ALCHEMY Jurnal Penelitian Kimia

Official Website: https://jurnal.uns.ac.id/alchemy

ISSN 1412-4092
EISSN 2443-4183

Improved Physico-Mechanical Properties and Microstructure of Modified Kenaf Fiber Reinforced Composites

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DOI: 10.20961/alchemy.21.1.92620.64-71

Received 25 August 2024, Revised 22 October 2024, Accepted 8 November 2024, Published 28 March 2025

Keywords:

composite; kenaf fiber; propionic anhydride; vinyl ester; surface modification.

ABSTRACT. This study examined the properties of chemically modified kenaf fiber using propionic anhydride as reinforcement in vinyl ester composites. The composites reinforced by unmodified and modified kenaf fibers were fabricated using 10, 20, 30, 40, and 50% (w/w) via a hand lay-up technique. This study's characterization included water absorption, thickness swelling, tensile and flexural strengths, and morphological analyses. The optimum physical, mechanical, and morphological properties were observed from the composite with 40% modified kenaf fiber. The optimized composite showed a considerable enhancement in the water absorption, tensile strength, and flexural strength of 1.82%, 54.90 MPa, and 55.85 MPa, respectively. These findings were supported by the fracture morphology after the mechanical test, exhibiting an enhanced interfacial modified kenaf fiber-matrix bonding. The improved characteristics of the composite were ascribed to the impact of the modification process within the fiber-matrix structures. Modified kenaf fiber via propionylation can be applied as a reinforcement material in the vinyl ester matrix.

INTRODUCTION

Kenaf fiber, a biofiber, has been widely utilized as an alternative to fossil-based fiber. It has inherent benefits and unique features, including low density with good strength and high thermal stability, which make it a favorable lignocellulosic bioresource for reinforcing agents in biocomposite materials (Sapiai *et al*., 2020). Since biofiber is renewable, biodegradable, and non-toxic, it is increasingly prevalent in various industrial applications. The development of kenaf fiber as a reinforcing material in composite applications has been extensively studied (Yusuff *et al*., 2020). However, the inherent hydrophilicity of kenaf fiber presents a primary drawback when used as a reinforcing agent (Prakash and Viswanathan, 2019). Combining hydrophilic kenaf fiber with hydrophobic matrix results in poor functional characteristics in composites, which is a novel challenge.

The inherent moisture absorption behavior of kenaf fiber presents a disadvantage as a reinforcing agent (Venkatasudhahar *et al*., 2021). It remains a challenge to overcome the weak functional features of biocomposite materials caused by hydrophobic matrices with hydrophilic biofiber. Enhancing the chemistry and structure of cell walls through surface modification of biofiber can increase the functional characteristics of biocomposites (Birnin-Yauri *et al*., 2017). In an earlier study, Cavdar *et al*. (2014) examined the impact of using propionic anhydride to modify aspen wood flour in a chemical modification on the composite network. The strength features of the composites fabricated by surface modification with the anhydride showed a discernible improvement. Compared to the unmodified composite, the modulus of the composite increased by 23% due to the propionylation on wood flour.

Furthermore, Gallardo-Cervantes *et al*. (2021) reported that the compatibility of biocomposites was enhanced through the chemical modification of agave biofiber using propionic anhydride. When comparing propionylated fiber biocomposites to unmodified biocomposites, the modified fiber's flexural strength and water barrier increased by 35% and 170%, respectively. In a previous study, the propionic anhydride modification on kenaf

Cite this as: Mistar, E. M., and Muhammad, M., Improved Physico-Mechanical Properties and Microstructure of Modified Kenaf Fiber Reinforced Composites, 2025. *ALCHEMY Jurnal Penelitian Kimia*, 21(1), 64-71. https://dx.doi.org/10.20961/alchemy.21.1.92620.64-71.

fiber with the introduction of bio-nanocarbon in the vinyl ester resulted in a significant improvement in tensile strength (63.91%) and flexural strength (49.61%) (Rizal *et al*., 2021).

