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Experimental Investigation of Hot Alkaline Treatment on Strength Characteristics of Cantala Fiber Reinforced Composites and Microcrystalline Cellulose

Sakuri Sakuri[1*], T.D. Susanto[1], Bambang Sugiantoro[1], and M.Agus Shidiq[2]

[1] *Department of Mechanical Engineering STT Wiworotomo, Purwokerto, Indonesia*
[2] *Department of Mechanical Engineering Panca Sakti, Univercity Tegal, Indonesia*

*Corresponding author: sakuridahlan@stt-wiworotomo.ac.id
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Abstract

This study was carried out to determine the effect of hot alkali treatment on cantala fiber and the addition of microcrystalline cellulose (MCC) on the density, tensile strength, flexural strength, water absorption, and Scanning Electron Microscopy (SEM) observations on composites. Cantala fiber was soaked with sodium hydroxide and 6 wt% distilled water for 8 hours at hot alkali temperatures of 40 ^{0}C, 60 ^{0}C, and 80 ^{0}C. The unsaturated polyester (UPRs) and MCC were mixed using a magnetic stirrer at a speed, temperature, time, and composition of 250 Rpm, 40 ^{0}C, 30 minutes, and 5% using the vacuum infusion method. The results showed an increase in composite tensile and flexural strengths after hot alkali treatment by 27.91% and 31.41% at 40 ^{0}C. SEM observations showed a stronger bond between the fiber and the matrix. The results of the water absorption test showed that the absorption decreased after the hot alkali treatment.

Keywords: Hot Alkali; Cantala Fiber; Microcrystalline Cellulose; Composite

1. Introduction

The use of natural fibers, including those derived from flax, abaca, kenaf, coconut, banana, etc, are used as a substitute for the synthetic type, and these are continuously utilized as a composite reinforcement (Salmah *et al.,* 2013). However, using those with short life cycles has led to the reduction of using wood, such as hemp, kenaf, sansevieria, pineapple, cantala fibers, etc. The utilization of natural fibers as composites is perceived as an effort to regulate consumables in automotive components, especially in developed countries, namely Japan, Germany, France, and the United States (Rahmat *et al.,* 2015).

Cantala (agave cantala) is an Indonesian tropical plant cultivated in the coastal regions. Its fiber is usually harvested at seven months when it is 90 cm long by farmers in Kulon Progo Yogyakarta. This is harvested by cutting the base of the cantala in a diretiing. Cantala fiber contains 64.23 % cellulose, 29.87 % hemicellulose, and 6.8% lignin, making it a suitable composite reinforcement. (Sakuri *et al.,* 2020a).

The advantages of this natural fiber are its abundance, affordability, easy to obtain, low density, renewable and recyclable, and environmentally friendly. In addition, it also has several disadvantages, such as poor adhesion to polymers (Dittember *et al.,* 2012), high water absorption, and low thermal stability. Various methods have been used to improve natural fibers' properties and weaknesses, such as alkali treatment (Sakurai *et al.,* 2020a), silane, permanganate, and smoking (Sakurai *et al.,* 2020b). According to Xue *et al.* (2019), alkaline treatment improves the mechanical properties of jute fiber laminated composites. Irrespective of the various surface treatments, it is *the best and is widely adopted in several preliminary studies* (Ariawan *et al.,* 2020). The variation of alkali treatment is % wt NaOH content and immersion time, making it paramount to carry out another analysis on heat treatment with temperature variants. Several surface methods have been carried out to reduce hemicellulose, lignin pectin, and other impurities. The reduction of amorphous content led to an increase in the shear strength of the interface between the matrix and the fiber, thereby producing better interlocking.

The application of hot alkali treatment on natural fibers increases the cellulose content. It eliminates the less effective components in determining the interfacial strength, namely hemicellulose, pectin, and lignin. This maximizes the wettability of the matrix, as well as increases the interfacial strength. It was discovered that the cantala fiber-reinforced composite has better mechanical properties. In an effort to realize this, the alkali treatment was applied at varying temperatures of 40 ºC, 60 ºC, and 80 ºC, with an immersion time of eight hours. Furthermore, microcrystalline cellulose (MCC) was used to fill up the pores as well as improve the mechanical characteristics of the cantala fiber-reinforced composite. Study was to determine the effect of hot alkali treatment on cantala fiber and addition of microcrystalline cellulose (MCC) on density, tensile strength, flexural strength, water absorption and Scanning Electrone Microscopy (SEM) observations on composites.

