THE EFFECT OF ALKALI AND FUMIGATION TREATMENTS ON KING PINEAPPLE FIBER PROPERTIES AND INTERFACIAL BONDING OF KING PINEAPPLE FIBER/UNSATURATED POLYESTER ON MICROCRYSTALLINE CELLULOSE REINFORCED COMPOSI

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THE EFFECT OF ALKALI AND FUMIGATION TREATMENTS ON KING PINEAPPLE FIBER PROPERTIES AND INTERFACIAL BONDING OF KING PINEAPPLE FIBER/UNSATURATED POLYESTER ON MICROCRYSTALLINE CELLULOSE REINFORCED COMPOSITE

碱和熏蒸处理对菠萝王纤维性能的影响以及菠萝王纤维/不饱和聚酯对微晶纤维素增强复合材料的界面结合的影响

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Abstract

This study aims to determine and compare the effect of alkaline and fumigation treatment on the properties of King pineapple fibers (KPF) and the interfacial bond between King pineapple fibers/ unsaturated polyester and microcrystalline cellulose. Chemically, King pineapple fiber was extracted and dried at room temperature. The fiber was made using the alkaline method by soaking in 5% NaOH for 4 hours, 6 hours, 8 hours, and 10 hours. The fumigation treatment method was carried out for 0 hours, 4 hours, 8 hours, and 12 hours. The single fiber tensile strength test results showed an increase of 51.99% after the fumigation treatment and 59.62 % after the alkaline treatment. The increase in interface shear strength (IFSS) was 32.12% after fumigation treatment and 47.38% after alkaline treatment. The increase in fiber tensile strength and interfacial shear strength (IFSS) is caused by the loss of hemicellulose, pectin, lignin, and other impurities. These results indicate that the alkaline treatment is still better than the fumigation treatment.

Keywords: King pineapple fiber, alkali, fumigation, microcrystalline cellulose

摘要 本研究旨在确定和比较碱处理和熏蒸处理对菠萝王纤维（KPF）性能以及菠萝王纤维/不饱和

聚酯与微晶纤维素之间的界面结合的影响。化学上，王菠萝纤维在室温下被提取和干燥。使用碱性方法通过在 5%氢氧化钠中浸泡 4 小时、6 小时、8 小时和 10 小时来制造纤维。熏蒸处理方法为 0 小时、4 小时、8 小时、12 小时。单纤维拉伸强度试验结果表明熏蒸处理后增加 51.99%，碱处理后增加 59.62%。熏蒸处理后界面剪切强度（国际食品安全局）增加 32.12%，碱处理后增加 47.38%。纤维拉伸强度和界面剪切强度（国际食品安全局）的增加是由半纤维素、果胶、木质素和其他杂质的损失引起的。这些结果表明碱处理仍优于熏蒸处理。

关键词: 國王菠蘿纖維，鹼性，熏蒸，微晶纖維素

I. INTRODUCTION

Increased world population growth resulted in reduced world oil reserves and increased material demand. The use of synthetic materials as reinforcement in composites was a common thing. The process and production in the manufacture of synthetic materials continue to result in waste, resulting in environmental pollution [1]. It takes serious efforts to replace synthetic materials with materials that can be degraded by nature. Using natural fibers as a substitute for synthetic materials in making composites as an alternative is a brilliant idea that must be developed [2].

Those composite materials made from natural fibers can still be recycled even though they have the best properties [3]. The use of natural fibers as reinforcement in composites was used to reduce costs, material weight, increase biodegradability, increase durability and mechanical strength [4]. Other advantages of natural fibers are abundance, biodegradability, and high toughness [5]. In addition, natural fibers provide adverse consequences such as poor adhesion properties of polymers [6]. The hydroxyl (-OH) and polar groups in the fiber will inhibit the binding of hydrophobic polymers and reduce the effectiveness of the loads transferred between the texture and the matrix [7]

The interface characterization and wettability of natural fibers affect the constituent components of the fiber, namely chemical and physical properties [8] and the low adhesion properties of polymers [9]. Modifications of natural fiber treatments are often carried out, such as silane, alkaline, permanganate treatments [10]. Alkali treatment is one of the cheapest and most environmentally friendly ways to improve mechanical properties and interface bonding of natural fibers as it does not require toxic organic chemicals [11]. Fumigation treatment has improved the interfacial bond between the fiber and matrix [12, 13]. Alkali and fumigation treatments are used to reduce wax coating on fibers such as hemicellulose, pectin, lignin, and impurities. Treatment modifications were also carried out to improve the surface fiber, increase fiber strength, and improve the interface bond between the fiber and matrix for better composite mechanical properties [14, 15].

