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The Impact of Alkaline Treatment On the Mechanical and Physical Properties of Kenaf Core Fibre Reinforced Vinyl Ester Composites

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ABSTRACT

Nowadays, natural fibre has a high demand in any application development. The use of natural fibre in various applications worldwide is one of the alternative ways to replace synthetic fibre. Currently, polymer composites using natural fibre as reinforced materials are trending in the aeronautic and automotive industries. Therefore, this research focuses on the impact of surface modification on kenaf core fibre reinforced vinyl ester composites towards mechanical and physical properties. From the obtained results, the modified fibre has good strength and modulus, and also physical properties, such as water absorption and density. The flexural strength of modified fibre improved to 55.47%, while the modulus increased to 83.87% as compared to the unmodified fibre. For tensile strength, the modified composites improved by 58.02%. The modified fibre shows improved water resistance, as clarified by the water absorption analysis. The surface modification of kenaf core fibre using alkaline treatment achieved good results compared to unmodified fibre in enhancing the interlocking between fibre/matrix, reducing certain chemical content, and reducing the hydrophilicity of the composites.

Keywords: Alkaline treatment, kenaf fibres, mechanical properties, polymer matrix composites

1. INTRODUCTION

In this modern world, Malaysia is trying to grow in parallel with developed countries by applying green technology in every marketing and production sector. Green technology is known as the technology that can be recycled or reversed and also used to develop products, equipment, and systems to conserve the natural environment and resources, thus minimises and reduces the negative impact of human activities. Recently, automotive, construction, sports, and leisure sectors, and other mass production industries are focusing on sustainable and renewable reinforced composites by using natural fibres. The addition of reinforcements, such as fibres and fillers into polymer composites, extends the use of fibres and improves the properties of composite components parallel with the requirements of engineering applications [1].

Nowadays, synthetic fibres like glass, carbon, and aramid are widely used as polymer-based composites due to their good properties and high stiffness. However, these fibres have severe disadvantages in terms of their biodegradability, initial processing costs, recyclability, energy consumption, machine abrasion, and health hazards. Negative environmental impacts have

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changed the attention from synthetic fibres to natural/renewable fibres. The successful use of these fibres depends on their best structural and mechanical properties [2].

This research focused on kenaf core [3], which is known as *Hibiscus cannabinus L* [4]. This *Malvaceae* family is a yearly plant that originally came from central Africa and can be found in wild tropical and subtropical Asia [5]. Kenaf core has consistency in terms of geometry and smooth surface compared to the well-known kenaf bast.

Among natural fibres, kenaf is comparatively commercially available and economically cheap among others [6]. Kenaf fibres have interesting mechanical properties, as shown in Table 1 [7].

Table 1 Mechanical Properties of Kenaf Fibre

Sources	Density (g/cm^3)	Tensile Strength (MPa)	Elastic Modulus (GPa)	Elongation at Break (%)
S. Screenivasan [8]	1.2-1.45	930	53	1.6
N. Saba [6]	1.45	930	53	1.6
H. M. Akil [1]	-	930	53	1.6
C. Ververis *et al*.[9]	-	930	-	1.6
A. B. Maslinda [10]	-	930	53	1.6
M. R. Nurul Fazita [5]	-	295-930	25-53	1.50-6.90

To enhance the matrix-fibre adhesion, chemical treatments using sodium hydroxide (NaOH) are used by increasing the roughness by cleaning the fibre surface from any impurities [11] and enhancing the interfacial bonding [4] between the fibre and the matrix to achieve the best results in mechanical and physical properties [6]. Table 2 shows the summary of several researchers using different chemical concentrations and immersion times. In this paper, we studied the treatment of kenaf core fibre with NaOH concentration 6% for 24h in way to modified the surface characterization of natural fibre. This paper aims to investigate the effect of chemical treated kenaf fibres on mechanical and physical properties of kenaf fibre reinforced vinyl ester composites. The effect of alkaline treatment for surface modification has been studied in this paper.

Table 2 Summary of NaOH Treatment for Different Researchers

Sources	NaOH Concentration	Time Immersion	Time to Dry
S. A. N. Mohamed [4]	6%	-	-
V. Fiore [12]	6%	48h and 144h	100˚c at 6h
A. Oushabi [13]	0%, 2%, 5% (optimum), 10%	-	-
R. Mahjoub [7]	5% (optimum), 7%, 10%, 15%	1h, 3h and 24h	-
A. M. M. Edeerozey [14]	3%, 6% (optimum), 9%	3h	Room temperature 24h
S. Scrrnivasan [8]	9%	-	-

2. MATERIAL AND METHODS

2.1 Material Preparation

Vinyl ester (VE) obtained from Polymer Technology Pte. Ltd. (Singapore) was used in this research. The density, heat distortion temperature (HDT), viscosity, and glass transition temperature of VE are 1.6 g/cc, 1,208 °C, 400 cps, and 104.44–143.34 C, °respectively. Methyl ethyl ketone peroxide (MEKP) was used as a hardener. Kenaf core fibre received from Lembaga Kenaf Malaysia was sieved and controlled with a diameter of 0.2 μm in powder form. For kenaf surface modification, kenaf fibre was treated using alkali solution with 6% concentration. The fibre was immersed at room temperature for 24 h. After that, the fibre was heated at 104 °C to remove moisture.

