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Characterization Studies on *Vetiveria Zizanioides* Natural Fiber and Graphene Filler Reinforced Nano Polymer Composite Material

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ABSTRACT

In this work, Vetiveria zizanioides (vetiver) fiber-reinforced epoxy matrix was developed with three fiber weight fractions (5, 10, and 15 wt.%) and with 3 wt.% of graphene (Gr) as filler material. This composite could replace asbestos-based braking material in automotive brake linings, as asbestos materials are carcinogenic and cause environmental issues. Hand lay-up and compression molding process was used to fabricate the natural fiber composite. The composite material was characterized by tensile strength, impact strength and flexural strength. It is found that, with increase in fiber content, mechanical properties tend to improve when compared with unreinforced epoxy polymer. The natural hydrophilic vetiver fibers increased absorption of water and thickness swelling was observed for all the composites. When compared with 3% Gr-reinforced composite, 15% vetiver fiber-reinforced epoxy matrix along with 3% Gr shows increase in tensile, flexural, and impact strength by 82.66, 34.25, and 157.14%, respectively, with 29.03% reduction in % elongation. A remarkable increase in water absorption was recorded with the highest fiber content.

Keywords: *Vetiveria zizanioides* fiber; Epoxy resin; Graphene; Water absorption; Compression molding.

1. INTRODUCTION

Developing biodegradable materials with environmental consciousness is the prime objective of scientists and researchers while searching out for newer materials for engineering applications. In this framework, natural fiber reinforced composite is the ideal choice in material science for applications that require improved mechanical properties, enhanced wear and corrosion resistance with light weight. With higher fracture toughness, high strength-to-weight ratio and non-corrosive nature, fiber integrated polymer composites are preferred over traditional materials. Natural fibers are widely used as reinforcements but the choice of selecting the matrix is a tedious task (Boey *et al.* 2022). Natural fibers are a potential alternative for synthetic fibers used in composites as reinforcement. The length of fiber plays an important role in composites towards their strength, especially when the interfacial adhesion is weak (Faheed *et al.* 2024). The 10% sugarcane bagasse introduced in polyester matrix with varying fractions of rice husk filler has been evaluated for their tensile and flexural strength. The matrix with higher filler content lowers the bonding of matrix with bagasse (Hemnath *et al.* 2021). Effect of vetiver fiber on different matrix materials (polypropylene and polyethylene) has been reported by varying the fiber content and the amount of filler (coconut shell powder 10% to 25%). Vetiver reinforced composite containing 20% of the filler showed overall higher tensile strength, impact strength, flexural strength, bending strength and hardness (Babji *et al.* 2020). Shahzad *et al.* (2017) reported that natural fiber composite materials have certain limitations that can be addressed by integrating them with synthetic fibres. The use of surface treatment on natural fibres significantly enhances the bonding between fibres and matrix, which increases mechanical strength and lowers moisture absorption. Dinakarraj *et al.* (2020) fabricated natural composite filled with rice bran particles, reinforced with woven banana fiber fabricated by hand lay-up approach with compression molding and performed micro-milling studies for application in automotive industries. Jena *et al.* (2022) studied the erosive wear behavior of red mud and vetiver fiber-strengthened thermosetting polymer composite prepared by hand lay-up by varying the percentage of vetiver grass and red mud. They found that the wear resistance significantly improved and the material exhibited a semi-ductile behavior during the addition of red mud. A polyester matrix (75%) with vetiver fiber reinforcement (25 wt.%) has been prepared by compression molding approach, where fibers were chemically treated with alkali, peroxide, and benzoyl chloride. It was confirmed that benzoylation of vetiver fibers improved the compressive, tensile and impact strengths of composites by 56.78, 113, and 95%, respectively (Vinayagamoorthy *et al.* 2019). Vinayagamoorthy *et al.* (2014) developed vinyl ester-based composite reinforced with vetiver, woven jute and glass fibres as reinforcements. They identified that by replacing 15% of the glass fibres with natural fibres, the flexural behaviour of the material improved without losing the composite properties.

In this work, a novel natural fiber reinforced polymer matrix composite was prepared by hand lay-up approach followed by compression molding technique. Three different fiber volume fractions were considered with a constant filler reinforcement (Gr) to enhance the strength and stiffness of the composite. The developed composite material was characterized for its mechanical properties (tensile, impact and flexural) and water absorption percentage of the fabricated composite was determined. All the tests adhered to the ASTM standards. The novel material fabricated can be an ideal choice for brake pad material against carcinogenic asbestos material.

