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Effects of Alkali (NaOH) Treatment of Banana Bunch Fiber (*Musa Paradisiaca*) on the Tensile Properties of Banana Bunch Fiber/Unsaturated Polyester Composites

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ABSTRACT

The increase in environmental awareness worldwide has driven the design of environmentally friendly materials, making this research on the effects of alkali treatment on banana bunch fiber composites particularly significant. The detrimental environmental impact has shifted attention from synthetic fibers to natural fibers. Natural fiber composites have attracted the attention of material engineers due to their ability to decompose, abundant and sustainable availability, low density, and high specific strength. Many sources of natural fibers have been used for composite reinforcement, such as jute, kenaf, sisal, coconut fiber, pineapple fiber, and banana stem fiber. Fiber treatment is necessary to enhance the compatibility between polymer matrix and natural fibers. The most commonly used method is alkali treatment using a sodium hydroxide (NaOH) solution. This research, which investigates the effect of alkali concentration (NaOH) on the properties of unsaturated polyester reinforced with Kepok banana (Musa paradisiaca) bunch fiber, significantly contributes to the global shift towards environmentally friendly materials, a movement that you, as researchers and professionals, are actively part of. Banana bunch fibers were treated with alkali solutions at 2, 4, 6, and 8% concentrations for 2 hours. The fiber content in the matrix was 40%. The tests that were conducted included Fourier Transform Infrared (FTIR) spectroscopy, X-ray diffraction (XRD), tensile testing, and scanning electron microscopy (SEM). It was found that alkali treatment improved the tensile strength and elastic modulus of unsaturated polyester/banana bunch fiber composites. The 2% alkali treatment provided the highest tensile strength and elastic modulus, increasing by 92.13% and 273%, respectively. FTIR, XRD, and SEM analysis confirmed the optimal results of the 2% alkali treatment.

1. Introduction

The increased environmental awareness worldwide has encouraged the design of environmentally friendly materials. Synthetic fibers such as glass, carbon, and aramid are widely used in polymer composites because they have high stiffness and strength. However, synthetic fibers have serious disadvantages in biodegradability, high pre-processing costs, recycling, energy consumption, machine abrasion, and health hazards [1]. The adverse environmental impacts have shifted attention from synthetic fibers to natural fibers. The introduction of biofibers, such as natural fibers from renewable resources, has attracted attention for use as reinforcement in polymer

composites to provide environmental benefits concerning biodegradability and utilization of renewable materials [1].

Natural fiber composites interest materials engineers because they are biodegradable, abundant, sustainable, low-density, and have high specific strength. According to Sivaranjana & Arumugaprabu [2], composites produced from natural fibers have attracted the interest of researchers because of their good strength, lower cost, and, more importantly, their easy availability. Because of these advantages, natural fiber composites will be widely developed. However, natural fibers have shortcomings that must be overcome using appropriate solutions. Natural fibers are hydrophilic because they contain lignin and hemicellulose. According to Zaman & Ruhul [3], the main disadvantages of natural fiber-reinforced composites are high moisture absorption properties, poor wettability, and poor fiber-matrix adhesion. Chemical treatment must be carried out to minimize poor compatibility between hydrophobic thermoplastics and hydrophilic natural fibers and to improve these composites' mechanical properties. Lignin and hemicellulose have a predominantly amorphous structure, so they provide less reinforcement than cellulose, which has a predominantly crystalline structure. Lignin and hemicellulose hinder the bond between the polymer matrix and cellulose, so they must be reduced or removed from the fiber as much as possible.