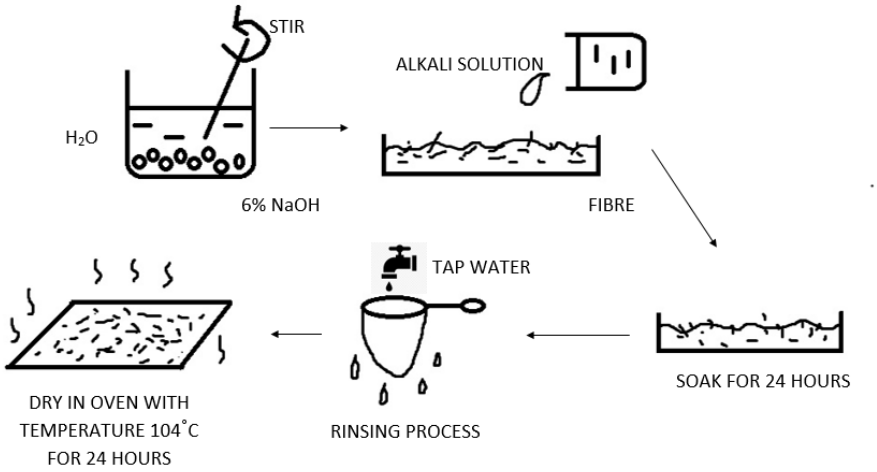


Figure 1. Preparation of composites.

2.2 Preparation of Composites

The composites were prepared using the law of mixture formula. Table 3 presents the weightage of the fibre and matrix to form composites. The composites were divided into three classes: neat polymer (100% vinyl ester without any fibre), treated, and untreated kenaf fibre.

Table 3 Parameter of Composites

Elements	Weight of Composites (g)	Weight of Fibre (g) 5 wt%	Weight of Matrix (g) 93 wt%	Weight of MekP (g) 2 wt%
Neat Polymer	100±5	-	98 ± 5	2±0.5
Untreated	120±5	6±5	111.6±5	2.4±0.5
Treated	150±5	7.5±5	139.5±5	3±0.5

The fibre and the matrix were mixed using a mechanical stirrer and poured into a mould using hand lay-up process. The composites were cured at room temperature and then heated at 110 °C for 24 h to ensure the sample is well cured. The hand lay-up process is a well-known process for thermoset polymers, as shown in Table 4.

Table 4 Thermoset Process

Sources	Type of Matrix	Thermoset Process Technique
R. Mahjoub [3]	Polyester/Epoxy	Wet hand lay-up
K. V. Krishna [11]	Polyester/Epoxy	Resin casting
S. A. N. Mohamed [4]	Vinyl Ester	Hand lay-up/spray up
M. R. Nurul Fazita [5]	Epoxy/Vinyl Ester/Polyester	Hand lay-up
A. B. Maslinda [10]	Epoxy	Vacuum infusion
V. Fiore [12]	Epoxy	Vacuum bagging/ hand lay up
N. Razali [13]	Vinyl Ester	Wet hand lay-up

2.3 Mechanical Testing

The tensile test was carried out following ASTM D3039 (Tensile Properties of Polymer Matrix Composite Materials). The measurement of each sample for the test is 150 mm × 10 mm × 3 mm. The test was conducted using a universal testing machine (Model: Instron 887) equipped with a 5 kN load cell and crosshead speed of 1 mm/min.

The flexural test was performed following ASTM D790-03 (Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials). The measurement of each sample for the test is 100 mm × 10 mm × 3 mm. The test was conducted using a universal testing machine (model: Instron 5585) equipped with a 5 kN load cell and crosshead speed of 1 mm/min. The span length was 50 mm.

2.4 Physical Properties

Five samples of treated and untreated kenaf fibre reinforced vinyl ester was prepared for moisture content analysis. All samples were heated at 110 °C in an oven for 24 h to evaporate water molecules trapped between the fibre linkage. For water absorption analysis, five samples of treated and untreated kenaf fibre reinforced vinyl ester were prepared, where all samples were immersed in water for 24 h and the weight was recorded every 2 h of immersion time to investigate the composite behaviour. Meanwhile, the values of density were calculated by determining the values of mass and volume for five samples of treated and untreated kenaf fibre reinforced vinyl ester.

2.5 Scanning Electron Microscopy (SEM)

For this research, morphological studies were done in detail on the fractured surface of the tensile test sample using scanning electron microscopy (SEM). The fibre loading of kenaf mixed with vinyl ester to form a composite was 5 wt. %. Prior to the testing, the sample was coated with platinum to achieve a better result as the machine offers good electrical conductivity.

