the integration of bio-fillers in bamboo composite presents a promising avenue for enhancing its material properties in eco-friendly applications. Motivated by these observations this study aims to explore the potential of waste-derived chicken eggshell bio-fillers in developing bamboo composite and enhance its mechanical performance, specifically by investigating their effect on tensile, flexural, and impact strengths. By developing hybrid bamboo composites with varied eggshell filler contents, this work seeks to optimize the synergies between bamboo fibers and eggshell bio-fillers. Tensile, flexural, and impact tests are conducted as per ASTM standards. Fractographic analysis of SEM micrographs was performed on fractured tensile samples to understand filler interaction and failure mechanism.

MATERIALS AND METHODS

Materials

Bamboo fibers, a natural reinforcement material known for its rapid growth and low density, bring exceptional mechanical properties. In the present work, bamboo fiber is employed as the main reinforcement. Woven Bamboo fiber was procured from the Sreenath weaving Industry in Rajasthan, India. The matrix phase is thermosetting epoxy resin. Yuji Marketing, Bengaluru, supplied Epoxy resin from Atul India Pvt Ltd. Eggshell powder is chosen as a bio filler to be incorporated in base bamboo composites. Repurposing these waste eggshells can be a cost-effective alternative to conventional fillers. The selection of these materials is driven by a strategic consideration of their unique properties and the potential benefits they offer to the final composite.

Filler preparation

In the present work waste chicken eggshells are utilized to prepare bio filler. To derive bio-filler from eggshells, discarded chicken eggshells were collected from the local market. To remove remnants and any other organic content these collected eggshells were first thoroughly washed and then sun-dried for 24 hours. Then they are coarse-grinded using a kitchen mixture. The coarse eggshell particles are further processed by ball milling to obtain fine particles and then sieved to get the eggshell powder of the desired particle size.

Fig.1 shows the materials used for composite fabrication and Tab. 1 lists the details.

Composite Preparation

In this work, bamboo composites were fabricated by integrating the hand layup with compression molding. A temperature-controlled hydraulic operating compression molding equipment shown in Fig. 2 is used which consists of heated platens to apply heat and a hydraulic press to apply pressure. A base composite (without filler) having bamboo fiber embedded in

the epoxy matrix was first prepared. Then hybrid composites were developed by keeping bamboo fiber wt% constant and varying filler content. Tab. 2 shows the details of both unfilled and filled bamboo composites prepared.

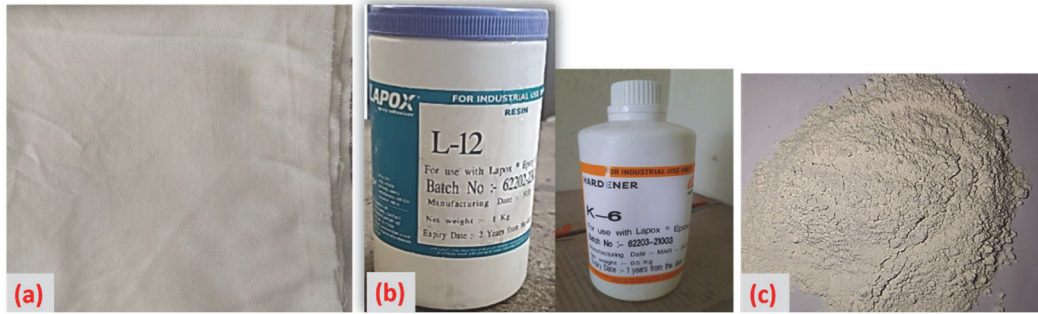


Figure 1: (a) Bamboo woven fiber (b) Epoxy with hardener (c) Eggshell powder.

Material	Details
Fiber	Bamboo fabric, Twill weave, 125 gsm
Matrix	Epoxy - Lapox L12 with K6 Hardener
Filler	Chicken Eggshell powder, Particle size: 60 μm

Table 1: Materials details.



Figure 2: Compression molding equipment.

Specimen Code Name	Bamboo fiber wt %	Epoxy wt %	Eggshell powder (ESP) wt %
B1	35	65	0
B/E2	35	63	2
B/E4	35	61	4
B/E6	35	59	6

Table 2: Composite configuration.

