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Effect of Silane Concentrations in Hardener on the Mechanical Properties of Jute/Epoxy Composites

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ABSTRACT

Natural fibre-reinforced composites are sustainable and biodegradable materials that have potential in industry. However, hydrophilic natural fibres have issues with compatibility with hydrophobic matrix, causing poor mechanical properties. The application of silane coupling agents on fibre reinforcement has been well documented in the literature to enhance fibre/matrix interface properties in composites. In this paper, we investigated the effects of varying silane modifications of woven jute/epoxy composites in the hardener approach. The experimental works began with applying different concentrations of (3-aminopropyl) triethoxysilane (APTES); 1.5, 3.0, and 4.5 wt.% into the amine hardener. The composites were fabricated using the resin infusion technique. The panels were tested for moisture absorption, flexural properties and fracture toughness. The moisture absorption study reveals that the silane addition to jute/epoxy composites results in higher moisture content (3.51%-3.67%) after 5 days of immersion. The diffusion coefficient, *D* was calculated, and the value consistently decreases with increasing silane concentrations from the highest 2.65 x 10^{-7} mm^2/s to the lowest 1.937 x 10^{-7} mm^2/s. All silane-treated composites resulted in remarkable degradation in flexural strength and flexural modulus. The flexural strength and flexural modulus reduction were 16.3% - 39.2% and 4% - 15%, respectively. In contrast, the fracture toughness of composites increased significantly from 4.1 MPa√m to 6.2 MPa√m after 1.5 wt.% silane modifications in the hardener. It was found that increasing concentrations of silane do not necessarily enhance the fracture toughness of composites. Except for the improved fracture toughness, the obtained results indicated that the use of silane in the hardener approach has a negative effect on the moisture uptake resistance and flexural properties, which may be attributed to the ineffective fibre/matrix interfacial bonding.

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INTRODUCTION

Natural fibres have been integral to human civilisation, and their use has surged in research and industry due to their cost-effectiveness, lightweight nature, renewability, and lower energy consumption. Sourced from agricultural waste like banana, coir, and rice husk, these fibres, including jute, flax, ramie, and hemp, offer numerous benefits over synthetic alternatives as reinforcement in polymer composites. Jute (Corchorus olitorius L.) stands out for its favourable mechanical properties, energy absorption capacity and decent fire resistance, making it essential for applications in industrial and sustainable uses (Chourasia et al., 2025). Belonging to the Liliaceae family, jute fibre mainly consists of 61% - 73% cellulose, 13.6% - 23% hemicellulose, and 12% - 16% lignin, along with small amounts of pectin, fats and waxes in their internal structures (Flores et al., 2024).

Thermosetting-based polymers, such as epoxy resin, are often chosen as the matrix material in the fabrication of fibre-reinforced polymer (FRP) composites due to their excellent mechanical and electrical performances. The mechanical performance of an FRP composite is influenced by several factors such as the strength of fibre and matrix, fibre orientation, fibre volume fractions and the interfacial bonding between fibre and matrix. Natural fibres are hydrophilic lignocellulosic materials, making them incompatible with the most hydrophobic non-polar thermoset materials such as epoxy resin. As a result, poor surface adhesion properties, which are caused by insufficient wetting, are the main reason for weak stress transfer in natural fibre polymer composites, limiting the application in demanding applications (Sepe et al., 2018).

Weak fibre/matrix interface adhesions can be improved by modifying the natural fibre surfaces, such as alkaline, silane, ultraviolet, acetylation, and benzoylation (Varma & Chandran, 2025). Research shows that the fibres treated with these chemical treatments increased their mechanical properties and reduced moisture content. A coupling agent is a chemical process where the improvement of fibre/matrix interfacial adhesion and compatibility is achieved by creating a chemical bridge between the reinforcement and the matrix (Xie et al., 2010). In one method, natural fibres were treated directly with silane solutions such as aminopropyltriethoxysilane (APS) and (3-aminopropyl) triethoxysilane (APTES). The established literature presented that the overall mechanical properties, moisture uptake resistance and thermal properties of natural composites were significantly improved.