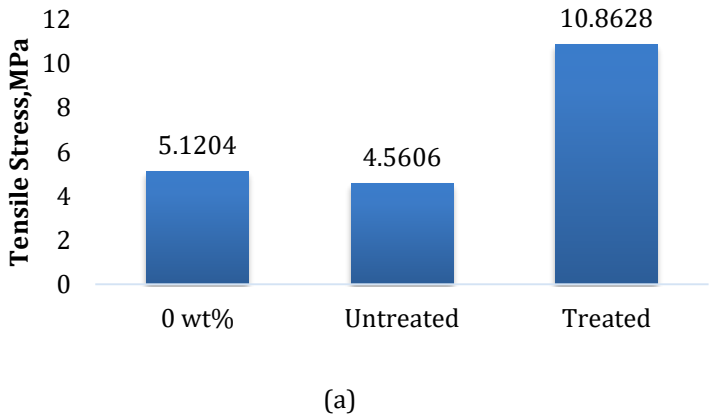
3. RESULTS AND DISCUSSION

Alkaline treatment was performed as the surface modification method of kenaf fibre to enhance the interfacial bonding effectiveness between the fibre and the matrix [2]. This treatment removes all impurities in the fibre and enhances the surface roughness to achieve good interlocking bonding. The results from good bonding will reflect the performance of the mechanical and physical properties of the composites.

3.1 Tensile Test

Ververis *et al*. [9] stated that the tensile test could be represented as one type of stress that works in one direction. The test can indicate whether the samples have good interfacial bonding or vice versa. The tensile test can be conducted to determine the strength, elastic modulus, and strain to failure of composites [10].

From Figures 2 (a) and (b), the treated kenaf reinforced polymer indicates the highest strength compared to the untreated sample. The mechanical properties of the modified fibre are better than unmodified fibre composites [14]. As the fibre is in powder form, the orientation of the fibre when mixed with polymer is in a three-dimensional (3D) case or known as isotropic, which means it does not have a specific fibre orientation. Therefore, it can be concluded that the mechanical properties of the treated composites subjected to surface treatment [14] are better than the untreated composites. The results for the tensile test shown in the bar graph indicate that the treated kenaf fibre obtained the highest value compared to neat polymer and untreated kenaf fibre. The bonding coverage can be improved using a smaller size of fibre, which will have a good surface area-to-volume ratio. Bio. Li [15] conducted an experiment on volume distribution and aspect ratio, and reported that an increase in the aspect ratio would reflect better results in strength.



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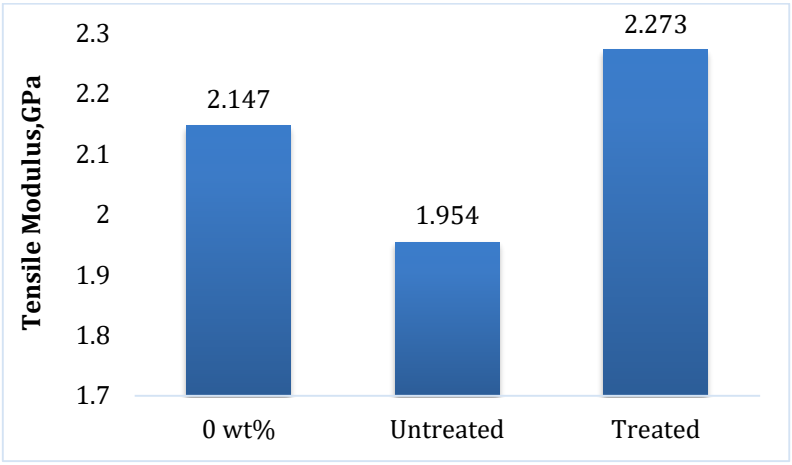


(b)

Figure 2. (a) Tensile strength of kenaf fibre reinforced vinyl ester and (b) tensile modulus of kenaf fibre reinforced vinyl ester.

The increasing trend of tensile stress and modulus shows that the treated composites have good interfacial bonding between the fibre and the polymer and also good stress dispersion of load towards the composites compared to neat polymer. The good interfacial bonding and lower void content of the samples can transfer the stress applied during the tensile test effectively [13]. The treated composites showed increased mechanical interlocking of fibre/matrix due to alkaline treatment. The higher tensile strength is due to the removal of impurities or voids in the fibre [16]. Clean surfaces of treated fibre produce better adhesion between two phases (fibre and matrix), which reflects enhanced mechanical properties [12].

The fibre acts as a reinforcement agent by stopping crack propagation [13] while stress is applied during testing. The tensile strength and tensile modulus of the treated composites increased by 58.02% and 14.03%, respectively. From the observation, crack propagated mostly at the end tab and not at the gage length.

From Table 5, the treated sample indicates lower data compared to the theoretical data [8]. The difference between the theoretical and experimental data of tensile strength is 75.86% and 49.49% for tensile modulus. The difference between these values may be due to several factors, such as testing speed, voids and impurities that might not be completely removed during cleaning, fibre condition during mixing, and different volume percentages of reinforcement [13].