A mild steel mold of size 300 mm x 300 mm and 6mm thick was used for composite plate fabrication. The required number of bamboo mats were cut to the mold size as per the requirement (Fig. 3 a). Initially required quantity of eggshell powder (ESP) was mixed with epoxy resin using a mechanical stirrer. Hardener was added to this mixture in a 10:1 ratio. Then, this

resin-filler mixture was sprayed onto the bamboo mat and kept within the mold. A hand roller is used to spread the resin mixture uniformly over the bamboo fiber surface and ensure proper wetting of fiber (Fig. 3 b). Each layer of the bamboo mat is impregnated with a resin mixture till the calculated weight fraction is achieved. The mold was then sealed and placed in compression molding equipment as shown in Fig. 3 (c). The equipment shown in the figure consists of heated platens to apply heat and a hydraulic press to apply pressure. For preparing bamboo composites a pressure of 150 bar and a temperature of 70°C was applied. Maintaining a moderate temperature of 70°C prevents any possible degradation of bamboo fibers and allows epoxy for better flow and distribution around the bamboo fibers without premature curing. Whereas applying a pressure of 150 bar ensures that the epoxy resin penetrates and wets the bamboo fibers thoroughly. This ensures uniform fiber wetting and compacts the material to eliminate air pockets, minimizing void formation. Composite plates were then dried at room temperature for the next 24 hours. After curing composite plates will be removed from the mold, and any excessive material will be trimmed (Fig. 3 d). From the fabricated 6 mm thick composite plates, tensile specimens of 250 mm x 25.4 mm, flexural specimens of 150 mm x 25.4 mm, and impact specimens of 65 mm x 12.5 mm size were prepared using a conventional cutting machine as per the testing standards requirements.



Figure 3: (a) Steel mold with bamboo fiber (b) Wetting of fibers (c) Compression molding (d) Composite plate.

Mechanical tests

Mechanical characterization studies for the fabricated bamboo composites were performed by conducting tests, as illustrated in Fig. 4. The testing samples prepared were kept initially in an airtight polythene bag to avoid moisture contact. At the time of testing, these samples were taken out, and tests were conducted at normal testing conditions at room temperature. For each composite composition, the mechanical tests were performed on a set of 5 samples, and average values were considered for result analysis. The tensile test and flexural tests were conducted on 20 kN load cell capacity UTM (Zwick/Roell, Germany). Tensile test was performed as per the ASTM 3039 standard with a span length of 150 mm, applying a 2 mm/min cross-head speed. The flexural test was conducted according to ASTM D 790 standard employing a 3-point bending test setup. Izod impact test impact was conducted for a notched specimen as per ASTM D 4812 standard using 25 J capacity impact test equipment.

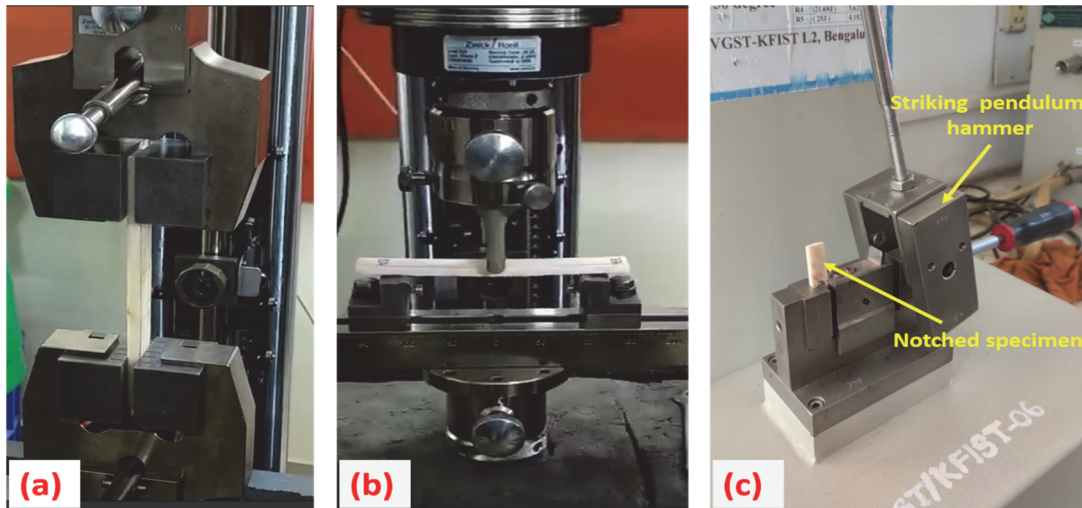


Figure 4: Composite Testing: (a) Tensile test (b) Flexural test (c) Impact test.

