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Characteristics of Lantung Fiber and the Effect of Alkali Treatment and Water Absorption on the Mechanical Properties of Lantung Fiber Reinforced Composites

Hendri Hestiawan[1*], Zuliantoni[1], NurulIman Supardi[1], Sudibyo[2]

[1]Study Program of MechanicalEngineering,FacultyofEngineering, University ofBengkulu, Bengkulu, 38112, Indonesia

[2]Research Unit for Mineral Technology, National Research and Innovation Agency, Lampung, 35361, Indonesia

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Abstract

This study aims to investigate the effect of alkali treatment and water absorption on the mechanical properties of composites reinforced with lantung fibers. Lantung fibers were alkali-treated for one and two hours at room temperature using 4% and 6% sodium hydroxide solutions. Composite samples reinforced with lantung fibers were prepared by the hand lay-up method. The composite samples were soaked in distilled water at 60°C for 1080 hours or 45 days. All samples were subjected to tensile tests, flexural tests, and impact tests based on ASTM D638-02, D790-02, and D5942-96 standards. Alkali treatment was proven to be effective in improving the mechanical properties of lantung fiber reinforced composites. Lantung fibers soaked in 4% sodium hydroxide solution for 2 hours gave the highest mechanical properties.

The initial absorption process of lantung fiber reinforced composites soaked in distilled water at 60°C was found to approach the Fickian diffusion behavior. Following an hour-long immersion in a 6% sodium hydroxide solution, lantung fibers had the lowest diffusion coefficient of 9.15×10^{-12} m^2/s and the highest water content of 4.89%. The mechanical properties of lantung fiber reinforced composites showed insignificant decrease in impact, flexural, and tensile strength after water absorption so that lantung fiber is recommended for use in wet environmental conditions.

Keywords: Alkali treatment, hand-lay technique, lantung fiber, mechanical properties, water absorption.

1. Introduction

These days, engineers and researchers are searching for materials that can change synthetic fibers to improve life cycle performance and prevent environmental harm. Because of their affordability, light weight, and even environmental friendliness, natural fibers have recently been used as reinforcement in composite materials[1],[2], [3], [4]. Numerous studies have been carried out to investigate the potential of natural fibers as reinforcement in composites that have not yet been discovered [5]. The fact that natural fiber is extremely susceptible to the effects of outside variables, such as water, is one of its many disadvantages.

For long-term use in outdoor applications, information regarding how moisture affects the mechanical qualities of natural fiber reinforced composites is crucial. No research has been done on the relationship between moisture resistance and mechanical properties at various temperatures. Because of the organic qualities of natural fibers and their higher water absorption behavior, several earlier studies have demonstrated that moisture degrades the mechanical properties of larger natural fiber-reinforced composites when compared to synthetic fiber-reinforced composites[6]. Synthetic fibers also have better mechanical properties and are more resistant to moisture. Some synthetic fibers are even more mechanically superior to metals. Conversely, synthetic fibers are environmentally harmful and non-renewable [7].

The interface bond between the matrix and the reinforcement is a critical area that can be affected by the manufacturing process. A decrease in composite properties will occur if this interface bond is not managed properly [8], [9]. One way that can be done is by performing alkali treatment. Alkali treatment is a common method to improve the chemical composition of natural fibers using sodium hydroxide (NaOH) solution [10], [11]. Among the chemical treatments used to increase the interfacial strength between the fibers and the matrix, the alkali treatment is the most economical method [12]. Alkalization of natural fibers can improve the mechanical properties of the fibers so that knowledge of what happens structurally is very important. There are three variables involved during the alkali treatment process, namely the concentration of the solution, the duration of immersion

* Corresponding author e-mail: hestiawan@unib.ac.id.

and the temperature of immersion. These treatment variables are set differently according to the type of fiber by researchers. An attempt to investigate the effect of immersion time during alkali treatment on the concentration of alkali and a certain immersion time on the fiber was carried out by Symington et al. [13] and Boopathi et al. [14]. Symington et al. [13] set the immersion duration at 10, 20 and 30 minutes where the concentration of the alkali solution was set as constant. The tensile strength of all the fibers studied was found to show a decreasing pattern as the immersion time increased. While Boopathi et al. [14] investigated the physical, chemical and mechanical properties of Borassus fruit fibers treated with alkali. The fibers were treated with varying alkali concentrations of 5%, 10%, and 15% with immersion time of 30 minutes at room temperature and found that the 5% alkali concentration produced the highest fiber tensile strength. Optimization of process parameters in the process of selecting the right concentration and time for alkali treatment will determine the balance between the input and output of the material strength obtained [15].