Table 5 Theoretical Results of Tensile Strength and Tensile Modulus of Kenaf Fibre Reinforced Vinyl Ester Treated with NaOH Solution [8]

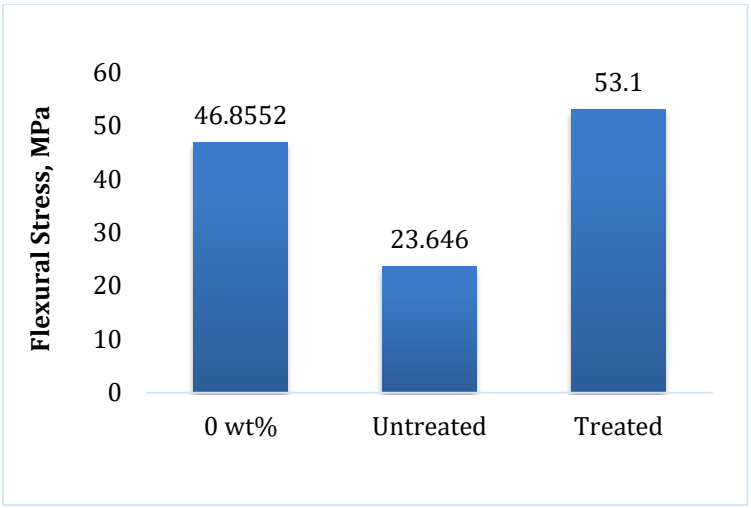
Composites	Tensile Strength, MPa	Tensile Modulus, GPa
Kenaf Reinforced Vinyl Ester (Theoretical)	40-50	4-5
	75.86%	49.49%
Kenaf Reinforced Vinyl Ester (Experimental)	10.8628	2.273

N. Razali *et al*. [13] stated that a good tensile test result depends on more effective and uniform stress distribution between fibres and polymers. Lower tensile stress indicates the weak ability of fibre to transfer the load from one point to another [9]. The crack propagation may be deformed only at one point as the stress is not distributed to other parts.

3.2 Flexural Test

Flexural testing is conducted to determine the strength and ability of a material to resist deformation under loading before reaching the breaking point [13]. This technique evaluates the modulus elasticity in bending, flexural stress, and strain by setting up the material on a support beam under two supports and a load is applied at a point [17].

The test determines whether the composites can withstand the bending load and deformation before it breaks the structure [10]. Alkaline treatment affects the results of flexural strength and modulus [12].



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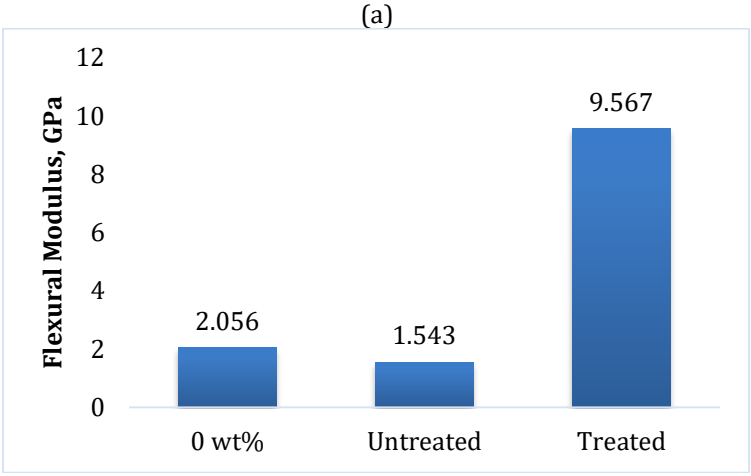


(b)

Figure 3. (a) Flexural strength and (b) flexural modulus of kenaf fibre reinforced vinyl ester.

Figures 3 (a) and (b) show that the treated fibre has the highest flexural strength and modulus than the untreated fibre, indicating that the alkaline treatment can enhance the flexural strength of the composites. The difference in percentage of flexural modulus and flexural strength of the treated composites increased to 83.87% and 55.47%, respectively. The untreated composites have lower data, which might be due to poor dispersion of fibre towards the matrix, leading to weak load transfer, and also due to voids that still present on the natural fibre [13]. The higher the bending capacity the composites can withstand, the better the mechanical interlocking between the kenaf and vinyl ester [10]. Good mechanical interlocking can be obtained when the surface modification is performed on the fibre surface. The treatment can improve the compatibility between the reinforcement and the matrix.

The addition of kenaf fibre improves the stiffness of vinyl ester polymer matrix [13]. The modulus value becomes higher for the treated composites due to the good mechanical interlocking between the fibre and the matrix. The comparison of experimental and theoretical results is presented in Table 6 [8]. The experimental value for flexural modulus is higher than the theoretical value, reflecting that the composite process is done perfectly. The differences between the theoretical and experimental data of flexural strength and flexural modulus are 24.14% and 47.74%, respectively. The correct testing method also reflects good experimental data, such as correct values for crosshead usage and load applied. To conclude, when surface modification is done correctly, the void content can be removed perfectly and produce better results in mechanical properties due to enhanced interfacial bonding between the fibre and the matrix.