2. Materials and Methods

2.1 Materials

Cantala fiber obtained from PT Rami Jaya Kulon Progo Yogyakarta Indonesia was processed by rettting. Microcrystalline cellulose (MCC), with a density of 1.54 gram/cm³, was derived from PT Sigma Aldrich Jakarta. PT Justus Kimia Raya Semarang got unsaturated Polyester and Catalyst (methyl Ethyle Ketone Peroxide (Mekpo). Sodium hydroxide (NaOH) and aquades were obtained from TJ Kimia shop, Purwokerto, Central Java.

2.2 Methods

Hot alkali treatment was prepared by mixing 6% wt NaOH with distilled water. Cantala fiber was soaked in a container containing a mixture of NaOH and aquades for eight hours and at varying immersion temperatures of 40 ºC (HA 40), 60 ºC (HA60), 80 ºC (HA 80), and variations without treatment (UT). Afterward, it was cleaned with tap water continuously until it had a pH of 7. In addition, the cleaned fiber was dried at room temperature for four days.

2.3 Composited Molding Process

The composite molding process was carried out by randomly placing the cantala fibers that had been treated with hot alkali and without treatment along 10 mm on the mold. The fraction applied included 30%, 65%, and 5% by volume of fiber, matrix, and microcrystalline cellulose, respectively. Furthermore, MCC and unsaturated polyester were mixed using a magnetic stirrer at a speed of 250 rpm and a temperature of 40 ºC, for 30 minutes by applying the Taguchi method (Sakurai *et al.,* 2020c). Finally, MCC and UPRs that had been mixed perfectly and were ready to be poured into molds were added to methyl ethyl ketone peroxide (mekpo) 1 % wt as a hardener.

The molding procedure involves using vacuum infusion, while the mold is left for three hours before being processed. Afterward, it is removed from the oven after being left for 120 minutes at 60 ºC

2.4 Density Test

Density testing was carried out by cutting a portion of the composite and then weighing it in air and water using the following equation.

$$\rho = \frac{W_1}{W_o + W_1} x \rho a \quad \text{(Alhijazi } et\ al.\text{, 2020)}$$

ρ   = composite density gram/cm³
W1 = mass of composite in air (gram)
Wo = mass of composite in water (gram)
ρa = density of water in the room 0.9978 gram/cm³

Figure 1. The process of hot alkali on fiber and its results

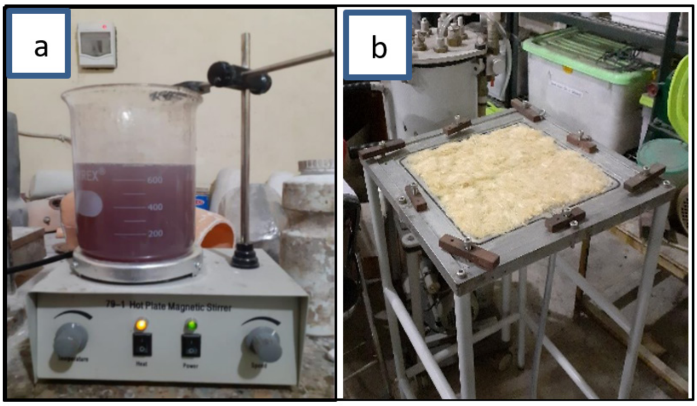

Figure 2. (a) magnetic stirrer, (b) Composite molding process

2.5 Tensile and Flexural Strength.

The implementation of tensile and flexural strength using the Universal Machine Test was carried out at the Sebelas Maret University, Surakarta, Indonesia. The tensile and bending strength testing of composite specimens involves the uses of ASTM D638-03 2003 and ASTM D 790-03 2003 standards, respectively. These tests were performed on composites with hot alkali treatment of 40 ^{0}C, 60 ^{0}C, 80 ^{0}C, and without treatment (UT).

2.6 Observation Scanning Electron Microscopy (SEM)

Microscopic SEM was carried out at the University of Malang Indonesia, Integrated Mathematics and Natural Sciences Faculty. SEM observations were performed using an instrument model from JEOL with the code JSM-610 PLUSS/LV to capture 2-dimensional images. These were used to monitor hot alkali-treated fibers and composite fractures from the test results.