2. Materials and Methods

2.1 Material Selection

Epoxy resins are reactive substances that must be cured by polymerization with co-reactants in order to make them useful products (Merline *et al.* 2013). Due to their high reactivity and strong bonding capabilities with fibres and thermoset resins, they produce composite materials with outstanding features compared to most thermosets when mixed with glass, carbon, or aramid fibres. Wet resin processes require direct mixing of reinforcing fibres with epoxy resin. This can be achieved manually through the application of resin and reinforcement to a properly treated mold (Sukanto *et al.* 2021). Epoxy resin undergoes a transformation during curing from its initial low molecular weight to its thermoset state, which consists of a three-dimensional network (Shundo *et al.* 2022). Graphene is composed of a monolayer of carbon atoms that are strongly connected in a hexagonal arrangement, creating a honeycomb lattice. It is an allotrope, with its atoms connected by a bond length of 0.142 nm (Mbayachi *et al.* 2021). The excellent natural mechanical characteristics of graphene, including its strength and toughness, are key factors that distinguish graphene as both a standalone material and a reinforcing component in composites. This is due to the stability of sp2 bonds that constitute the hexagonal lattice and withstand various deformations within the same plane (Keyte *et al.* 2019). Graphene offers the highest thermal conductivity compared to all other known materials. Graphene, due to its ultra-thin and robust structure with minimal surface energy, is a highly suitable option as a solid lubricant for wear resistance applications (Wang *et al.* 2022). Vetiver is a perennial bunchgrass of the family Poaceae and native to India. It can grow up to 5-6 ft forming a wide clump. The stems are tall and rigid with thin and long leaves. The roots of vetiver extend vertically to a depth ranging from 7 to 14 ft. Vetiver fibres are naturally found in muddy and aquatic environments. The stems were cut to produce vetiver fibre as illustrated in Fig. 1. The stem was chopped into desired size, thoroughly cleaned with water to remove dust and mud and dried for 6 hours in sunlight. The dried vetiver stems were boiled at 100 °C in 1 liter of water containing 15 grams of NaOH for 6 hours in order to separate the fibers. After cooling, they are dried again.

Fig. 1: ***Chrysopogon zizanioides*** **plant, its stem and extracted fiber**

2.2 Fabrication of composite

The composite employed in this study was fabricated using the hand layup process. The test specimen was prepared using vetiver fibre (VF) with 270 × 270 × 3 mm size. The composite is composed of three layers. The fibre mats were treated with epoxy resin of LY556 grade (Hemachandra Reddy *et al.* 2023). Methyl ethyl ketone peroxide (MEKP) was used as the catalyst to achieve strong and efficient binding during the mixing of epoxy resin and graphene powder. Epoxy resin and MEKP were mixed in a ratio of 10:1 (Muniappan *et al.* 2020; Vetre Selvan *et al.* 2021) and graphene was added to each sample of about 3 g to impart strength. First, the VF was positioned in a die impregnated with epoxy resin on both sides and then resin was applied over which another set of VFs was placed and so on. A blend of hardener, epoxy resin and graphene was applied on each side of VF. After arranging the fibers, the setting was placed in compression molding machine and 100 bar pressure was applied at 120 °C for making the composite void free and then cured for about 2-3 hours. Each sample was developed with identical orientations of about 90° and the samples were subjected to extensive analysis.

3. RESULTS AND DISCUSSION

After fabricating the different volume fractions of the composite, the specimens were subjected to tensile strength, impact strength, flexural strength and water absorption tests to determine their suitability for engineering applications. ASTM D638-10 standard was adopted for tensile test with dimensions of 165 × 10 × 3 mm. ASTM D790 was adopted for performing the 3-point flexural test with specimen dimension of 127 × 13 × 3 mm. For determining impact strength, ASTM D256-

10 standard with specimen dimension of 65 × 13 × 3 mm was used. For identifying water absorption property of natural fiber reinforced composite, ASTM D5229 standard was adopted with specimen size of 100 × 100 × 3 mm. The percentage absorption was calculated using Eq. (1). The specimens were cut as per the required dimensions using water jet machine (WJM). Fig. 2 shows the optical images of the fabricated composite.