Table 6 Theoretical Flexural Strength and Flexural Modulus of Kenaf Fibre Reinforced Vinyl Ester Treated with NaOH Solution [8]

Composites	Flexural Strength, Mpa	Flexural Modulus, GPa
Kenaf Reinforced Vinyl Ester (Theoretical)	60-80	3-5
	24.14%	47.74%
Kenaf Reinforced Vinyl Ester (Experimental)	53.1	9.567

3.3 Water Absorption

Voids are removed during alkaline treatment, including the chemical content of natural fibre, such as hemicellulose, lignin, cellulose, and any other impurities. This process enhanced the bonding between the fibre and the matrix, fibre wetting, and mechanical interlocking [17].

The most critical issue that natural fibre needs to overcome is its hydrophilic nature. The hydrophilic characteristic of natural fibre can cause the absorption of water in fibre, the growth of fungi on natural fibre surface, the swelling of natural fibre, the change of dimension, and rotting due to fungi attacks. The hydrophilicity comes from lignocellulose that strongly polarised with hydroxyl groups. If the wetting of the fibre-matrix occurs, the weak interfaces between the two phases will occur. The swelling that occurs due to the absorption of water will lead to poor mechanical properties of the composites. Many surface modifications are available, such as alkaline treatment. The alkaline treatment indicates better wetting between the fibre and the matrix compared to untreated composites [8].

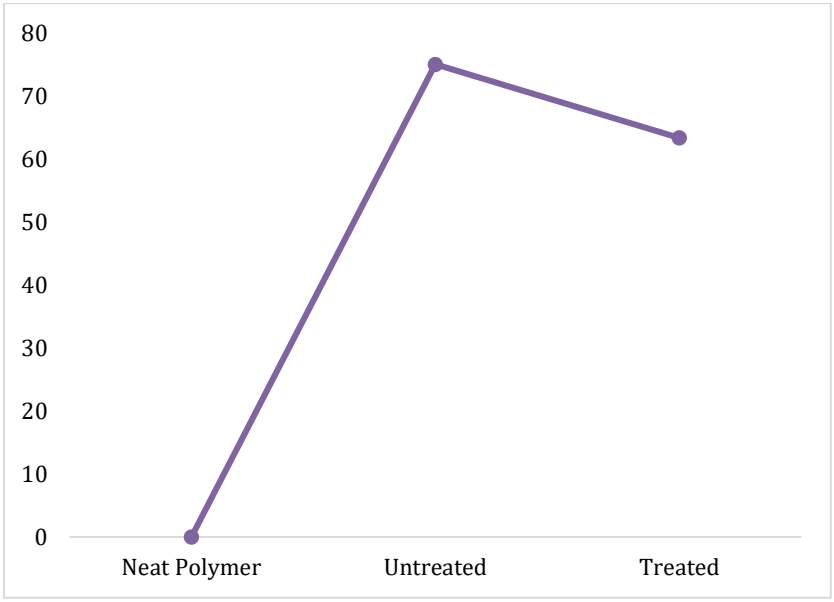


Figure 4. Percentage of water absorption in 24 h.

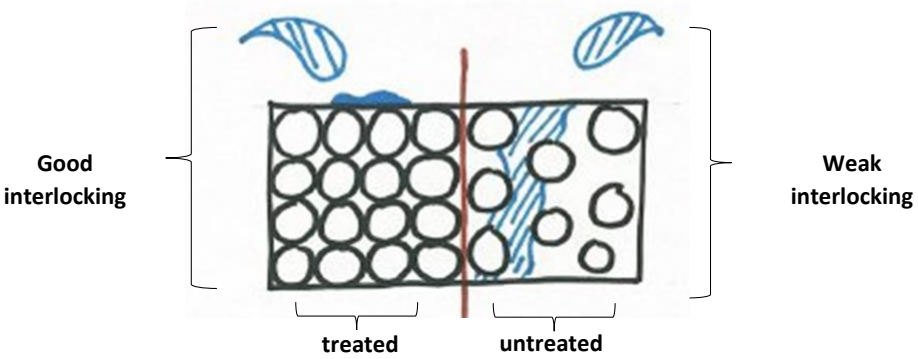
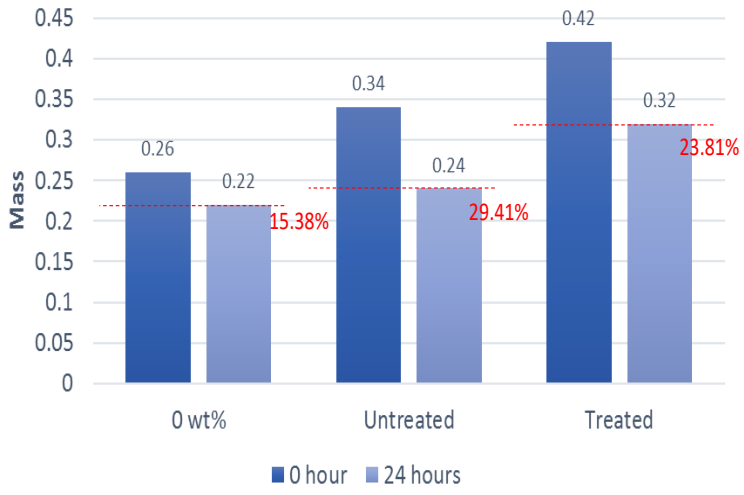


Figure 5. Different penetration of water between treated and untreated composites.

