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Impact of marble powder addition on the mechanical properties of glass-epoxy composites

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Abstract

The demand for enhanced operational performance in advanced science and technology has driven the use of materials with superior properties. To address this need, the industrial application of polymer composite materials filled with cost-effective industrial waste, as an alternative to expensive ceramic fillers, has gained significant attention. In this study, a novel class of glass fiber-reinforced epoxy composites incorporating varying weight fractions of marble powder was developed using a simple hand layup technique. Mechanical property tests were conducted to evaluate the performance of these composites. The results indicate that the new hybrid composites exhibit improved impact resistance and hardness compared to unfilled composites. However, tensile and flexural strengths decreased, likely due to the presence of marble particles and surface voids, which create stress concentration zones that promote crack propagation in the matrix as filler content increases.

Keywords: Marble powder, epoxy, composite, alkali treatment, mechanical properties

Introduction

Polymer matrix composite materials reinforced with fibers combine two or more distinct elements at the macro scale, along with varying chemical compositions, offering significant potential to replace conventional materials [1]. Modifying the polymer matrix by incorporating different filler contents in varying proportions remains a key challenge in meeting diverse application demands. The inclusion of particulate fillers has been shown to enhance the performance of composites for structural and industrial applications, making it a topic of substantial interest. However, the use of inorganic industrial waste as a filler in polymer matrices has been largely underexplored. Recycling or reusing such inorganic waste is crucial to promoting sustainability and reducing environmental impact. In this context, the present work endeavors to effectively utilize marble particulate as a filler to develop epoxy-based hybrid composites with enhanced properties.

Literature Review

A significant volume of solid waste is generated as a by-product of mineral resource utilization to meet the growing demands of the civil engineering and construction industries [2]. The urgent need to reutilize these solid wastes is driven by both environmental and economic considerations. Developing new composites with a focus on strength, cost efficiency, and eco-friendliness has become a key area of research [3]. Fly ash, a primary industrial waste produced by coal combustion in thermal power plants, contributes substantially to industrial pollution and incurs high storage costs [4]. Previous studies have explored the use of marble waste as a partial replacement for natural aggregates in concrete production [5], while the effects of various ceramic fillers on the mechanical properties of composite materials have also been investigated [6]. Recycling offers a sustainable solution by preserving natural resources and reducing industrial waste through methods like landfilling during composite fabrication. Adding fillers to polymer matrices not only enhances the physical and mechanical properties of composites but also lowers production costs [7]. This growing demand for inorganic fillers has spurred the development of innovative composite materials. Researchers have explored diverse filler materials, such as cenospheres and fly ash, to improve composite properties [8-10]. Over the past decades, significant research efforts have focused on using ceramic fillers in composites [13-15]. For instance, Samal *et al.* [11] demonstrated that reinforcing materials like glass fibers, carbon fibers, and aramids enhance the strength, stiffness, and moisture resistance of composites. Marble powder, an industrial by-product generated during the cutting of ornamental stones in the stone processing industry, has emerged as a promising filler for polymer composites. Its homogeneity, uniform distribution, and chemical stability make it a viable alternative to conventional ceramic fillers. While marble powder has been partially used as a replacement for cement, its potential as a filler in glass fiber composites remains underexplored.

This study aims to investigate the use of marble powder as a filler in epoxy-based glass fiber composites. The physical and mechanical properties of the developed composites are evaluated, with a focus on the effect of incorporating different weight percentages of marble powder on their mechanical performance.

Materials and Methods

Materials

In this study, the primary constituents of the composites are epoxy resin, glass fiber, and marble powder, utilized as the matrix, reinforcement, and filler materials, respectively. The epoxy resin (LY 556) and hardener (HY 951) were procured from Ciba Geigy Ltd., India, while the bi-directional glass fiber mat was supplied by Saint Gobain Ltd. The epoxy resin has a density of 1.10 g/cm³ and a modulus of 3.42 GPa, whereas the glass fiber possesses a density of 2.59 g/cm³ and a modulus of 72.5 GPa. Marble powder, with a density of 2.68 g/cm³ [12], was sourced from a local stone processing industry.