Composite or fiber fractures were mounted on aluminum stubs with a platinum-coated sputter and observed for one minute at a pressure of two bars. SEM was used to observe the tensile test results, fracture, and cantala fiber after and without treatment.

2.7 Composite Water Absorption

The water absorption test was used to determine the ability of the composite to absorb liquid materials within a certain time. Incidentally, this increased the weight, volume, and bonding ability of its structure. The water absorption test involves the use of ASTM D 5229 in accordance with the following formula

$$\text{Water Absorption} = \frac{W - D}{D} x100\,\% \quad \text{(Alhijazi \textit{et al.}, 2020)}$$

W = Wet weight (grams)
D = dry weight (grams)

3. Results and Discussion

3.1 Density composite

The density test results of the cantala fiber reinforced composite with hot alkali treatment and without treatment, as well as the addition of 5% MCC with UPRs matrix, are shown in Table 1.

The test results showed that the density of the composite with alkali fiber treatment increased compared to the specimen without treatment. The composite density of the flax fiber with alkali treatment and resin matrix yielded 1.20 grams/cm^3 (Pandita *et al.,* 2014). Regenerative natural fibers treated with a 6% alkaline and polylactic acid (PLA) matrix resulted in a composite density of 1.32 grams/cm^3, while the untreated one was 1.28 grams/cm^3 (Ramamoorthy *et al.,* 2015). Changes in composite density were strongly influenced by the hot alkali treatment, which triggered the degradation of amorphous compounds such as hemicellulose, pectin, and lignin.

The increase in density was due to the fiber undergoing a new structural change from the cellulose II component, which was more stable than the unstable cellulose I (Jakob *et al.,* 2004). It was also caused by removing non-cellulosic materials with low density (Vandhini *et al.,* 2019). The test results stated that the hot alkali treatment increased density. However, the composite treated with hot alkali at 40 ^{0}C and excessive heating of the fiber decreased density.

3.2 Results of Tensile Strength and Modulus of Elasticity

Figures 3 and 4 showed the results of the tensile strength and the modulus of elasticity of the composites treated with hot alkali and the untreated specimen.

Test results showed an increase of 28.43% in the tensile strength of the composite after it was treated with hot alkali. Interestingly, this was also influenced by the addition of microcrystalline.

Table 1. Composite Density test results

No.	Code	Density (gram/cm^3)
1	UT + MCC + UPRs	1.19 ± 0.02
2	HA 40 + MCC + UPRs	1.22 ± 0.02
3	HA 60 + MCC + UPRs	1.21 ± 0.03
4	HA 80 + MCC + UPRs	1.21 ± 0.02

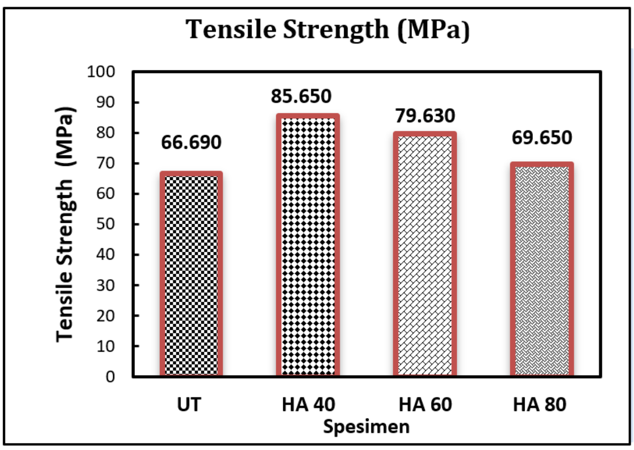

Figure 3. Tensile strength test results

The highest increase occurred in the HA 40 treatment, and afterward, it decreased due to the defibrillation of the cantala fibers due to heating and excessive concentration (Santos *et al.,* 2013).

The tensile strength of the composite also increased after king pineapple fibers were subjected to alkaline treatment due to loss of hemicellulose, pectin, lignin, and other impurities. (Sakuri *et al.,* 2021). In addition, Eleocharis Dulcis (purun rat) fibers also increased after the alkali treatment due to its coarseness, and this caused the interfacial bond between the matrix to be stronger (Haryanti *et al.,* 2020).