Mendong grass and salacca zalacca fibers were subjected to surface impurity removal using 5% NaOH by Suryanto et al. [16] and Ariawan et al. [17], respectively. After treating hemp fiber with different concentrations of NaOH solution, Suardana et al. [18] discovered that, when compared to other alkali treatments, 4% NaOH solution applied for an hour had the highest tensile and flexural strengths. Lantung fiber research is still in its infancy and has not been thoroughly examined. The purpose of this study is to look into how alkali treatment and water absorption affect the mechanical properties of lantung fibers reinforced composites.

2. METDODOLOGY

2.1. *Materials*

Lantung fibers were gathered from different local suppliers. Vynilester resin (Ripoxy), methyl ethyl ketone peroxide, and promoter X were purchased from Justus Kimiaraya Ltd. Lantung fibers were obtained from the bark of lantung tree (Artocarpus elasticus), as shown as Fig. 1.The process of processing lantung fiber begins with selecting a 2-year-old lantung tree (1a). Lantung fiber is taken from the bark of the lantung tree which is peeled from the trunk until it becomes a sheet shape (1b). Then the lantung bark is washed with running water and occasionally brushed on the inside to remove tree sap and dirt that is still stuck to the lantung bark (1c). The lantung bark is then dried in the open air until it dries completely until lantung fiber is obtained (1d).

2.2. *Fiber Treatment*

The alkali treatment in this research used a concentration variation of the sodium hydroxide solution of 4 and 6% for an hour and two hours at room temperature. The sodium hydroxide solution was neutralized numerous times for 30 minutes after the lantung fibers were soaked, and the fibers were then dried for 48 hours at room temperature to eliminate any remaining free water [19]. Alkali treatment of lantung fiber is expected to produce changes in adhesion parameters between the matrix and fiber in the composite [20].

Figure 1. Lantung fiber processing process

(a) Lantung tree; (b) Skinning the bark; (c) Washing the bark; (d) Lantung fiber

2.3. Composite Manufacturing

Composite samples have been produced using hand lay-up technique as shown in Fig. 2. Prior to composite fabrication, lantung fibers were placed in an oven at 100ºC for an hour to remove moisture in the fibers. To remove the specimen easily, the mold surface was treated with mold release wax. The ratio of vinylester resin and catalyst was 100:1 (v/v). The sample was allowed to cure for an hour at room temperature before being demolded. Furthermore, the sample was kept at room temperature on a level surface for about a day [21]. Subsequently, the material was sliced and shaped into test specimens for tensile, flexural, and impact strength in compliance with ASTM standards.

Figure 2. Hand lay-up technique

2.4. Fiber Density Measurement

A bundle of lantung fibers density is lower than that of the sample when it is weighed in both air and the liquid used to moisten it. Use ethanol liquid (density: 0.789 g/cm^3) as the solvent. An analytical balance with a resolution of 0.0001g is used for the weighing procedure. To calculate sample density (ρf), use the ASTM standard of D 3800, as follows.

$$\rho f = \frac{(M_3 - M_1)\,\rho l}{((M_3 - M_1) - (M_4 - M_2))} \qquad (1)$$

where ρl is the liquid's density, M_1 and M_2 are the wire's weights in air and liquid, respectively, M_3 and M_4 are the wire's weights plus the object's weights in air and liquid, respectively. The data from the nine examined samples were averaged.

2.5. X-ray Diffraction

The fiber crystallinity index (CI) is determined by the Segal equation in the context of the X-ray diffraction (XRD) test[22].

$$CI\ (\%) = \frac{(I_{002} - I_{amp})}{I_{002}}\, x\ 100\% \qquad (2)$$

where CI is the crystallinity index, I_{amp} is the diffraction intensity at 2θ at an angle of 18°, which represents the amorphous material in cellulose fibers, and I_{002} is the maximum intensity of the 002 grating diffraction field at 2θ, which occurs between 22° and 23°. An X-Ray Rigaku Mini flex 600 with copper as a radiation source (λ = 1.54), 30 mA of current, and 30 mA of voltage were employed in the XRD test. Every specimen was scanned at a speed of 2°/min within an angle range of 2θ, 10–40°.