Many natural fiber sources have strengthened composites, such as jute fiber, kenaf, sisal, coconut fiber, pineapple fiber, and banana stem fiber. Fiber treatment must be carried out to increase the compatibility of polymer matrices and natural fibers. The most common method is alkaline treatment using sodium hydroxide (NaOH) solution. Yudhanto et al. [4] researched the effect of surface treatment of sisal fiber with a 5% NaOH solution. This research used the variation of soaking time, namely 0, 2, 4, 6, and 8 hours. This research found that the soaking time affected the tensile strength of the sisal fiber. If the fiber was soaked for too long, the tensile strength of the sisal fiber decreased. Maryanti et al. [5] conducted research by varying the concentration of NaOH solution in coconut fiber. The highest tensile strength value of the coconut fiber-polyester composite occurred in the composite with fibers treated with 5% NaOH, namely 97.356 MPa. Paundra et al. [6] researched a hybrid of banana stem fiber and areca nut fiber by varying the ratio of banana stem fiber and areca nut fiber with a fixed volume fraction of 30% to see the effect on tensile strength. The maximum tensile strength was obtained at a ratio of banana stem fiber to areca fiber of 1/1 or 15%/15%. Barrera-Fajardo et al. [7] investigated the alkali treatment of banana and coir fibers at 5% concentration to reinforce polylactic acid and unsaturated polyester resin. They found that the alkali treatment significantly affected the elastic modulus but not the tensile strength and elongation.

Banana plants grow widely in tropical areas. The Kepok banana plant (*Musa paradisiaca*) is a germplasm widely distributed in Indonesia. Banana bunches are part of the banana tree, which has quite a lot of fiber. The existence of Kepok bananas in Indonesia is very abundant. Banana bunches in Indonesia are still categorized as organic waste, and to the best of the authors' knowledge, research in using this bunch of fiber for reinforcing polymeric materials is rarely reported. This research aims to determine the effect of alkali (NaOH) concentration in banana bunch fiber on the tensile properties of an unsaturated polyester matrix reinforced with banana bunch fiber. Fourier Transform Infrared (FTIR) and X-ray diffraction tests were carried out on the fibers.

2. Research Methodology

2.1. Materials

The material used was polyester resin type Yukalac 157 BQTN-eX as a matrix with a Methyl Ethyl Ketone Peroxide (MEKPO) catalyst. Kepok banana bunch fiber (Musa paradisiaca L.) was used as reinforcement. The properties of Yukalac 157 BQTN-EX polyester resin can be seen in Table 1.

Table 1. Properties of YUKALAC 157 BQTN-EX polyester resin [8]

Properties	Value
Specific gravity (25°C)	1.10 ± 0.02
Viscosity (Poise, at 25°C)	4.5 – 5.0
Thixotropic index	> 1.5
Gel time (minutes, at 30°C)	20 – 30

Properties	Value
Curing condition	+ MEKPO = 1 part
Storage life at 25°C in the dark (months)	< 6
Flash Point Range, °C	26 – 27

2.2. Fiber Extraction

Banana bunch fiber extraction was done by peeling the skin of the banana bunch and cutting the banana bunch into four parts. The banana bunches were then soaked in water for 3-4 days. Fiber separation was carried out while the banana bunches were wet. Fiber extraction was carried out mechanically by pressing and pulling the fiber. The fiber obtained was then dried before fiber treatment was carried out.

2.3. Fiber Treatment

Fibers were treated with NaOH solutions with 2, 4, 6, and 8% concentrations. Fiber without NaOH treatment was used as a control. The fiber was soaked in NaOH solution for 2 hours and washed several times with water until the fiber was no longer slimy. After that, the fiber was dried in the sun for several days and stored in an airtight plastic bag to avoid absorbing water vapor from the air.

Some fibers were taken to test fiber density (ρ). The fiber was weighed with a 0.01 g digital scale to determine its weight. Then, the fiber was put into a measuring cup with a capacity of 5 ml filled with a certain volume of water (initial volume). The increase in volume, as the difference between the final volume and the initial volume, was the fiber volume, according to formula (1).

$$\rho = \frac{m}{V_1 - V_2} \quad (1)$$

With: m = fiber mass, V_1 = initial water volume, and V_2 = final water volume

2.4. Specimen Making

The manufacturing method used was the press molding method. The fibers were arranged unidirectionally in the fixture and then placed in the mold. The volume fraction of fiber used was 40%. Before molding, the mold was smeared with mirror glaze so that the composite did not stick to the mold and was easy to remove. Polyester resin and hardener were mixed with a weight ratio = 100:1 in a container. Then, the mixture was stirred until homogeneous and vacuumed for ± 3 minutes so the air bubbles disappeared. The resin was then poured into the prepared mold. The mold was then closed and pressed using a press machine. The composite was left for 24 hours until the resin was completely hardened (cured).