The application of MCC increases tensile strength because it can fill up empty holes due to its small size. MCC, with a small grain size, was able to cover all the voids in the composite. Its addition reduced the void area in the composite that needed to be filled by the matrix or fiber, thereby increasing the density and tensile strength values (Kiziltas *et al.,* 2019). The modulus of elasticity and tensile strength of hot alkali-treated composites are shown in Figure 4.

The elastic modulus test results showed an increase of 36.55% after hot alkali treatment was applied. The graph shows an increase in the composite's elastic modulus and tensile strength. The test results show that the elastic modulus graph is comparable to the tensile strength (Jakob *et al.,* 2004).

3.3 Result of flexural and modulus elastisiticity.

The flexural strength test results showed an increase after the treatment with hot alkali, considering that it reduced the amorphous content in cantala fiber. The maximum increase was 98.89 MPa, therefore it was raised by 31.41%. This was due to reduced fiber impurities such as hemicellulose, pectin, and lignin and is in line with the alkali treatment, which increased flexural strength (Sakurai *et al.,* 2020b). The addition of MCC also boosted flexural strength because this pure cellulose filled the voids in the composite (Chabros *et al.,* 2020). The research on adding MCC to pure unsaturated polyester stated that it increased the tensile and flexural strength of the composite (Sakurai *et al.,* 2019).

Figure 6 shows the results of the elastic modulus analysis of the composites' flexural strength.

The elastic modulus was increased after the hot alkali treatment. The highest results were obtained in HA 40, and this decreased thereafter. However, this depicts a graph that is proportional to the flexural strength, which is proportional to the elastic modulus results. (Sakuri *et al.,* 2019).

The flexural strength test results are shown in Figure 5.

