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INVESTIGATION INTERFACIAL SHEAR STRENGTH AND MECHANICAL PROPERTIS OF ALKALI TREATED HONEY PINEAPPLE FIBER/ MICROCRYSTALLINE CELLULOSE COMPOSITE

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Abstract

The research objective was investigate the effect of alkali treatment on thermal stability, interfacial shear strength, mechanical properties and waterabsorption in composite of honey pineapple fiber (HPF). HPF was not given and treated by soaking in a mixture of 6% wt sodium hydroxide. Mixing between Unsaturated Polyester (UPRs) and Microcrystalline Cellulose (MCC) applied the Taguchi test with 5% MCC, 250 RPm, 40 oC temperature, and 30 minutes. The composites were mold using the vacuum infusion method and curing at 60 o C for 120 minutes. Interfacial shear strength test showed 9 hours of immersion, gave strength of 3.49 MPa, an increase of 37.39%. Tensile strength increased by 30.4%,the flexural strength of the composite increased by 51.08%. The results of the SEM test showed the fiber untreated was still smooth and there were several interface gaps. The water absorption test showed that the untreated fiber had a higher absorption rate.

Keywords: *Honey Pineapple Fiber, Microcrystalline Cellulose, Interfacial Shear Strength, Mechanical Properties.*

1. INTRODUCTION

Honey pineapple plants in Indonesia were found in Purbalingga, precisely on the slopes of Mount Slamet, Central Java, Indonesia. Honey pineapple plants were harvested at the age of 18-24 months, most of which were ripe. The shape of the honey pineapple leaves was pointed at the ends, has a blackish green color, and the leaf length was between 55 - 75 cm [1]. Honey pineapple fiber (HPF) was taken from the leaves by manual extraction. The process was used for components attached to cellulose fibers, namely pectin, lignin, hemicellulose, and other impuritis without damaging the cellulose fiber. Utilization of natural fibers such as honey pineapple as a substitute for synthetic fiber continues to be developed, because natural fibers have advantages such as unlimited availability, biodegradability, and high toughness [2]. Low prices, environmentally friendly, renewable, energy-efficient, high rigidity, difficult to decompose, types and variations of waste as part of evaluating the use of natural fiber in applications [3].

The disadvantages of honey pineapple fiber as well as natural fibers are high polarity, non-abrasive, and less compatible with polymers [4]. Various approaches were used to

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improve the characteristics of natural fibers such as fumigation [5], silane treatment [6], permanganate treatment [7]. Alkali treatment has shown an increase existence between cantala fiber and unsaturated polyester, thereby increasing the strength of the prosthetic socket composite [8]. Simultaneous modification of the fiber surface improves the wetting of the fiber with the matrix and promotes better mechanical properties between the banana-coir fiber and the matrix on automotive application [9]. Surface modification of pineapple leaf fiber (PLF) was used to increase the strong adhesion between fiber and polymer.[10]

Alkali treatment can change the crystalline structure of cellulose to be smaller due to the loss of amorphus in the fiber. Alkali treatment was able to increase the interfacial bond between the fiber and the matrix because the fiber surface was rougher due to the loss of fiber amourphages such as pectin, lignin, and hemicellulose and increased mechanical properties of the composite. [11]. MCC added to the matrix can increase the tensile strength and modulus of elasticity considering that MCC is a pure cellulose particle and is micro-sized so that it can fill holes in the composite [12]. The addition of MCC was able to increase thermal stability, reduce the coefficient of thermal expansion, have a more pronounced bond effectiveness, and increase shear strength. [13]. This research is still very relevant to test the increase in the interfacial bond strength in the fiber and the addition of MCC to increase the tensile strength of the composite.

2. MATERIAL AND METHOD

2.1. MATERIAL

Honey pineapple fiber was obtained from local farmers in Purbalingga, Central Java, Indonesia. Fiber extraction from the honey pineapple tree trunk system and washed with water to clean and dried at room temperature. Sodium hydroxide (NaOH) and aquades of 98% purity were obtained from TJ Kimia Purwokerto Indonesia Store. Unsaturated polyester with Yukalac BQTN 157 series and Methyl ethyl ketone perioxide as catalyst was purchased from PT Justus Kimia Raya Semarang Indonesia. Microcrystalline cellulose (MCC) 310697 series, size 20 micro, density 1.56 gram/cm3 obtained from PT Sigma Aldrich Jakarta Indonesia. HPF leaves are retting manually to get the fiber.