The King pineapple (Agave Cantula Roxb) is a tropical plant well-known in Indonesia's Java and South Sulawesi regions as a tropical plant taken regularly through the leaves. The fiber of the King pineapple is planted regularly by farmers and harvested after it reaches nine months with a height of 90 cm. The fibers are harvested by cutting the base of the leaves, and this is done mechanically. King pineapple fiber has 64.23% cellulose, 29.87% hemicellulose, and 6.8% lignin, which is useful for reinforcing composites [12]. But there has not been much research on the use of King pineapple fiber as reinforcement in composites. This study will compare the ability of alkaline treatment and fumigation to morphology, density, surface energy, crystallinity index, tensile strength of single fibers, and strength of sharing interface (IFSS) by adding microcrystalline cellulose. The alkaline treatment and fumigation of the King pineapple fibers can significantly increase the tensile strength of the single fiber and the interface shear strength between the fiber and the matrix.

II. METHODS/MATERIALS

A. Materials

King pineapple fiber was obtained from CV "Kencana Jaya" Magelang (Central Java, Indonesia), fiber was extracted from the leaves of the King pineapple tree through a mechanical retting system, and the fiber was dried under room temperature. Then, sodium hydroxide (NaOH) with a purity of 98 % and Aquades were collected from Merck. Inc (Jakarta, Indonesia)

for the treatment of fiber alkalis. Unsaturated polyester (UPRs) of Yucalac BTQN 157 and methyl ethyl ketone peroxide were obtained from Justus Kimia Raya.Inc (Semarang, Indonesia). Microcrystalline cellulose (MCC) was gathered from the sigma Aldric agent (Jakarta, Indonesia), and coconut skin was obtained from the local market in Purwokerto, Indonesia.

The King pineapple (KP) Plant and King pineapple fiber (KPF) resulted from manual retting treatment were shown in Figures 1a and 1b.

Figure 1a. KP plant

Figure 1b. KP fiber

B. Methods

1) Alkali Treatment

The fiber was soaked with sodium hydroxide (NaOH) of 5 % wt concentration for 0 h, 4 h, 6h, 8 h and 10 h at room temperature. The fiber was rinsed with tap water to clean the fiber from the alkaline solution until pH ∽7 was reached. The fibers were dried at room temperature for 24 h and heated in an oven at 60°C for 10 hours.

2) Fumigation Treatment

The fiber was extracted from King pineapple leaves by mechanical retting and dried at room temperature for five days. The fiber was fumigated through a chimney of 800 mm x 800 mm x 1500 mm. Fumigation was done by burning coconut skins with the temperature controlled between 60°C and continuously for 4 h, 8 h, and 12 h. The King pineapple fiber from fumigation was cooled with room air for 24 hours before proceeding to a sample. The process of fumigation and burned coconut skin was shown in Figures 2.a and 2.b.

Figure 2a. Coconut skin burning model

Figure 2b. Coconut skin

The research treatment table was given in nomenclature, as shown in Table 1.

Table 1.
Nomenclature fumigation and alkali treated fibers

No.	Treatment type	Description	Code
1	Untreated	Fiber without treatment	UF
2	Fumigation	Fiber fumigation 4 h	FU4
3		Fiber fumigation 8 h	FU8
4		Fiber fumigation 12 h	FU12
5	Alkali	Fiber alkali 4 h	KL4
6		Fiber alkali 6 h	KL6
7		Fiber alkali 8 h	KL8
8		Fiber alkali 10 h	KL10

3) Density Testing

Fiber density testing uses ASTM 792-13 the year 2013. The method used with the Precisa XT220 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 specific gravity (g/cm^3) in the air and b is the specific gravity (g/cm^3) in fluids. Tests were performed at room temperature using biodiesel with a density of 0.867 g/cm^3.

4) Fourier Transform Infrared Spectroscopy (FTIR)

FTIR was used to determine the functional group of fibers that have been treated and fibers that were not treated. Fiber is mashed and mixed with (KBr) in a ratio of 1: 20 with transmission spectra recorded at 400 cm^{-1} to 500 cm^{-1} wavelengths. There were about 24 scanners with

2 cm^{-1} resolution using Shimadzu IR Prestige 21 (MIPA Laboratory-Universitas Sebelas Maret, Indonesia).

5) X-Ray Diffraction

The structure of cellulose from treated and untreated King pineapple fiber was analyzed by X-rays at room temperature (Integrated Laboratory of Diponegoro University, Indonesia). Using CuKα radiation (λ = 1.54 A), the intensity of CuKα radiation recorded from 2θ = 100° to 900° had 20 steps and a voltage of 30 kV current of 30 mA. The crystallinity index (Cr.I) and degree of crystallinity (% C) were calculated by the Segal method, according to Equation 2 [16].

$$Cr.I. = \frac{I_{(002)} - I_{am}}{I_{(002)}} x100\% \quad (2)$$

where $I_{(002)}$ is the intensity of the sample peak based on the Miller Index (002) at an angle of 2θ ranging from 22° to 23°, and I_{am} *is* the minimum intensity of the non-crystal content, which showed the peak at ($2\theta = 18°$).