Before composite fabrication, the epoxy resin and hardener were thoroughly mixed in a 10:1 ratio. The marble powder was dried in an oven and sieved to a particle size range of 90–120 µm. The detailed composition of the marble powder is provided in Table 1 [13].

To enhance the mechanical and tribological properties of the composites, alkali treatment was employed as an effective chemical modification method [14]. The dried marble particles were immersed in a 5% NaOH solution for 1 hour at room temperature (30°C). Subsequently, the treated particles were washed several times with distilled water, neutralized with acetic acid, and rinsed again with distilled water. Finally, the neutralized particles were dried in an oven at 100 °C to eliminate moisture. This process removed impurities and improved interfacial adhesion by enhancing the O-H group presence, thereby increasing the hyperbolic and interfacial bonding properties of the treated material [15], as described in Equation 1.

XRD analysis of marble powder

Philips X-Ray Diffractometer was used for examining the crystalline phase. The test was carried out using Cu Kα radiation.XRD analysis of the marble powder is shown in Figure 1. [13]

Table 1: Chemical composition of waste marble powder [13]

Ceramic Oxides (%)	Marble powder
MgO	0.4
CaO	51.7
SiO_2	0.18
Al_2O_3	0.67
Fe_2O_3	0.44
K_2O	0.21
SO_3	0.08
LOI	46.04

LOI-Lost on Ignition

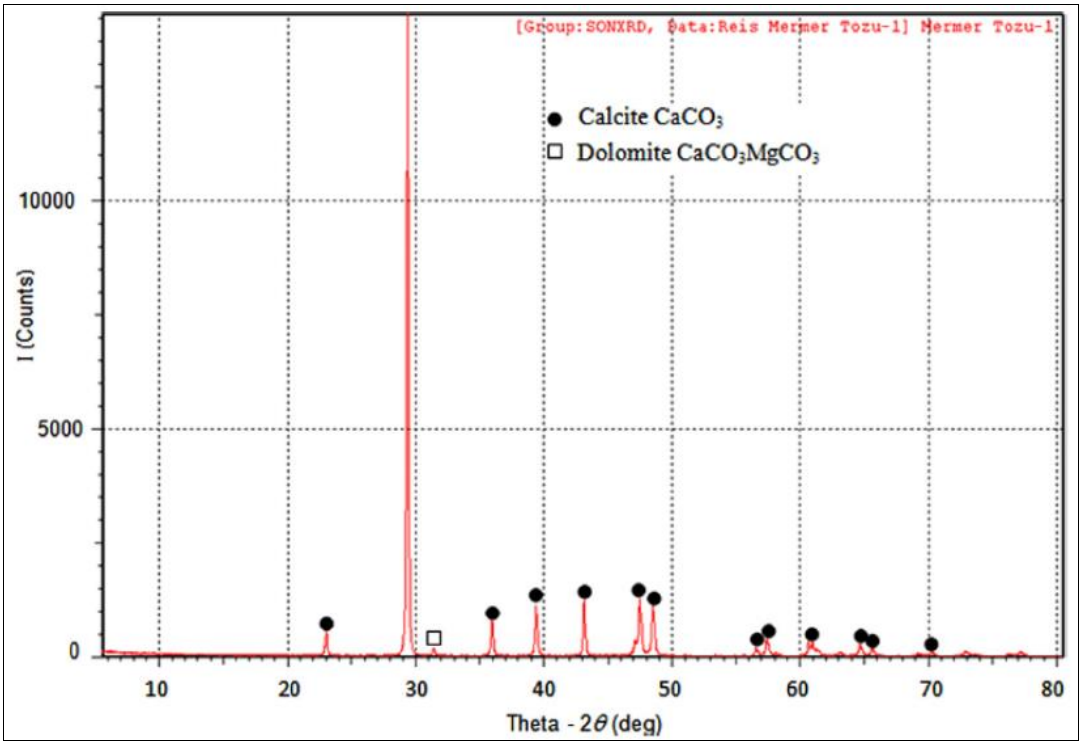

Fig 1: XRD analysis of waste marble powder

Composite Fabrication

The composites are fabricated by the conventional hand layup technique at room temperature afterwards light compression moulding. A number of composite samples are prepared with different weight proportions of marble powder as represented in Table 2. Finally, the composites were cut into the required dimensions as per the standards for the evaluation of physical, mechanical properties.

