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EXPERIMENTAL STUDY ON WOVEN RAMIE FIBRE EPOXY COMPOSITE WITH SILANE-TREATED GROUNDNUT SHELL POWDER AS A FILLER MATERIAL

Summary

This study focuses on the effect of using silane-treated groundnut shell powder (GSP) as a filler material in varying weight proportions (1 %, 3 %, 5 %, and 7 % wt.) in the fabrication of ramie fibre-reinforced epoxy composites. This study also deals with the mechanical, thermal, and hydrophobic properties of ramie fibre-epoxy composites. A biological waste filler made from groundnut shell (Arachis hypogaea L.), which incorporates cellulose, hemi-cellulose, and lignin, is surface-treated with silane (3-aminopropyltriethoxysilane) using the wet solution technique. Ramie fibre-epoxy composites were created using hand layup and ambient temperature curing. The highest tensile strength of a composite made with 5 wt. % GSP particles in an epoxy matrix is 171 MPa; the maximum flexural strength is 228 MPa, the Izod impact toughness is 6.7 J; and the micro-hardness is 91 Shore-D. Although the thermal stability rises as the filler loading increases, nanocomposites also show a nearly similar tendency toward thermal stability at higher loadings. The silane-treated GSP contributed to an improvement in wear resistance of the composite specimens ERG1, ERG2, ERG3, and ERG4 compared to the untreated ones. The composite specimens (ERG4) with more filler showed greater water absorption. After 45 days of immersion, the ERG4 specimens show a 17 % moisture absorption (the untreated specimen) and a 15% moisture absorption (the treated specimen).

Key words: *Groundnut shell, Epoxy composite, Mechanical, thermal and hydrophobic properties*

1. Introduction

Biological substances have been widely used as reinforcements in polymer frameworks instead of non-degradable reinforcements like those made of carbon, glass, or aramid; this is due to their low density, excellent mechanical properties, abundance in nature, and biodegradability [1]. Green or bio-composite materials are those that contain one or more phases that have a natural or biological origin. Cow dung, marine shells, pulverised nuts, rice husk ash, and egg shell ceramic powders are typical examples of significant natural filler particles. Similar to natural fibres, jute, flax, ramie, kenaf, hemp, coir, and banana fibres find numerous applications [2]. Composites, made by adding natural fibres to a polymeric matrix as reinforcements, are used to make boat hulls, surfboards, sporting goods, swimming pool linings, construction panels, and automobile bodywork. The use of natural fibres not only lowers the cost but also prevents garbage and environmental pollutants from being dumped in the open [3]. One of the most often used polymers is epoxy, which exhibits excellent performance qualities, including superior mechanical and electrical properties, good corrosion resistance, strong adhesion, and minimal shrinkage after curing [4]. Ramie has higher specific stiffness (120 GPa) and specific strength (600 MPa.cm^3/g) than other natural fibres [5]. The use of ramie fibre in composite materials shows great promise for Indonesia, one of the nations that produces ramie [6]. Ramie has a particular rigidity even near that of glass fibre, making it a possible alternative to glass fibre in a number of applications, including body armour, socket prostheses, and civil uses [7]. Numerous useful components found in peanut shells have proven to be safe for humans [8]. In comparison to fibres made from bamboo, hemp, kenaf, coconut coir, and sisal, groundnut shell fibre has a significantly higher lignin concentration [9].

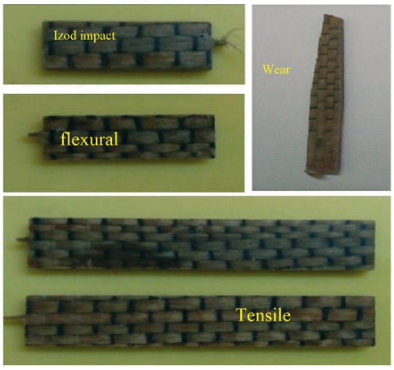
Only when the adhesion to the polymer matrix of these add-ons is enhanced can the performance of added plant-based fibres and other supplied fillers be improved. Therefore, before incorporating the cellulose-rich plant fibres and ceramic-turned-bio particles into the polymer matrix composite, they must first have their surfaces treated [10]. Yamoum et al. [11] investigate the impact of peanut powder on the mechanical, thermal, and biodegradable characteristics of polylactic acid composites. In comparison to pure epoxy resins, the composites with peanut shell powder exhibited improved mechanical and thermal characteristics. The surface of peanut shell powder (PSP) was altered by Prabhakar et al. [12] using alkali treatments at concentrations of 2, 5, and 7 w/v %, and the composites were created by altering the weight fractions of the filler between 5 and 15 wt%. The PSP-loaded epoxy composite with 7% alkali treatment had the best mechanical properties, while that with more bio-filler added had greatly increased tensile strength. The characteristics of an epoxy polymer composite reinforced with ramie fibre were investigated by Lokesh et al. [13]. The composite made of ramie, glass, and Kevlar has the maximum levels of various characteristics. A barrier between the fibres and the matrix occurs at ramie fibres in tiny pieces, mimicking the bonding factor.