6) Scanning Electron Microscopy (SEM) Analysis

Scanning electron microscopy testing using JSM-610 PLUS/LV model instrument from JEOL to capture two-dimensional images of King pineapple fiber surface with treatment and without treatment. (Integrated Laboratory of Diponegoro University, Semarang Indonesia). King pineapple fiber was mounted on a piece of aluminum coated platinum and observed for 1 minute at a pressure of 2 bar.

7) Thermogravimetry Analysis

TGA testing used Perkin Elmer Pyris Diamont TGA 6 Analyzer (MIPA Laboratory Sebelas Maret University Surakarta Indonesia) to test the stability of King pineapple fiber (KPF) samples with and without treatment. All samples were scanned at elevated room temperatures from 30°C to 600°C at a speed of 100°C/min. The TGA test was carried out in a nitrogen environment.

8) Contact Angle and Surface Energy

The contact angle measurement starts with stretching the fiber and dropping the liquid on the fiber surface, and an image was taken and measured with a microscope. The liquid was controlled 0.5 ml with a dropper. Ethylene glycol and distilled water were used to investigate the surface characteristics of King pineapple fiber. Surface energy was measured by underlying the contact angle using the Owens and Wendt method according to equation 3 [17].

$$\gamma_L(1+\cos t\theta) = 2\sqrt{\gamma_S^d}\sqrt{\gamma_L^d} + 2\sqrt{\gamma_S^p}\sqrt{\gamma_L^p} \quad (3)$$

where d is the energy-dispersive subscribe (mNm $^{-1}$), p is the polar surface energy (mNm $^{-1}$), s is the solid state, and L is the liquid state. King pineapple fiber image was taken using DF Plano microscope 1X-4 macro microscope (Mechanical Engineering Laboratory of Sebelas Maret University Surakarta, Indonesia).

9) Single Fiber Testing

King pineapple fiber was attached to paper with a 10 mm x 60 mm size and was given a hole in the middle of the paper. The fiber was clamped at both ends. Before testing the fiber, its diameter was measured at all three ends with a microscope. Testing was done by pinning both ends of the testing machine and cut paper on both sides. Testing using ASTM C155-03-2003 was carried out at the Textile Laboratory of the Islamic University of Indonesia, Yogyakarta, Indonesia. The distance between the two clamps was 50 mm with a speed of 250 mm/minute for 30 repetitions.

10) Interfacial Share Strength (IFSS) Testing

Interfacial shear strength testing (IFSS) was done by attaching 60 mm long cantula fiber to a mixture of unsaturated polyester, microcrystalline cellulose, and methyl ethyl ketone peroxide (Mekpo) catalyst. King pineapple fibers were glued to cardboard with a hole in the middle and dried for 120 minutes. The clamping distance is 50 mm with a 250 mm/minute pull speed, and 30 repetitions are performed.

Paper from both sides was cut to get a maximum pull. The fiber was pulled until the bond between UPRs + MCC and fiber regardless. The calculation of the test was based on the release of fibers from the matrix. Fiber diameter is measured on the top, middle, and bottom sides. The Tenso model 300 type 168 E newton unit textile pulling machine was used for tests at the textile laboratory of the Islamic University of Indonesia, Yogyakarta, Indonesia.

III. Results and Discussion

A. Density

Density test results showed that the fumigated fiber has increased in density proportional to the fumigation time. Density increased due to loss of impurities in the fiber and reduced moisture content in the fiber. The increase in temperature due to fumigation results in degradation of the amorphous structure of the fibers and rearranging the fiber structure. The color of the fibers getting black is proportional to the duration of fumigation treatment. Alkaline test results on the fiber showed an increase in density in the fiber. This increase in density is proportional to the

length of time of fiber soaking and alkali concentration [18].

The loss of amorphous material and the formation of a denser structure can decrease fiber volume and increase fiber weight. The increase in density was due to the removal of the low-solid non-cellulose material. The alkaline treatment has formed a new structure for the cellulose II component, which is more stable and compact when compared to cellulose I. But the length of immersion time and too much alkaline concentration can result in decreased density; fiber was seen to be damaged because it will break the hydroxyl bonds of hemicellulose, defibrillation, and fiber will break. [19]. The results of the fiber density test are shown in Table 2.