2.5. Testing

1) Fourier Transform Infrared (FTIR) spectroscopy

FTIR spectroscopy was performed with a PerkinElmer Spectrum IR Two test equipment in a % transmittance mode of 4000-500 cm^{-1}. The amount of fiber was ± 10 grams from each sample in powder form.

2) X-ray diffraction

X-ray diffraction was carried out with an analytical PAN test equipment type x'Pert3 Powder with Cu, 40 kV, and 30 kV radiation sources. The materials tested were banana bunch fibers without treatment (0% alkali treatment) and those treated with NaOH (2%, 4%, 6% and 8%). The 2theta (2θ) test angle is 5-90°.

3) Tensile testing

Composite testing was carried out using a Tensilon tensile tester with a load cell capacity of 10 kN. The displacement speed was 5 mm/min. An extensometer with a measuring length of 50 mm was used to measure the elongation of the specimen. Tensile test specimens referred to ASTM D3039 (see Fig. 1). Three specimens were tested. After the specimen fractures, the test was stopped, and the tensile forces were recorded in the data logger.

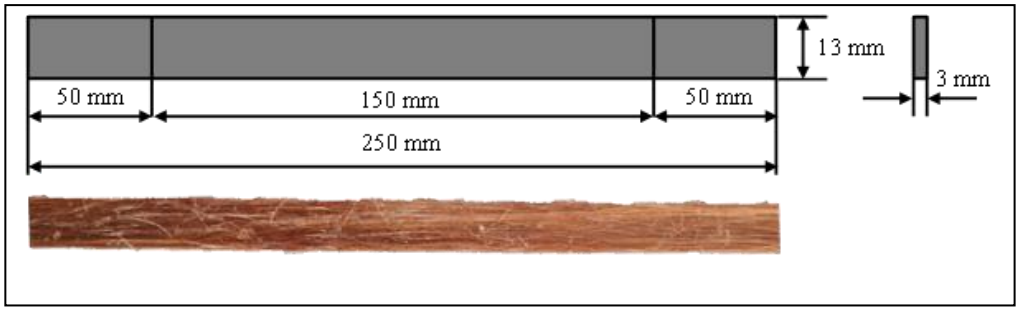

Fig. 1. Tensile test specimens according to ASTM D3039.

4) Scanning electron microscopy of fracture surfaces

Scanning electron microscope testing was carried out using a JCM-7000 NeoScope. The specimens tested were fracture results from the tensile test. Before testing, the specimen was coated with a thin layer of gold (coating) for 4 minutes to increase its conductivity. The operating voltage of the electron microscope was 5.0 kV

3. Results and Discussion

3.1. FTIR Spectroscopy

The FTIR test results are shown in Fig. 2. In the FTIR test, wave numbers were found to be 3300, 1724, 1631, 1365, 1235, and 1029 cm^{-1}. Based on [9,10], the wave number of 3300 cm^{-1} was related to the OH stretching in lignin and the absorbed water. The wave number of 1724 cm^{-1} corresponded to the C=O stretching in hemicellulose. The peak at wave number 1631 cm^{-1} was related to the OH bending of water absorbed in the amorphous cellulose structure and C=C vibrations in the aromatic framework. The peak at wave number 1365 cm^{-1} was related to CH bending in lignin. The peak at wave number 1235 cm^{-1} was related to CO (lignin) stretching. The peak at wave number 1029 cm^{-1} corresponded to the deformation of aromatic C–H planes, guaiacyl, and C–O of primary alcohols in lignin and C–O stretching in cellulose. The peak intensity at almost all wave numbers decreased with increased alkali concentration (NaOH). However, the wave number peaks 1724, 1365, and 1235 cm-1 were the most visible, corresponding to hemicellulose for the first and lignin for the last two. Starting from a 2% NaOH concentration, the peak at wave number 1724 cm^{-1} disappeared, indicating that hemicellulose was almost completely lost, while the peaks at wave numbers 1365 and 1235 cm^{-1}, which were related to lignin only partially disappeared because the peak intensity only decreased but did not disappear completely. This was in line with the statement of Sugiman et al. [11], which stated that when alkali treatment was carried out at high and low temperatures, the decrease in peak intensity at each concentration observed was reduced but did not disappear completely. This identified that hemicellulose was easier to remove with alkali than lignin. Meanwhile, according to previous research [12], using NaOH solution caused the loss of most of the C=O groups in a hemicellulose component, which indicated that hemicellulose was easily lost using alkali treatment compared to lignin.