2.6. Scanning electron microscope

Morphological study of lantung fibers used scanning electron microscopy (SEM) images at 12 kV accelerating voltage using a JEOL model. Before recording, a thin layer of gold-palladium was coated on the fiber surface in a vacuum chamber to increase the conductivity of the fiber. SEM pictures were taken for all samples to determine the microstructural changes that occurred with each alkali treatment.

2.7. Water Absorption Behaviour

Water absorption tests were performed in accordance with ASTM D570-98 to investigate the water absorption behavior of lantung fiber-reinforced composite materials. All samples were dried in an oven set to 100°C for an hour before being allowed to cool to room temperature before being soaked in water. Determine the sample's starting weight (Wo) by weighing on a digital balance with a 1 mg accuracy. Three specimens measuring 25.4 mm x 76.2 mm x actual thickness were submerged in distilled water at 60°C to investigate water absorption. After about 45 days, the procedure is repeated, weighing the sample (weight) at regular intervals until the saturation limit is reached. Plotting the square root of the water immersion time against the percentage increase in sample weight at each time point (t) was done. Equation (3) calculates the difference between the sample's weight gain (Wt) when it is dry and after it has been submerged in water for a period of time (t) as follows [23].

$$\mathrm{W_t}(\%) = \frac{(\mathrm{W_t} - \mathrm{W_o})}{\mathrm{W_o}} \mathrm{x}100 \qquad (3)$$

The weight increase of the specimen due to water absorption was expressed by two parameters, namely maximum water content (M_m) and diffusivity (D), as shown in equation (4) [24].

$$\mathrm{W_t} = \mathrm{M_m}\left\{1 - \frac{8}{\pi^2}\exp\left[1\left(\frac{\mathrm{Dt}}{\mathrm{h^2}}\right)\pi^2\right]\right\} \qquad (4)$$

where t was the time and h was the sample's thickness. The slope at the initial point of the water absorption curve can be used to determine diffusivity.

2.8. Mechanical Test

The tensile test on lantung fiber reinforced composite was performed both prior to and following the water immersion test using a servopulser machine based on ASTM D638-02 and a test speed of 5 mm/min[25]. For every case, three specimens were assessed in order to determine the average value. The samples were subjected to flexural tests before and after immersion according to ASTM D790-03 using a three-point bending apparatus and a crosshead speed of 5 mm/min. The flexural tests were performed using a Torsee universal machine. The test terminates upon accomplishment of 5% deflection by the specimen or its breakage prior to 5% deflection [26].The samples were 1/16 thicker than the support span, and the thickness to support span ratio was 1:16. The dimensions of there ctangular samples were 80 x 10 x actual thickness. The arithmetic mean of the results from testing three specimens would be determined. A Charpy impact testing machine, which is based on ASTM 5942-96 standards, was used for the impact test. A Charpy impact test machine was used to perform the impact test in accordance with ASTM 5942-96. Notches were absent from every impacted specimen. The measurements of a typicals ample were 75 x 10 x actual thickness. For each sampleset, at least three samples were analyzed and the average value was recorded. To determine how water absorption affects the mechanical properties, all impact and bending samples were collected and dried in an oven at 100 °C for one hour.

3. Result and Discussion

3.1. Fiber Density Measurement

Density measurements for untreated and alkali-treated lantung fibers at room temperature are shown in Fig. 3. The density of lantung fibers was determined to be 1.451 g/cm^3 through the application of the Archimedes method. The density of lantung fiber is in the range of hemp fiber (1.401 g/cm^3) [27] and ramie fiber (1.5 g/cm^3) [21], when compared to a number of other natural fibers. Density measurements for untreated and alkali-treated lantung fibers at room temperature are shown in Table 6. The density of the alkali-treated fibers is higher because the alkali treatment effectively dissolves non-cellulose components such as hemicellulose, lignin, impurities in the fiber surface which have low density compared to cellulose itself. As a result, the fiber volume decreases and has an impact on increasing its density. The highest density was obtained in alkali-treated fibers at concentrations of 4% for 2 hours, which was 1,512 g/cm^3. Similar results were obtained in the study of salaccazalacca fiber density conducted by Ariawan et al., where salaccazalacca fiber treated with alkali showed a higher density than fiber that was not treated [17].