RESULTS AND DISCUSSIONS

Mechanical test results obtained for all bamboo composite samples are presented in Figs. 5-7. The results of filled bamboo hybrid composites are compared with unfilled base bamboo composites for analysis.

Tensile test results

As shown in Fig. 5, the base composite exhibited a tensile strength of 39 MPa and a modulus of 670 MPa. The findings indicated that the incorporation of eggshell powder enhanced the tensile characteristics of bamboo composites. As the quantity of eggshell powder increased, there was a corresponding increase in tensile strength up to an addition of 4 wt % filler, further increase did not yield advantageous results and a decrease in strength was recorded. The highest strength increment was achieved for 4 wt% ESP filler inclusion which exhibited a tensile strength of 44.2 MPa and modulus of 750 MPa. At 6wt % addition strength started to decrease slightly to 42.6 MPa which was still higher than base composite strength.

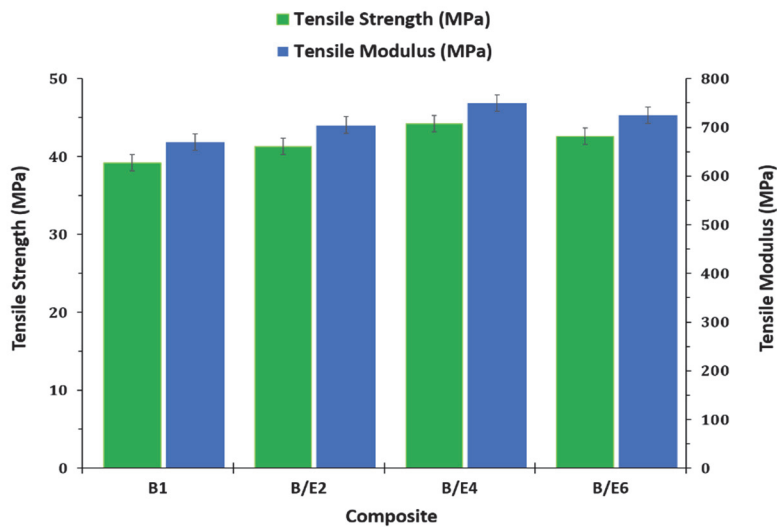


Figure 5: Tensile strength and modulus of bamboo composites .

Flexural test results

The results of the flexural tests are presented in Fig. 6. A comparable characteristic to that observed in tensile testing was noted in the flexural behavior of bamboo composites upon the integration of eggshell powder. ESP filler inclusion improved



flexural properties. At a filler concentration of 2 wt% slight increase in flexural strength was observed while a noticeable enhancement in flexural strength was observed for a 4 wt% filler. In comparison to base composite which had a flexural strength of 70 MPa and modulus of 4100 MPa, the highest flexural strength of 81.5 MPa and a modulus of 4600 MPa were achieved for 4 wt% ESP filler. The incorporation of rigid eggshell powder results in an increase in the stiffness of the epoxy matrix, thereby contributing to an elevation in its strength and modulus. At 6 wt% ESP filler concentration, a slight decline in strength was observed, resulting in a value of 78 MPa; however, this value remained superior to that of the base composite.

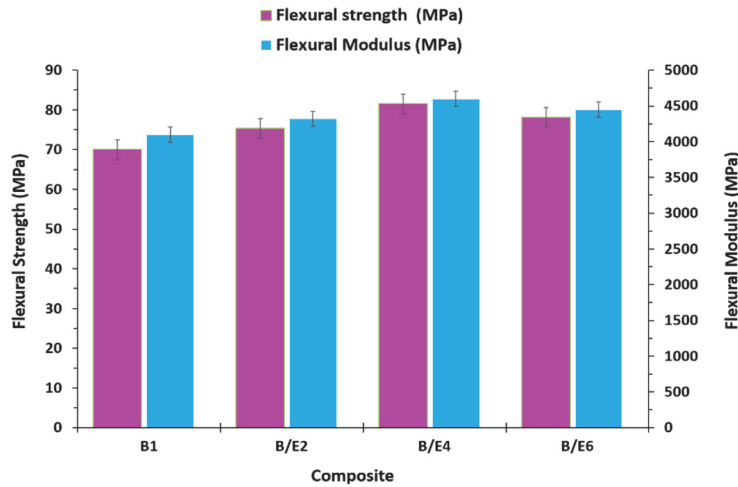


Figure 6: Flexural strength and modulus of bamboo composites.