Fig. 1. Honey Pineapple plants

Fig.2. Honey Pineapple fiber

2.2. METHODS

HPF fibers were soaked in a mixture of distilled water and sodium hydroxide (NaOH) with a concentration of 6% wt for 0, 3, 6, 9, and 12 hours at room temperature. The HPF fibers were cleaned with running tap water to pH ± 7. The HPF fiber at room temperature was dried for 24 hours and put in an oven at 60 ^{o}C for 10 hours.

2.3. FIBER AND COMPOSITE DENSITY

Fiber density testing uses ASTM 792-13 the year 2013. The method used with the Precisa XT 220 A Balance (Indonetwork Jakarta, Indonesia) by comparing the weight in fluids and the air, using the formula:

$$Density = (g/cm^3) = \frac{a}{b+a} x.\delta.f \quad (1)$$

Where a, is spesific gravity (g/cm^3) in the air and b, is the specific gravity (g/cm^3) in fluid. Test were performed at room temperature using biodiesel with a density of 0.867 g/cm^3.

2.4. X-RAY DIFFRACTION

The X-ray diffraction test of honey pineapple fiber in the cellulose structure was measured at room temperature, carried out at the Integrated Laboratory of Diponegoro University, Semarang Indonesia. X-ray diffraction uses CuK radiation or n = 1.54 A and the radiation intensity was recorded from 2 theta = 1000 in 2 theta steps with a voltage of 30 Kv and a current of 30 mA. Calculation of crystallinity index (Cr.I) and degree of crystallinity (%) can be calculated by the Segal method as in the following equation:

$$Cr.I = \frac{I_{002} - I_{am}}{I_{002}} x\,100\,\% \quad (2)$$

The peak sample intensity was based on the Miller index (002) with 2 theta angles ranging from 22 degrees to 23 degrees. The intensity of the non-crystalline content is at the peak angle of 2 theta = 18 degrees.

2.5. THERMOGRAVIMETRI ANALYSIS

The Thermogravimetry (TGA) analysis technique uses the Perkin Elmer Pyris diamont TGA 6 analyzer model at the Integrated MIPA Laboratory, Sebelas Maret Surakarta Indonesia. All HPF samples were scanned at room temperature increasing from 30^o C to 600^o C at a rate range of 100^o C/min. The TGA test was carried out in a nitrogen environment.

2.6. INTERFACIAL SHEAR STRENGTH (IFSS)

The interfacial shear strength (IFSS) testing technique was carried out by attaching 60 mm long HPF to a mixture of MCC, Unsaturated polyester (UPRs) and Mekpo. The HPF was attached to a cardboard with a hole in the middle and dried for 120 minutes. The clamping distance from the hole is 50 mm with a tensile speed of 250 mm/min.The single fiber tensile test was repeated 30 times. Paper from both sides of the hole is cut for maximum traction. The HPF is pulled until the HPF is released from the Matrix. The diameter of the HPF is measured at the top, middle, and bottom sides. The test used a textile pulling machine model Tenso 300 with type E newton unit at the Textile Laboratory of the Indonesian Islamic University Yogyakarta Indonesia.

2.7. COMPOSITE FABRICATION

Composite production begins by cutting the honey pineapple fiber 10 mm long and installing it randomly on the molding site. Composite molding with variations of 65% matrix, 30% fiber and 5% MCC. Unsaturated polyester was mixed with MCC in a container and spun at 250 Rpm, at 40^{o}C for 30 minutes [14]. The ready matrix was added with 1% methyl ethyl ketone peroxide catalyst and stirred. Put unsaturated polyester and MCC with additional

catalyst in the mold using a vacuum infusion system. The composite molding results were put in an oven with a temperature of 60^{o} C for 120 minutes and cut the specimens.

2.8. TENSILE, FLEXURAL STRENGTH AND MODULUS ELASTICITY

The ASTM D638-03 (2003) standard was used for testing the tensile strength of composites and the ASTM D790-03 (2003) standard was used for flexural strength. Tensile strength and flexural strength testing using Universal Testing Machine. Tensile strength and flexural strength tests were carried out at the Materials Laboratory of Sebelas Maret University, Surakarta Indonesia. Tensile and flexural strength specimens were tested for 5 repetitions.