In another approach, silane coupling agent modification in epoxy resins was reported to modify their mechanical properties, thermal stability and flame resistance. In their publications, Chruściel & Leśniak (2015) stated that adding silane modification to epoxy resin has beneficial effects on decreasing the viscosity of the epoxy system, and increasing impact strength with no adverse effect on the mechanical and thermal properties of the epoxy resins. Wang et al. (2012) showed that the adhesion properties of silane-modified epoxy polymer had higher lap shear strength than unmodified epoxy polymer. In the case of natural fibre composites, Michelena et al. (2017) explored the use of 3-(trimethoxysilyl)propylamine silane modification with 1.5 wt.% concentrations in hardener for flax/epoxy composites. They reported that the longitudinal and transverse properties of silane-modified composites were higher than the untreated composites. The study has demonstrated that silane modification in hardener can eliminate the need for direct chemical pre-treatment of fibres and produce composites with optimum mechanical properties. In similar studies, Rajan et al. (2018) investigated the γ-aminopropyltriethoxysilane (APTES) coupling agents into epoxy resins in the case of viscose fabric/epoxy composites. Their findings indicated that APTES silane-modified composites with 2 wt.% and 5 wt.% exhibit improved tensile strength compared to untreated composites.

While silane coupling agents have shown promise in enhancing this fibre/matrix interface in the literature, their effectiveness in resin modification needs further exploration in the case of jute fibre composites for developing high-performance and sustainable composites for various industrial applications. Therefore, this research aims to investigate the impact of adding silane coupling agents in the hardener

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before composite fabrications. Silane-modified jute/epoxy composites with varying silane concentrations were also determined.

METHODOLOGY

Materials

The reinforcing materials, woven jute fibre were purchased from a local online store. The ply thickness of the fabric was between 0.60 mm to 0.9 mm. The fabric was cut into 290 mm (warp direction) x 190 mm (weft direction) sheets before composite fabrications. A low-viscosity, two-part resin system of Bisphenol A diglycidyl ether (DGEBA) epoxy resin (Miracast 1517A) and amine-curing hardener (Miracast 1517B) that cured at room temperature was supplied by Miracon Sdn Bhd. The silane coupling agent used in this study was (3-aminopropyl) triethoxysilane (APTES) provided by Sigma Aldrich.

Preparation of Silane Modification in Epoxy Resin Systems

The composites were prepared in three different silane concentrations in the hardener by weight percentages (wt.%): 1.5 wt.%, 3.0 wt.%, and 4.5 wt.%, respectively. The process began with adding silane liquid into the hardener in a plastic cup and then stirring thoroughly for about 3 minutes with a glass rod. The epoxy resin was later mixed with the hardener/silane mixtures by a weight content ratio of 100:30 and stirred in the same manner until homogeneous mixtures were achieved. The mixture was placed into a degassing chamber to eliminate air bubbles for about 4 minutes and was ready for the resin infusion process.

Composites Fabrications

The resin infusion method was employed to produce composite panels using a stainless-steel mould. The mould was prepared by applying tacky tape along its edges and lined with peel-ply sheets to facilitate easy removal of the composite later. Four (4) layers of jute fibre laminates were stacked on top of the peel ply and then covered with another layer to ease the peeling process at the end. To ensure even distribution of the resin, a resin floor mesh was placed on top of the peel ply. Each layer was securely sealed with tape to maintain the position of the layers. PVC spiral tubes were positioned at the top of the layers as inlets, and PVC tubes were placed at the bottom of the layers as outlets. The entire setup was then covered with a vacuum bag sealed with tacky tape. Epoxy resin was infused in the laminates with the assistance of a vacuum pump. The resin infusion process is finished when all the fibre laminates are completely soaked with epoxy resin. The composite laminates were cured for 24 hours at room temperature. The composite plates were 3.2–3.6 mm thick. As tabulated in Table 1, the fabricated composite laminates were designated as JE (without silane modification in the hardener), JES 1.5 (1.5 wt.% silane modification in the hardener), JES 3.0 (3.0 wt.% silane modification in the hardener) and JES 4.5 (4.5 wt.% silane modification in the hardener).