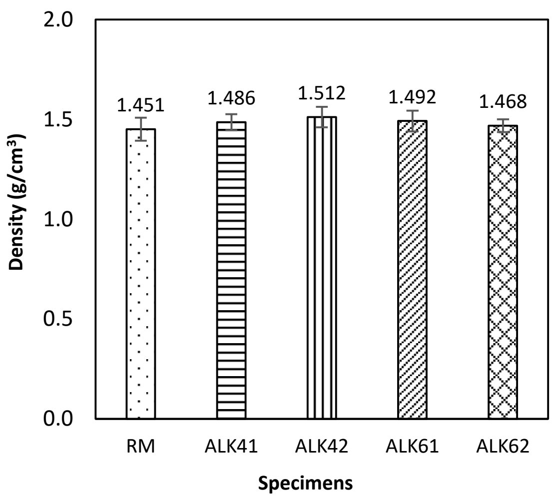

Figure 3. Density of lantung fiber before and after alkali treatment

3.2. X-ray Diffraction Observations

Fig. 4 displays the results of the XRD test. Fig. 3 illustrates that there are three primary intensity peaks in all lantung fibers, which are located at 2θ angles of around 16, 22, and 35°. These peaks, shown by reflection points (1 0 1), (0 0 2), and (0 4 0), are linked to the crystal structure of cellulose I. Almost the same peak intensity was obtained in the research of Hakim, et al.[28] which uses sugar palm bunch, namely at 2θ angles of around 16.0, 22.2, and 34.5°.

The spectrum of alkali-treated lantung fiber shows that the cellulose I profile at an angle of 2θ = 22o has a sharper peak compared to lantung fiber without treatment (RM). This indicates that by eliminating certain chemical components including hemicellulose, pectin, and lignin, the alkali treatment of cellulose can increase its crystallinity. The cellulose content rises as a result, making the peak at the reflection point (0 0 2) sharper. In the immersion process in alkali treatment, cellulose I transforms into Na-cellulose I, as described by Nishiyama et al. [29].The presence of NaOH solution during the alkali treatment process causes the loss of some amorphous parts in the fibers that have a lower molecular weight than cellulose so that there is no barrier to the entry of NaOH solution. Thus, the amorphous part is more easily hydrolyzed during the treatment process and is expected to increase the crystallization index of cellulose [30].

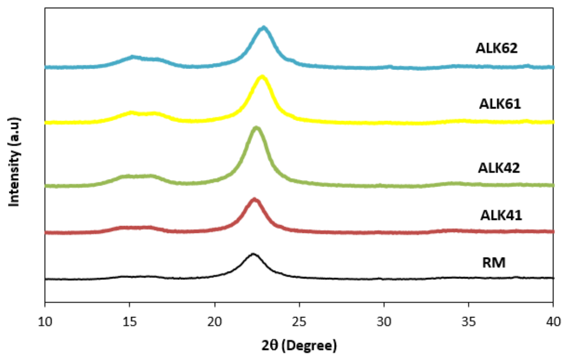

Figure 4.X-Ray diffraction spectra of lantung fiber

Table 1 displays the findings of the lantung fiber's crystallization index calculation. Compared to other fibers, lantung fiber treated with alkali at a concentration of 4% NaOH for two hours (ALK42) exhibited greater peak intensity, measuring 85.42%, or 5.61% more than untreated lantung fiber (RM). The crystallization index of jute fibers alkali treated in 5% NaOH for 2 hours increased by 15% compared to untreated jute fibers [31].The rise in the crystallization index in the alkali-treated lantung fiber indicates that the alkali treatment can eliminate amorphous substances like hemicellulose, lignin, and other non-cellulosic substances, increasing the proportion of crystalline substances like cellulose on the fiber surface [32]. Meanwhile, the 6% alkali treatment (ALK61 and ALK62) showed a lower crystallinity index due to the disintegration of excessive cellulose content when the alkali concentration increased, resulting in a reduction in crystal area [33][22].