The water absorption and moisture content of fibre reinforced polymer composites have been investigated. As seen in Figure 4, the value for neat polymer is 0% as the polymer can resist water better compared to the fibre due to the hydrophobic properties of polymer plastic. Unmodified kenaf core fibre has a wetting problem compared to modified fibre. The unmodified fibre might have higher cellulose content, which attracts more water molecules. The high cellulose content allows more water to penetrate the fibre-matrix interphase, which can lead to cracking in microstructure and swells. The longer the immersion time, the larger amount of water will penetrate the interphase of the composites through the microcracks and the detachment between the fibre and the matrix will occur. The water absorption behaviour of the fibre reinforced composites needs to be properly analysed, especially for outdoor products [10]. Akil *et al*. [1] stated that water absorption might occur due to the fibre dispersion in the matrix, fibre permeability, and void content temperature. Neat polymer can resist water well because the polymer is hydrophobic, hence the polymer cannot react with hydroxyl groups, in contrast to natural fibre [10], as illustrated in Figure 5. The modified composites can resist water well due to the alkaline treatment that roughens the surface and enhances the bonding between the fibre and the polymer; therefore, the composites can resist water molecules in the long term.

3.4 Moisture Content

As for moisture content, the composites and neat polymer were heated in an oven at 110 °C for 24 h. When the composites are exposed to a humid environment and contact with the fibre, the hydrogen bond breaks and the hydroxyl group will form new hydrogen bonds with water molecules. Neat polymer samples have no value because vinyl ester resins are hydrophobic, where plastics cannot absorb water, as opposed to natural fibre properties. Figure 6 (a) shows the percentage of hydrogen molecules in composites prior to heating in the oven, which released hydrogen particles. The moisture content for untreated composites (29.41%) is higher than treated composites (23.81%). This finding proves that untreated composites absorb more moisture than treated composites. Furthermore, as the treated composites have a lower percentage than the untreated composites, air or liquid molecules cannot penetrate the sample due to the good mechanical interlocking between the fibre and the matrix.



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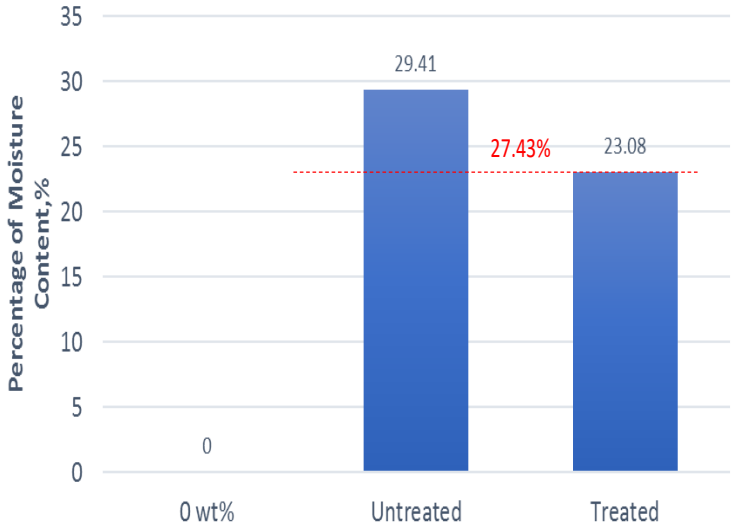


(b)

Figure 6. (a) Initial moisture content and final mass and (b) percentages of moisture content for composites and neat polymer after 24 h.

The fewer liquid molecules that can penetrate into the samples, the more stable the sample. This characteristic can help to prevent the samples from swelling and failing for a short period. Alkaline treatment can enhance the bonding well and increase the time for liquid molecules to penetrate into the sample; hence, the sample has better resistance to liquid molecules.

3.5 Density

A density test is conducted to check the weight of the composites in order to produce a high-performance product with light weight.

Table 7 Parameter of Composites (2)

Elements	Weight of Composites (g)	Weight of Fibre (g) 5 wt%	Weight of Matrix (g) 93 wt%	Weight of MekP (g) 2 wt%
Neat Polymer	100±5	-	98 ± 5	2±0.5
Untreated	120±5	6±5	111.6±5	2.4±0.5
Treated	150±5	7.5±5	139.5±5	3±0.5

Table 7 presents composites of different weight with the same optimum weight percentage of 5 wt. %. From the table, the neat polymer has 100 g of composites, the untreated sample is 120 g of mixture composites between the matrix and the fibre, and the treated sample has 150 g of mixture composites between the matrix and the fibre.