Table 2: Hybrid composition of the composites

Composites	Composition
EGM0	Fiber (40 wt%) + Epoxy (60 wt %) + Filler (0 wt%)
EGM1	Fiber (40 wt%) + Epoxy (55 wt %) + Filler (5 wt%)
EGM2	Fiber (40 wt%) + Epoxy (50 wt %) + Filler (10 wt%)
EGM3	Fiber (40 wt%) + Epoxy (45 wt %) + Filler (15 wt%)

Experimental Details

Test of Density, Micro hardness, Impact and Tensile Properties

The theoretical density of developed composites can be found out by using the equation stated by Agarwal and Broutman [2].

$$\rho_{ct} = \frac{1}{(W_f/\rho_f)+(W_m/\rho_m)+(W_p/\rho_p)} \quad (1)$$

According to Archimedes principle the actual density (ρ_{ce}) of prepared samples can be found out experimentally.

The void content of respective composite samples can be calculated using following equation:

$$V_v = \frac{\rho_{ct} - \rho_{ce}}{\rho_{ct}} \quad (2)$$

Digital Leitz micro-hardness tester is used in the observation to measure the micro-hardness of the composites. A diamond shaped indenter, in the form of right pyramid is pressed over the sample to get the square impression on the composite surface. The Vickers hardness number is found out using the equation:

$$H_V = 0.1889\frac{F}{L^2} \quad (3)$$

$$L = \frac{X+Y}{2} \quad (4)$$

Where:
L=Diagonal of the square impression (mm)
X= Horizontal length (mm)
Y=Vertical length (mm)

The tensile test was carried in a universal testing machine Instron 3369 as per ASTM-D-3039-76 standard using dog-bone specimen. The dimension of the specimen for the test is 150 mm × 10 mm × 4.5 mm, with the 4.5-mm thickness being maintained for all the samples. Geometry configuration for tensile test is shown in Figure 2. The test is carried out with a cross head speed of 10 mm per minute.

The inert laminar shear strength of the specimens is also conducted using UTM Instron 3369. The test is conducted as per ASTM-D-2344-84 standard in cross head speed of 2mm/min with a sample of dimension 60 mm × 10 mm × 4.5 mm. The geometry of the sample is shown in Figure 3. The ILSS is calculated from the following equation:

$$ILSS = \frac{3P}{4bt} \quad (5)$$

$$FS = \frac{3PL}{2bt^2} \quad (6)$$

Low velocity impact tester was used for conducting the impact test of the prepared samples (ASTM D 256). A pendulum impact tester is used to determine the impact strength of the specimen by crushing the V-notched specimens with a pendulum type hammer, recording the exhausted energy relating to the dimensions of the specimen. The composite samples with dimension 64 mm × 12.7 mm × 4.5 mm is considered for the test as shown in Figure 4.