2.9. SCANNING ELECTRON MICROSCOPY

Scanning electron microscope (SEM) observation using the instrument model JSM - 610 PLUS/LV from JEOL. SEM was carried out at the Integrated Mathematics and Natural Sciences Laboratory, State University of Malang. SEM testing was used to capture 2-dimensional images of untreated and alkaline-treated composite fractures. Indonesia. The composite with small dimensions was mounted on a platinum-coated sheet of aluminum and observed carefully for 1 minute at a pressure of 2 bar. The SEM test was carried out after the tensile strength test to determine the fracture results in the specimen.

2.10. WATER ABSORTION COMPOSITE

The water absorption test on the composite was used to test the ability of the composite to absorb water at a certain time. Water absorption in the composite will result in an increase in the weight, volume, and bonding ability of the composite structure. Water absorption testing using ASTM D 5229 with the formula:

$$\text{Water absortion} : = \frac{W - D}{D} x100\% \quad (3)$$

W = Wet weight (gram)

D = Dry weight (gram)

3. RESULTS AND DISCUSSION

3.1. FIBER DENSITY

Tests for the density of HPF treated and untreated were given the results in Table 1

Table 1. Fiber Density

No.	Code	Descryption	Density (g/cm^3)
1	UT	Fiber without treated	1.09 ± 0.02
2	AT 3	Fiber treated alkali 3 h	1.23 ± 0.03
3	AT 6	Fiber treated alkali 6 h	1.31 ± 0.04
4	AT 9	Fiber treated alkali 9 h	1.37 ± 0.05
5	AT 12	Fiber treated alkali 12 h	1.38 ± 0.06

The results of the density test showed that the HPF after being given treatment experienced an increase in weight compared to before treatment. The increase in HPF density was in line with the length of immersion time and the concentration of NaOH. [15]. The increase in density is due to the loss of dense and low non-cellulose material [16]. Alkali treatment has formed a new structure of the cellulose II component which was more stable and compact when compared to cellulose I [17]. The increase in fiber density due to alkaline treatment was found in flax fiber [18], borassus fiber [19], and cantala fiber increased by ± 30% [20]. Alkaline treatment of kenaf fiber can increase fiber density and decrease fiber volume because the fiber diameter was reduced due to the loss of amorphous and other impurities in the fiber.

3.2. X-RAY DIFFRACTION

It can be seen in Figure 3 that the honey pineapple fibers have formed with 3 large peaks at 15.2 degrees and 22.12 degrees, and 44 degrees. The first peak represents the region or part associated with the 110 and 002 crystal planes [21], while the 040 peak represents the final diffraction peak of plant fibers in the [22] crystal plane. Cellulose structure is seen at peaks between 22 degrees to 23 degrees which displays the original cellulose structure. The structure of the HPF can be seen on the diffatogram in both valleys between the two theta peaks of about 18 degrees. Observing the test results in the X ray diffraction HPF fraction observed 3 peaks of the highest and lowest intensity as shown in Figure 3.