The literature search has shown that only groundnut shell powder epoxy composites and ramie fibre epoxy composites have been studied. However, no literature report is available on the combination of ramie fibre as a reinforcement and groundnut shell powder as a filler material used in the fabrication of epoxy composites. To fill in the aforementioned research gap, the current study aims to investigate the effect of varying weight proportions of silane-treated groundnut shell powder (1 %, 3 %, 5 %, and 7 % wt.) used as a filler material in the fabrication of ramie fibre-reinforced epoxy composite. In addition to that, it also focuses on the mechanical, thermal, and hydrophobic properties of ramie fibre-epoxy composites.

2. Materials and Methods

India places second in the world for producing groundnuts. The outer covering of a groundnut is referred to as the shell and the entire fruit as the pod. The hemicellulose ratio in groundnut shells ranges from 18 to 20 %, cellulose from 34 to 36 %, lignin from 29 to 31 %, and ash from 4 to 6 %. Sisal has less hemicellulose than groundnut shells do. Groundnut shells that had been cleaned and dried were first washed with water to get rid of sand and other impurities. After that, the washed shells were chemically treated with the acid hydrolysis silane treatment (4 weight %) to remove the soluble extractives and to modify the untreated groundnut. Following the surface treatment, acetic acid and distilled water were used to neutralise the groundnut shells until all chemicals were removed. The shells were then solar-dried for 10 days at a temperature of 40 ± 5 °C and then crushed to fine particles using a hammer mill for one hour. Then, 0.5 to 1.5 mm BS sieves were used to separate the particles.

Finished Composite



Test Specimens

Ramie woven mat fibre

Fig. 1 Photographs of finished laminate and test specimens

Table 1 A list of specimens, their designations and compositions

Specimen	Specimen designation	Epoxy (Vol. %)	Ramie fibre (Vol. %)	GSP (wt. %)
Pure epoxy	E0	100	0	0
Epoxy+ramie fibre composite	ER	60	40	0
Epoxy+ramie fibre reinforced with 1% GSP composite	ERG1	59	40	1
Epoxy+ramie fibre reinforced with 3% GSP composite	ERG2	57	40	3
Epoxy+ramie fibre reinforced with 5% GSP composite	ERG3	55	40	5
Epoxy+ramie fibre reinforced with 7% GSP composite	ERG4	53	40	7

The composites employed in this experiment were created using a hand layup technique. A mould with particular dimensions was used. A layer of wax was applied to the mould in order to take the sample out easily. Groundnut shell powder and epoxy were mixed using mechanical stirring for 4 hours without interruption at a constant 500 rpm speed. To get rid of the surplus resin, three laminae of 40 % ramie fibre were put together one at a time and meticulously compressed. The composites were cured for around 24 hours at room temperature. A similar fabrication procedure carried out under the same conditions was presented in the study of Vrgoč et al. [14]. Figure 1 shows the photo images of the finished laminate and mechanical test specimens prepared. The surface quality of the cured composites was carefully inspected. The margin and fill flaws were identified in order to prevent a flawed specimen-making procedure. Table 1 lists out the list of specimens fabricated along with their composition and designation assigned.

3. Characterisation

For physical/mechanical characterisation, specimens of sufficient dimension were cut using a diamond cutter in accordance with ASTM (D-3039 - Tensile test, D-790 - Flexural test, and D-256 - Impact test) standards. Utilizing an all-purpose testing device, the tensile and flexural characteristics were evaluated (Instron 1195, CIPET, Chennai, India). Similar to that, a micro impactor was used to evaluate the impact of composite materials in line with ASTM D 256. (Krystal Equipments India Pvt. Ltd). Utilizing an ULTRA-55 Field Emission Scanning

Electron Microscope, morphology of the composite was studied (FE-SEM). Thermograms were acquired using a Perkin Elmer Pyris1 TGA thermogravimetric analyser. The water absorption test results of the composite were calculated using a semi-micro weighing balance, the MSA 225S - 100 DA type. The water absorption of the composite materials has been measured using the ASTM standard test technique ASTM-D570.