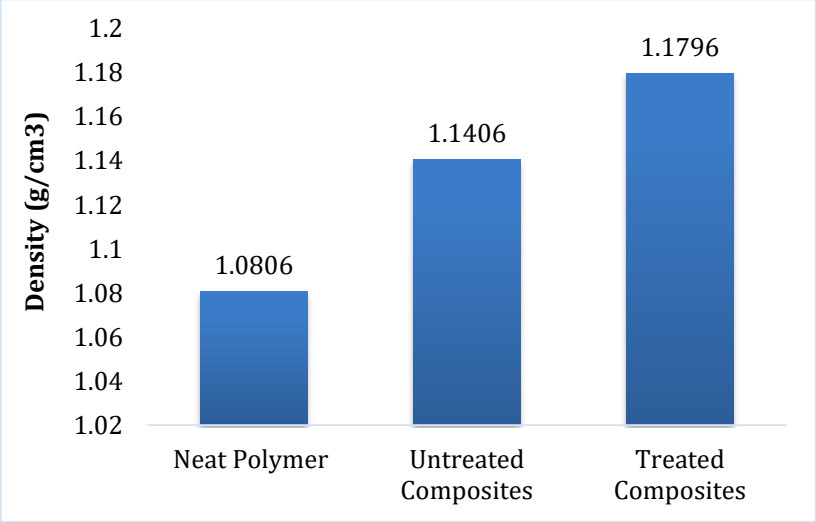


Figure 7. Density of sample composites.

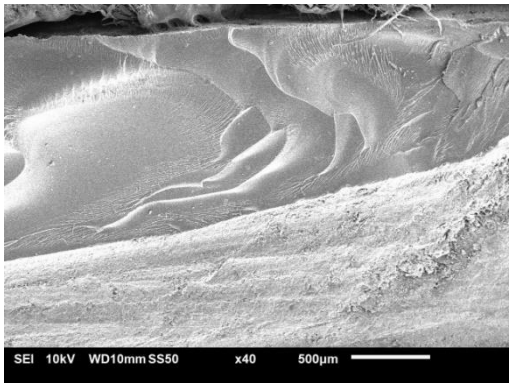
From the data, the increase of the weight of fibre will increase the weight of composites, which is parallel with the amount of density calculated. The optimum fibre weight percentage must be correct to ensure all fibres can react with the mixture of the matrix applied.

3.6 Scanning Electron Microscope (SEM)

The SEM results of the failed samples were observed to evaluate the adhesion/interfacial bonding between the fibre and the polymer. Figure 8 (a) indicates smooth neat polymer surface defects. From the observation, vinyl ester resins are compared to untreated and treated composites that are brittle. The fibre as a reinforcement material improves the hardness of the composites. Meanwhile, the rigidity of the fibre limits its stiffness.

Figure 8 (c) and Figure 9 (c) of untreated composites show lower fibre pull-out than treated composites. This is due to the weak bonding between the fibre and the matrix. As a result, when a load was applied during the tensile test, the fibre could not withstand the high load and off from the grip of the matrix. Compared to the treated fibre in Figure 8 (b) and Figure 9 (b), the adhesion is strong between the matrix and the fibre. Thus, there is higher fibre pull-out left on the surface than the untreated fibre. R. D. Santos stated in their paper regarding the best result obtained in the water absorption profile and improved adhesion bonding of fibre after surface treatment of sisal fibre [18]. The good interaction between the fibre and the matrix produced good experimental data of tensile and flexural tests [13].

Bubbles that appeared on the composite surface for treated and untreated composites are due to the processing method and curing process. The sample composites are not vacuumed to release the air trapped at the surfaces. Hence, the air is trapped between the fibre and the matrix.



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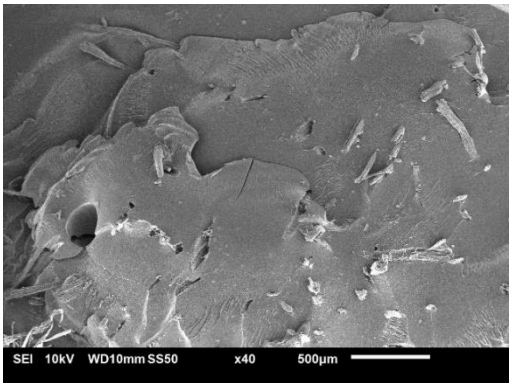

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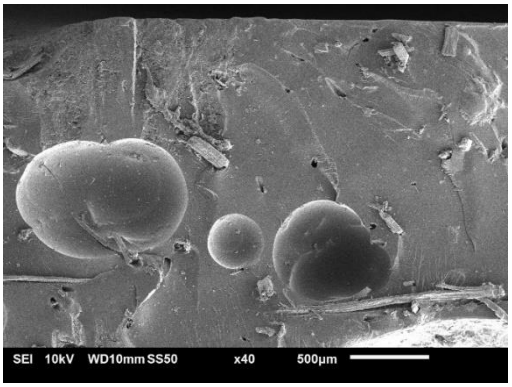

(c)

Figure 8. (a) Neat polymer at 40× magnification, (b) treated composites at 40× magnification, and (c) untreated composites at 40× magnification.