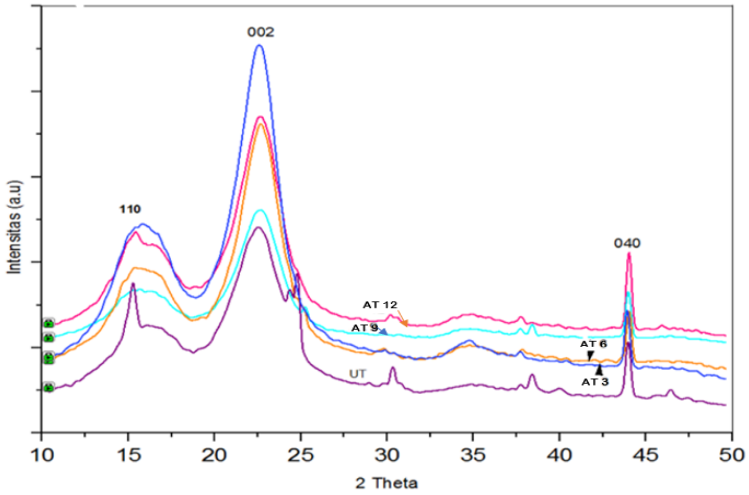


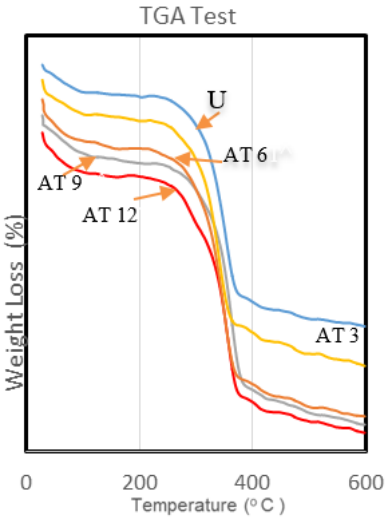
Fig. 3. X-ray diffraction test

Figure 3 . showing untreated fiber (UT), the cellulose structure was at the peak of the diffraction angle of 21.24 degrees with an intensity of 1175, and the amorphous part or valley was at the diffraction of 18.68 degrees with a crystallinity index of 54.55%. The AT 3 treatment showed the presence of a diffracted cellulose structure at an angle of 22.66 degrees with an intensity of 2242. Amorphous at an angle of 18.84 degrees with an intensity of 639 and a crystallinity index of 71.48%. In the AT 6 treatment, the cellulose structure was seen at a diffraction angle of 22.68 degrees with an intensity of 1725, and amorphous at an angle of 18.66 diffractions and resulted in a crystallinity index of 74.64%. The diffatogram of the cellulose structure in the AT 9 treatment occurred at an angle of 22.72 degrees with an intensity of 945, while the amorphous diffraction was at 18.56 with a crystallinity index of 75.87%. The decrease in crystallinity index occurred in AT 12 treatment with a crystallinity index of 70.25%. X-ray diffraction test concluded that alkali treatment was able to increase the crystallinity index by 21.32%. The increase in crystallinity index indicates that alkaline

treatment of kenaf fiber has been able to reduce hemicellulose, pectin, lignin, and other amorphous elements.

3.3. THERMOGRAVIMETRY ANALYSIS (TGA) AND DERIVATIVE TGA

The results of the TGA and Derivative TGA tests were shown in Figure 4a & 4b. as a basis for conducting the analysis.



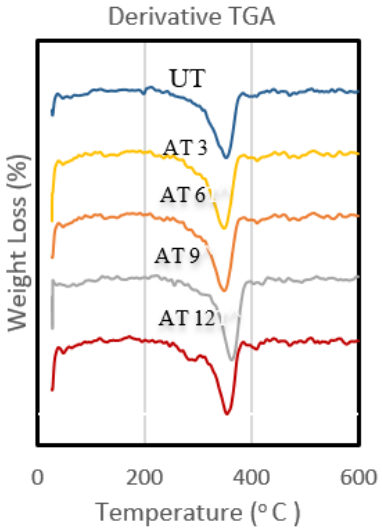


Fig 4a. Thermogravimetry Analisis **Fig4b**. Derivative TGA

Fibers without alkali treatment showed a weight loss of 7.68% at temperatures below 100 °C. Alkaline treatment for 2 hours gave a weight reduction of 6.78% and for 12 hours experienced a weight reduction of 5.82%. Rasyed noted there was a 12% weight reduction associated with water evaporation for bamboo pulp [23]. This desorption was related to the evaporation of water that occurs between temperatures of 25 - 150 °C [24]. Decomposition occurred at the HPF at a temperature of 248 °C and ended at a temperature of 389 °C, a weight loss of 45.23% occurred. The complete calculation of thermogravimetry analysis can be seen in Table 3.