4. Results and Discussion

4.1 Mechanical Properties

Table 2 shows the mechanical properties of the GSP-reinforced ramie fibre-epoxy composite, with both untreated and surface silane-treated forms of GSP. The mechanical properties, such as tensile strength, flexural strength, impact strength, and hardness, are measured and the results are shown in Figure 2. The tensile and flexural strengths of pure epoxy resin (E0) are 63 MPa and 112 MPa, respectively. This is due to the extreme brittleness of the matrix phase, which causes plastic deformation even at low stresses. The tensile strength and flexural strength of the epoxy-ramie fibre composite (ER) have improved by 112 % and 53.5 %, respectively, when the pure epoxy is supplemented with 40 vol. % of ramie fibre. The greatest load is absorbed by the ramie fibre, which also protects the epoxy matrix from degradation and reduces stress intensity. As a result, the composite would be able to tolerate a bigger load impact since the fractures would not acquire enough energy to expand farther. The load bearing ability of ramie fibres has been improved by the small addition of groundnut shell powder to the composite specimen ER. As the wt. % of the filler material (GSP) progressed from 1 % to 5 % for both the untreated and treated specimens, the tensile and flexural strengths of the composite specimens improved. Similar observations were made by Obasi [15] With the loading of filler material limited to 5 %, the ERG3 specimen exhibits the best mechanical properties of all the specimens.

Table 2 Mechanical properties of pure epoxy and the GSP-reinforced ramie fibre-epoxy composites

S. No	Composite Designation	Tensile Strength (MPa)		Flexural Strength (MPa)	
		Untreated	*Treated*	*Untreated*	*Treated*
1	**E0**	63±0.87	-	112±0.79	-
2	**ER**	134±1.07	-	172±1.60	-
3	**ERG1**	146±0.97	185±1.58	201±0.89	253±1.78
4	**ERG2**	162±1.06	190±0.83	212±0.84	268±1.71
5	**ERG3**	171±0.98	210±0.84	228±0.91	291±1.77
6	**ERG4**	168±1.01	205±0.96	224±0.86	275±1.81

S. No	Composite Designation	Izod Impact (J)		Hardness (Shore-D)	
		Untreated	*Treated*	*Untreated*	*Treated*
1	**E0**	0.31±0.01	-	86±1.14	-
2	**ER**	4.7±0.11	-	86±0.91	-
3	**ERG1**	6.0±0.14	6.2±0.07	88±0.89	89±0.87
4	**ERG2**	6.4±0.12	6.8±0.06	89±0.95	91±0.91
5	**ERG3**	6.7±0.08	7.1±0.09	91±0.91	92±0.89
6	**ERG4**	6.5±0.10	7.0±0.07	90±0.88	91.5±0.9

The highest tensile strength of a composite with 5 weight % GSP particles in an epoxy matrix is 171 MPa, the maximum flexural strength is 228 MPa, the Izod impact toughness is 6.7 J, and the micro-hardness is 91 shore-D. GSP particles in microcracks absorb a significant amount of energy, improving the fracture resistance of the composite. This improvement is the result of the improved adhesion and even dispersion of the additive. Similar outcomes were also observed for the other mechanical parameters, including hardness and Izod impact toughness. When GSP particles are present, the cross linking density is reduced, resulting in a larger free volume. The enlarged free volume allows the molecules to expand freely during the impact test in which rapid stress is applied. It was discovered that the tensile and the flexural strength of the composite specimen ERG4 had decreased due to the high amount of GSP particles (7 wt. %) that form clusters and reduce the load bearing effect. The uniform load-sharing effect of the composites is hampered by the development of intercalated structure of groundnut shell particles.