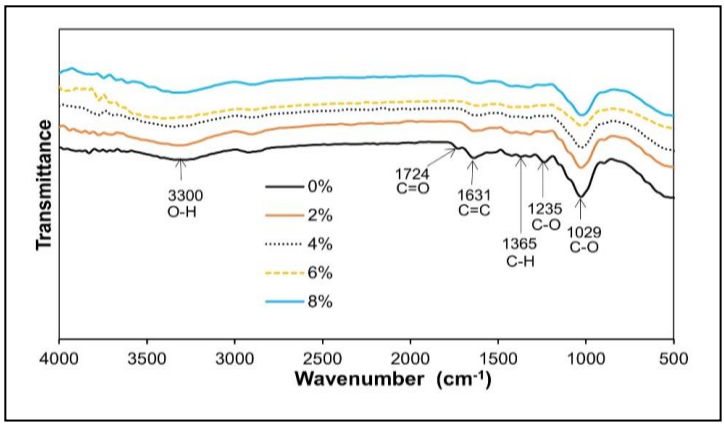

Fig. 2. FTIR of banana bunch fiber.

3.2. X-Ray Diffraction (XRD)

Fig. 3 shows the XRD spectrum of banana bunch fibers that were not alkali-treated (0% alkali treatment) and those that were treated with alkali (2, 4, 6, and 8%). This curve had 2 main intensity peaks, namely at angles 2θ around 15.5º and 22.8º. These peaks are attributed to the 101 and 002 crystallographic planes [13,14]. The peak at 15.5º indicates amorphous hemicellulose, while the peak at 22.8º indicates the presence of crystalline cellulose. The angle 2θ = 22.8º had a sharper peak than the angle 15.5º. This was in line with the statement of Hestiawan et al. [15], which stated that the spectrum of lantung fiber treated with alkali shows a cellulose profile one at an angle of 2θ = 22º and has a sharper peak compared to lantung fiber without alkali treatment.

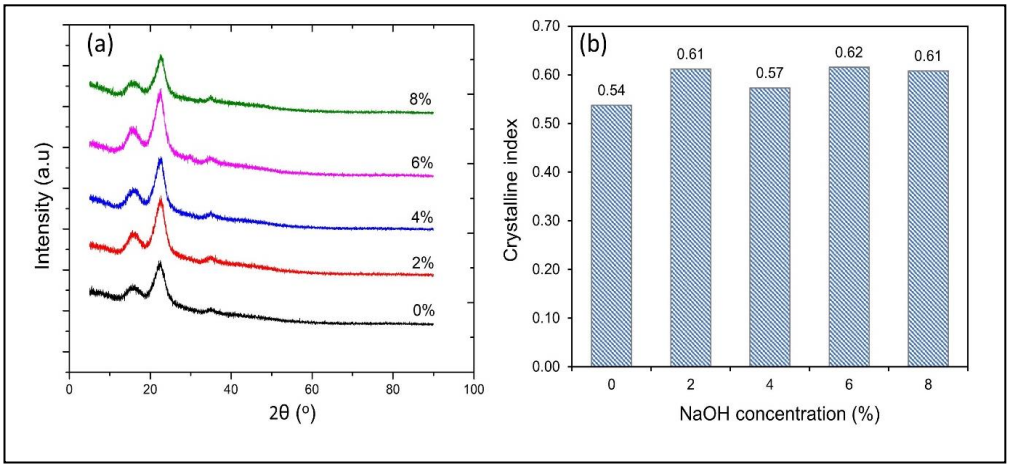

Fig. 3. (a) XRD curves and (b) Crystalline index of banana bunch fiber without treatment and with alkali treatment.