Therefore, it is pivotal to modify kenaf fiber with propionic anhydride to increase its hydrophobicity and compatibility with the vinyl ester matrix, thereby improving the functional qualities of the biocomposites. The present work aimed to prepare and characterize the composite fabricated by adding unmodified and modified propionic anhydride kenaf fibers to vinyl ester with varying fiber loadings. Characterization of the obtained composite examines the functional properties, including the mechanical, physical, and morphological behaviors, to observe the impact of the chemical modification on the composite's interaction.

RESEARCH METHODS

Materials and Chemicals

Kenaf fiber mats utilized in this research were sourced from Lamongan, East Java Province, Indonesia. The fibers underwent chemical modification using propionic anhydride, a method that has been detailed in previous work (Mistar and Muhammad, 2023). For this experiment, kenaf fiber mats measuring 200 mm × 200 mm (length × width) were placed within a Soxhlet extractor. The specimens were then subjected to drying in an oven at 110 °C overnight. Afterward, the dried samples were cooled to room temperature inside a desiccator before being weighed. Five sets of these fiber mats were subsequently immersed in a flask containing a propionic anhydride solution. The modification process was conducted under controlled temperature and time conditions to ensure precision. Post-treatment, the specimens underwent ethanol rinsing for three hours to remove any residual acid from the fiber surface. Following this, the fiber mats were oven-dried once again at 80 °C overnight. In this study, the modified kenaf fiber mats, which demonstrated optimal weight gain and desirable morphological characteristics when processed at 100 °C for 200 minutes, were employed as reinforcement materials. Additionally, vinyl ester and methyl ethyl ketone peroxide (MEKP; $C_8H_{18}O_6$) were procured from a supplier in Aceh Province, Indonesia. Other chemicals, including propionic anhydride ($C_6H_{10}O_3$), acetone (C_3H_6O), toluene (C_7H_8), ethanol (C_2H_5OH), and cobalt naphthenate ($CoC_{22}H_{14}O_4$ 6.0%), were obtained from Sigma Aldrich (St. Louis, MO, USA).

Fabrication of Composites

The prepared composites of vinyl ester reinforced by unmodified or modified kenaf fibers were fabricated through 10, 20, 30, 40, and 50% (w/w) of kenaf fiber loadings and recorded as UK10, UK20, UK30, UK40, UK50, MK10, MK20, MK30, MK40, and MK50, respectively. This study used the MEKP of 1.5 w/w% and cobalt naphthenate of 0.2 w/w% as hardener and accelerator, respectively. The vinyl ester-catalyst-hardener mixture was blended in a plastic container. The mixture was poured onto fractions by weight loading the unmodified or modified kenaf fibers and molded into a flat surface stainless steel mold through a hand lay-up technique. The mold with the mixture was subjected to 8 MPa pressure on the plate of a pressing machine until it was cured at ambient temperature. The prepared composites were stored in a desiccator for further analysis. Both unmodified and modified kenaf fibers reinforced the composites.

Water Absorption and Thickness Swelling Characterizations of Composites

To investigate the impact of water absorption on the composites, the samples were submerged in distilled water at room temperature for 24 hours. The analysis followed the ASTM D570 standard for all tested composite specimens. Each composite type had five square samples prepared to measure the water absorption percentage. The rate of water absorption was calculated by Equation (1).

$$\text{Water absorption (\%)} = \frac{w_2 - w_1}{w_1} \times 100 \quad (1)$$

W_1 is the weight of the composite before immersion, and W_2 is the weight of the composite after immersion.

In this study, ASTM D570 was used to conduct the thickness swelling analysis for all composite samples. Five composite samples were tested to determine the thickness and swelling of each composite. In brief, the samples were soaked in distilled water for 24 hours at room temperature. The thickness of the sample before and after water immersion was recorded. The thickness swelling percentage was determined using Equation (2).

$$\text{Thickness swelling (\%)} = \frac{T_2 - T_1}{T_1} \times 100 \quad (2)$$

T_1 is the thickness of the composite before soaking, and T_2 is the thickness of the composite before soaking.