Table 1. Details of modification and designations of composites

Silane	Description	Designation
0 wt.%	Without silane modification in the hardener	JE
1.5 wt.%		JES 1.5
3.0 wt.%	With silane modification in the hardener	JES 3.0
4.5 wt.%		JES 4.5

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Testing Procedures

Moisture absorption test

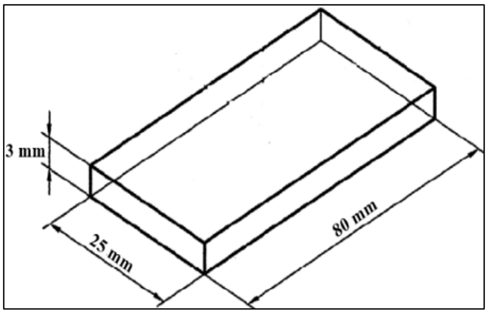
The amount of moisture uptake is tested using this test. The samples were prepared as per ASTM D570 standard requirements. The samples were cut into dimensions of 80 mm in length and 25 mm in width, as shown in Fig. 1(a). The initial weight of each sample was recorded before immersion in distilled water. Subsequently, the samples were removed from the water, wiped with tissue paper, and weighed using a high-precision analytical balance. This procedure was repeated at regular 24-hour intervals for one week. Fig. 1(b) shows the setup for the moisture absorption test. Three (3) samples were tested for each configuration, and the average moisture uptake was recorded. The amount of moisture absorbed in a specimen is calculated using Equation 1.

$$M_t(\%) = \frac{(w_0 - w_t) \times 100}{w_0} \tag{1}$$

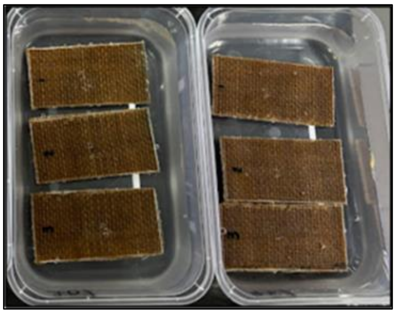
The weight gain from water absorption can also be expressed by the diffusion coefficient, *D*. The *D* was computed by the following equations (Dhanunjayarao et al., 2022):

$$\frac{M_t}{M_{eq}} = 4\left(\frac{D}{\pi h^2}\right)^{1/2} \tag{2}$$

where M_{eq} is the equilibrium of moisture content, M_t is the moisture content at time, t and h is the sample thickness.



(a)



(b)

Fig. 1. Woven jute/epoxy composites (a) sample geometry and (b) sample undergoing moisture absorption testing.

Flexural test

The flexural properties of jute/epoxy composites were determined using the ASTM D790 method. A three-point bending force was applied to the samples with a support span of 60 mm, allowing for a 10% tolerance of the support span. Composite panels were cut to dimensions of 80 mm in length, 12.7 mm in width and 3 mm in thickness as shown in Fig 2(a). Flexural composites were tested on the universal testing machine equipped with a 10 kN load cell at room temperature. The crosshead speed was maintained at 2 mm/min from the beginning of the test up to failure. Five (5) specimens were tested in each configuration, and the mean values of the flexural strength, flexural modulus and flexural strain were reported. Fig 2(b) shows the setup for the flexural test.

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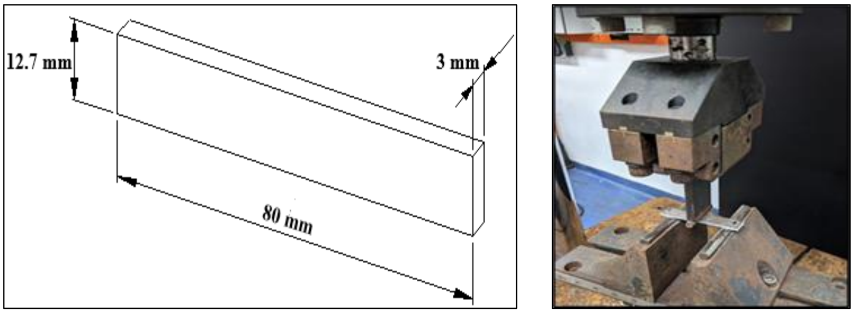


(a) (b)

Fig. 2. Woven jute/epoxy composites (a) sample geometry and (b) sample undergoing flexural testing.