Table 3. Thermogravimetri Analysis

Treatment	Code	30 - 200° C Final temp./ Wt loss (%)	200 – 400° C. Initial temp./Wt loss (%).	400 - 600° C Initial temp./ Wt loss (%).
Untreatment	UT	88.82/7.68	267.53/58.34	424.45/14.45
Alkali	AT 3	92.81/6.78	269.34/54.78	419.23/12.45
Alkali	AT 6	94.43/6.18	271.45/53.12	427.85/10.83
Alkali	AT 9	95.84/5.88	273.43/50.36	418.34/9.39
Alkali	AT 12	98.38/5.82	276.87/50.91	414.56/9.67

The second stage of treatment sign fiber decomposed at a temperature of 267.53 °C with a weight reduction of 58.34 %. This decrease was smaller than the study result [25] of 56.9% in cantula fiber. The second stage of decomposition occurs at temperatures between 150 – 380 °C [26]. The degradation of HPF in stage 2 between 200 - 300 °C reflects the degradation of hemicellulose and some lignin [27]. The Peak decomposition occurred at a temperature of 269.34 °C and a weight loss of 54.78% occurs at AT 3. Decomposition of AT 12 occurs at a

temperature of 276.87 ºC and a weight loss of 38.91%. The degradation in step 2 was observed by [28] in sisal fibers and in agave augustifola fibers [29]. HPF degradation before and after alkali treatment showed that the fiber after treatment was able to increase the value of thermal stability due to the loss of hemicellulose, lignin, and pectin in the fiber [30]. The third decomposition was related to the degradation of hemicellulose and lignin. Decomposition of UT fiber was 14.45% and fiber with AT 12 treatment resulted in a weight loss of 9.67%. The DTGA graph shows the peak of desorption of water absorption at To, T2 degradation of hemicellulose and peak position of T3 degradation of lignin [31].The results of the DTGA graph are as shown in Table 4.

Table 4. Derivative TGA Alkali Treatment

Treatment	To (ºC)	T2 (ºC)	T3 (ºC)	Residue
UT	55. 10	354.21	437.34	21.04
AT 3	58.67	364.91	464.08	25.23
AT 6	58.93	354.78	467.06	27.54
AT 9	60.43	359.98	478.34	29.82
AT 12	58.78	354.23	474.92	26.82

The residue of the untreated fiber showed a smaller amount than the fiber after treatment. Fiber residue without treatment was 21.04% and after 9 hours alkaline treatment was 29.82 %.

3.4. IFSS (INTERFACIAL SHEAR STRENGTH)

The IFSS test was carried out by attaching kenaf fiber to a mixture of UPRs and Microcrystalline cellulose and pasting it on thick paper (manila). The test paper is made a hole in the middle and if the test is carried out the paper will be cut on the right and left sides so that only fibers are drawn. The fiber was pulled with a testing machine to free it from its matrix bonds. The IFFS test was successful if the HPF was detached from the matrix bond. The average diameter of the HPF was calculated to determine the shear stress as shown in Figure 5.

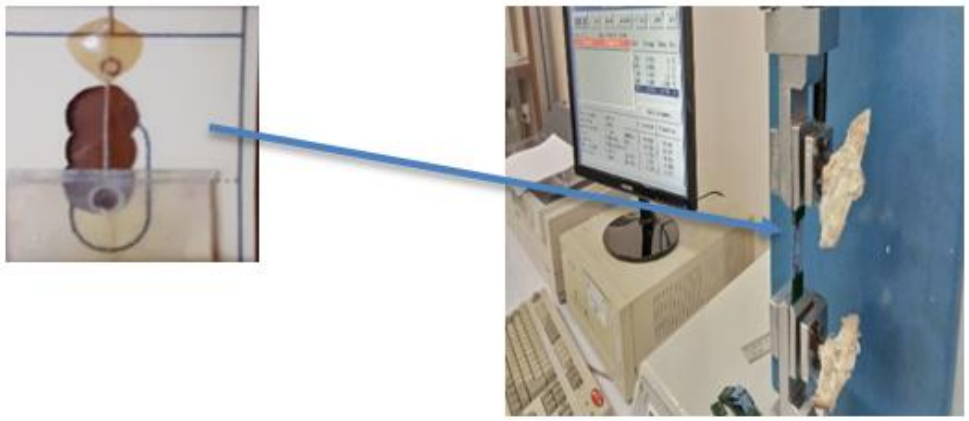


Fig.5. IFFS Test

The results of the IFSS test on untreated fiber showed a figure of 2.51 MPa. This result is higher than the test result and lower than the research result. After the test, the shear force

increased after alkaline treatment. The increase in shear force occurred with the TA 3 treatment 3.28 MPa and the highest test results were obtained in the TA 9 treatment of 3.49 MPa. The test results were as shown in Fig 6.