Impact test results

Findings from the impact test are presented in Fig. 7. The base composite exhibited an impact strength of 61 J. In case of hybrid bamboo composites, there was a modest increase to 63 J observed for a lower filler content of 2 wt%. Further addition of ESP filler content led to a diminution in impact strength, reaching 60 J for a 4 wt% filler. The lowest impact strength of 57 J was recorded for the 6 wt% filler which was lower than the base composite. This decline can be attributed to incorporation of inorganic filler like eggshell powder which possesses a high concentration of CaCO_3 , the material tends to become stiffer and reduces the material's ability to absorb energy during impact. Higher content of rigid eggshell filler particles would make the composite brittle, and more susceptible to crack propagation and failure under impact loading.

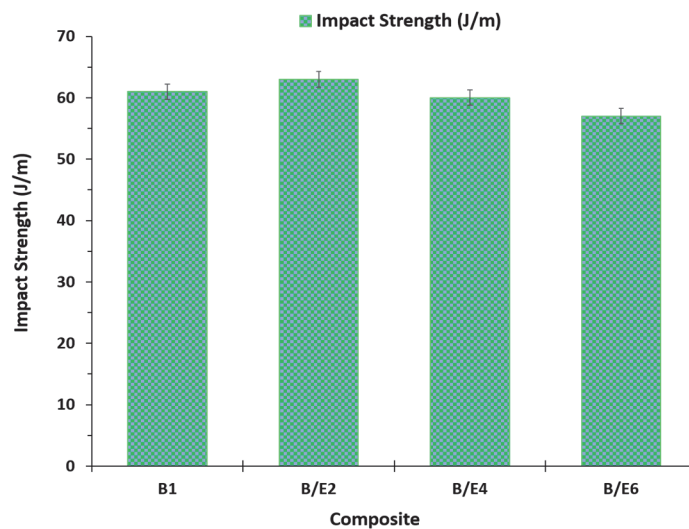


Figure 7: Impact strength of bamboo composites.

These improvements observed in strength characteristics of hybrid bamboo composites can be mainly attributed to the inherent characteristics of eggshell powder which primarily consists of CaCO_3 . The eggshell inclusion improves the interfacial

adhesion, which aided in better stress transfer between bamboo fibers and epoxy matrix, improving load-bearing capacity, and resulting in higher strength. Modulus also increases due to the inherent stiffness of eggshell powder, enhancing the stiffness of the overall composite. However, at a higher filler loading of 6 wt%, the reduction observed might be due to filler uneven distribution or agglomeration leading to poor bonding and microcracks that can offset the improvement. These findings suggest that the incorporation of ESP filler enhances mechanical strength of bamboo composites; however, there exists a threshold for the filler quantity addition, beyond which no further enhancement in strength is observed. Similar observations were made in other studies involving natural fibers incorporated with eggshell filler resulting in improvement of their mechanical properties. Tab. 3 shows a comparison of results obtained for different natural fiber composites incorporated with optimum eggshell filler content. The tensile and flexural strength of coir fiber composites improved with the inclusion of eggshell filler. However higher filler content resulted in a strength decline. The highest strength values were obtained for 6 wt% ESP filler exhibiting a tensile strength of 34.6 MPa and flexural strength of 48.5 MPa. Adding 2.5 wt% of ESP filler to banana-epoxy composite also enhanced its tensile and flexural properties. As illustrated in Tab. 3, these findings establish the significance of ESP filler in enhancing the mechanical properties of natural fiber composites, with bamboo-epoxy composites demonstrating superior performance compared to banana and coir fiber composites. The outcome of this study highlights the better synergies obtained with bamboo fibers and eggshell bio-fillers reinforcement and ESP can be considered as a potential filler with bamboo composites.

Composite Material	Eggshell wt %	Tensile Strength (MPa)	Flexural Strength (MPa)	Reference
Coir fiber/Epoxy	6	34.6	48.5	[19]
Banana fiber/Epoxy	2.5	31.2	33.6	[20]
Bamboo fiber/Epoxy	4	44.2	81.5	Present work

Table 3: Comparison with other works

Fractography: SEM micrograph analysis of composites

A fractographic study of the bamboo composite samples after tensile test was done using ZEISS SEM equipment operating at a voltage of 15 kV. Micrographs of fractured specimens are presented in Figs. 8 (a-c). Fig. 8 (a) displays an SEM micrograph of the base composite without filler. The presence of voids is fairly recognizable and fiber detachment and traces of fiber pull out were evident in the fractured samples. The traces of fiber pull-out emphasize poor interfacial bonding. Insufficient wetting of bamboo fiber causes poor interfacial bonding between with epoxy matrix which leads to fiber debonding and fiber pull-out from the epoxy matrix as seen in the SEM image. This eventually results in poor stress transfer as fibers get detached from the matrix, resulting in lower load-bearing capability. The micrograph shows failure was caused mainly by fiber detachment and significant fiber pull-out, suggesting weak fiber-matrix adhesion. Fibers pulling out without breaking suggests that the load transfer between bamboo fibers and the epoxy matrix is insufficient, leading to premature failure.