Table 2.
Test results of King pineapple fiber density

No	Code	Treatment	Density (g/cm^2)
1	UF	Untreated	1.087 ± 0.03
2	FU4	Fumigation 4 h	1.143 ± 0.02
3	FU8	Fumigation 8 h	1.146 ± 0.03
4	FU12	Fumigation 12 h	1.148 ± 0.04
5	KL4	Alkali 4 h	1.228 ± 0.03
6	KL6	Alkali 6 h	1.270 ± 0.04
7	KL8	Alkali 8 h	1.310 ± 0.05
8	KL10	Alkali 10 h	1.320 ± 0.03

B. X-Ray Diffraction

X-ray diffraction test of King pineapple fiber with treated and without treated was based on a crystal lattice method with a gap when diffraction occurs. The incident ray and diffracted beam will have the same wavelength as the X-ray wavelength. The α fiber structures were observed and analyzed in these cellulose samples. The X-ray diffraction curve determined the crystallinity of α cellulose crystals with the Segal method, which we used with the Original Pro 2020b and obtained the following graph:

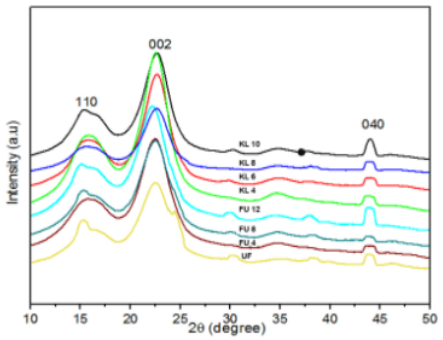

Figure 3. X-ray diffraction test results

Figure 3 shows x-ray diffraction results from King pineapple fiber structure without treatment, with fumigation, and alkaline treatment. King pineapple fibers form 3 large peaks with peaks between 15°, 22, 12°, and 44°. The two initial peaks show areas related to the crystal fields 110 and 002 [20]. At the same time, the final diffraction peaks of King pineapple fibers showed 040 crystal fields [21]. The amorphous portion of the King pineapple fiber structure is shown by the valley diffractogram between two peaks with an area of 2θ around 180. The cellulose structure is shown by the diffraction peaks in the range between 22° – 23°, which is the character of the original cellulose [22].

X-ray diffraction test results with the peak and diffraction angle of the King pineapple fiber, with the highest intensity and the lowest intensity peak, are given in Table 3 below.

Table 3.
Results of X-ray diffraction testing

Material Code	I (oo2)		I (am)		Index crystallinity (Cr.I) (%)
	Peak 2θ (o)	Intensity (Cps)	Peak 2θ (o)	Intensity (Cps)	
UF	22.31	1162	18.88	470	59.55%
FU4	22.46	1734	18.40	648	62.62%
FU8	22.36	1562	18.80	572	63.43%
FU12	22.22	1748	18.60	668	61.78%
KL4	22.58	2238	18.80	638	71.49%
KL6	22.67	1726	18.94	440	72.65%
KL8	22.72	940	18.36	274	70.85%
KL10	22.58	1480	18.44	434	70.67%

The results showed that fumigation treatment could increase the crystallinity index in King pineapple fiber. The fiber crystallinity index without treatment was 59.55% after the fumigation treatment increased to 63.53% for the fumigation treatment for eight hours. Heat treatment of fibers contributes to the increase in the crystallinity index; this was due to the degradation of the amorphous structure of the fibers and the rearrangement of the crystal structure [23]. The degradation process also makes the cross-sectional area smaller. The

treatment of liquid fumigation on sago fibers showed an increase in crystallinity after 4 hours of treatment [24].

The increase in the crystallinity index on alkali treatment was higher to 72.65% for six hours' soaking. The increase in crystallinity in the alkaline treatment shows a decrease in the composition of the amorphous fibers due to the alkaline treatment [25]. Partial removal of cementing materials such as lignin will lead to better packaging of the transformed cellulose chain from the form I to form II [26]. Increased crystallinity index on King pineapple fiber due to fumigation and alkali treatment shows that hemicellulose, lignin, and other dirty elements have been lost or reduced in the fiber. King pineapple fiber will be cleaner with alkali and fumigation treatment, which can be seen from the results of SEM morphology.

C. Scanning Electron Microscope

SEM testing used the Jeol JEC 3000 FC type with a 20 kV, a magnification of 150 times, a capture time of 50 seconds; the results are shown in Figure 4 below.