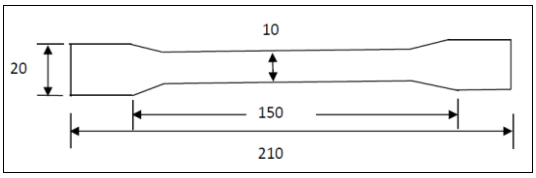

Fig 2: Composite sample for tensile test

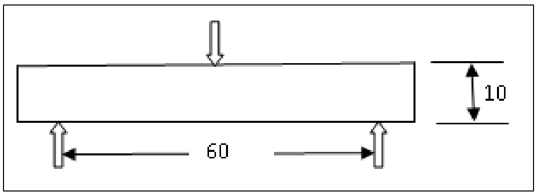

Fig 3: Composite sample for Flexural test

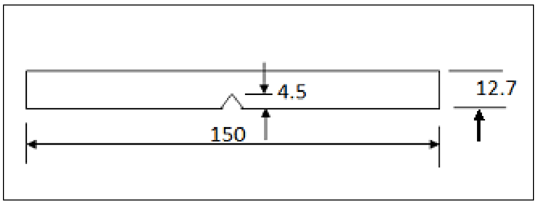

Fig 4: Composite sample for Impact test

SEM examinations

The surface of the specimen was examined by scanning electron microscope (Hitachi Model S-3400N) PC-Based Variable Pressure Scanning Electron Microscope. A thin film of gold was coated on the specimen by sputtering technique to enhance the conductivity.

Results and Discussion

Density and Void content

The composite specimens, along with their corresponding theoretical and measured densities and volume fractions of voids, are summarized in Table 3 and illustrated in Figure 5. As shown in Table 3, the sample without filler (EGM0) exhibits the lowest void content compared to other samples. The formation of porosity in composites, caused by air-filled cavities, is an inherent challenge during the fabrication process.

A higher void content typically leads to increased sensitivity to water dispersion and weathering, as well as a reduction in fatigue strength (Agarwal and Broutman, 1990) [16]. Nevertheless, the presence of voids remains an unavoidable phenomenon when using the simple hand lay-up technique for composite preparation.

Table 3: Theoretical and measured densities of composite samples

Composite samples	Theoretical density (gm/cc)	Measured density (gm/cc)	Void Content (%)
EGM0	1.40	1.42	1.40
EGM1	1.44	1.48	2.70
EGM2	1.48	1.54	3.89
EGM3	1.52	1.61	5.59

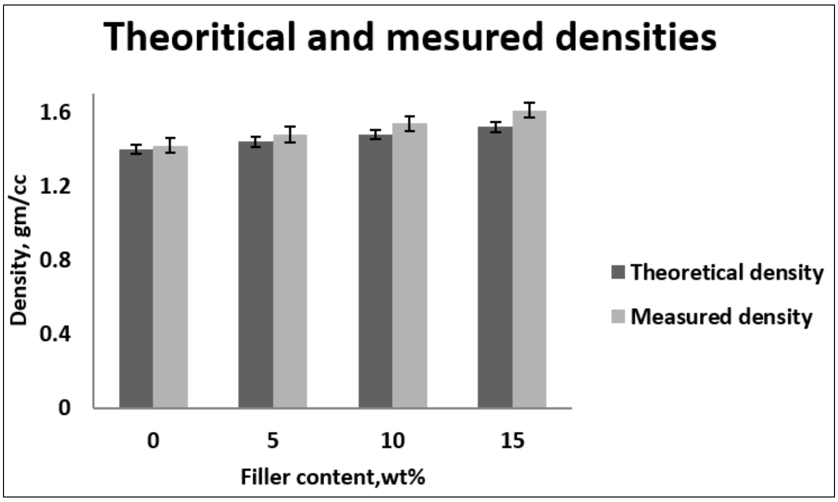

Fig 5: Theoretical an measured densities of composite samples

Micro hardness, Tensile Strength, Flexural strength and Impact strength

The hardness values of the prepared composite specimens are presented in Table 4. It is observed that the addition of marble filler has a marginal impact on the micro-hardness of the composites. Among all the samples, EGM2, containing 10 wt.% marble powder, demonstrates the highest hardness value of 49 HV, as depicted in Figure 4. Typically, the addition of oxide ceramic fillers like marble particles is expected to enhance the mean hardness of the composites. However, the experimentally measured values deviate from this expectation. The minimal effect of marble fillers on the hardness of the composites can likely be attributed to the presence of voids and pores, which undermine the potential reinforcement provided by the filler. Tensile properties were evaluated using an Instron 3369 machine at room temperature with a gauge length of 50 mm and a crosshead speed of 10 mm/min. Each sample was tested five times, and the average values were recorded. The tensile strength results for glass-epoxy composites filled with marble powder (0 wt.% to 15 wt.%) are presented in Table 4. The results reveal a notable decrease in tensile strength from 363.27 MPa for the unfilled composite (EGM0) to 281.64 MPa for the composite with 15 wt.% filler (EGM3), as illustrated in Figure 2.