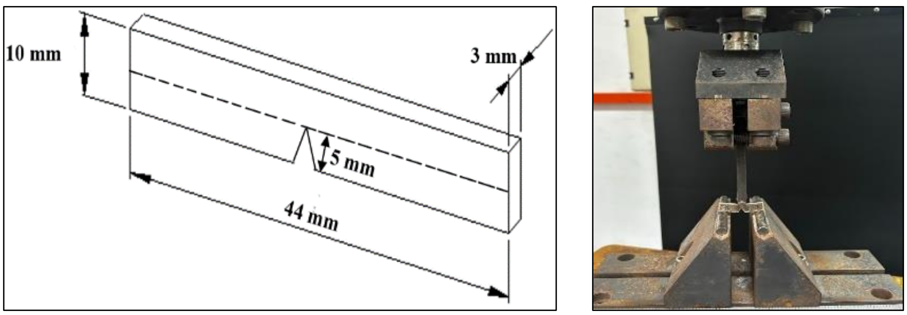
Fracture toughness test

The fracture toughness, K_{IC} of jute/epoxy composites was determined by 3-point bending configurations using single-edge notch bend (SENB). Composite panels were cut into a dimension of 44 mm (length) x 10 mm (height) following ASTM D5045. A notch with a depth of 5 mm at the halfway point of the sample's length of the SENB specimens was machined using a specimen notcher machine, as shown in Fig. 3(a). The tests were conducted under displacement control mode with displacement rates of 1 mm/min using a UTS machine equipped with a 10kN load cell, as shown in Fig. 3(b). An average of 5 specimens were tested for each configuration. The K_{IC} values were determined using the following relationship recommended by the ASTM testing standard:

$$K_{IC} = \left(\frac{P_Q}{BW^{1/2}}\right) f(x), \quad W = 2B \tag{3}$$

$$f(x) = 6x \frac{[1.99 - x(1-x)(2.15 - 3.93x + 2.7x^2)]}{(1+2x)(1-x)^{3/2}}, \quad x = a/W \tag{4}$$

where K_{IC} is a fracture toughness, $f(x)$ the shape factor, P_Q the peak load, B the specimen thickness, W the specimen width, W the crack length.



(a) (b)

Fig. 3. Jute/epoxy composites (a) sample geometry and (b) sample undergoing fracture toughness testing.

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RESULTS AND DISCUSSIONS

Moisture Absorption Behaviour

Fig. 4 illustrates the moisture content of various samples over a five-day immersion period for all composites. Overall, the data indicates a consistent increase in moisture content across all samples over the five days. The data also showed that the samples that were not silane-modified, JE exhibited the lowest moisture content at 3.0%. In contrast, the silane applications in the resin samples such as JES 1.5, JES 3.0 and JES 4.5 absorbed higher moisture at around 3.5%, an increase of ~0.5% by the time of 5 days of immersion time. It is known that surface modifications with silane in the fibre have produced composites with less hydrophilic and consequently, less moisture uptake in the water absorption test as shown in previous research (Dessie et al., 2022). In fibre modification, silane plays a crucial role as a water repellent by effectively altering the surface properties of cell walls. Silanization of fibres creates strong hydrogen and covalent bonds between silanol (Si-OH) groups and the hydroxyl groups of the fibres. This process reduces interactions between the hydroxyl group and water molecules (Xie et al., 2010). In the present investigations, however, silane modifications in the resin have worsened the water uptake in the composites as shown in Fig. 4. The possible explanation is that higher moisture uptake is due to a less effective interface between the fibre and the matrix (Islam et al., 2024). A weak fibre/matrix interface might introduce defects such as micro-cracks or hairline cracks, void content and gaps (Ghori et al., 2022). Consequently, moisture could infiltrate and occupy these defects.

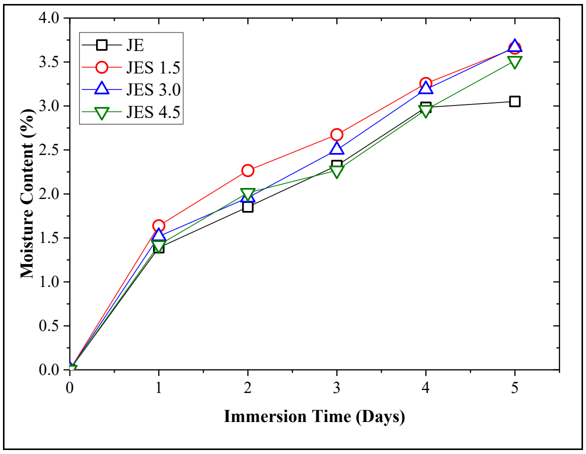

Fig. 4. Moisture content against immersion time.