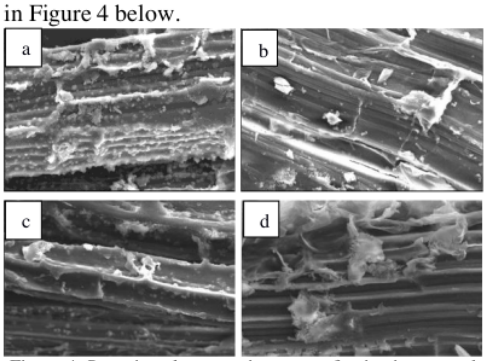

Figure 4. Scanning electron microscope, fumigation treated samples: a) Untreated; b) Fumigation treated 4 h; c) Fumigation treated 8 h; d) Fumigation treated 12 h

The test results showed that untreated fibers were still in the tissue structure, fibrils were still bound, and were wrapped by other substances such as pectin, lignin, hemicellulose, and other waste. The fiber was clean in the four- and eight-hour treatment fumigation because the bonding (cementing) began to disappear. The removal of pectin, wax, oil, and other extractive substances from the fiber resulted in smooth surface topography and better surface adhesion between the fiber and the matrix to improve mechanical properties and interfacial bonding. Heat treatment of kenaf fibers makes the surface of the fiber cleaner due to the loss of impurities such as wax on the fibers [27]. The heating treatment of the fibers makes the fibers cleaner due to the loss of hemicellulose and lignin in the fibers [28]. The longer the fumigation, the more the fume will stick to the fiber surface, which results in the fiber getting dirty, as shown in Fig. 5.d. SEM test results of the King pineapple fibers with alkali treatment as show in Figure 5.

Untreated fibers indicated that fibers are still in the tissue structure, and fibrils were still bound and wrapped by other substances such as pectin, lignin, hemicellulose, and other waste. The four-hour alkali treatment showed the fiber was clean enough because the bonding had begun to disappear. In the six-hour alkali treatment, the fibers appear cleaner with the removal of hemicellulose, pectin, wax, oil, and other extractive substances from the fiber, able to produce a smooth surface topography and offer better mechanical adhesion between surfaces of the fiber and the matrix so that mechanical properties can be improved. The alkaline treatment of banana fibers had significantly made the surface smoother because it has lost dirt, fat deposits, and wax [29].

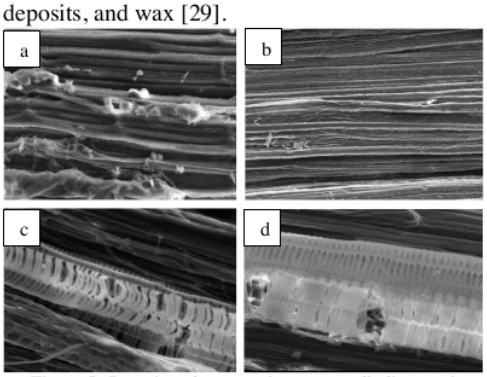

Figure 5. Scanning electron microscope, alkali treated samples: a) Alkali treated 4 h; b) Alkali treated 6 h; c) Alkali treated 8 h; d) Alkali treated 10 h

The removal of surface dirt on the fibers results in the surface of the fibers becoming rougher and beneficial for adhesion between the matrix and fibers provide facilities for mechanical interlocking and bonding reactions [8]. The removal of lignin, wax, and grease produced a rough surface topography offering better inter-surface mechanical adhesion properties between fibers and matrices to improve mechanical properties. Alkaline treatment with immersion for 8 and 10 hours SEM results showed that the fiber was damaged in the hemicellulose bonds, and the damage was getting worse after 10 hours of treatment. The alkaline treatment with the concentration of immersion time and excess NaOH resulted in the fiber surface being depleted of non-cellulose/hemicellulose and lignin materials. [15]. The immersion time and the excess alkaline

concentration result in more severe damage to the surface due to the corrosive effect of the alkalis, as shown in Figures 5c and 5d.

D. Fourier Infrared Transmittance Testing

FTIR was used to determine the functional groups of fibers that are treated and not treated. The fiber was crushed like powder by finely ground will be mixed with potassium bromide (KBr) in a ratio of 1:20, and the spectral transmission will be recorded with wavelengths between 400-500 cm^{-1} with scanning 24 times and a resolution of 2 cm^{-1} using FTIR type Shimadzu IR prestige 21. FTIR graph as seen below:

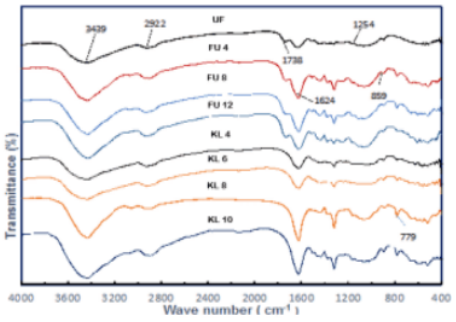

Figure 6. Fiber test results with FTIR

The sample spectra without treatment (UF) and fumigation treatment showed a shift in peak intensity from 3443 cm^{-1} to 3432 cm^{-1} for FU4, 3429 cm^{-1} for FU8, and FU12. While the alkali treatment shifted from 3443 cm^{-1} to 3432 cm^{-1}, this shows that some free -OH groups contribute to chemical reactions CO_2. [30]. Then the band 2922 cm^{-1} was a characteristic stretching vibration (celling vibration) of cellulose/hemicellulose. Table 4 shows the peak taking of each functional group based on several studies and FTIR testing guidelines.