Table 1. Crystallization index of lantung fiber

Specimens	I_{amorf} (cm^{-1})	I_{002} (cm^{-1})	Crystallization Index (%)
RM	1533	7594	79.81
ALK41	1715	9742	82.4
ALK42	2283	15661	85.42
ALK61	2119	12788	83.43
ALK62	1972	11442	82.77

3.3. *Scanning Electron Microscope Studies*

Fig. 5 present SEM photographs of untreated and alkali treated lantung fiber. The highly uneven surface of untreated lantung fiber (RM) indicates the existence of impurities in the fiber gaps. On the surface of lantung fibers treated with alkali at concentrations of 4% and 6% NaOH for an hour (ALK41 and ALK61), these impurities were still discernible in lower amounts. Mean while, on the surface of lantung fiber that was treated with alkali with a concentration of 4% and 6% NaOH for 2 hours (ALK42 and ALK62), these impurities were no longer visible. This suggests how contaminants on the fiber surface can be successfully removed by alkali treatment [34], [35]. The loss of impurities on the fiber surface produces a rougher fiber surface so that it will have better interfacial bonding [36].

However, the lantung fiber surface of that was treated with alkali with a concentration of 6% NaOH for 2 hours (ALK62) appeared to have a smaller fiber size compared to the alkali treatment with a concentration of 4% NaOH for 2 hours (ALK42). This shows that the alkali treatment has reached its saturation point, resulting in fiber degradation and damage to the fiber surface. According to Kaima et al. [37], using excessive concentrations of alkali solutions can harm the fiber structure and reduce its tensile strength.