The tensile, flexural, impact, and hardness characteristics of the ramie fibre-epoxy composite have been proven to be improved by the silane surface-modified groundnut shell powder particles for all the specimens. When compared to the untreated groundnut shell powder dispersed in the ramie fibre-epoxy composite, the tensile strength, flexural strength, Izod impact toughness, and hardness of the silane-treated ERG3 composite increased by 17 %, 26 %, 6.2 %, and 2.2 %, respectively. The good load-sharing capacities of the particles, which may restrict the tendency of the crack to open, are responsible for this improvement. The silane substance covers the particle during the epoxy matrix phase, preventing it from being drawn to surrounding particles and thus fostering uniform dispersion. The homogeneous dispersion of silane-treated GSP particles results in a low propensity for clustering and a high affinity for the matrix. The silane treatment encourages the elimination of some lignin and hemicellulose, which causes the structure of GSP to develop holes or voids. These spaces make it easier for the resin to penetrate and diffuse into the structure of the filler while also increasing its wetness. This encourages effective interaction and stress transmission between the two phases, which leads to superior mechanical qualities. The silane treatment and exfoliation processes will eliminate the free OH functional group [16]. As a result, the surface-treated groundnut shell powder boosts molecular flexibility and energy absorption while reducing the cross-linking density of epoxy resin. Similar results have been obtained by Rajmohan [17] with the alkali treatment of some natural fibres. In the epoxy matrix, the amine-grafted GSP disperses more evenly and does not form any clusters. Because of the decreased stress intensity component, there will not be any disruptions to the load distribution over the matrix phase. Lowering the stress intensity factor significantly enhanced the capacity of the composite to support loads; some of the above results have been correlated with the study of Rajeshkumar et al. [18].

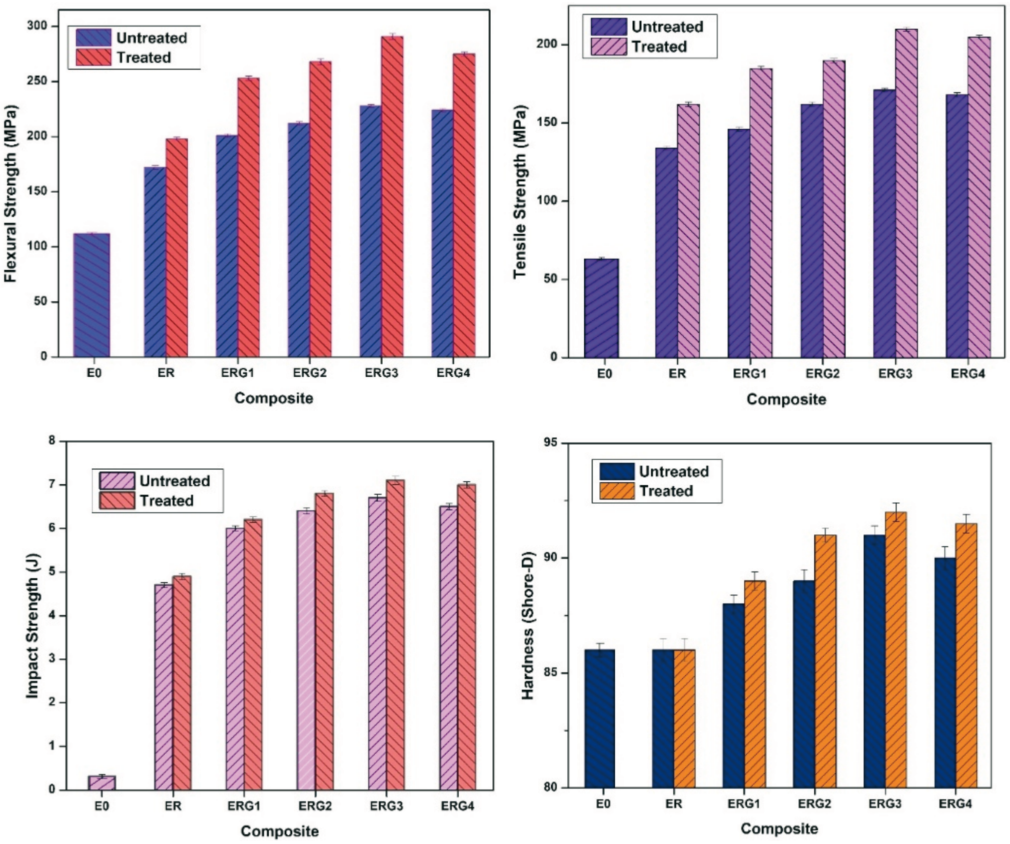


Fig. 2 Mechanical properties of epoxy resin and the GSP-reinforced ramie fibre-epoxy composite