The crystallinity index (CI), as in Fig. 3b, was calculated using the Segal method [16]. This method considered the intensity of the 002-crystal peak (I_{002}) and the amorphous peak (I_{am}) with the formula as in equation 2.

$$CI = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \quad (2)$$

As shown in Fig. 3b, alkali treatment with a NaOH concentration of 2% has a higher value than other fibers, namely 61%, and an increase of around 12.96% compared to fibers without treatment. The increase in crystal index is probably caused by the loss of hemicellulose and lignin so that the cellulose chain packaging becomes tighter and denser [9]. By increasing the NaOH concentration to 8%, CI did not increase significantly, indicating that 2% NaOH treatment effectively removed lignin and hemicellulose from banana bunch fiber.

3.3. Tensile Properties of Composites

Fig. 4 shows the tensile strength vs. NaOH concentration. Based on the graph in Fig. 4, it can be seen that alkali treatment tends to increase the tensile strength of the composite. The highest tensile strength was obtained in banana bunch fibers that underwent alkali treatment at a concentration of 2% NaOH, namely 38.10 Pa, an increase of 92.13% compared to those without alkali treatment. Increasing the NaOH concentration to more than 2% gave a slight decrease compared to the 2% concentration, but based on standard deviation, the tensile strength in the 2-8% treatment did not show a significant difference. The function of NaOH solution is to remove lignin, hemicellulose and other impurities in banana bunch fibers. The level of non-polar lignin was reduced, and the non-polarity of banana bunch fiber also decreased. The reduction in lignin resulted in good adhesion between the matrix interface and the filler in the resulting composite. This aligns with previous research [17] that the NaOH solution could remove hemicellulose, impurities, and lignin.

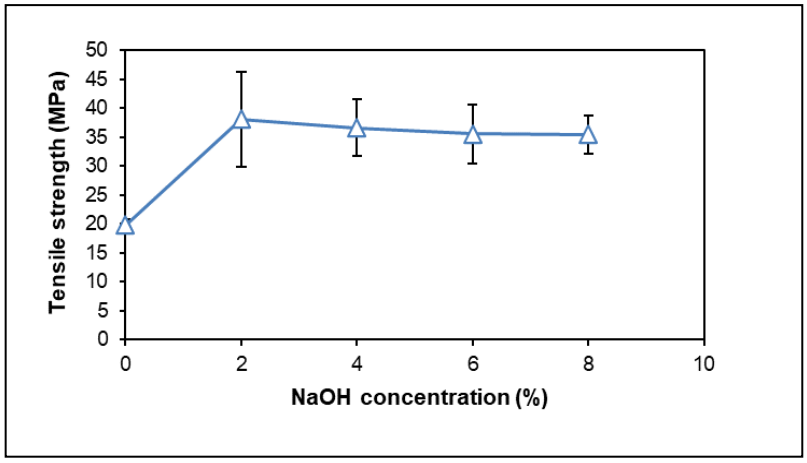

Fig. 4. Tensile strength of banana bunch fiber.