Tensile and Flexural Properties of Composites

A tensile analysis was conducted to evaluate the mechanical properties of the prepared composites, including tensile strength (TS), tensile modulus (TM), and elongation at break (EAB), following ASTM D638 standards. Rectangular specimens measuring 120 mm × 15 mm × 5.5 mm were precisely cut using a circular saw. The testing procedure employed an INSTRON 5582 universal testing machine (Norwood, MA, USA) equipped with a 10 kN load cell. A total of five specimens were tested, and the resulting data were presented as mean values accompanied by their respective standard deviations.

A flexural test was performed to assess the flexural strength (FS) and flexural modulus (FM) of the composites in accordance with ASTM D790 standards. Rectangular specimens measuring 160 mm × 20 mm × 5.5 mm were prepared for testing. The analysis was conducted using an INSTRON 5582 universal testing machine (Norwood, MA, USA) equipped with a 10 kN load cell. Five composite specimens were tested for each sample type, and the resulting data were recorded as mean values accompanied by their respective standard deviations.

Morphological Analysis of Composites After Tensile Test

The fracture morphology of the composite samples after tensile testing was analyzed using a field emission scanning electron microscope (FESEM, FEI Quanta FEG 650, Thermo Fisher Scientific, Eindhoven, The Netherlands). For specimen preparation, the samples were affixed to aluminum (Al) stub holders using double-sided copper (Cu) tape. To improve the electrical conductivity of the samples, a thin platinum (Pt) coating was applied using a Quorum Technologies Q150T sputter coater.

RESULTS AND DISCUSSION

Water Absorption and Thickness Swelling Characterizations

The water absorption percentage and thickness swelling of the kenaf fiber composites are illustrated in Figure 1. As the fiber content increases, water uptake shows a declining trend. This reduction is attributed to the presence of additional fibers within the composite matrix, which limits water penetration. Moreover, the water absorption characteristics of kenaf fiber-reinforced composites are influenced by factors such as the exposed surface area, void content, and fiber permeability.

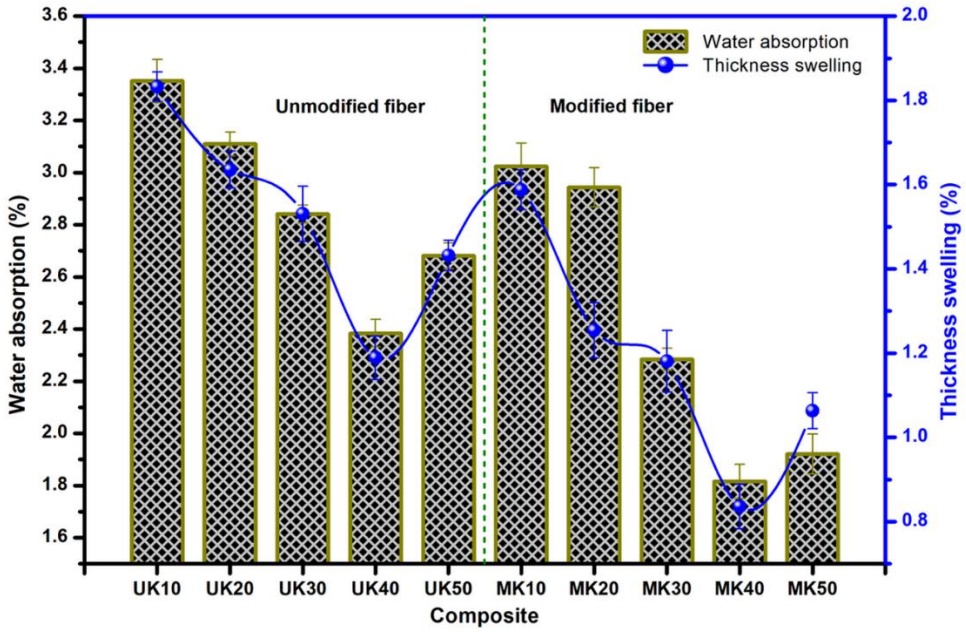

Figure 1. Water absorption and thickness swelling of composites reinforced by unmodified and modified kenaf fiber loadings.