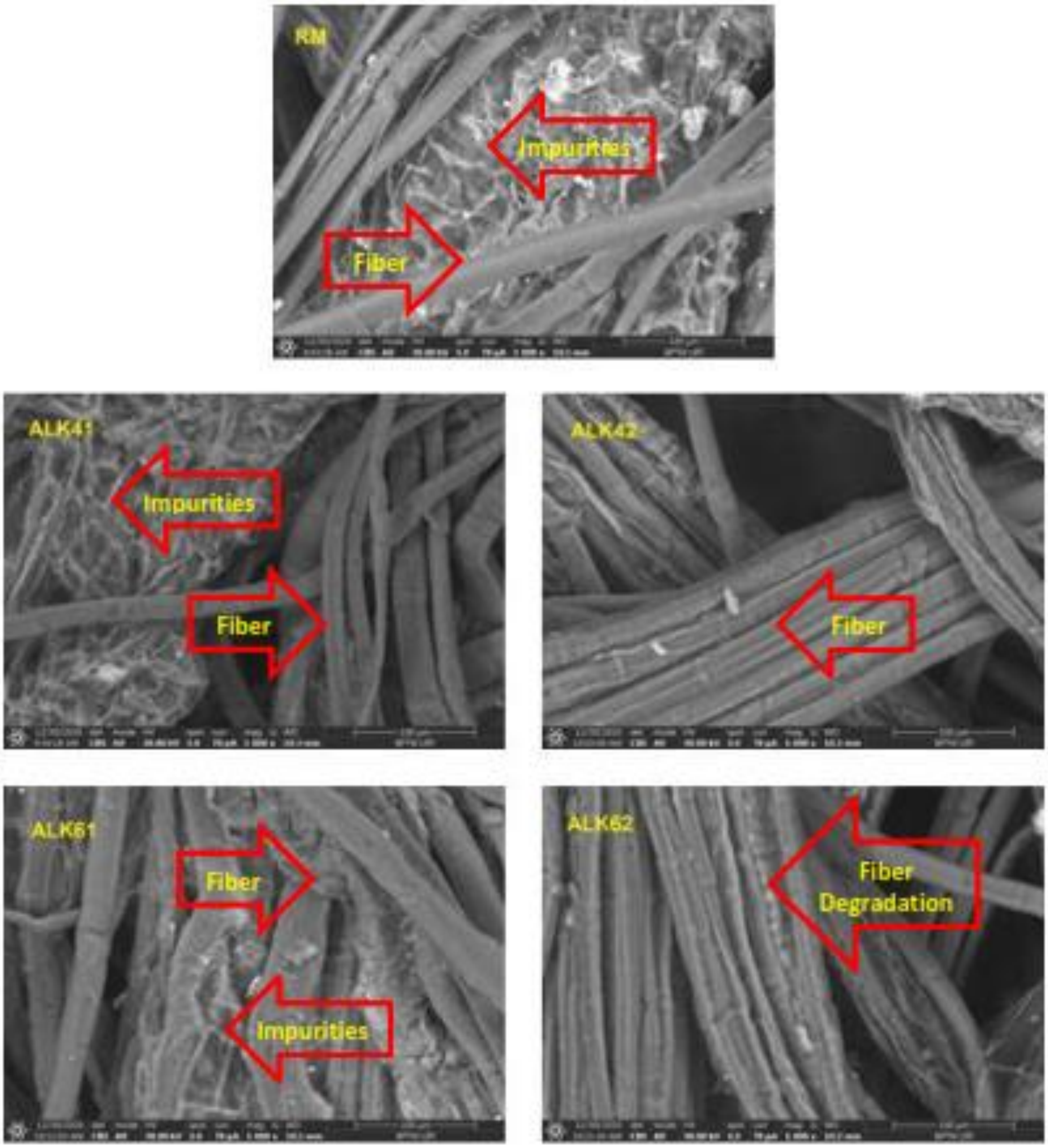

Figure 5. SEM image of the lantung fiber’s surface

3.4. Water Absorption

The percentage of water absorption for all samples is presented in Fig. 6. The rate of water absorption varies and depends on the alkali treatment applied to the composite. Every sample showed the characteristic linear Fickians behavior, which slows and approaches saturation over time. Numerous research on the behavior of natural fiber reinforced composites in terms of moisture absorption have also revealed similar behavior[23], [38]. It is evident how alkali treatment affects the absorption of water. The percentage of water absorbed by samples that have undergone alkali treatment is lower than that of untreated ones[39], [40]. The interfacial bond between the fibers and the matrix is affected by the alkali treatment, which can prevent liquid from penetrating the composite. In comparison to other alkali-treated samples, the samples that were immersed in a 6% NaOH solution for an hour had the least amount of water absorption. Natural fibers treated with alkali are able to increase the interfacial bond between the fiber and the matrix due to the presence of more polar reactive functional groups on the fiber surface so that the composite absorbs less water [40].

Based on the results of water absorption tests, it is known that alkali treatment effectively reduces the maximum water content (Mm) and diffusivity (D) for composite samples as shown in Table 2. The maximum water content and lowest diffusivity in samples without treatment are 8.56% and 148.86x10^{-12} m^2/s, respectively. Meanwhile, for samples treated with alkali, the maximum water content and lowest diffusivity were obtained in samples soaked in 6% NaOH solution for one hour (ALK61) which were 4.89% and 9.15x10^{-12} m^2/s, respectively.

The explanation for this is that the fibers with alkali treatment have stronger interface connections with the matrix. By removing impurities, hemicellulose, pectin, and lignin from the surface of the fiber cell walls, natural fibers treated with alkali can strengthen the adhesive link between the fiber and the matrix, which enhances the mechanical properties of natural fiber composites [41], [42]. Nayak and Ray [43] reported similar results, noting that the atmosphere remained somewhat aerated even after the addition of nanoparticles to the production process and that minute air bubbles persisted in the viscous epoxy/nanoparticle mixture.

The water content of the matrix and interphase must differ. The localized stress and strain that the composites experience are caused by the discrepancy in volumetric expansion between the fiber interphase and matrix. Furthermore, water that has been absorbed might move to other areas of the composites due to capillary action at the fiber/matrix contact. The capacities of the epoxy matrix to expand, plasticize, hydrolyze, and form fissures as a result of absorbed moisture enhances water diffusion in composites. Moisture hence reduces the elasticity, viscosity, and mechanical properties of polymers [44]. During the first part of the test, weak bonding points between neighboring layers of a specimen allow water molecules to enter and quickly fill internal cavities. Water absorption ends when internal cavities are completely filled with water. The polymer components of the specimen alter chemically in the interval. Water molecules will continue to be absorbed by polymer complexes containing hydrophilic groups [45].