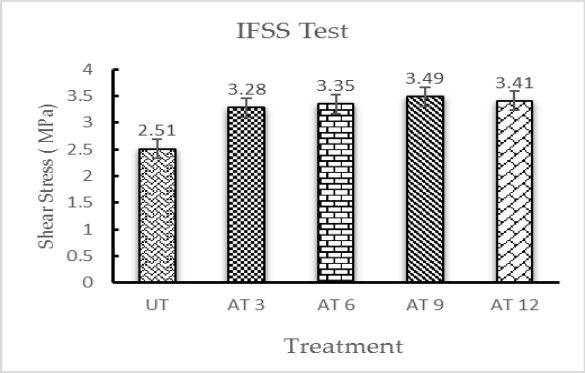


Fig. 6. Result IFSS test

An increase in IFSS also occurred in alkaline treatment with a concentration of 2% NaOH on cantala fiber with an immersion time of 20 hours from 2.44 MPa to 3.49 MPa [27]. and from 2.49 to 3.67 MPa [8]. Typha fiber with 5% NaOH alkaline treatment also increased from 1.48 to 3.05 MPa. [32]. The results above show that the alkali treatment makes the surface roughness of the fiber due to the reduction of hemicellulose, lignin, wax, and pectin so as used to increase the interfacial tensile strength between the HPF and the matrix [33]. Alkali treatment has provided stronger hydrogen bonds and is able to increase the interfacial adhesion between the fiber and the matrix so as to increase the tensile strength of the composite. [34]. The results of the IFSS test showed that the alkaline treatment of AT 9 was better, this was supported by the results of TGA showing a lower rate of desorption and decomposition than UT, AT 3, AT 6, and AT 12..

3.5. COMPOSITE DENSITY

The results of the density test on HPF-reinforced composites showed an increase after alkaline treatment. Table 5 shows the results of the composite density test.

Table 5. Composites Density

No.	Code	Descryption	Density (g/cm^3)
1	UT	Fiber untreated	1.29 ± 0.02
2	AT 3	alkali 3 h + MCC	1.38 ± 0.03
3	AT 6	alkali 6 h + MCC	1.45 ± 0.04
4	AT 9	alkali 9 h + MCC	1.47 ± 0.05
5	AT 12	alkali 12 h + MCC	1.46 ± 0.06

Base treatment with longer immersion resulted in an increase in the density of the composite, this was because the fiber density also increased. Composites reinforced with polylactic acid with regenerative cellulose reinforcement with 6% NaOH resulted in a density of 1.32 g/cm^3, while for 10% NaOH the density was 1.31 g/cm^3 and fiber composites without treatment were 1.28 g/cm^3. Density with 12 hours treatment decreased, this was due to the polymorphic transformation of cellulose 1 to cellulose 2, so that the area of the crystal fiber decreased which resulted in a decrease in density [35]. The flax fiber-reinforced composite with a resin

matrix yielded a density of 1.2 g/cm^3. [36]. The addition of MCC to the composite has a density effect because the MCC density value was higher. The addition of MCC to the composite will reduce the void area that should be filled by the matrix or fiber so that the density value increases [37].

3.6. TENSILE STRENGTH

Tensile strength was tested using ASTM D 638-03 along with its modulus of elasticity. The tensile strength of the untreated honey pineapple fiber reinforced composite resulted in a strength of 52.63 MPa. Tensile strength increased after alkali treatment for AT 3 of 69.78 MPa and the highest with 9 hours of alkaline treatment of 78.89 MPa. The graph of the test results as shown in Fig 7.