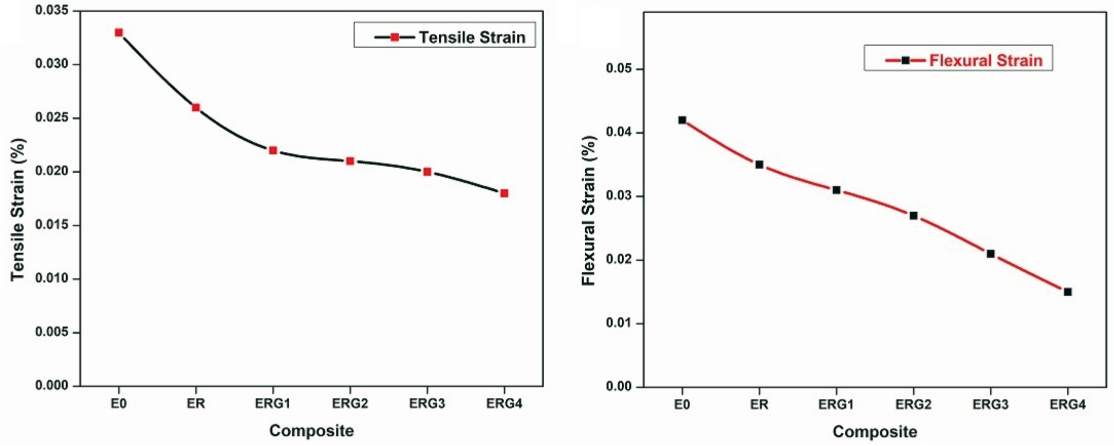


Fig. 3 Tensile and flexural strain of epoxy resin and the GSP-reinforced ramie fibre-epoxy composite

Figure 3 shows the variation in tensile strain and flexural strain among all the GSP-reinforced ramie fibre-epoxy composite. The results showed that both the tensile and the flexural strain were reduced as the weight proportions of groundnut shell particle increased, i.e., the ERG4 specimen exhibited the least strain value. The reinforcement acts as a barrier and resists the deformation of the composite, which also reduces the overall strain level.

4.2 Thermal Properties

The thermal gravimetric analysis (TGA) thermographs of pure epoxy and groundnut shell powder-reinforced ramie fibre-epoxy composite specimens are shown in Figure 4.

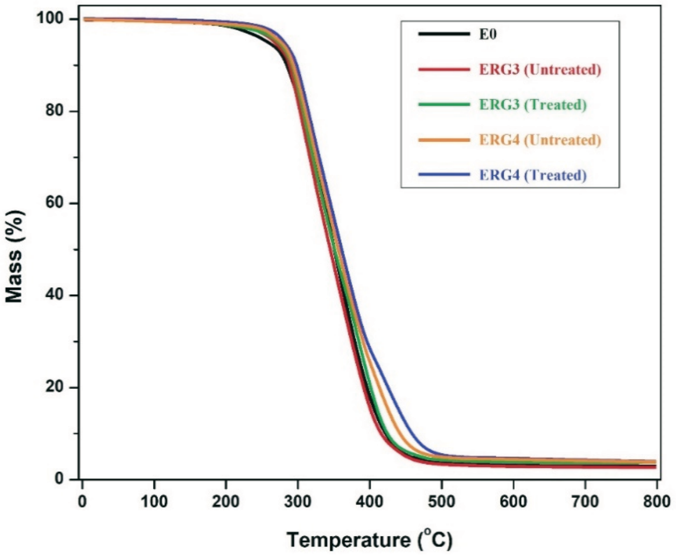


Fig. 4 TGA thermographs of pure epoxy and groundnut shell powder-reinforced ramie fibre epoxy composite specimens

The thermo-gravimetric curves of all specimens have two deterioration phases. The first is related to the evaporation of moisture from the specimens, while the second is due to the polymer chain scission. The pure epoxy resin has two degradation phases, with maximal breakdown temperatures of 290 °C and 480 °C. The first phase was most likely caused by the breakdown of low molecular weight components such as diluents or matrix pendant chains, whereas the second phase showed the deterioration of pure epoxy main chains. It should be noted that the deterioration of ERG3 and ERG4 specimens has been delayed or that the thermal stability of ERG3 and ERG4 has been somewhat enhanced. The other composites show a similar trend in their behaviour as that of the pure epoxy except for the marginal difference in their corresponding temperatures. Ikladious et al. [19] plotted similar TGA curves and explained them in their study on eco-friendly composites based on peanut shell powder / unsaturated polyester resin.