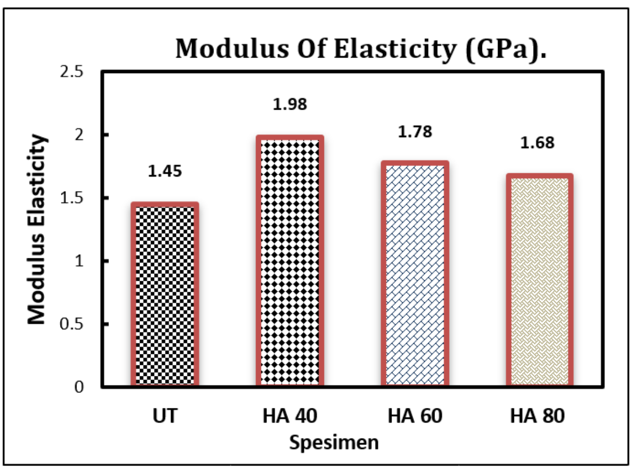

Figure 4. Modulus elasticity of tensile strength

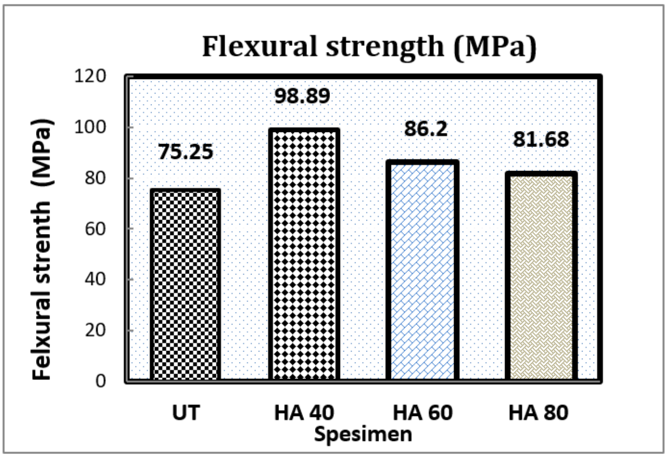

Figure 5. Flexural Strength Results

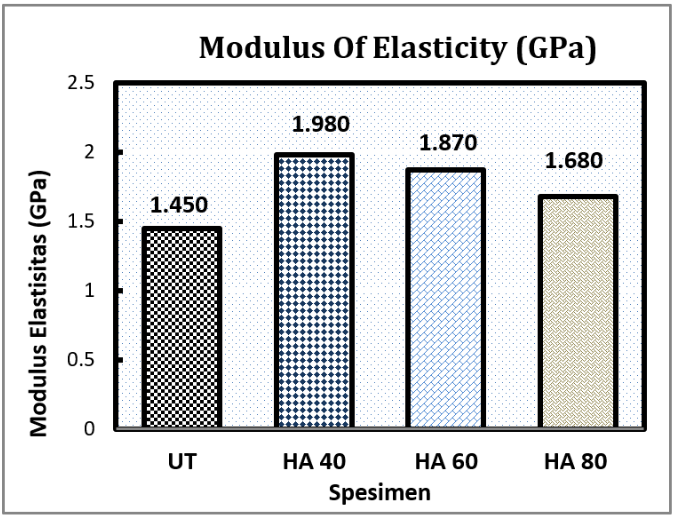

Figure 6. Elasticity Modulus flexural strength.

3.4 Observation Scanning Electron Microscopy (SEM) and Cellulose Content

SEM observations of cantala fiber with and without hot alkali treatment are shown in Figure 7

The results of SEM scanning were obtained at a voltage, magnification, and time of 20 Kv, 450 x, and 50 seconds. The untreated fiber was in a fibril network, still bound and wrapped by other substances such as pectin, lignin, hemicellulose, and impurities.

The fiber looked cleaner when subjected to hot alkali treatment for eight hours, and the binding started to disappear at a heating temperature of 40 ^{0}C, as shown in Figure 7b. This is also detected by reducing hemicellulose, pectin, wax, oil, and other extractive substances.

The subjection of salak fiber to alkali treatment has significantly made the surface smoother due to the loss of dirt, fat deposits, and wax (Ariawan *et al.,* 2020). In addition, the structure was altered and comprised of monofilaments almost not bonded to each other with extremely little lignin content (Gomes *et al.,* 2007).

Based on the SEM results, hot alkali treatment at 60 ^{0}C and 80 ^{0}C showed that the hemicellulose bonds of the fibers were damaged. In addition, hot alkali treatment with concentrated soaking time increases the surface of the fiber, resulting in the depletion of non-cellulosic or hemicellulose and lignin materials (Akhtar *et al.,* 2016). The longer the immersion in the concentrated alkali, the more the damage surface severity due to its corrosive effect and overheating, as shown in Figure 7c. and d.

The results of the microscopic observations of the cantala fiber-reinforced composites with and without hot alkali HA 40 treatment are shown in Figures 8 and 9. Microscopic observations were made with Scanning electron microscopic (SEM) to test for tensile strength.

The microscopic observations of composite fractures reinforced with untreated cantala fiber (UT) and the addition of MCC proved that the condition of the fiber is smooth and dominated mainly by its pull-out and debonding. According to Aydin *et al.* (2011), the pull-out event shows that the mechanical strength of the composite is lower than the fracture. The number of debonding and facial fractures in the composite indicates that the fiber contains a lot of lignin, pectin, hemicellulose, and other impurities (Astika *et al.,* 2014). Interfacial fractures in the composites depict the fibers' hydrophilic properties were still present. Cui *et al.* (2020) stated that this resulted in a poor interfacial bond between the fiber and the matrix.

Microscopic observations of the cantala fiber-reinforced composite subjected to 40 ^{0}C hot alkali treatment and the addition of MCC are shown in Figure 8. The results showed that the fiber broke and the tight bonds had more fractures than the pull-out and interphase. Although, this indicates that the bond strength between the fiber and the matrix is better. However, this is due to reduced hemicellulose, pectin, lignin, and other impurities. These results indicate that the bond strength between the matrix and fiber was better. This is caused by the decrease in the amorphous content of the cantala fiber. When subjected to hot alkali treatment, the fiber becomes coarser and cleaner due to the loss of impurities such as wax, pectin, and hemicellulose. However, excessive heating of the fiber leads to defibrillation of the excess hemicellulose element, which decreases the tensile and flexural strengths of the composite (Kaewkuk *et al.,* 2020).

The microscopic observations made with SEM reinforced the density test results depicting that the fiber was getting cleaner due to loss of amorphous content, thereby increasing its density. This also results in coarser fibers, and the interfacial bond becomes stronger, leading to increased tensile strength and flexibility. Furthermore, the addition of MCC to the micro size was able to close the cavities in the composite as well as increase the tensile strength and composite.