Table 4.
FTIR testing guidelines

Wave	Functional Groups	Ref.
3443	OH stretching vibration from cellulose	[31]
2925	CH Symmetrical stretching (waxes and oils)	[20]
1731	> C = O stretching of a carboxyl acid or ester	[20, 31]
1320	Si-O- cellulose	[32]
1158	C-O stretching of acetyl (lignin)	[33]
1060	Si-O-Si stretching	[32]
780	Si-OH (Silanol Group	[33]

This band shifted after the fumigation treatment with twin peaks became 2934 cm^{-1} and 2901 cm^{-1} for FU4 and 2932 cm^{-1} for FU8, while FU12 became 2930 cm^{-1}. In the KL4 treatment, the band moves to 2924 cm^{-1} and decreases to 2900 cm^{-1} for KL10. This indicates the existence of vibration stretching -OH and between groups of intra-molecular hydrogen bonds.

The peak at 1738 cm^{-1} for cantula fiber was the aromatic skeletal vibrations and carbonyl groups where lignin and hemicellulose may still be present [20]. This peak shifts after fumigation to 1735 cm^{-1} for FU4, 1722 cm^{-1} for FU8, and 1731 cm^{-1} 1 for FU12. In 1623 cm^{-1} is C = C aromatic aromatic fiber without treatment and shifts to 1625 cm^{-1} for FU4, 1622 cm^{-1} for FU8 and 1621 cm^{-1} for FU12. As for the alkali band treatment, there was not much change. Before the fiber was treated, the peak Si-O cellulose was at 1320 cm^{-1} and was not changed much after treatment. The peak of 1161 cm^{-1} is C-O stretching acetyl in the untreated fiber, and it becomes 1158 cm^{-1}, which tends to disappear at its peak increasingly. The peak 1060 cm^{-1} was Si-O-Si stretching at the peak before treatment and shifted to 1058 cm^{-1} after FU8 and FU12 [34]. After six-hour alkali treatment, the peak shifted to 1060 cm^{-1}, and after 10 hours, the peak shifted to 1058 cm^{-1}.

E. Thermogravimetric Analysis Results

Thermogravimetric analysis (TGA) curves and TGA derivative analysis without treated and by treated were showed in Figure 7. Untreated fibers (UF) appear to lose weight between 5.7% at temperatures below 100°C. This loss was related to the evaporation of water on the surface of the fiber. As for the fumigation and alkali treatment, the weight loss was smaller; this shows that the King pineapple fiber with the treatment made the fiber more hydrophilic. The temperature decomposition of UF was obtained at 210°C with a weight reduction of 5.9% (Figure 7).

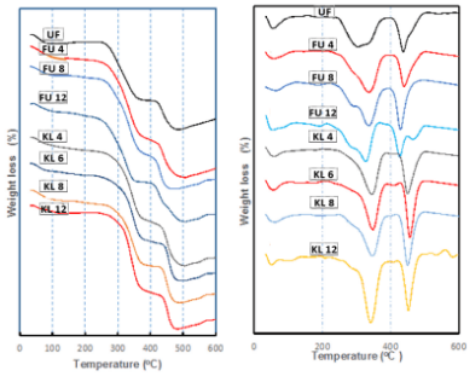

Figure 7. TGA and TGA derivative test results

After fumigation and alkali treatment, the initial temperature decomposition shifts higher at

± 250°C. This increase was caused by removing an amorphous substance with a value more sensitive to heat than the crystal element [35]. The fumigation showed the fume layer of the fumigation system covered the fiber section, especially in FU8 and FU12. The untreated fiber (UF) at a temperature of 300°C experiences a 45 % weight loss. After fumigation treatment, the peak shifted to 338°C for FU4 and 340°C for FU12. The peak temperature of fumigation fiber cellulose decomposition shifted to a higher temperature position; this can be attributed to the residual layer of smoke on the surface and increased heat resistance and fiber decomposition. As for the alkali treatment, the peak weight loss occurs at 348°C for KL4 and 352 for KL8. This was closely related to the removal of most amorphous material. A similar study was observed by [36] on sisal fibers. The second part of the temperature of 300°C to 400°C was related to cellulose decomposition, at temperatures between 400°C to 600°C associated with combustion and the degradation of the material in the previous stage related to the decomposition of lignin [31].