Furthermore, the inclusion of ramie fibre and groundnut shell powder had a significant impact on the early, fast, and ultimate mass loss of the composite. According to research in natural fibres, this variance was linked to the breakdown of the fibre components, notably hemicellulose, cellulose, and lignin. The inclusion of groundnut shell powder particles contributes to the intercalation/exfoliation of the epoxy matrix, resulting in a strong barrier effect that, to some extent, prevents thermal degradation. As a general conclusion, thermal stability improves with filler loading, although nanocomposites demonstrate almost the same trend in terms of thermal stability at higher loadings.

4.3 Wear Properties

The specific wear rate and coefficient of friction (COF) of pure epoxy resin and all composite specimens were determined and plotted, as shown in Figure 5. Pure epoxy has the highest specific wear rate of 0.016 mm^3/Nm and a COF of 0.74. Pure epoxy is brittle by nature, so when it is exposed to a wear-loading test, the surface of the epoxy adheres to the abrasion disc, resulting in a greater material loss. Nevertheless, when ramie fibres of a significant volume are mixed in the resin, they reduce the wear rate and COF marginally, as shown for the ER composite specimen. The ERG3 specimen exhibits a high hardness value which yields a lower specific wear rate; this correlation is in agreement with the studies of

Mikušova et al. [20]. This reduction in the specific wear rate was due to the reduced frictional force between the disc and the specimen and the load bearing capacity of the fibre. It is observed that as the percentage of loading of groundnut shell powder increased from ERG1 to ERG4 composite specimens, the wear rate and COF were reduced. The frictional wear resistance improved as the filler loading increased; similar results were found and interpreted in the study of Aly et al. [21]. The reason for this is that the GSP particles act as a hindrance, improve abrasion resistance, and protect the surface of the composite from wear or material loss.

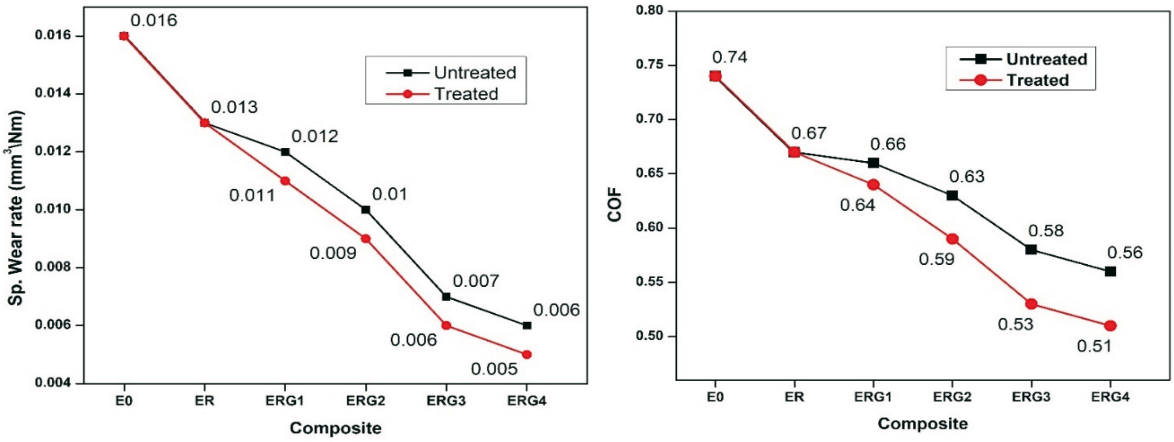


Fig. 5 Specific wear rate and coefficient of friction (COF) of epoxy resin and all composite specimens

However, the surface-treated composite showed a reduction in specific wear and COF compared to the untreated composite specimens. The silane-treated GSP showed an improvement in wear resistance for the composite samples ERG1, ERG2, ERG3, and ERG4 compared to the untreated ones. This improvement in wear resistance occurs because of the effective particle dispersion due to the reaction of the NH2 functional group on the particle surface, improvement in sliding forces, and achieved uniform dispersion in the matrix phase. Thus, the uniform dispersion of GSP particles in the matrix hinders the removal of matrix material and reduces the abrasion losses such as adhesion and wear losses.