In this study, the 40% kenaf fiber composite showed a lower water uptake. It was probably due to the decrement of available voids in the composite network, which contributed to reducing exposure surface to water

(Alamri and Low, 2012). The obtained results indicated that water absorption of modified composites provided superior water resistance compared to unmodified composites. However, modified composites containing 40% fiber presented the lowest percentage of water absorption at 1.82%. Furthermore, the thickness swelling of the composites exhibited a decreasing trend with increasing kenaf fiber content. This reduction was attributed to the incorporation of kenaf fibers within the matrix network. The most significant reduction in swelling behavior was observed at a fiber loading of 40%, where composites reinforced with unmodified and modified kenaf fibers showed reductions of 1.19% and 0.84%, respectively.

Modifications using propionic anhydride could decrease the microvoids in the fiber-matrix structure and enhance interfacial fiber-matrix adhesion. Similar findings were reported in a previous study, where it was observed that surface modification of kenaf fiber contributed to greater compatibility of the fiber-vinyl ester matrix (Gallardo-Cervantes *et al*., 2021). However, such property improvement decreased by 50% at fiber loading due to agglomeration of the fiber. This might weaken the interfacial bonding between kenaf fiber and vinyl ester, leading to increased water absorption and thickness swelling to the composite structure.

Tensile Analysis

Figure 2 renders the tensile properties of the composites, including TS, TM, and EAB. The tensile features of unmodified and modified fiber-reinforced composites increased by up to 40% of fiber loading. The composite with 40% kenaf fiber loading was the optimum reinforcement level since further incorporation of 50% fiber decreased the tensile behaviors of the composites. This was ascribed to the maximum reinforcing impact given by the optimum volume of fiber fraction.

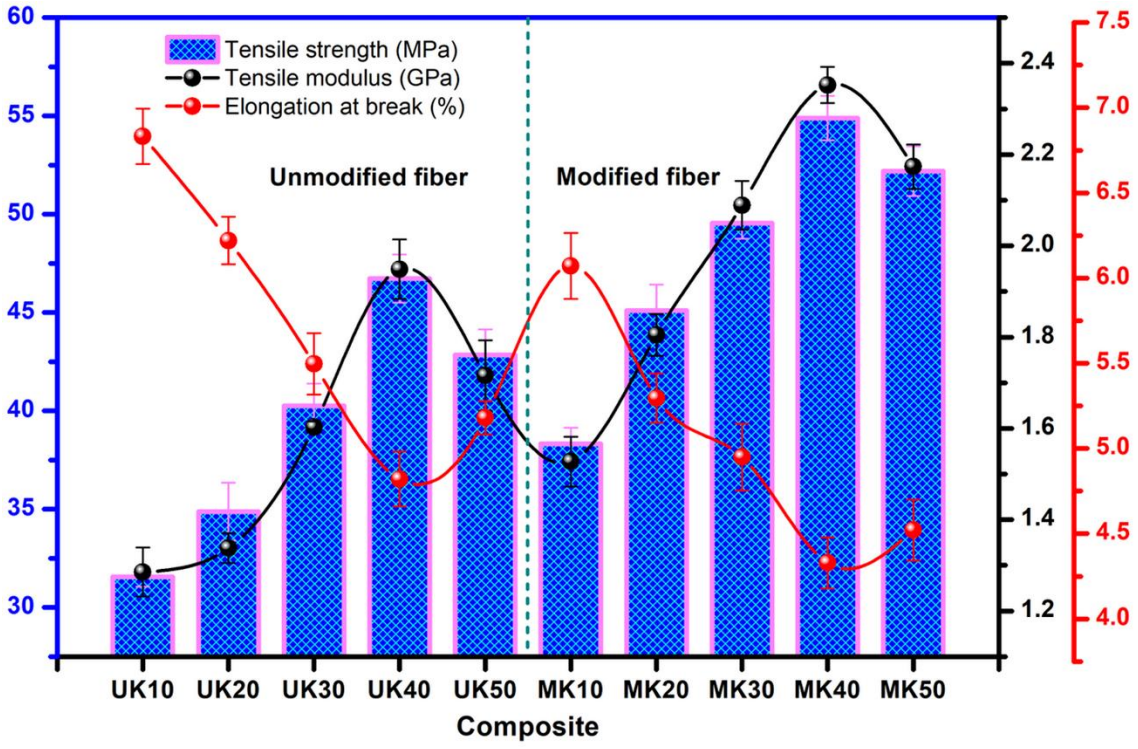

Figure 2. Tensile strength, tensile modulus, and elongation at break of unmodified and modified kenaf fibers reinforced composites at different fiber loadings.