The TGA derivative curve shows that the decrease in the maximum weight of UF fibers at a temperature of 438°C loses weight by 25%, for fumigation treatment, this does not make a lot of shifts of 445°C loses weight of 28% at FU4, whereas for the alkali treatment it showed a temperature shift up to 453°C for KL6. This finding showed that alkali and fumigation treatment resulted in significant thermal stability. Alkali and fumigation treatment made coating were able to prevent structural damage and increase the thermal stability of fiber.

F. Contact Angle and Surface Energy

The contact angle and surface energy tests were carried out with droplets on fibers stretched using partial drops of water and ethylene glycol.

The recapitulation results of contact angle and surface energy testing can be seen in Table 5. below.

Table 5.
Results of contact angle and surface energy

Treatment	Contact angle Water (deg)	Contact angle Ethylene Glycol (deg)	Polarity mN/m	Dispersion mN/m	Surface energy mN/m
UF	72.78	71.36	63.24	1.04	64.28
FU4	64.45	61.44	72.28	0.56	72.84
FU8	63.83	60.68	73.56	0.65	74.21
FU12	64.67	62.09	73.21	0.76	73.97
KL4	69.89	68.63	68.34	1.2	69.55
KL6	65.51	64.54	74.54	1.62	76.16
KL8	68.37	67.784	72.83	1.75	74.58
KL10	67.76	66.41	71.96	1.25	73.21

Evaluations of King pineapple fiber wettability, polar energy, dispersion energy, and surface energy are discussed. Total surface energy is the amount of polar energy and energy dispersion.

Table 5 above shows that the King pineapple fiber without treatment (UF) has a greater contact angle when compared to the fiber contact angle after treatment. So that the total surface energy of the fiber before fumigation treatment was lower than after fumigation, both using equates and ethylene glycol. The increase in total surface energy indicates a change in fumigation time. These results indicate that the fumigation time affects the reduction of acid and base increase in the fiber and plays an important role in intensifying the wetting of King pineapple fibers. Surface roughness improved wetting properties and can determine liquid dispersion on the surface. [37].

The highest surface energy is obtained by treatment for six hours of alkali treatment and the lowest treatment by three hours. Whereas alkali treatment can improve on surface energy when compared to before treatment. The results of alkali treatment with 5% NaOH comply with the test carried out on kenaf fiber for 5 hours soaking [18]. These results indicate that the fiber was hydrophobic with a low level of polarity, which results in the fiber having low wettability. The low level of wetting of the fiber was caused by the amount of wax and dirt that was still often attached to the fiber.

G. Single Fiber Tensile Strength Test Results

King pineapple fiber diameter was measured three times using a microscope. Single fiber tensile strength test results were analyzed with Minitab, using the Weibull distribution with

Anderson Darling value of 0.33; these results are shown in Figure 8.

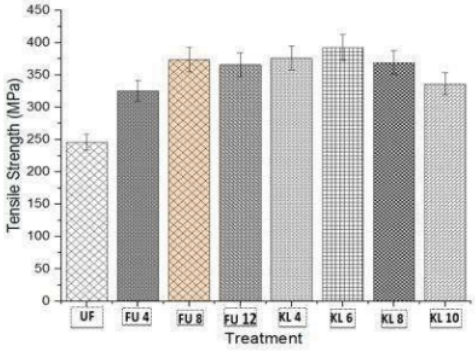

Figure 8. Single tensile fiber test results

The test results showed an increase in the tensile strength of the fibers due to fumigation treatment. The increase in fiber tensile strength after fumigation for 8 hours was 51.99% from the previous treatment (UF) of 245.87 MPa. Fumigation treatment for 12 hours decreased tensile strength. The increase in the tensile strength of the fibers is caused by the loss of hemicellulose, pectin, lignin, and other impurities.

The loss of hemicellulose and impurities in the fiber increased the crystallinity index and the tensile strength of the cantula fiber. The increase in tensile strength of the single fiber on fumigation was due to a decrease in hemicellulose and lignin levels in the fiber [13]. This increase was associated with the element carbonyl (carbon monoxide) in the fiber of the King pineapple due to the fumigation treatment [12].