In Figure 6a one can see that the pure epoxy resin exhibits a flat wear track surface, indicating the extreme brittleness of the resin. The surface shows more criss-cross patterns, which indicates the three body abrasive wear loss mechanisms. From Figure 6b one can notice the fibre debris for the untreated ERG3 specimen, which indicated the removal of fibre at the time of the abrasion process. However, in the silane-treated ERG3 specimen (Figure 6c), no such debris was found. There are also no broken fibre traces. This indicated improved adhesion and an improved sliding surface. Therefore, the addition of stacked silicate groundnut shell particles of notable volume to the resin marginally improved the wear resistance.

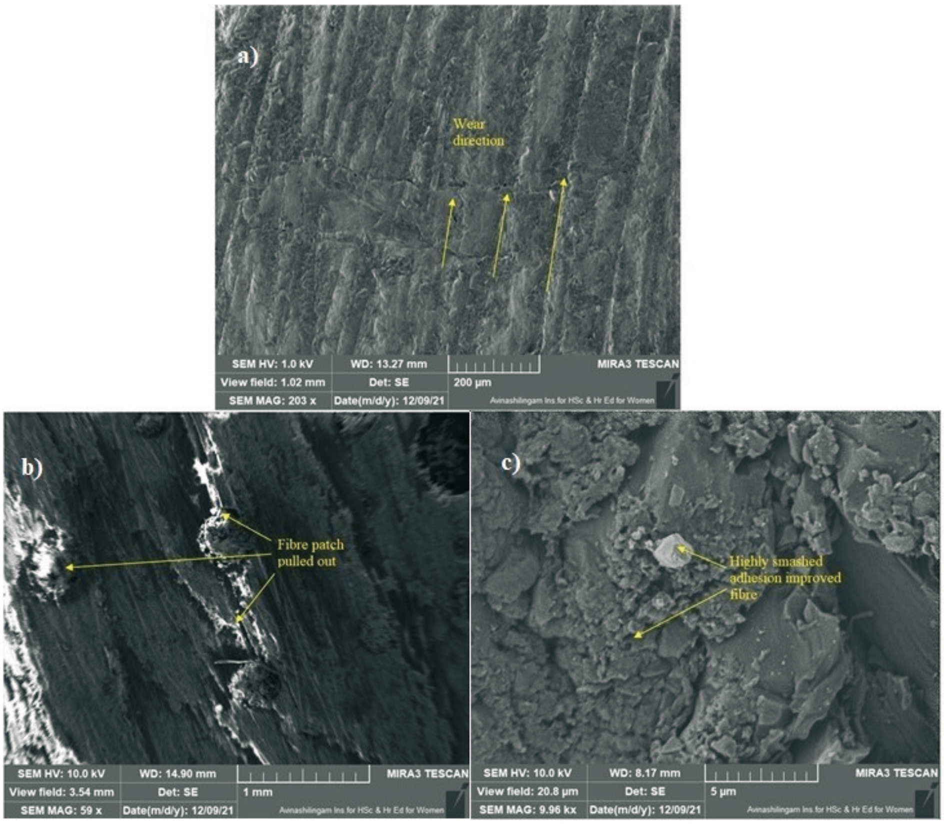


Fig. 6 SEM image of (a) Pure epoxy (E_0) (b) Untreated ERG3 (c) Silane-treated ERG3 specimen

4.4 Water Absorption Test

The usual behaviour of any composite regarding water absorption is fast absorption followed by a progressive increase until an equilibrium condition is reached. Natural fibres are hydrophilic by nature, which means they absorb water; however, research into their water absorption behaviour is of critical importance. Since pure epoxy comprises only polymer molecules, it does not change in terms of water absorption. Following a Fickian diffusion process, the water absorption process in all specimens is linear at first, then it slows and achieves saturation after a lengthy period. The inclusion of ramie fibre in pure epoxy affected its behaviour in the ER specimen because natural fibre is hydrophilic and swells by absorbing water. Cracks usually form in a brittle matrix, such as that of epoxy resin, as a reaction to swelling, resulting in a bigger movement of water across the fibre and matrix interface. A capillarity process is also activated; water molecules travel across the interface of the fibre and matrix, resulting in increased diffusivity.