Fig. 5 shows the elastic modulus vs NaOH concentration. Like the tensile strength, the elastic modulus significantly increased after the fiber was treated with 2% alkali solution, increasing by 273% compared to without treatment. After 2% alkali treatment, the elastic modulus tended to decrease, but the elastic modulus was still higher than without alkali treatment. The alkali treatment makes the fiber surface rough and compatible with the matrix and provides good bonding. This causes better load transfer and produces sound reinforcement. However, excessive alkali concentrations tended to damage the fibers and caused a decrease in the mechanical properties of the fibers and composites. According to previous research [18], who researched the effect of alkali treatment on date palm fiber, the optimum modulus was achieved at 5% by weight of NaOH, where the modulus decreased in solutions with higher concentrations. This shows that excessive alkali (NaOH) treatment can cause the fibrils to break in the fiber, reducing the elastic modulus's value. This is also in line with the statement of previous research [19], which stated that excessive alkali administration caused fibril rupture, which reduced the tensile strength of the fiber and its elastic modulus.

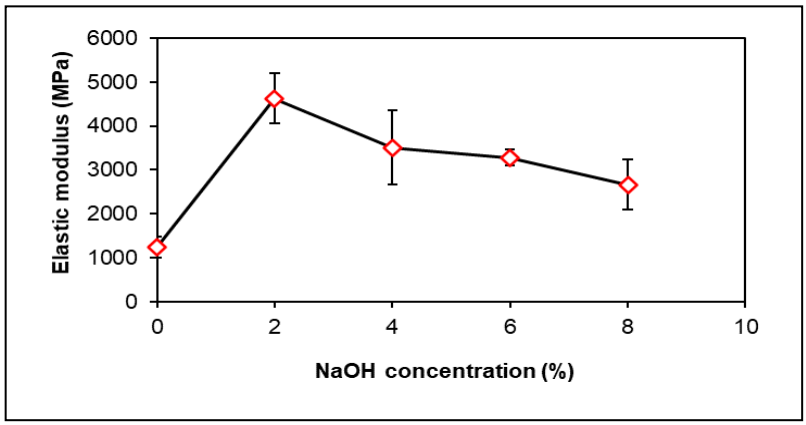

Fig. 5. Modulus of elasticity of banana bunch fibers.

The fracture strain of the banana bunch fiber composite is presented in the graph in Fig. 6. In this graph, the strain at fracture of the banana bunch fiber specimen does not have an obvious trend with the increase of alkali concentration. The highest strain at break was 0.0258, obtained at a concentration of 8%, while the lowest value was obtained at 2%, which was 0.0148. Strain describes the change in the relative length of the tensile test material relative to its initial length. An increase in strain value can reflect the elasticity or ability of a material to stretch before experiencing permanent deformation. Factors such as material properties, temperature, and pressure can influence the value of a strain to change over time in a tensile test. According to Nesimnasi et al. [20], the release of the bond between the fiber and the matrix caused by the tensile stress resulted in the fiber and matrix not withstand a given load. Low stress in a matrix can produce long fiber strains, so the fracture strain of the composites is also high.

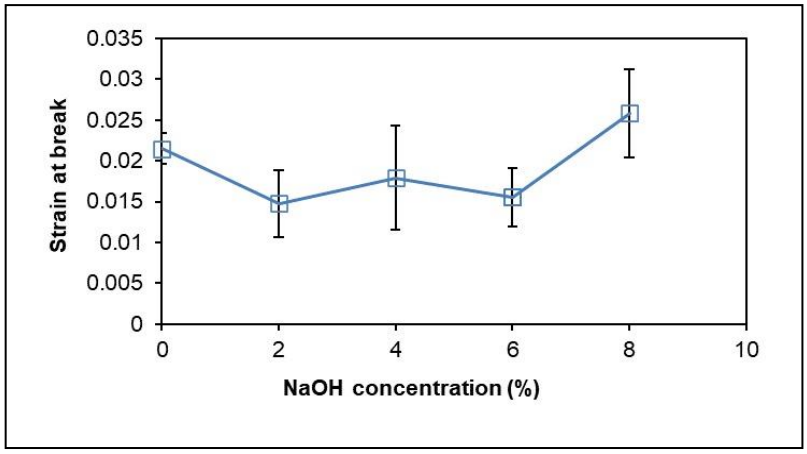

Fig. 6. Strain at fracture of banana bunch fibers.