Treatment of liquid fumigation on sago fiber for 1 hour showed an increase in tensile strength of 38.92% compared to before treatment [24]. A decrease in fiber tensile strength on FU12 treatment can be caused by the remaining stuck fume seen in SEM results. The alkali treatment showed that KL6 had the highest tensile strength of fibers, up 59.6% of the untreated fibers. This increase caused by alkali treatment can increase fiber crystallinity due to the removal of hemicellulose and other impurities due to the alkali process. The highest tensile strength for KL6 is under research [38] alkali treatment for sisalana fibers. Sansevieria fiber with a concentration of 5% NaOH can increase the tensile strength from 48.05 MPa to 71.60 MPa [39]. Hemp fiber treated with 5% NaOH increased tensile strength by 19% and modulus of elasticity by 68%. [40]. The alkaline treatment can clean the surface of the amorphous fibers, thereby increasing the tensile strength and bonding strength between the fibers and the matrix [41]. The alkaline treatment at KPF resulted in a single fiber tensile strength which was 7.61% higher than the fumigation treatment

H. Interfacial Shear Strength (IFSS) Test Results

The interfacial shear strength test (IFSS) was based on the release of fibers from the bonding, measured the average diameter, and was calculated based on the shear stress value. Removing non-cellulosic material in the fiber expands the surface and increases the interfacial bond between the matrix and the fiber [8]. The Interfacial shear strength for fiber untreated (UF) makes 2.49 MPa. The four-hour diving fumigation treatment increased the IFSS to 3.04 MPa, whereas the FU8 treatment showed interfacial shear strength between the matrix and fiber increased to 3.29 MPa. The increase in shear forces after the fumigation treatment shows a reduction in lignin and hemicellulose so that the fiber surface becomes rough [12]. The increase in IFSS shows that the interface adhesion between fibers and matrices was caused by loss of amorphous fibers, reduced fiber diameters, and cross-sectional area the larger the fiber surface; therefore, the fiber and matrix compatibility increases.

The FU12 treatment shows the strength of the interface bonding has decreased because the remaining smoke increasingly wrapped the fiber, so the interfacial bonding between the fiber and the matrix was reduced. The highest increased for fumigation treatment was 36.14% for FU8. The shear strength of the interface between the KPF/ unsaturated polyester and microcrystalline cellulose as a function of the treatment is shown in Figure 9.

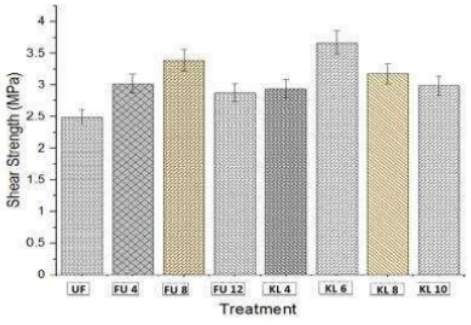

Figure 9. Interface shear strength test results

The IFSS test results showed an increase in adhesion between the fiber and the matrix in all alkaline treatments. The increase in interface shear strength is due to a decrease in surface

roughness and cleaner fiber. IFSS increased in fibers by alkali treatment from UF of 2.49 MPa to 2.94 MPa for Al 3 treatment and in Al 6 by 3.67 MPa. Increased shear forces in alkaline treatment of typha fibers with a concentration of 5% NaOH from 1.48 MPa to 3.05 MPa [42]. Coconut fibers soaked with NaOH at a concentration of 5% for 72 h increased IFSS by 55.60% [43]. The condition of increasing the interfacial shear force on the fibers is due to increased surface roughness; the amount of hemicellulose, lignin, and pectin was reduced after alkaline treatment [42].

Alkali treatment of KL8 and KL10 shear strength of fiber and matrix interfaces decreased to 3.12 MPa and 2.99 MPa. Immersion for more than six hours indicates that the bonding of the interface decreases; this is because the fiber has defective damage. The highest increase in shear strength of the KPF / UPRs + mcc interface was 47.38% in the KL6. treatment. A similar treatment was done to increase the bonding interface between sisalana and PLA and sisalana and PP [44]. These results indicate that the alkaline treatment has better interfacial bond strength than the fumigation treatment.

IV. Conclusion

The fumigation and alkali treatments can improve the properties of King pineapple fibers and the adhesion strength of the KPF/Unsaturated polyester and microcrystalline cellulose. Fumigation and alkali treatment can increase density, tensile strength of fibers, thermal stability, interface bonding, and shear strength. Materials other than cellulose such as hemicellulose, pectin, lignin, and other impurities can be removed with both treatments, which are confirmed by the results of FTIR and TGA tests.

The fumigation treatment produced the highest surface energy of 74.21 mN/m, and the alkali treatment increased the surface energy to 76.16 mN/m. The highest thermal stability was obtained at 453°C for alkali treatment with a soaking time of 6 h. The highest increase in the interface shear strength between the fibers and the matrix obtained 3.67 MPa at six h of alkaline treatment. This study indicates that the alkaline treatment has a better effect compared to the fumigation treatment on King pineapple fiber.

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