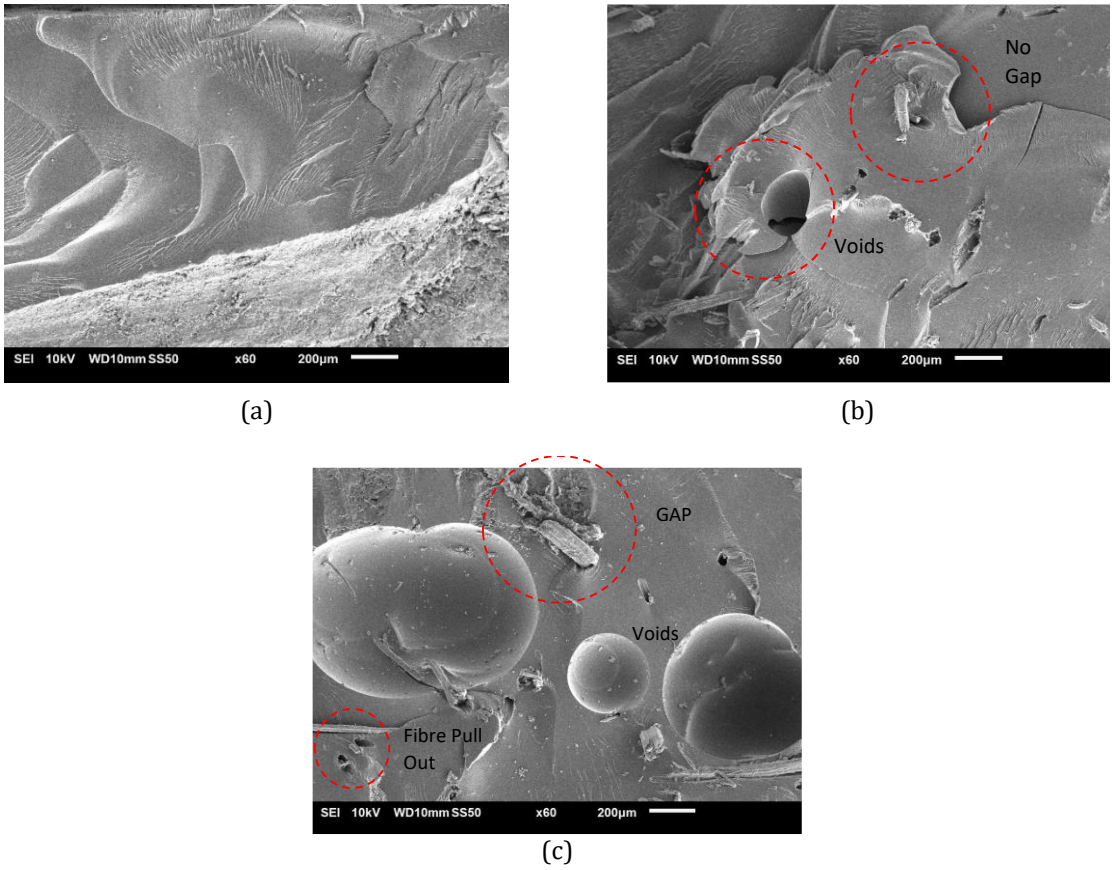

Figure 9. (a) Neat polymer at 60× magnification, (b) treated composites at 60× magnification, and (c) untreated composites at 60× magnification.

4. CONCLUSION

For many years, researchers have tried to discover the correct way to produce high-performance materials using natural fibre as the reinforcement material. Many methods have been used to process natural fibre by combining it with polymer matrix. This research focuses more on the effect of using alkaline treatment towards kenaf fibre reinforced composites on the mechanical, physical, and morphological properties.

In this experiment, the optimum weight percentage (5 wt. %) of kenaf fibre was measured and blended with the specific weight of vinyl ester, and three different samples were produced: neat polymer (0 wt. % of fibre), treated composites, and untreated composites. Based on the comparison between treated and untreated samples without alkaline treatment, it is shown that treated kenaf fibre could achieve better results.

The effect of alkaline treatment with 6% concentration of NaOH can enhance the interlocking effect between the matrix and the fibre, reduce the chemical content of natural fibre, especially lignin, hemicellulose, and cellulose, and remove the impurities of natural fibre that may affect the performance of the composites. The surface treatment also enhances mechanical properties, such as tensile strength and flexural strength.

For future study, the improvement on handling the reinforced and matrix process should be studied more to prevent the failure of the samples and results. The study about the optimum weight and concentration of NaOH should also be emphasised to thoroughly clean natural fibre surfaces to reduce the number of voids and impurities. The optimum immersion time of kenaf

fibre should be investigated more to ensure that the chemical content of natural fibre can be reduced to achieve good bonding between the matrix and the reinforcement material. In conclusion, further study should be done so that high-performance composites can be produced without facing any problems.

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