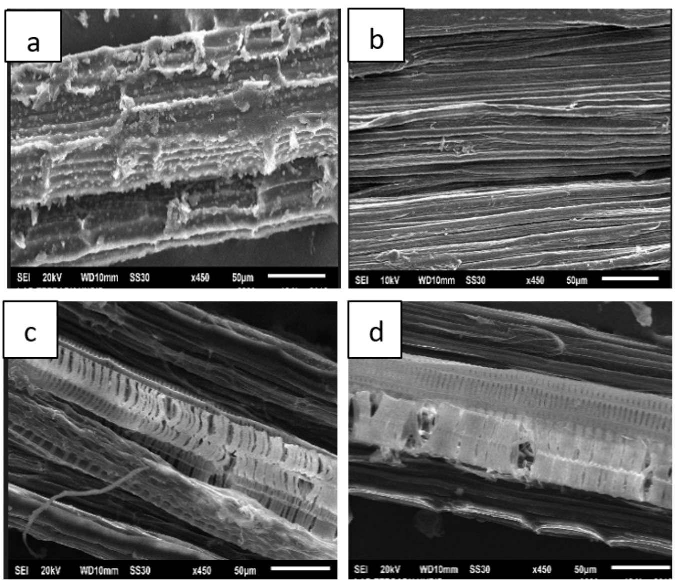

Figure 7. Observation Microscopic SEM of cantala fiber

The results of the observations carried out with SEM exhibited an increase in the cellulose and pentosan contents (SIN 14-0444-1989, SIL 0443-81) of the cantula fiber, as shown in Table 2.

The results showed an increase in the cellulose content after being subjected to hot alkali treatment. This is because the amorphous content in the cantala fiber had been lost. After the alkaline treatment, an increase and a decrease were detected in both the cellulose and hemicellulose contents (Raharjo *et al.,* 2018). Hot alkali testing carried out on jute fiber led to a decrease in the hemicellulose and other amorphous contents (Wang *et al.,* 2019). Therefore, the treatment increased the cellulose content and, at the same time, reduced that of the hemicellulose and other impurities.

3.5 Water Absorption Composite

The results of the water absorption test carried out on the cantala fiber reinforced composite without treatment, and the specimens treated with hot alkali and MCC are shown in Figure 10.