Filled composites (EGM1, EGM2, and EGM3) consistently exhibited lower tensile strength compared to the unfilled composite (EGM0). Similar trends were reported in previous studies using glass-epoxy-rice husk composites [10]. The reduction in tensile strength is attributed to the increased filler content (5 wt.% to 15 wt.%), which leads to poor adhesion between the matrix, reinforcement, and filler. This behavior can primarily be explained by the increase in void content with higher filler loading, which weakens the interfacial bond strength between the filler particles and the resin matrix.

Additionally, the irregular, sharp edges of the marble filler particles create stress concentrations under tensile loading. These stress points promote de-bonding, preventing the fillers from effectively carrying the load, which ultimately diminishes the composite's strength as filler content increases [17].

Thomason *et al*. [18] suggest that reinforcement, such as glass fiber, in a composite restricts matrix (resin) deformation, resulting in reduced tensile strain values. Consequently, this restraint leads to an improvement in the tensile modulus of the composite. In the current study, the tensile modulus of marble-filled composites increased from 5.16 GPa to 7.51 GPa, as shown in Table 4. While the presence of marble filler in glass-epoxy composites leads to a reduction in tensile strength, it simultaneously enhances the tensile modulus despite the presence of long glass fibers as reinforcement. This behavior highlights the contrasting effects of filler addition on the composite's mechanical properties. Composite materials used in structural applications are prone to failure, necessitating the development of advanced composites with improved flexural properties. Interlaminar shear stress refers to the stress generated between the layers of laminated composites. The interlaminar shear strength or flexural strength values obtained from the three-point bend test for glass-epoxy composites filled with marble powder (0 wt.% to 15 wt.%) are presented in Table 4 and illustrated in Figure 6. The results indicate that adding marble filler causes a gradual reduction in the flexural or interlaminar shear strength of the glass-epoxy (EGM) composite, decreasing from 387 MPa to 323 MPa, as shown in Figure 6. This decrease can be attributed to the presence of micro-sized filler particles and voids within the composite, which lead to localized high stresses. These stress concentrations cause relative deformation (delamination) between the layers, thereby reducing the overall flexural strength. Impact load refers to a suddenly applied force with a specific initial velocity. The material's resistance to breakage under such conditions is known as its impact strength. In polymer matrix composites, impact strength is a key mechanical property [19]. Table 4 presents the measured impact energies of various hybrid composite samples. The results indicate that the impact strength of glass-epoxy composites improves with the addition of marble particulates as a filler. However, the improvement in impact resistance is marginal when using marble filler. In structural design, composite materials are often subjected to impact loads, making impact strain energy an important consideration in selecting materials for various engineering applications. The findings of this study suggest that marble powder can serve as an effective filler material for producing high-strength (impact-resistant) composites.

Table 4: Composite samples with their mechanical properties

Composite samples	Mean hardness (H_v)	Tensile strength (MPa)	Tensile modulus (MPa)	Flexural Strength (MPa)	Impact energy (J)
EGM0	36	364	5.17	386	0.82
EGM1	41	329	6.28	362	0.91
EGM2	48	301	6.73	348	0.97
EGM3	45	280	7.54	322	1.05