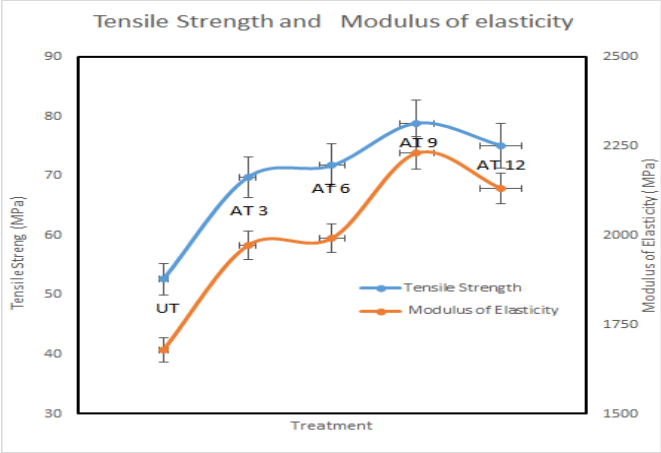


Fig. 7. Tensile strength and modulus of elasticyti

The tensile strength of the composite increased until the AT 9 treatment was 79.82 MPa. The increase in the tensile strength of the composite was due to the strength of the interfacial bond between the fiber and the matrix. The increased tensile strength was also due to the MCC mixture which is pure cellulose. MCC is able to fill the micro holes in the composite so that the strength increases [38]. After AT 9 treatment, the tensile strength decreased. The decrease in tensile strength is caused by the length of immersion time which affects HPF defibrillation [8]. The graph of the tensile test results and the modulus have similar results according to the test [19].

3.7. FLEXURAL TEST.

The results of the flexural strength test on HPF and MCC reinforced composites are shown in Figures 8.a and 8.b.

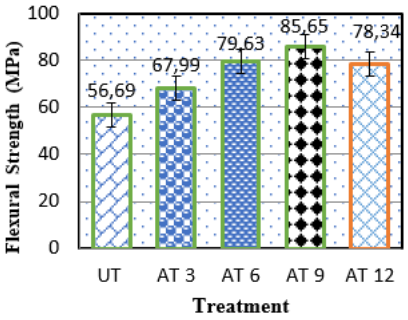


Fig. 8.a. Flexural Tensile

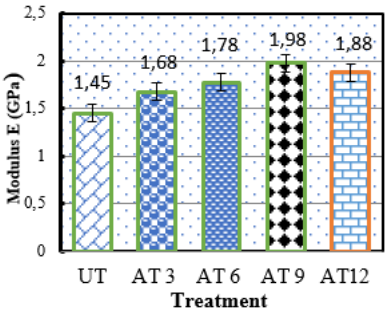


Fig. 8.b. Modulus elasticity.

In Figure 8.b, it can be seen that the flexural strength of the composite reinforced with HPF without treatment was 56.69 MPa and the AT 9 treatment was 85.65 MPa, which experienced an increase in flexural strength of 51.08%. The yield of flexural strength is higher than the composite reinforced with kenaf fiber and epoxy matrix. [39]. Research on composites reinforced with flax fiber and bioepoxy showed a flexural strength of 90.2 MPa.[40]. This increased flexural strength was due to the clean HPF due to alkali treatment due to the loss of amourphus such as hemicellulose, pectin, and lignin. The increased flexural strength was also caused by the addition of MCC as pure cellulose in the composite. The micro MCC size has been able to close the hole in the composite. The test results on the graph and the modulus of elasticity show results that were proportional to the flexural strength [19].

3.8. OBSERVATION SCANNING ELECTRONE MICROSCOPY

The fracture of the tensile strength test results for HPF and MCC reinforced composites was observed by SEM. The use of SEM to observe the broken fiber, porosity, fiber pulled, and the quality of the interfacial bond between the fiber and the matrix. SEM observations as shown in Fig. 9.