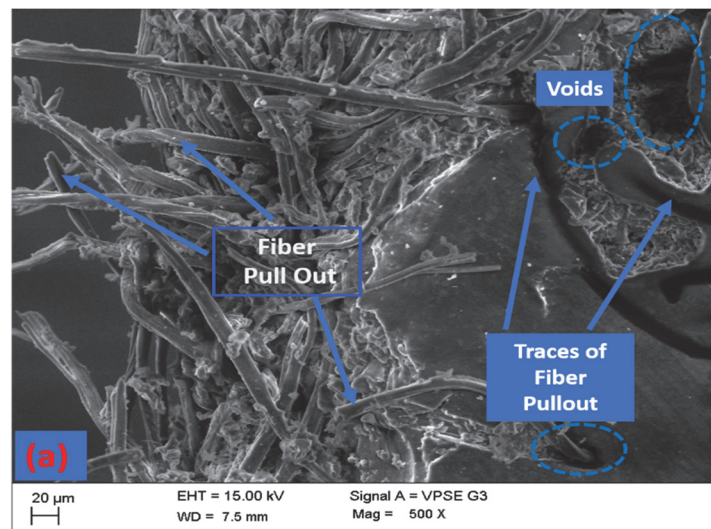


Figure 8 (a): SEM image of bamboo composite without filler.

Fig. 8 (b) shows an SEM micrograph of a composite fractured specimen with 4 wt% eggshell filler. The micrograph reveals presence of eggshell particles embedded in epoxy matrix, appearing to be evenly distributed. This ensures sufficient wettability of bamboo fibers and better load transfer mechanisms by enhancing the interaction between the bamboo fibers and matrix. Enhanced interfacial bonding results in efficient stress transfer and improves strength. A micro-crack was observed in the matrix interphase; however, it appears to have a not prominent role in further crack initiation and failure. The well-dispersed eggshell particles might help in restraining the growth of such cracks by improving the toughness of the composite. Further, a reduction in fiber pullouts from the matrix and the occurrence of more fiber breakage was observed indicating effective stress transfer leading to strength increment of bamboo composite.

Fig. 8 (c) displays an SEM image of the fractured surface of bamboo composite with 6 wt% eggshell filler. Incorporating higher ESP filler content inclusion causes uneven distribution and reduced uniform dispersion of eggshell particles. As a result, the eggshell particles form clusters or agglomerates in certain regions as evident in the micrograph. This agglomeration of filler particles may form stress concentration points and weaken the composite interfacial bonding. As the stress is unevenly distributed around the agglomerates, it hinders effective stress transfer. Thus, incorporating excessive filler compromised the load-bearing capacity of fibers. Certain regions showed clear breakage of fibers. The agglomeration of fillers and subsequent fiber breakages due to a weakened fiber-matrix interface suggest a possible reduction in the strength of the composite. This aligns with the typical behavior observed in natural fiber composites where excessive filler content leads to a reduction in uniform dispersion, which weakens the strength of composite.

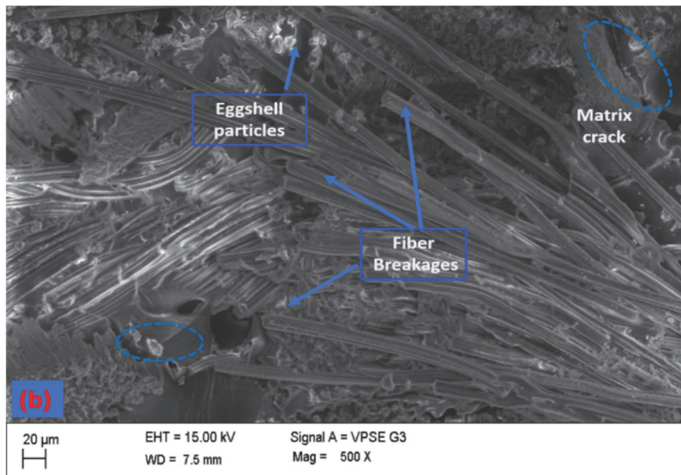


Figure 8 (b): SEM image of a bamboo composite with 4 wt% ESP filler.