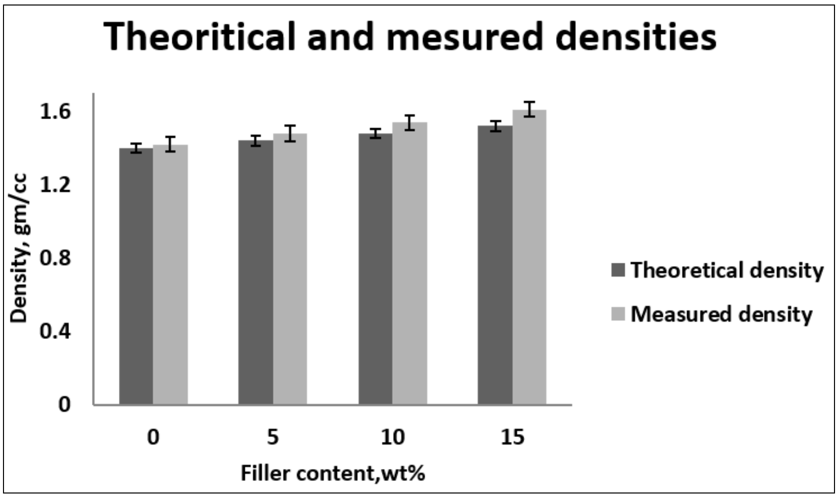

Fig 5: Theoretical an measured densities of composite samples

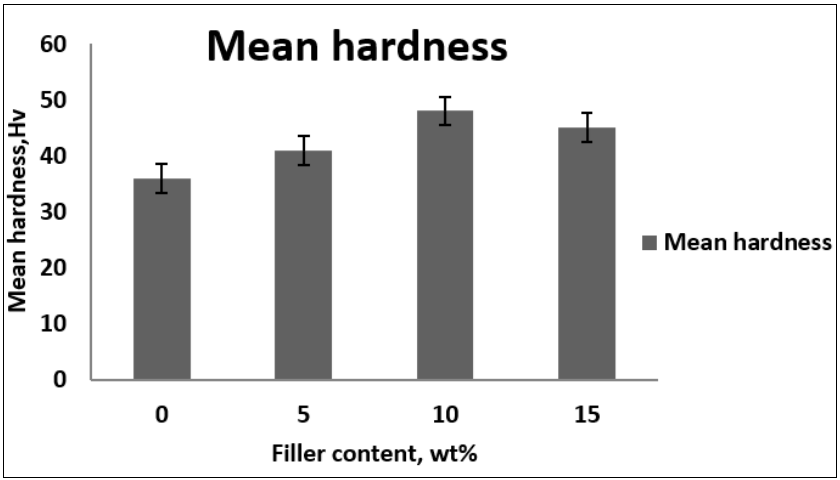

Fig 6: Mean hardness of composite samples

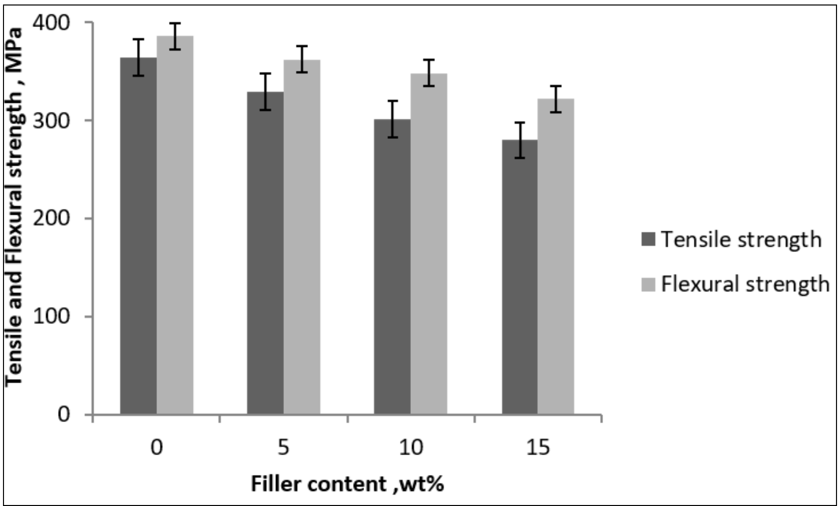

Fig 7: Tensile strength and Flexural strength of composite samples

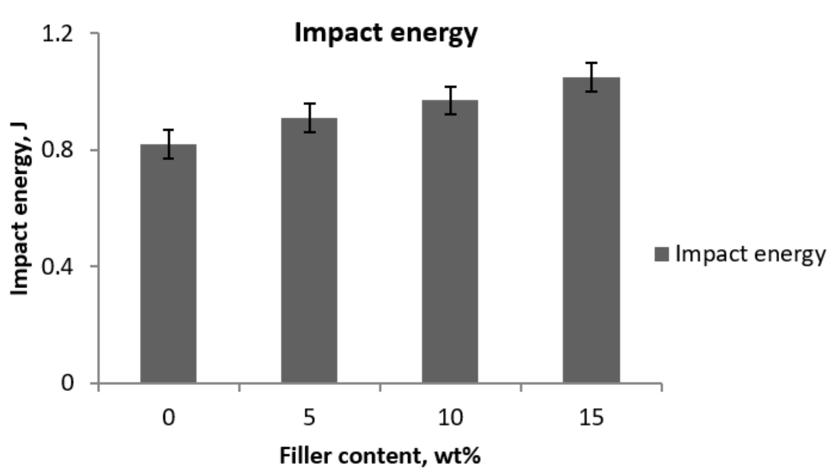

Fig 8: Impact energy of composite samples

Micro-structural observations: Figures 9(a-f) show the SEM micrographs of marble particle-filled glass-epoxy composites following tensile and impact tests. The smooth surface of the composite indicates good compatibility between the marble powder and the glass-epoxy matrix. After the impact test, complete fracture of the glass fiber in the specimen is shown in Figures 9(a-c). Figure 9(b) illustrates the fracture behavior of the fiber in both the linear and transverse directions. Similarly, after the tensile test, the fractured layers of the glass fiber mat are clearly visible in Figures 9(d-g). The delaminated layers of the marble-filled glass-epoxy (EGM1) lamina are shown in Figures 9(e-f). These images distinctly highlight the presence of marble particles within the composite. The maximum shear stress occurs between the layers, resulting in cracks along the mid-plane of the lamina, which causes delamination.