As shown in the curves in Fig. 4, the water uptake in jute/epoxy composites decreases with increasing silane concentration. The JES 4.5 plot shows the lowest moisture content across immersion time in days between 1.38% and 3.51%, while the JES 1.5 has the highest between 1.64% and 3.67%. Therefore, the data suggest that the moisture uptake improves with higher silane additions across the immersion time. A possible explanation for this phenomenon is that the silane treatment has begun to initiate a chemical reaction at the fibre surface when applied at higher concentrations, especially with a 3.0% concentration of silane. To support this argument, a diffusion coefficient, *D*, was calculated for every composite. The diffusion coefficient describes the ability of solvent molecules to move among the polymer segments (Kumar et al., 2022). In natural fibre reinforced polymer composites, moisture absorption mainly occurs through hygroscopic natural fibres rather than factors like the fibre-matrix interface and the matrix polymer. Many recent investigations have shown that the diffusion coefficient was lower for a silane treated on the fibre of composites compared to the untreated composites (Kusmono et al., 2020; Bollino et al., 2023;

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Chandekar et al., 2020). The comparison of the diffusion coefficient, *D* between the composites is illustrated in Fig. 5. It is seen that the *D* consistently decreases with increasing silane concentrations in the following orders: JE>JES 1.5>JES 3.0>JES 4.5. The values range from the highest 2.65 x 10^{-7} mm^2/s to the lowest 1.937 x 10^{-7} mm^2/s. This indicates that the hydrophilic (polar) end of the silane interacts with the surfaces of fibres. As silane concentration increases, the jute fibres gain a thicker water-repellent layer, resulting in a lower diffusion coefficient.

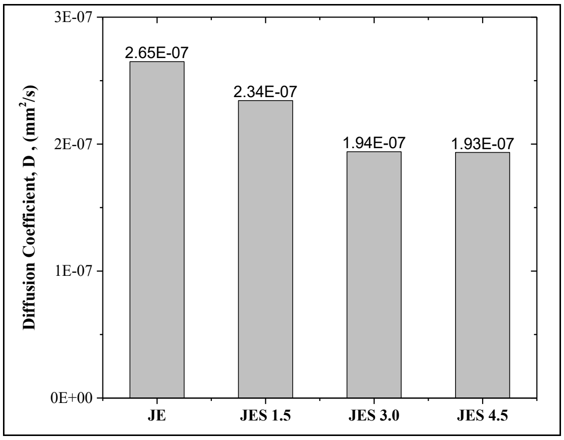

Fig. 5. Diffusion coefficient, *D* of jute/epoxy composites.

Flexural Properties

Fig. 6 displays the typical flexural stress-strain for the composites with and without silane modifications in the resin. The curves indicated that the overall tendency of the flexural stress-strain curves was not affected by the silane modifications. The curves exhibit a linear response, allowing measurement of flexural modulus within 0.1% - 0.5% in the flexural strain. The curves continued to follow a linear trend, followed by a sudden drop in flexural stress. This behaviour indicates that the composite had failed in brittle fracture. The curve showed that the JE had failed at the highest peak flexural stress with elongated flexural strain. Meanwhile, the silane-modified composites had experienced lower peak strength and strain values, suggesting the early onset of cracking.

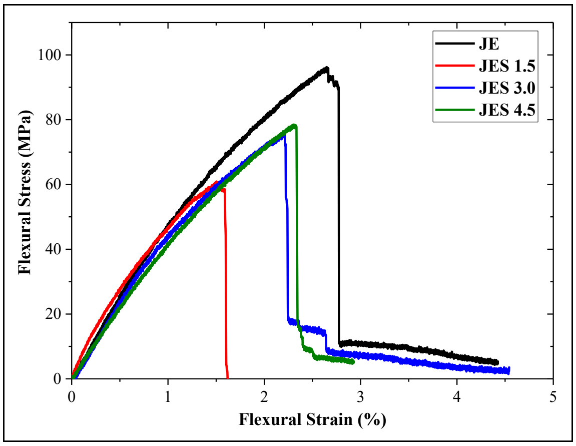

Fig. 6. Typical flexural stress-strain plot for jute/epoxy composites.

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The flexural strength and strength retention of the control group and silane-modified in the resin of jute/epoxy composites are presented in Fig. 7(a) and Fig. 7(b). The results show that the JE sample exhibits the highest flexural strength at 92.88 MPa, indicating superior performance in bending resistance. The bar charts show a clear indication that silane modification in the hardener causes a reduction in flexural strength between 20.1% and 39.2%. Interestingly, there is an improvement in flexural strength due to an increase in the amount of silane in the hardener. The silane modifications with 1.5 wt.%, 3.0 wt.%, and 4.5 wt.% have resulted in an increased flexural strength of 58.0 MPa, 74.7 MPa, and 79.9 MPa.