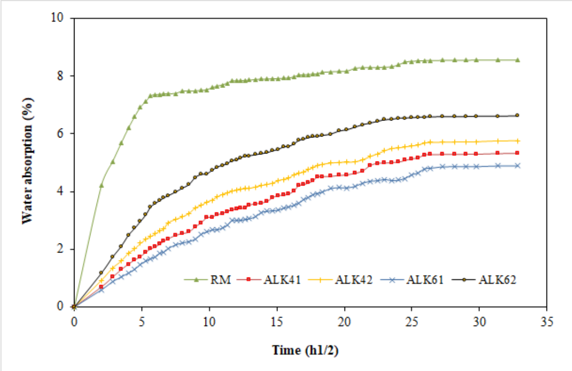

Figure 6. Water absorption curve of lantung fiber reinforced composites in distilled water at 60ºC

Table 2. Maximum water content (M_m) and diffusivity (D) of composite sample

Sample	M_m (%)	D (x 10^{-12} m^2/s)
RM	8.56	148.86
ALK41	8.32	16.24
ALK42	5.76	24.93
ALK61	4.89	9.15
ALK62	6.62	35.99

3.5. Effect of Water Absorption on Tensile Strength

The tensile strength of the composite samples before and after they were submerged in distilled water at 60°C is shown in Fig. 7. It is clear that the alkali-treated composites have more robust tensile bonding [46]. According to this table, all composite samples had greater tensile strength prior to immersion than they did following [47]. The composite samples that were submerged in 4% NaOH solution for two hours (ALK42) showed the highest tensile strength, measuring 60.2 MPa. and 57.2 MPa, respectively, before and after immersion. The decrease in tensile strength was not significant because it was below 5%. The damage mechanism in the composite occurs due to the tensile stress/strain given exceeding the maximum resistance of the composite so that the bond between the matrix and fiber is released and the matrix layer between the fibers is damaged [48].

Water absorption begins at the composite materials' outer layers and progressively diminishes as it moves into the matrix's center. Composite materials with high water intake tend to weigh wet profiles more, weaken them, and make them more prone to deflection and swelling, which puts additional load on nearby structures. The mechanical qualities of composite materials can be destroyed by freeze and unfreeze cycles, bending, buckling, and a higher likelihood of microbial inhabitation [49]. The increase in tensile strength is due to the addition of fibers, which have a higher tensile strength than neat epoxy and act as a carrier for the forces applied to the matrix. The composites' tensile strength was raised by applying chemical treatment at the fiber-matrix interface[50].

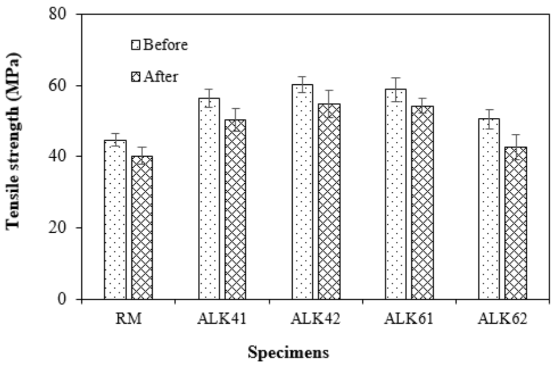

Figure 7. Tensile strength of composite before and after immersion

The tensile strength of composites tends to increase when alkali treatment is given to natural fibers, thus increasing the fiber-matrix bond [51]. Composites with reinforcing fibers in the direction of tensile force provide the highest tensile strength because the fibers are oriented in the direction of the applied force [52]. Narayana and Rao [53] reported that alkali treatment of natural fibers effectively improves the mechanical properties of composites if carried out at appropriate alkali concentrations and soaking times. The tensile strength of jute fiber reinforced composites treated with alkali increased with increasing concentration of NaOH solution from 1% to 5% and then decreased thereafter.

3.6. Effect of Water Absorption on Impact Strength

Impact strength of composites treated differently both before and after immersion is displayed in Fig. 8. The graph showed that lantung fibers' impact strength may be raised by treating them with alkali. After alkali treatment, impact strength considerably increased [54], [55]. The impact strength of the alkali treated samples with 6% NaOH solution for an hour (ALK61) gave the best results both before and after immersion compared to other alkali treated samples, namely 48.2 and 46.8 kJm^{-2} respectively.