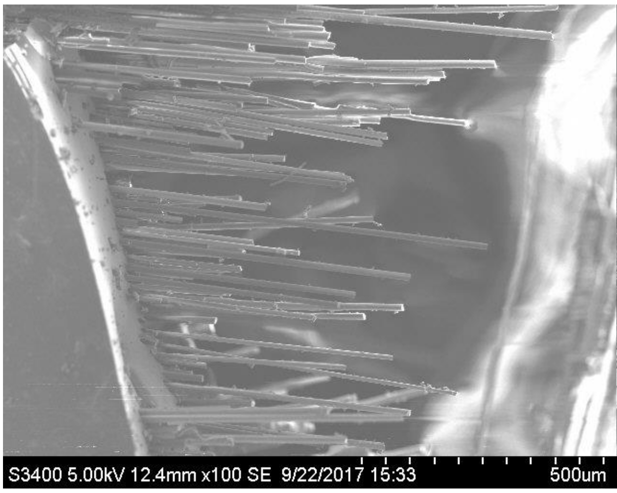

Fig 9(a): Micrograph of EGM0 sample after impact test

Fig 9(b): Micrograph of EGM1 sample after impact test

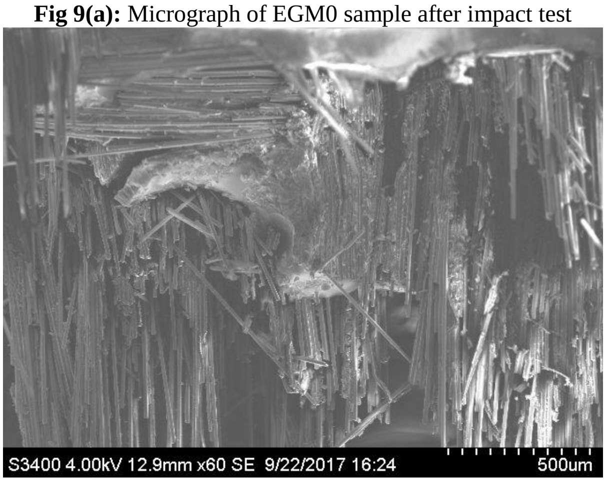

Fig 9(c): Micrograph of EGM2 sample after impact test

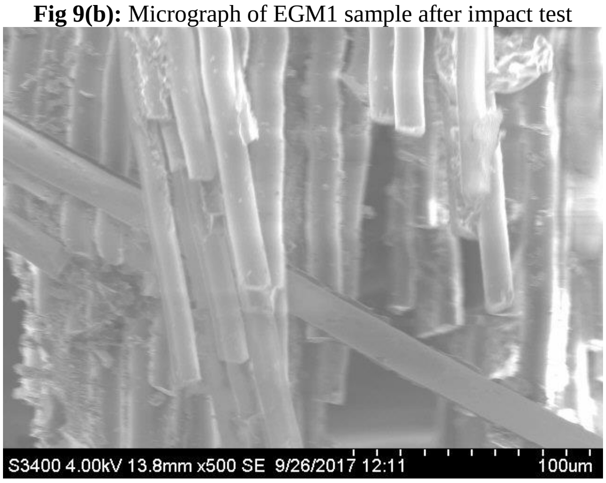

Fig 9(d): Micrograph of EGM0 sample after tensile test

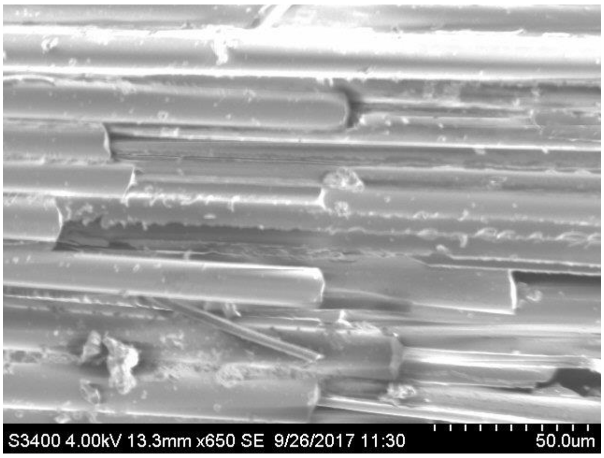

Fig 9(e): Micrograph of EGM1 sample after tensile test

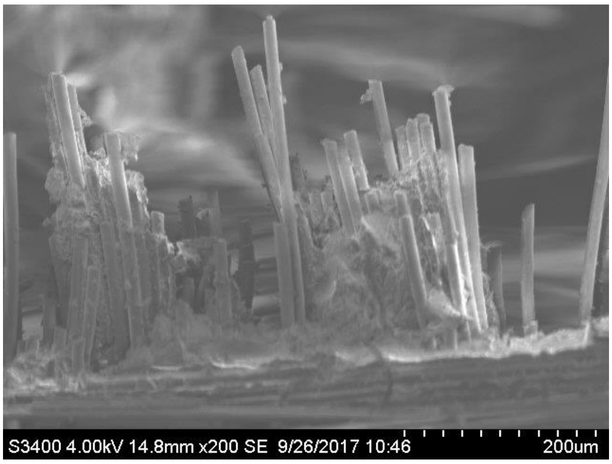

Fig 9(f): Micrograph of EGM2 sample after tensile test

Conclusion

The following conclusions can be drawn from our present experimental observations:

Epoxy-glass-fiber composites filled with marble powder can be successfully fabricated using the simple hand lay-up technique. Experimental observations indicate that the composite with 10 wt.% marble powder exhibits superior filler characteristics, achieving a maximum hardness of 49 HV among all the composites. The unfilled composite specimen (EGM0) demonstrates better tensile strength compared to the filled samples. However, the tensile modulus of the marble-filled composites increases from 5.16 GPa to 7.51 GPa. Additionally, the impact strength of the composites improves from 0.82 J to 1.05 J with the inclusion of marble powder. Morphological studies clearly reveal the fracture behavior of the various composites. Overall, these marble-modified hybrid composites show promise for use in low-cost construction materials, pipes, automobile parts, artificial limbs, and similar applications. Further research could explore the sliding, erosion, and abrasion wear behavior of these composite specimens to evaluate their performance under different conditions.

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