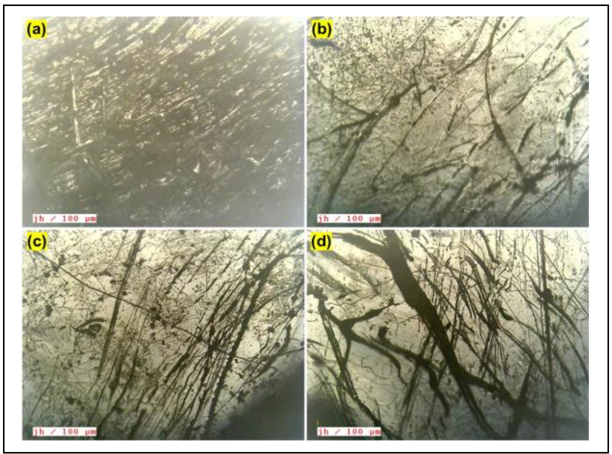

Fig. 2: Optical images of (a) ER+3%Gr (b) ER+3%Gr+5%VF (c) ER+3%Gr+10%VF (d) ER+3%Gr+15%VF

$$\left(\begin{matrix} \%\ moisture \\ absorption \end{matrix}\right) = \frac{\left(\begin{matrix} Weight\ after \\ immersion \end{matrix}\right) - \left(\begin{matrix} Weight\ before \\ immersion \end{matrix}\right)}{\left(\begin{matrix} Weight\ before \\ immersion \end{matrix}\right)} \quad (1)$$

Table 1 presents the properties of vetiver fiber reinforced graphene filled epoxy-based composites determined as per the adopted ASTM standards. The epoxy resin matrix was developed with three vetiver fiber weight fractions (5, 10, and 15 wt.%), keeping a constant 3 wt.% of graphene as filler material. It is observed that the mechanical properties tend to improve with the addition of graphene and vetiver fiber. Also, due to the absorption of water by both graphene and fibers, an increase in water absorption property of the composite was noticed (Mirabedini *et al.* 2020).

Table 1. Mechanical properties of vetiver fiber reinforced composites

Material	Tensile Strength (MPa)	% Elongation	Impact Strength (J)	Flexural Strength (MPa)	% of Water Absorption
Epoxy resin	28.45	3.3	0.408	92.39	0.3
ER+ 3%Gr	39.45	3.1	0.7	96.14	0.51
ER+3%Gr + 5%VF	55.94	2.9	1.1	106.54	0.81
ER+3%Gr + 10%VF	64.08	2.5	1.6	118.89	0.93
ER+3%Gr + 15%VF	72.06	2.2	1.8	129.07	1.02

Fig. 3 presents the tensile strength and % elongation of the fabricated specimens and the properties were compared with the base epoxy resin. It is found that, with the addition of graphene powder, tensile strength increases with reduction in % elongation. With the incorporation of VFs, further enhancement of tensile strength is attained with reduction in ductility. Inclusion of graphene particles and fibers inhibits dislocations during tensile test and thereby restricting the debonding of resins from fibers, which enhances the mechanical strength. Interfacial adhesion or bonding among the fibers and epoxy resin improves the tensile strength. However, the Young's modulus of the composite increases with higher fiber loading (Wang *et al.* 2024).

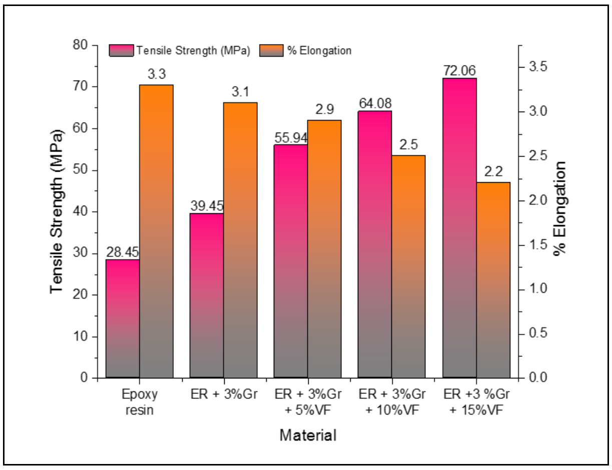

Fig. 3: Tensile strength and % elongation of fabricated materials

Impact and flexural strengths of the fabricated specimens are presents in Fig. 4. Observation shows that with the addition of graphene and VFs, the strength of the laminate gets increased due to the increase in load-bearing capacity of the composite. Most of the applied load is taken by the fibers rather than the matrix, which resists the breakage (Perry *et al.* 2022). With the incorporation of VFs, stress transfer and stiffness tend to increase in the composite. With the higher energy absorption capacity of VFs, the impact strength of the composites is higher (Stalin *et al.* 2022). Also, the crosslinking of the fibers will enhance the bonding within the matrix.