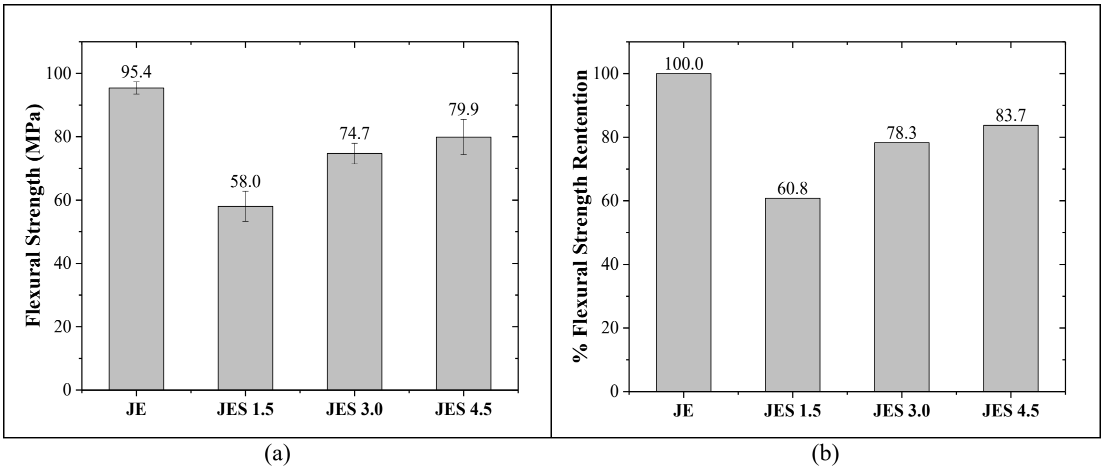

Fig. 7. Effect of silane modification in the hardener on (a) flexural strength and (b) % flexural strength retention.

Fig. 8(a) and Fig. 8(b) illustrates the flexural modulus and flexural modulus retention of control silane-modified in the hardener of jute/epoxy composites. Flexural modulus measures the material's stiffness during bending. The silane modifications also lead to degradation in the flexural modulus. Unlike flexural strength, it is interesting to observe that an increase in silane modification of the hardener has led to a decrease in the flexural modulus from 58.9 MPa to 52.1 MPa. From Fig. 7(b) and Fig. 8(b), it is clear that the degradation of flexural strength is more severe than that of flexural modulus; 39.2% for the former and 15.8% for the latter. It is not surprising because the flexural modulus is less sensitive to interfacial defects (Betanzos et al., 2016). The data on flexural properties indicates that silane applications at 1.5 wt.%, 3.0 wt.%, and 4.5 wt.% have negatively affected the fibre-matrix adhesion strength. This has caused deficiencies in load transfer bearing and resulted in lower flexural properties of jute/epoxy composites. The findings in this investigation contradict those of Michelena et al. (2017) and Michelena et al. (2022) which reported that stronger flax/epoxy composite structures were obtained with 1.5 wt.% silane treatments in matrix resins before composite fabrications. The likely reason is that the chemical link between the fibre surface and the epoxy matrix via a siloxane bridge is either absent or insufficient due to the silanization process in the matrix resins.

Fracture Toughness Properties

Fig. 9 illustrates the typical SENB load-extension traces showing the crack growth behaviour of jute/epoxy composites during the fracture toughness test. The jute/epoxy composites display a proportional increase in load with extension, indicating elastic deformation along the traces. The fracture mode of the composites is brittle, exhibiting slip-stick behaviour once a peak height is reached. It is interesting to note that, for all silane concentrations applied, the SENB load-extension curves display an increase in stiffness (slope of the initial linear part of the curve) over the control samples. An increase in the slope of SEB load-extension curves may be attributed to the weakened fibre/matrix interfacial bond, which is similarly