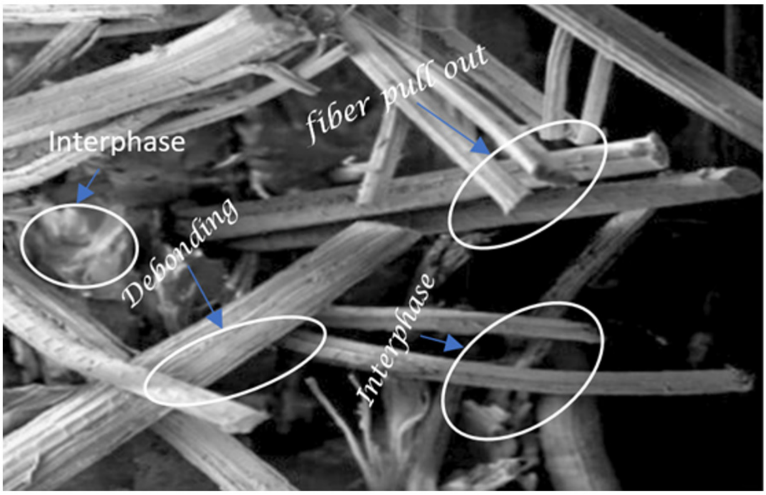

Figure 8. SEM Untreated fiber composite

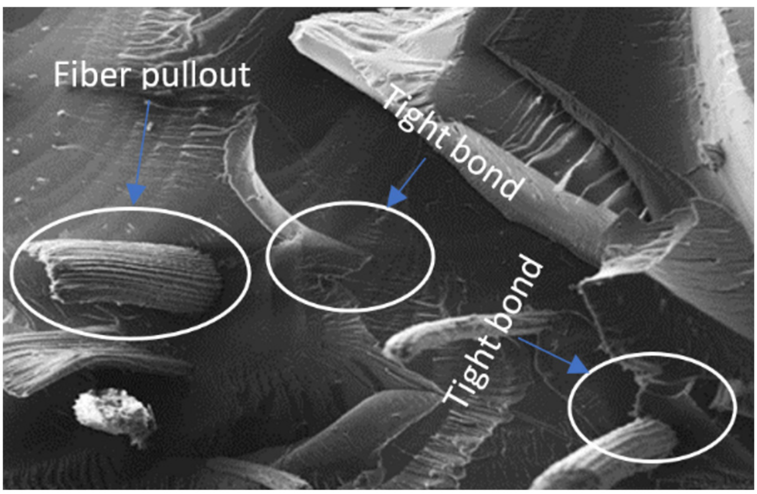

Figure 9. SEM HA 40 fiber composite

Table 2. Cellulose and hemicellulose content

Treatment	Cellulose (%)	Hemicellulose (%)
Untreated (UT)	64.17	24.31
Treatment HA 40	74.45	7.45
Treatment HA 60	79.67	4.34
Treatment HA 80	73.33	7.99

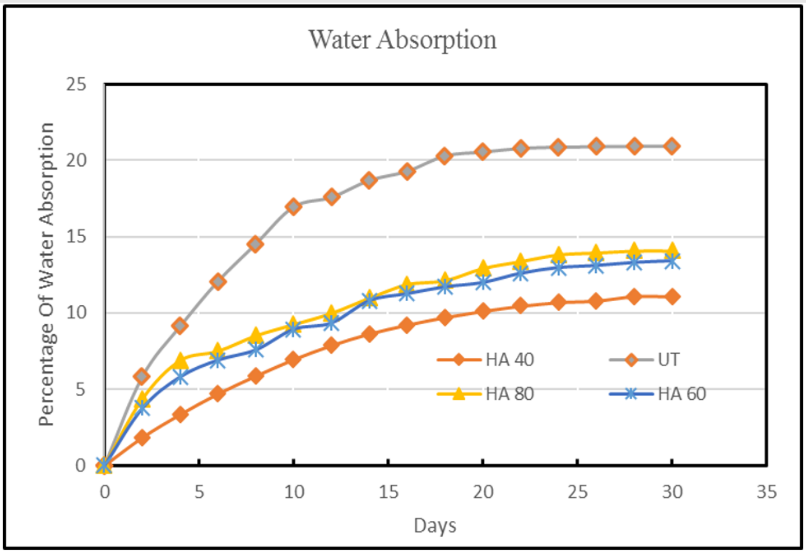

Figure 10. Water absorption

The test results show that water seeps into the composite, forming a linear graph, and slows down in the first and third weeks of immersion. Water absorption in the composite reaches the equilibrium point from the 28th to 30th day of immersion. Then, it slows down while the sample approaches saturation. This water absorption model is in line with the Fickian diffusion model and process (Munoz *et al.,* 2015). Meanwhile, the Fickian model is preceded by the rapid absorption of water, followed by saturation in the next stage (Salim *et al.,* 2020).

Natural fibers such as honey pineapple play a massive role in the absorption of fluids due to its nature which tends to be hydrophilic (Kord *et al.,* 2014). Hemicellulose absorbs water more easily than crystalline cellulose elements (Punyamurthy *et al.,* 2012). The removal or reduction of hemicellulose and lignin in HPF after treatment has been proven by X-ray diffraction, SEM, and IFSS tests. The higher the cellulose and hemicellulose contents in the composite, the greater the hydroxyl (-OH) percentage, which is the main contributor to water absorption (Hamdan *et al.,* 2019).

4. Conclusion

In conclusion, using hot alkali treatment on cantala fiber and adding microcrystalline cellulose (MCC) increased the tensile strength and elastic modulus by 28.43 % and 36.55%, respectively. This was mainly due to the cleaner cantala fiber before the treatment alongside the addition of microcrystalline cellulose. The flexural strength of the composite increased when subjected to the hot alkali and HA 40 treatment from 75.25 to 98.89 MPa. In addition, the elastic modulus and flexural strength increased proportionally after the specimen was subjected to the HA 40 treatment by 36.55%. SEM observations show that the fiber was getting cleaner and fractured with tighter bonds in the composite due to the hot alkali treatment. Furthermore, this treatment and the addition of MCC reduce water absorption.

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