The water absorption capacity of epoxy resin is negligible because of its hydrophobic nature (Gunwant *et al.* 2024). Natural fibers on other hand are hydrophilic in nature. Water absorption test was carried out for a time period of 24 hours at room temperature, where the composites were immersed in distilled water. The properties of water absorption and thickness were determined for all the specimens (Khan *et al.* 2020). Vetiver fibres contain lignin, cellulose, and hemicellulose. The hydroxyl group present in VFs react with hydrogen bond available in water molecules resulting in higher uptake of moisture by the composite

(Samir *et al.* 2022). Hence, it is observed from Fig. 5 that with increasing fiber loading, water absorption nature of the composite increases. Water penetrates the composite through the surface irregularities making the fibers to swell leading to matrix embrittlement (Mayya *et al.* 2021). Microcracks produce higher surface area at the matrix-fiber interface resulting in higher diffusion of water into the composite material (Huang *et al.* 2021).

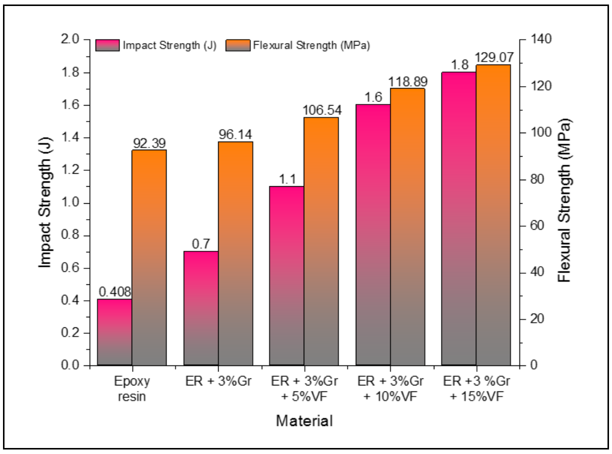

Fig. 4: Impact and flexural strengths of fabricated materials

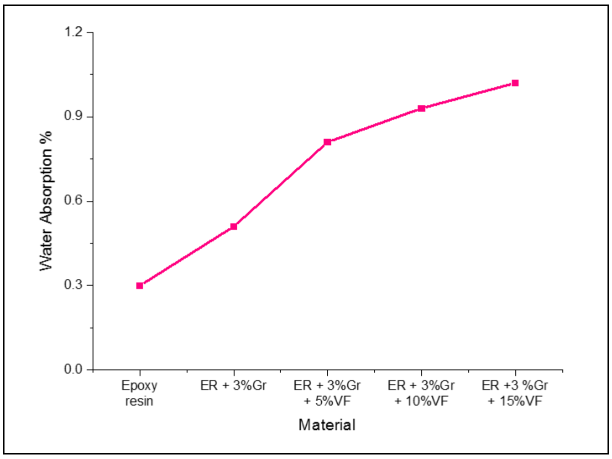

Fig. 5: Water absorption property of fabricated materials

4. CONCLUSIONS

A vetiver fiber reinforced graphene filled epoxy matrix composite was fabricated by an economical hand lay-up fabrication method. The mechanical and water absorption characteristics of the composite were studied and compared with those of pure epoxy resin. The outcomes of this study are:

- By incorporating VFs and graphene in epoxy matrix, tensile strength tends to improve with reduction in ductility as dislocation movements are inhibited by the filler and fiber reinforcements. This restricts the debonding of resins from fibers. Also, interfacial adhesion or bonding between resin and fibers improves the tensile strength.
- Impact and flexural strengths increased due to the higher load withstanding capability of the composite as most of the applied load are taken by the fibers rather than the matrix, which resists breakage. With the incorporation of VFs, stress transfer and stiffness tend to increase in the composite. With higher energy absorption capacity of VFs, the impact strength of the composite is higher.
- Due to the hydrophilic nature of VFs, increased absorption of water and thickness swelling have been observed in all the specimens. The hydroxyl groups present in VFs react with hydrogen bonds in water molecules leading to higher uptake of moisture by the composite. Thus, water penetration into the composites through the surface irregularities makes the matrix brittle. Microcracks increase the surface area at the matrix-fiber interface, leading to greater water diffusion into the composite material.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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