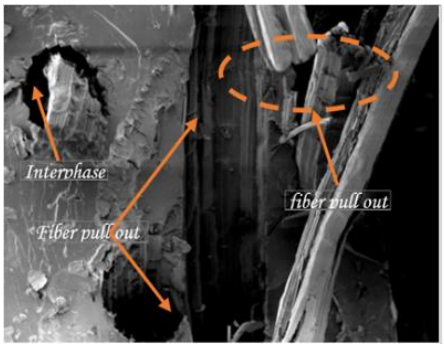


Fig. 9.a. SEM - UT

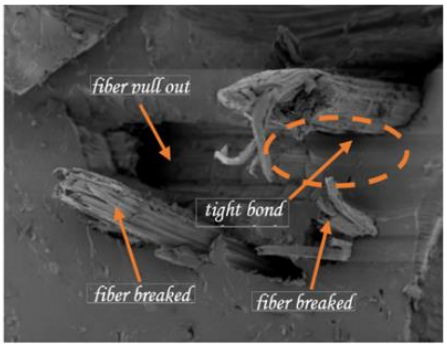


Fig.9.b. SEM – AT 9

The results of the composite test with untreated fiber showed the condition of the fiber that looked still smooth and there were several interphase gaps and was dominated by fiber pullout. Interphase is due to the fact that the fiber still contains hemicellulose, lignin, pectin, and other impurities. The interface in the composite can be caused by the difference between the hydrophobic properties of the polymer and the hydrophilic properties of natural fibers, thus causing poor mechanical interlocking bonds and fiber pullout fractures [41]. Fig 9b. The results of the SEM morphology of HPF-reinforced composites with AT 9 treatment. The results showed that the HPF broke in the interphase condition with tight bonds due to the reduced content of hemicellulose, pectin, and lignin. The reduction of impurities in the HPF results in an increase in the interfacial bond between the fiber and the matrix.

The loss of amorphous in HPF due to alkaline treatment resulted in coarser fibers and increased interfacial bonding which resulted in optimal transfer from matrix to fiber so that the composite strength increased. Adding MCC to the composite will close the cavities because of its small size so as to increase the strength of the composite. MCC in the composite produces a smooth surface structure due to the micro particle size. Adding MCC can increase crystallinity because there is no amourphus content and the bond between MCC-matrix was getting better so that it can increase mechanical strength.

3.9. WATER ABSORPTION IN COMPOSITES

Water absorption test on HPF and MCC composites as shown in Figure 10.

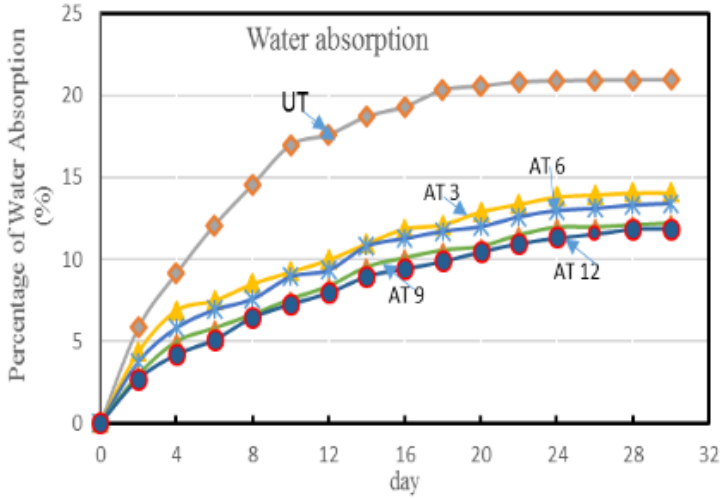


Fig. 10. Water absorption

The test results show that water penetrates into the composite forming a linear graph in the first week, and begins to slow down in the third week of immersion. Water absorption reaches a balance between 28 to 30 days of immersion. Water absorption will slow down and the sample will approach saturation condition. The water absorption behavior of this model can be said to be in line with the Fickian-type diffusion model and process [42]. The water absorption behavior of the Fickian model is preceded by rapid absorption of water and followed by saturation in the next absorption stage. [43]. Natural fibers such as honey pineapple play a large role in the ability to absorb fluids, due to the nature of natural fibers which tend to be hydrophilic . Hemicellulose absorbs water more easily than crystalline cellulose elements [44]. 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 content in the composite, the higher the hydroxyl (-OH) percentage and was the main contributor to water absorption.

4. CONCLUSION

Honey fiber pineapple used to strengthen polymer composites has low density, low cost, continuous availability, and lower solidity when compared to synthetic composites, thus providing advantages in commercial applications. Alkaline treatment of honey pineapple fiber can increase fiber density due to the loss of some amourpus in the fiber. The alkali treated fiber has better thermal stability. The interfacial bond of fiber after alkaline treatment showed an increase, due to the decrease in hemicellulose, pectin, lignin, and other impurities. Physical and mechanical properties improved due to alkali treatment and addition of microcrystalline cellulose. The results of water absorption showed that the fiber experienced a decrease in water absorption after alkaline treatment.

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