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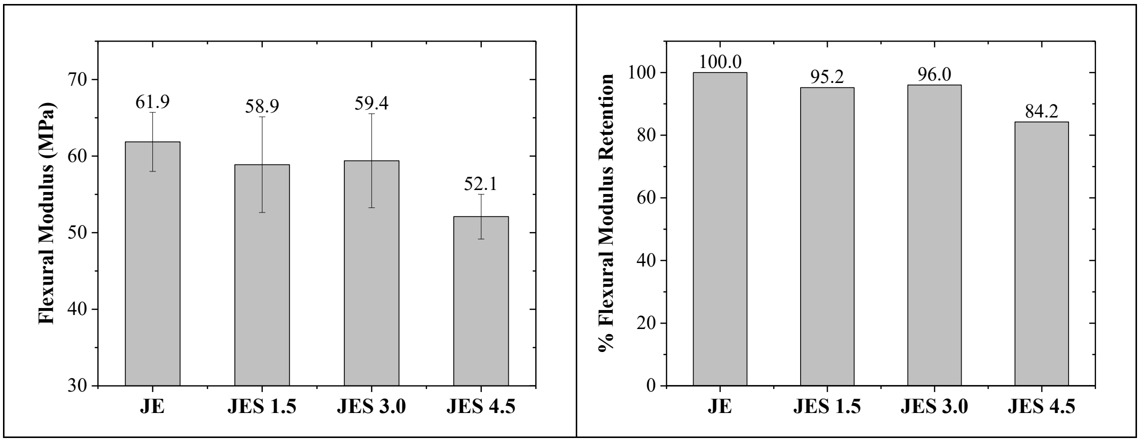
described by Deng et al. (2021). The load-extension curves of the SENB tests can effectively characterize material toughness. Tough materials require a significant amount of load to induce a complete fracture. This occurrence is evident as the silane-modified resin samples show a higher peak load than the control sample (JE).



(a) (b)

Fig. 8. Effect of silane modification in the hardener on (a) flexural modulus and (b) % flexural modulus retention.

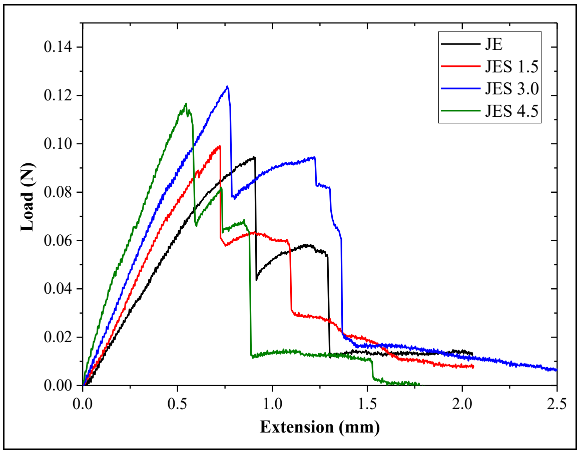


Fig. 9. Typical SENB Load-Extension plot for jute/epoxy composites.

Fracture toughness, K_{IC} is a measure of a material's ability to resist crack propagation, and higher K_{IC} indicate better toughness resistance of a material. Fig. 10(a) compares the fracture toughness with respect to silane content in the resin of jute/epoxy composites. It is worth noting that the fracture toughness of silane-modified composites was higher with the addition of silane in the hardener as a secondary treatment. The fracture toughness of the untreated JE composite is the lowest, presenting an average of 4.1 MPa√m, whereas the fracture toughness of the silane-modified JES 1.5 specimen has risen to 6.2 MPa√m. Nevertheless, increasing the silane concentration may not always result in better resistance to crack propagation. The fracture toughness of JES 3.0 and JES 4.5 specimens attained values between 5.3 MPa√m and 6.1 MPa√m, respectively. Fig. 10(b) illustrates the % fracture toughness retention of jute/epoxy composites. The silane modification in the hardener with 1.5 wt.%, 3.0 wt.% and 4.5 wt.% have shown