The results showed better tensile behaviors of the composites with modified fiber than unmodified ones. This was probably associated with higher interfacial fiber-matrix bonding as propionic anhydride modification of fibers provides greater mechanical interlocking because of better interfacial biofiber-matrix interaction (Emam and Shaheen, 2019). In this study, the composite having 40% modified fiber provided the optimum tensile strength, tensile modulus, and elongation at a break of 54.90 MPa, 2.35 GPa, and 4.33%, respectively. The obtained tensile results were supported by water barrier and morphology characterizations that showed the optimum properties of composite with 40% kenaf fiber, resulting in better stress transfer between the matrix and reinforcing fiber. These

findings are consistent with an earlier study observing that the stress concentration could be dispersed uniformly through kenaf fiber from the matrix as the chain mobility of the matrix was efficiently resisted (Chin *et al*., 2020).

Morphological Analysis

Figure 3 displays the tensile fracture morphology of unmodified and modified composites at 10%, 40%, and 50% fiber loadings. The compactness of the composites was found to improve with the addition of fiber loading. Figure 3(a) shows that interfacial voids and holes are responsible for the weak interfacial bonding. Figure 3(b) shows the surface morphology of incorporating 10% modified fiber in the vinyl ester composites. As seen in the figure, the effect of propionylation process of the fiber in the matrix structure formed a greater interfacial bonding with lesser microvoids compared to its counterpart at equal fiber loading. Although few available voids were observed in Figure 3(c), incorporating 40% unmodified fiber presented better interfacial bonding in the fiber-matrix structure.