3.4. Fracture Surface Analysis

Fig. 7 shows the scanning electron microscope images of the composite fracture surface without and with alkali treatment. In this study, 3 SEM images were taken, namely concentrations of 0%, 2%, and 8%, where only the clearest images were taken and showed matrix fractures in the banana bunch fibers.

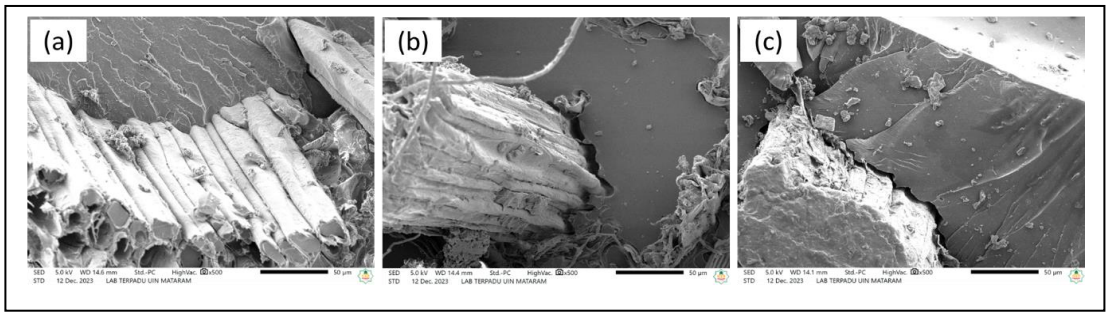

Fig. 7. Fracture morphology of banana bunch fiber composites (a) without treatment and with alkali treatment (b) 2% and (c) 8%, at 500x magnification.

In these observations, it was seen that there were differences between the morphology of banana bunch fibers before and after undergoing alkali (NaOH) treatment. The morphology of banana bunch fibers treated with 2% and 8% NaOH concentrations looked rougher than without treatment. Untreated fibers show a smooth morphology, indicating the presence of lignin and hemicellulose. As seen in Fig. 7a, the untreated fiber appeared covered by lignin and hemicellulose, so it did not provide a good bond with the matrix. After treatment (see Fig. 7b and 7c), the fibers appeared coarser and bonded well with the matrix. In the 2% alkali treatment, the banana bunch fiber was coarser and showed matrix strengthening by the fiber. Matrix fractures were smoother than without treatment, which showed that the fibers bore the load on the composite. The voids between the fiber and the matrix showed that the fracture process was not easy, starting from the fiber's strengthening process, the matrix cracking, the fiber breaking and debonding, and the voids forming. This was also experienced by composites with 8% treated fibers. However, because the reinforcement was lower than the reinforcement made with 2% treated fibers, the voids were narrower because the fibers were more easily pulled out after the matrix breaks. This is in line with the statement of Dewri and Gnanamoorthy [21], which stated that the morphology of a fiber that has undergone alkali treatment (NaOH) was rougher than fiber without alkali treatment, and the higher the NaOH content, the rougher the fiber.

4. Conclusion

Research on the alkali treatment effects of banana bunch fiber on the tensile properties of banana bunch fiber composites with an unsaturated polyester matrix has been carried out. It was found that the alkaline treatment of banana bunch fiber significantly increased the tensile strength and elastic

modulus of banana bunch fiber composite. Compared to the untreated fiber, the greatest increase in tensile strength and elastic modulus was obtained at an alkali concentration of 2%, approximately 92.13% and 273%, respectively. Increasing the alkali concentration to more than 2% decreased the tensile strength and elastic modulus relative to the 2% alkali treatment. The strain at break tended to be lower for higher-strength composites, indicating a good fiber-matrix bonding. Alkaline treatment of banana bunch fiber removed lignin and hemicellulose, thereby increasing the fiber crystalline index, as indicated by FTIR and XRD. Alkaline treatment also made the fibers rougher, thereby increasing the bond between the fibers and the matrix, as shown by scanning electron microscope results of the fracture surface of the specimen

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