

## Research Article

# Tension Strength and Fiber Morphology of Agave Cantala Roxb Leaves due to Liquid Smoke Immersion Treatment

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In this work, we investigate the fiber tensile strength of the Agave cantala roxb leaves based on the treatment of immersion in liquid smoke which was obtained from mature coconut shells. Four different time variations of treatments, namely, one hour, two hours, three hours, and four hours, have been carried out. After the immersion, we investigated the chemical contents of the Agave cantala using scanning electron microscope (SEM) which is equipped by energy dispersive spectroscopy (EDS). Furthermore, the tension strength is tested using tensile strength plus 50 N. The test is applied for a single Agave cantala fiber. The results of the tensile test of single fiber showed an increase in tensile strength when compared with fiber tensile strength without immersion. The tensile strength of a single fiber of Agave cantala leaves increased with increasing duration of immersion time. After reaching a certain strength, however, the strength of the fiber decreases. The tensile strength of a single fiber without immersion is 192.85 MPa and the highest is 670.82 MPa for Agave cantala after immersion for three hours, with an elongation of 0.68 mm. By increasing the tensile strength of Agave cantala through liquid smoke immersion treatment, it can potentially improve its performance as a material composite reinforcement.

## 1. Introduction

Increasing environmental awareness around the world has encouraged the design of environmentally friendly materials. The adverse environmental impacts have shifted attention from the use of synthetic fibers to natural fibers. Natural fibers have provided many advantages as composite reinforcement when compared with synthetic fiber reinforcement because of their price and density. As known, the price and the density of natural fibers are low. Moreover, they are easy to separate, widely available and renewable, biodegradable, and environmentally friendly [1–3]. Natural fibers contain lignocellulose which is hydrophilic because it contains many hydroxyl groups. The main limitation of using natural fibers as reinforcement for thermoplastic or thermosetting composites is that natural fibers are not compatible with hydrophobic composite matrices. This incompatibility leads to low interfacial adhesion between the

hydrophilic poles of the fiber and the hydrophobic poles of the matrix and difficulties in mixing due to the low wetting of the fibers by the matrix. This can result in the composite material having a weak interfacial strength [4–7].

This weakness can be overcome by providing chemical treatment, including alkali treatment which can remove some of the lignin, hemicellulose, wax, and oil, so that the surface of the fiber becomes rough, so there is a good compatibility when combined with the matrix [6, 8]. Voids affect the bonding between the fiber and the matrix, i.e., the gaps formed by the fiber and matrix. Not only so, the strength of the natural fiber-reinforced composite is influenced by several factors such as the shape of the fiber, the location of the fiber, the length of the fiber, and the bond between the fiber and the matrix [9]. Interfacial properties play an important role in analyzing the mechanical behavior of composites with natural fiber reinforcement. The strength of a biocomposite based on natural fibers is not only affected

by the interface conditions, namely, the bond between the fiber surface and the matrix, but is also influenced by the tensile strength of the single fiber itself [4, 10, 11].

Research on the characteristics of natural fibers is generally carried out experimentally through the study of several parameters such as physical, chemical, and mechanical properties of natural fibers whose origin is abundant in Indonesia, for example, king pineapple leaf fiber. It should be noted that king pineapple leaf fiber is the fiber obtained from *Agave cantala*. Hence, *Agave Cantala* hereafter is called king pineapple leaf fiber (KPLF). Given the great potential of king pineapple leaf fiber, it is necessary to strive to increase its role, not only as a traditional product but also to increase its function as a reinforcing material for natural fiber composites [12].

Based on the aforementioned facts, generally the surface treatment of natural fibers uses alkali and other chemical substances. In this work, the liquid smoke obtained from the distillation of burning coconut shells will be used as an alternative material for replacing alkali. The pH and the density of the liquid smoke are 2.429 and 1.026 gr/ml, respectively. The smoke from burning coconut shells contains carbonyl compounds, phenols, acetic acid, and other chemical compounds [13, 14]. The role of carbonyl groups is to improve the mechanical properties and the surface of the fiber to change the color and the phenol group to extend the shelf life of smoke because phenol is used as an antioxidant and antimicrobial so that the fiber can last longer [14], and acetic acid can break the lignin chain bonds and also penetrate the hemicellulose chain bonds and have an impact on the mechanical properties of the fiber increases [3, 13, 15]. The strength of composite materials with natural fiber reinforcement is generally determined by the chemical, physical, and mechanical properties of the fiber. Therefore, it is important to know the strength of the fiber before combining it with a matrix to understand how the final composite product behaves [14, 16]. Thus, the fiber with liquid smoke immersion treatment is predicted to play an important role in producing changes in the chemical, physical, and mechanical properties of the fiber. For this reason, research on natural cellulose fibers, especially KPLF, was carried out with the aim of revealing the effect of liquid smoke immersion treatment on the chemical properties and tensile strength of single fiber KPLF to be used as composite reinforcement.

The use of liquid smoke is very wide, such as in the food industry [17–19]. This liquid smoke is not only used as a food preservative but also as a flavor and aroma enhancer. Liquid smoke contains phenolic compounds and acids that are antimicrobial and antioxidant.