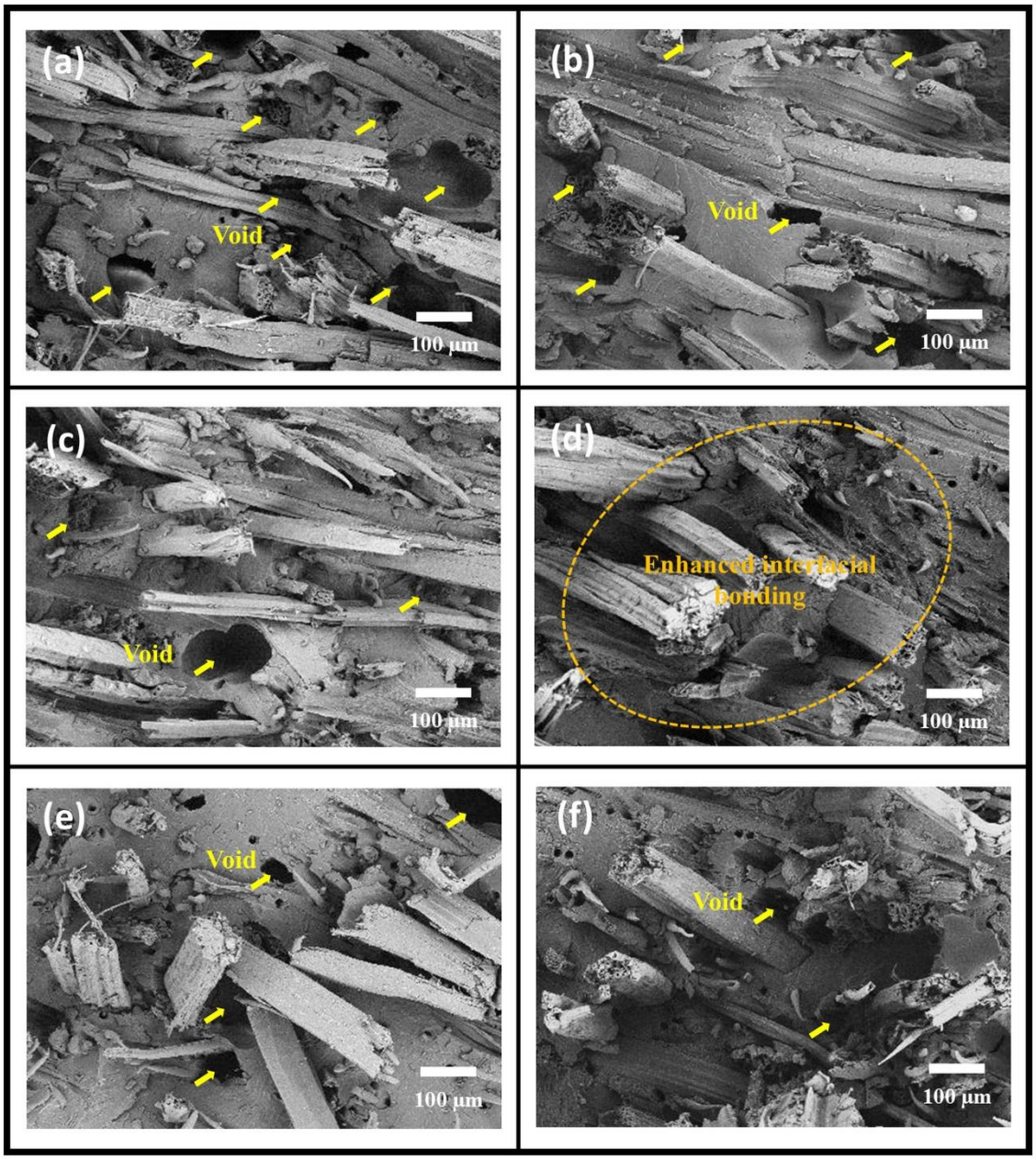

Figure 3. SEM micrographs of the tensile fracture surface of composites reinforced with (a) 10% unmodified fiber, (b) 10% modified fiber, (c) 40% unmodified fiber, (d) 40% modified fiber, and (e) 50% unmodified fiber, and (f) 50% modified fiber.

Interestingly, the well-embedded fibers and enhanced interfacial fiber-matrix bonding by introducing 40% modified fibers are exhibited in Figure 3(d). At the same fiber loading, propionic anhydride modification showed a favorable improvement in interfacial fiber-matrix bonding compared to those without modification. A previous study reported that waxy substances on the fiber are expunged during the modification process, allowing for better interfacial kenaf fiber-matrix interaction (Khalil *et al*., 2013). The anhydride modification improved the bonding, compatibility, and interfacial adhesion behaviors between the hydrophobic matrix and kenaf fiber, contributing to greater ultimate characteristics. Similar findings were also reported in the literature, revealing that high compatibility and miscibility between matrix and fiber enhance the interfacial fiber-matrix interaction (Durmaz *et al*., 2022). However, there was a slight decrement in interfacial interaction of the composite with 50% kenaf fiber loading, as observed in Figure 3(e-f). This was probably due to excessive fiber beyond the optimum loading, leading to twists and bends in the matrix during molding.

Flexural characterization

The results of FS and FM were improved by adding kenaf fiber-reinforced composites, as seen in Figure 4. Similar to tensile properties, modified composites showed higher flexural features than composites without modification. The propionic anhydride-based composites with 40% loading showed the highest FS and FM of 55.85 MPa and 5.72 GPa, respectively. This suggests that an improvement in the compatibility of the fiber-matrix structure was achieved. The flexural behaviors achieved in this work were higher than those observed in the literature (Khan *et al*., 2020).