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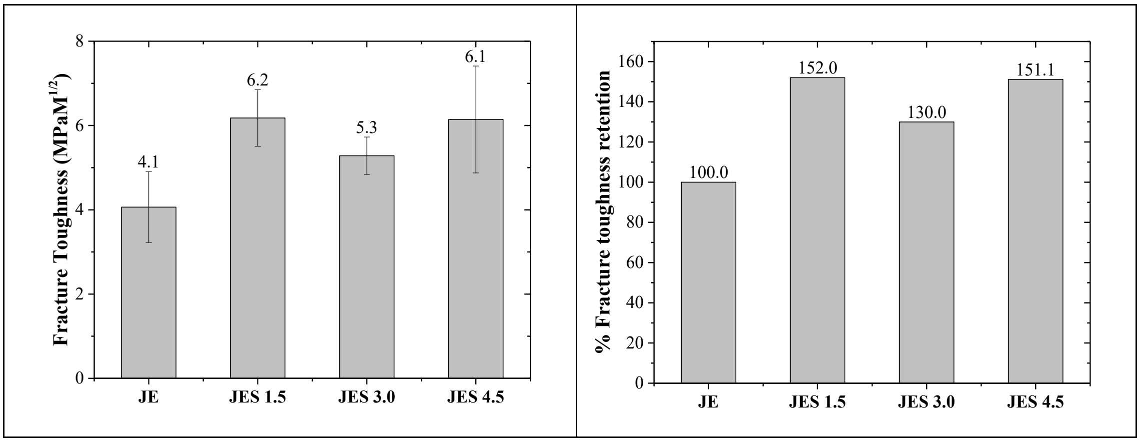
beneficial effects in improving fracture toughness. The fracture toughness of silane-treated jute/epoxy composites increases by as much as 52% compared to the JE specimen.



(a) (b)

Fig. 10. Effect of silane modification in the resin on jute/epoxy composites on (a) fracture toughness and (b) % fracture toughness retention.

It is reported in the works of literature that surface treatments on natural fibres cause inferior fracture toughness in composites due to enhanced interfacial bonding (Pickering et al., 2011; Kumar et al., 2024; Abdullah et al., 2016; Silva et al., 2006). The results of this investigation may imply that, contrary to expectations, the silane coupling agent did not enhance interfacial adhesion between the jute fibres and the epoxy matrix, as evidenced previously by poor flexural properties (Fig. 7 and Fig. 8). This would mean that silane application in the hardener methods appears ineffective at creating a chemical bridge between the reinforcement and the matrix. The increase in fracture toughness can be attributed to the debonding of fibres from the matrix, allowing for additional energy absorption during crack propagation (Pinto et al., 2014). Epoxy resin is a highly crosslinked polymer known for its brittleness, poor impact resistance and low fracture toughness (Chruściel & Leśniak, 2015). The silane modification in the hardener may toughen the cured epoxy polymer resin, which might be one possible explanation for the additional increment in its fracture energy. These results reflect those of Li and Xie (2009) who also claimed there is an improvement in the toughness of neat epoxy cured with silane as a liquid epoxy curing agent.

CONCLUSION

The current study aimed to determine the effect of silane coupling agent concentrations in the hardener (1.5 wt.%, 3.0 wt.%, and 4.5 wt.%) on the moisture content, flexural properties and fracture toughness of jute/epoxy composites. It was found that water uptake was higher in those silane-modified composites than in the control specimen in the five-day immersion experiment. Water content decreased as silane concentrations increased from 1.5 wt.% to 4.5 wt.%. The lower diffusion coefficient, *D* was found on specimens with higher silane modification from 2.65 x 10^{-7} mm^2/s to 1.937 x 10^{-7} mm^2/s. The jute/epoxy composite exhibited reduced flexural strength and flexural modulus following the silane modification in the hardener. While silane treatment negatively affected the moisture content and flexural properties of jute/epoxy composites, it significantly improved the fracture toughness as compared to untreated samples, with an improvement of 51%. The higher moisture intake, poor flexural properties, an increased fracture toughness directly result from ineffective silane interaction between the fibre reinforcement and the epoxy

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matrix, leading to inevitable fibre/matrix debonding. This study offers valuable insights for developing sustainable high-performance composites for industrial applications, highlighting the importance of carefully considering silane treatment processes to achieve the desired material properties.

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CONFLICT OF INTEREST STATEMENT

The authors agree that this research was conducted in the absence of any self-benefits, commercial or financial conflicts and declare the absence of conflicting interests with the funders.

AUTHORS' CONTRIBUTIONS

The authors confirm their contribution to the paper as follows: Manuscripts Preparation: Abdul Hakim Abdullah, Anis Zahrina Azwa, Nor Fazli Adull Manan. Data Collection: Abdul Hakim Abdullah, Anis Zahrina Azwa, Izdihar Tharazi. Result Analysis and Interpretation: Abdul Hakim Abdullah, Nor Fazli Adull Manan.

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