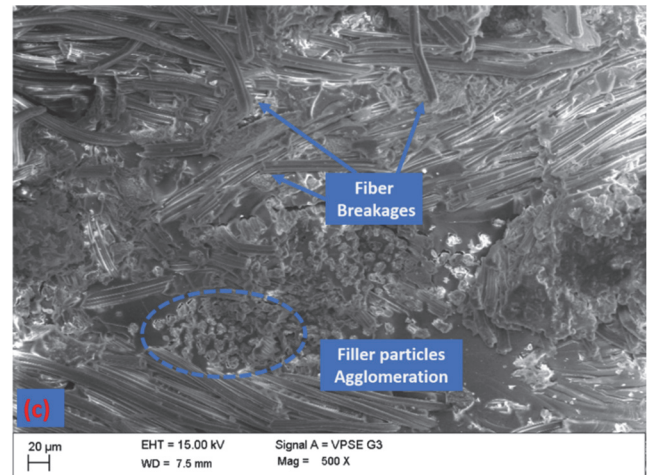


Figure 8 (c): SEM image of a bamboo composite with 6 wt% ESP filler.

This fractography analysis is in support of the observed enhancement in mechanical properties of developed bamboo composites incorporated with eggshell powder up to 4 wt%.

CONCLUSIONS

This research focused on developing hybrid composites by incorporating bio filler derived from waste eggshells and conducting experimental investigations on filler's influence on mechanical properties. The mechanical characteristics of bamboo composites involved conducting tensile, flexural, and impact tests. The main findings drawn from the present study are as follows:

- Experimental results revealed that inclusion of eggshell powder enhanced the tensile strength and flexural strength of bamboo composites by improving the load transfer between the fibers and the matrix. However, this improvement was seen up to 4 wt % filler addition, and at 6 wt% strength was decreased slightly.
- While the incorporation of ESP filler resulted in enhancement of tensile strength and flexural strength, it exerted an adverse influence on the impact strength of bamboo composites, as the presence of rigid eggshell particles diminishes the material's ability to absorb energy upon impact. Although a marginal improvement in impact strength was achieved initially at a lower filler concentration of 2 wt%, a subsequent increase to moderate to high filler levels of 4 wt% and 6 wt%, led to reduction in impact strength.



- Based on the results obtained, 4 wt% eggshell powder can be considered the optimal filler percentage for balancing tensile, flexural, and impact properties. At this level, the tensile strength increased by 13.5% and flexural strength improved by 16.4%, while the negative effect on impact strength remains manageable. Beyond 4%, agglomeration of the filler particles causes a decrease in mechanical properties.
- Fractographic analysis of SEM micrographs of the fractured bamboo-epoxy composite revealed several key features that provided insight into ESP filler interaction and failure mechanisms. At optimal filler loading of 4 wt%, images revealed the presence of eggshell particles embedded in the matrix, exhibiting well-dispersed ESP particles. This resulted in a strong interfacial bonding, contributing to an enhanced load transfer mechanism. Failure modes characterised by fiber breakages suggested effective stress transfer and composite is experiencing typical failure under load, where the stress transfer from the matrix to the fibers is causing them to reach their failure point. For higher filler addition at 6 wt%, SEM images revealed the agglomeration of ESP particles suggesting a potential reduction in material properties.

This study demonstrated that utilizing waste eggshells as fillers presents a viable alternative to conventional fillers without much compromising on mechanical properties. Moreover, repurposing of such waste reduces environmental impact and aligns with the principles of sustainability and circular economy. In summary, it can be inferred that eggshell powder offers a sustainable, cost-effective, and performance-enhancing filler for natural fiber composites, especially in eco-conscious industries seeking lightweight, durable, and biodegradable materials.

Further future research could be extended involving chemical treatment of eggshell powder to enhance the dispersion and interfacial adhesion mitigating issues related to agglomeration at higher filler contents. Also, add other micro/nanofillers to improve the impact strength. Additionally, based on the results obtained with eggshell particles as potential filler, conduct research work involving tests on vibration and wear properties, investigating moisture absorption characteristics, as well as explore the use of bio-based resins to create a fully green composite to further expand the scope of sustainable composite materials applications.

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