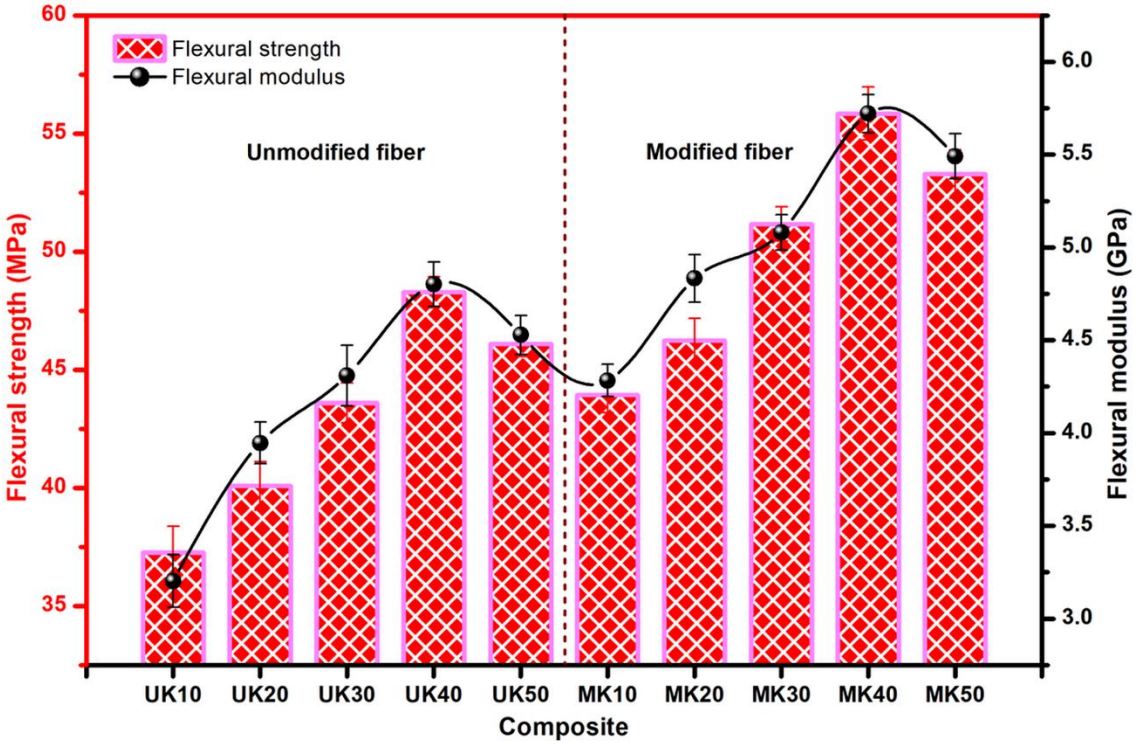

Figure 4. Flexural strength and modulus of unmodified and modified kenaf fibers reinforced composites at different fiber loadings.

A noticeable enhancement in the flexural behaviors of modified composites was probably due to hydrogen bonding within the composite network. Significant interfacial interaction allows good resistance to fiber pullout with less or no rupture, improving composites' flexural characteristics (Oushabi *et al*., 2017). The flexural results were confirmed by tensile, water uptake, and morphology analyses, which observed the superior features of 40% fiber-reinforced composite, leading to greater stress transfer between the kenaf fiber and matrix. However, the excessive loading of biofiber at 50% may lead to bending in the matrix during the molding process, which could affect the composites' strengths (Kumar *et al*., 2021) and rupture the fiber walls and surface. This is responsible for a stress concentration spot, leading to a decrease in the fiber-matrix interphase, which restrains the force to pull them apart.

CONCLUSION

Composites reinforced by unmodified and modified kenaf fiber were successfully fabricated and characterized. The modified fiber improved the characteristics of composites compared to unmodified composites. The enhanced functional properties included physical, mechanical, and morphological features, and the optimum results were observed in the modified composite containing 40% fiber loading. In this study, the optimum properties observed were a water absorption of 1.82% and thickness swelling of 0.84%, while the highest tensile and flexural strengths were 54.90 MPa and 55.85 MPa, respectively. The findings imply that modified kenaf fiber-reinforced vinyl ester composite has good prospects in composite applications, including storage, packaging, furniture, and automobile interiors.

CONFLICT OF INTEREST

There is no conflict of interest in this article.

AUTHOR CONTRIBUTION

EMM: Conceptualization, Methodology, Funding Acquisition, Project Administration, Resources, Supervision, Validation, Visualization, Manuscript Drafting, Manuscript Review, and Editing; MM: Investigation, Tools Software, Data Analysis, Manuscript Drafting.

ACKNOWLEDGEMENT

The authors would like to thank the Ministry of Education, Culture, Research and Technology of the Republic of Indonesia through Penelitian dan Pengabdian Bagi Dosen No: 055/LPPM-USM/VII/2024.

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