Compared to the treated specimens, all untreated composite specimens absorbed more water. The mixture of lignin and hemicellulose may be responsible for the greater water absorption and poor wetting of the filler particles by the polymer matrix, resulting in weak bond strength. Water absorption was increased in composite specimens (ERG4) with higher filler content. Water absorption was enhanced in composite specimens (ERG4) after 45 days of immersion. The untreated specimen absorbs water up to 17% of its weight, while the treated specimen absorbs water up to 15% of its weight. Silane treatment can lower the hydrophilicity of the GSP filler by reducing the hydroxyl groups in the molecules, leading to improved interfacial adhesion and, as a result, a decrease in the water absorption of the composites. The greater wetting of the composite with the silane-treated GSP filler in the epoxy matrix, where the epoxy blocks the channels inside the fibres and water cannot diffuse through it, is in good agreement with the studies of Kushawaha and Kumar [22].

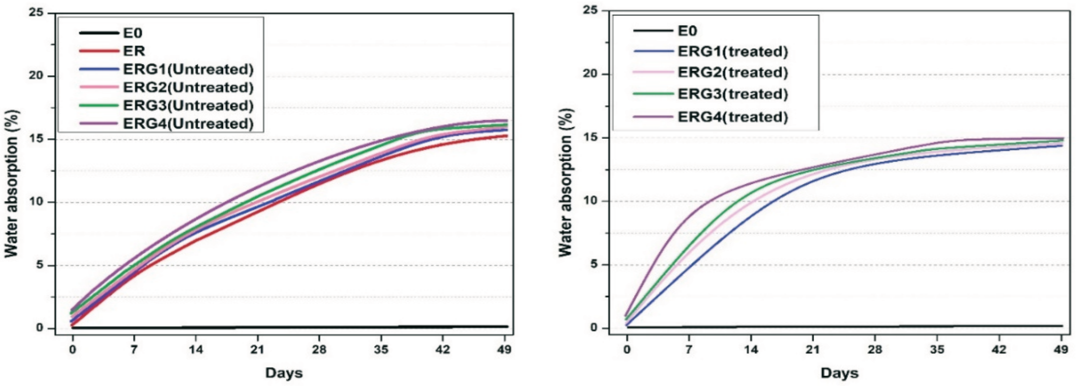


Fig. 7 Water absorption test for all untreated and treated composite specimens vs pure epoxy specimen

Table 3 Water absorption test readings for all untreated and treated composite specimens vs pure epoxy specimen

Composite	**Water absorption, %**	
	Mean Value	**Standard Deviation**
E0	0	0
ER	15.21	0.41
ERG1	15.71	0.35
ERG1 (Treated)	15.00	0.44
ERG2	16.00	0.47
ERG2 (Treated)	14.86	0.43
ERG3	16.15	0.39
ERG3 (Treated)	14.58	0.42
ERG4	16.50	0.44
ERG4 (Treated)	14.29	0.41

5. Conclusions

The major goal of this research was to create a composite out of agricultural waste such as groundnut shell. The following are the key findings of this study:

- A composite made of epoxy and ramie fibre reinforced with groundnut shell powder particles was successfully fabricated using a simple hand layup approach.
- Mechanical strength measurements revealed that increasing the load of the treated filler in the composites resulted in considerable improvements. Compared to specimens with untreated groundnut shell powder, the specimens with the silane-treated groundnut shell powder (with varied weight proportions of 1 %, 3 %, 5 %, and 7 % wt.) exhibited improved mechanical parameters, such as tensile strength, flexural strength, impact toughness, and micro-hardness. A composite made of ramie fibre reinforced with 5 wt. % silane-treated GSP particles and an epoxy matrix possesses the best mechanical properties.
- The thermal stability of all composites increased as a consequence of the GSP filler being added to the epoxy matrix, and better improvement was made by increasing the filler quantity.
- The silane-treated GSP contributed to an improvement in wear resistance of the composite specimens ERG1, ERG2, ERG3 and ERG4 over the specimens with the untreated GSP.

- Due to the alkali and silane treatments, the hydrophilicity of the filler has been reduced, resulting in a better water absorption resistance of the composites. Water absorption was enhanced in composite specimens (ERG4) after 45 days of immersion. The untreated specimen absorbs water up to 17% of its weight, while the treated specimen absorbs water up to 15% of its weight.

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