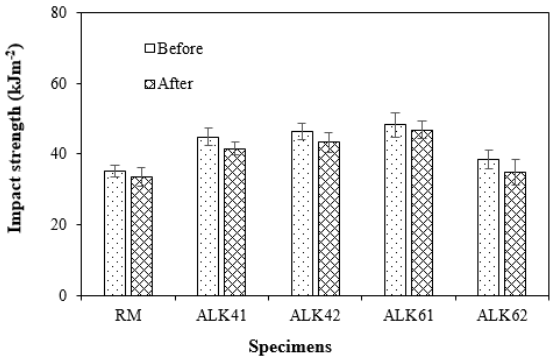

Figure 8. Impact strength of composite before and after immersion

The decrease in impact strength after water absorption was not significant because it was still below 5%. The impact strength of the composite treated with alkali with 6% NaOH solution for one hour (ALK61) gave the best results both before and after immersion compared to samples treated with other alkalis, which were 48.2 and 46.8 kJm-2, respectively. Alkali treatment is able to increase the homogeneity of lantung fibers and the interfacial bond between the matrix and fibers, so that the resistance of lantung fiber-reinforced composites to impact energy increases [56]. The impact strength of composites is affected by water absorption due to the dimensional instability of the composites and the degradation of the matrix-fiber interface, the impact strength decreases after water absorption [39].

3.7. Effect of Water Absorption on Flexure Strength

Fig.9 shows the flexural strength of the composite samples before and after immersion in distilled water at a temperature of 60°C. The alkali treated lantung fiber reinforced composite exhibited increased flexural strength both before and after immersion. According to Shifa et al.[39] and Mohanta and Acharya [57], alkali treatment makes the fiber surface rougher, which increases the interlocking bond between the fiber and the polymer matrix so that it can increase the flexural strength of the composite. The highest flexural strength was obtained in the composite treated with 4% alkali for 2 hours before and after immersion, which were 86.6 MPa and 82.4 MPa, respectively. The reduction in flexural strength was not significant because it was below 5%.

This study shows that water absorption by the composite reduces the flexural strength of each composite sample. The weak interaction between the fiber and matrix interfaces and the formation of hydrogen bonds between cellulose fibers and water molecules are responsible for the decrease in flexural strength after water absorption[58]. Compression, shear, and tension are the principal causes of failure in the bending or flexural test. This is because the fiber orientation in the composite system is short and perpendicular, which makes it simple for cracks and faults to form and ultimately cause failure. Where there is no supporting fiber or reinforcement to fight or prevent defects from showing through [59].

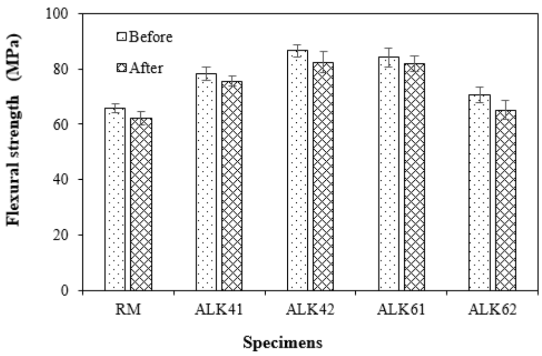

Figure 9. Flexure strength of composite before and after immersion

4. Conclusion

The behavior of alkali treatment and water absorption on the mechanical properties of lantung fiber reinforced composites prepared by hand-lay up technique was examined in this study. Alkali treatment was proven to be effective in improving the mechanical properties of lantung fiber reinforced composites. Lantung fibers soaked in 4% sodium hydroxide solution for 2 hours gave the highest mechanical properties. Lantung fiber reinforced composites soaked in distilled water at 60°C showed that the initial absorption process approached the Fickian diffusion behavior. There was the lowest diffusion coefficient of 9.15×10^{-12} m^2/s and the highest water content of 4.89% for an hour in lantung fibers soaked in 6% sodium hydroxide solution. The mechanical properties of lantung fiber reinforced composites showed insignificant decrease in impact, flexural, and tensile strength after water absorption so that lantung fiber is recommended for use in wet environmental conditions.

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