## 2. Materials and Methods

**2.1. Materials.** The material used in this study is known as “daun pondan datu” in the local language, and it was translated into English and became king pineapple leaves (daun is leaf, pondan is pineapple, and datu is king) which is actually *Agave cantala*. The materials were obtained from Tana Toraja regency which is one of the regencies in South

Sulawesi province. As mentioned above, this material will be further called KPLF. The selected samples of KPLF were soaked in freshwater for three weeks to make it easier for the fiber to be separated from the leaf flesh. The fiber is discharged mechanically from the sheet cells, and then the KPLF is cleaned of impurities with distilled water. The KPLF was cut into three parts, namely, the base, middle, and end, each with a length of 30 cm. As a test sample, the base of the KPLF is grouped to be treated with liquid smoke immersion with different time variations. In order to obtain the liquid smoke from coconut shell, the following procedures have been performed. Firstly, the mature coconut shells were selected. Then, coconut shell is put into a pyrolysis reactor to be heated at a temperature of 300–450°C for 5–8 hours. The result of this process is the liquid smoke in which it has pH of 2.429, density of 1.026 gr/ml, and viscosity of 1.45 Pascal seconds.

**2.2. Immersion Treatment.** The KPLF treatment process is by immersing the KPLF in a container that has been filled with liquid smoke. The KPLF treatment lasted for 1, 2, 3, and 4 hours, respectively. After the immersion time is reached, each KPLF sample is photographed to determine the color change due to immersion. The sample groups are presented in Table 1.

**2.3. Single Fiber Tensile Test.** KPLF with and without liquid smoke immersion treatment was used as a tensile test sample, namely, the fiber at the base with a sample length of 30 mm as shown in Figure 1. The KPLF diameter was measured with a digital optical microscope. The specimen was a separate fiber tensile test with ASTM 3379–02 test, and the tensile test was carried out using a tensile strength plus 50 N tensile testing machine. Each sample was tested five times, and the tensile strength of each specimen was automatically recorded on the monitor screen.

**2.4. SEM and Energy Dispersive Spectroscopy (EDS) Testing.** KPLF surfaces with and without liquid smoke immersion were tested with a scanning electron microscope (SEM) which is equipped by energy dispersive spectroscopy (EDS) facilities to detect chemical elements and compounds on the KPLF surface by firing X-rays at the position in which we want to know the chemical elements and compounds.

## 3. Results and Discussion

The liquid smoke immersion treatment clearly affects the color of the KPLF (see Figures 2(b) to 2(e)) compared with KPLF without liquid smoke immersion as shown in Figure 2(a).

The color of the KPLF surface is changed by immersing liquid smoke into a brownish to dark brown color because the liquid smoke from the distillation of coconut shell combustion contains carbonyl compounds [14, 20, 21], that contribute to color changes and transfer by fusion to the surface of the KPLF [14, 22, 23] and phenolic compounds

TABLE 1: King pineapple leaf fiber sample group name [15].

KPLF sample	Treatment
TP	Without immersion
P1J	1 hour immersion
P2J	2 hours immersion
P3J	3 hours immersion
P4J	4 hours immersion

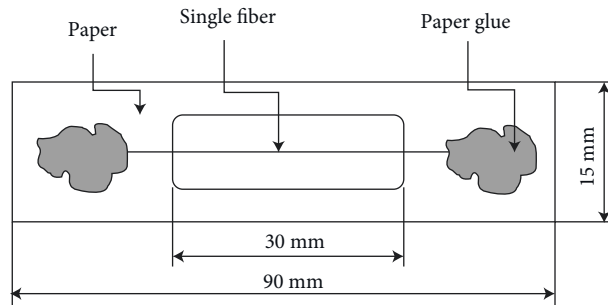


FIGURE 1: KPLF single fiber tensile test specimen.



FIGURE 2: Effect of liquid smoke immersion on the color change of KPLF: (a) TP, (b) P1J, (c) P2J, (d) P3J, and (e) P4J.

function as antioxidants and antimicrobials so that the smoked product lasts longer [14]. The surface color of the KPLF varies which depends on the liquid smoke soaking process time. The longer the immersion time is, the darker the surface of the KPLF will be [13, 14].

The results of the SEM and energy dispersive spectroscopy (EDS) tests show the mapping of the chemical elements contained in the KPLF before and after the liquid smoke immersion treatment, as depicted in Figures 3(a) to 3(e). Based on the results of the SEM and energy dispersive spectroscopy (EDS) tests, the chemical elements detected in the KPLF without liquid smoke immersion in Figure 3(a) and the KPLF undergoing liquid smoke immersion treatment, as shown in Figures 3(b) until 3(e), completely show the chemical elements accompanied by the percentage of Wt. The chemical elements in KPLF with and without liquid smoke immersion treatment were relatively constant, namely, O<sub>2</sub>, So, Mg, Po, Ph, Su, Fe, and Ch. However, Ca element decreased drastically after four hours immersion.

Unlike Si and Al elements in which their quantity of chemical elements Wt % increased until three hours immersion, but then they decreased after four hours immersion time. The details of these changes are shown in Table 2.

Table 2 reveals the results of the SEM (EDS) test which show that several chemical elements have increased, including Al which plays a role as a structure or framework in the fiber [23, 24], and is also supported by an increase in Si, while Mg acts as a binder. On the other hand, with the decrease of Ca element, it has an impact on changes in mechanical properties, namely, an increase in the tensile strength of KPLF.

Figure 4 shows a SEM photo of the KPLF surface, and the effect of liquid smoke immersion is visible because the KPLF surface without liquid smoke immersion in Figure 4(a) is very different when compared with the KPLF surface which is treated with liquid smoke immersion in Figure 4(b). Moreover, Figure 4 presents the KPLF surface with and without liquid smoke immersion treatment. The surface of

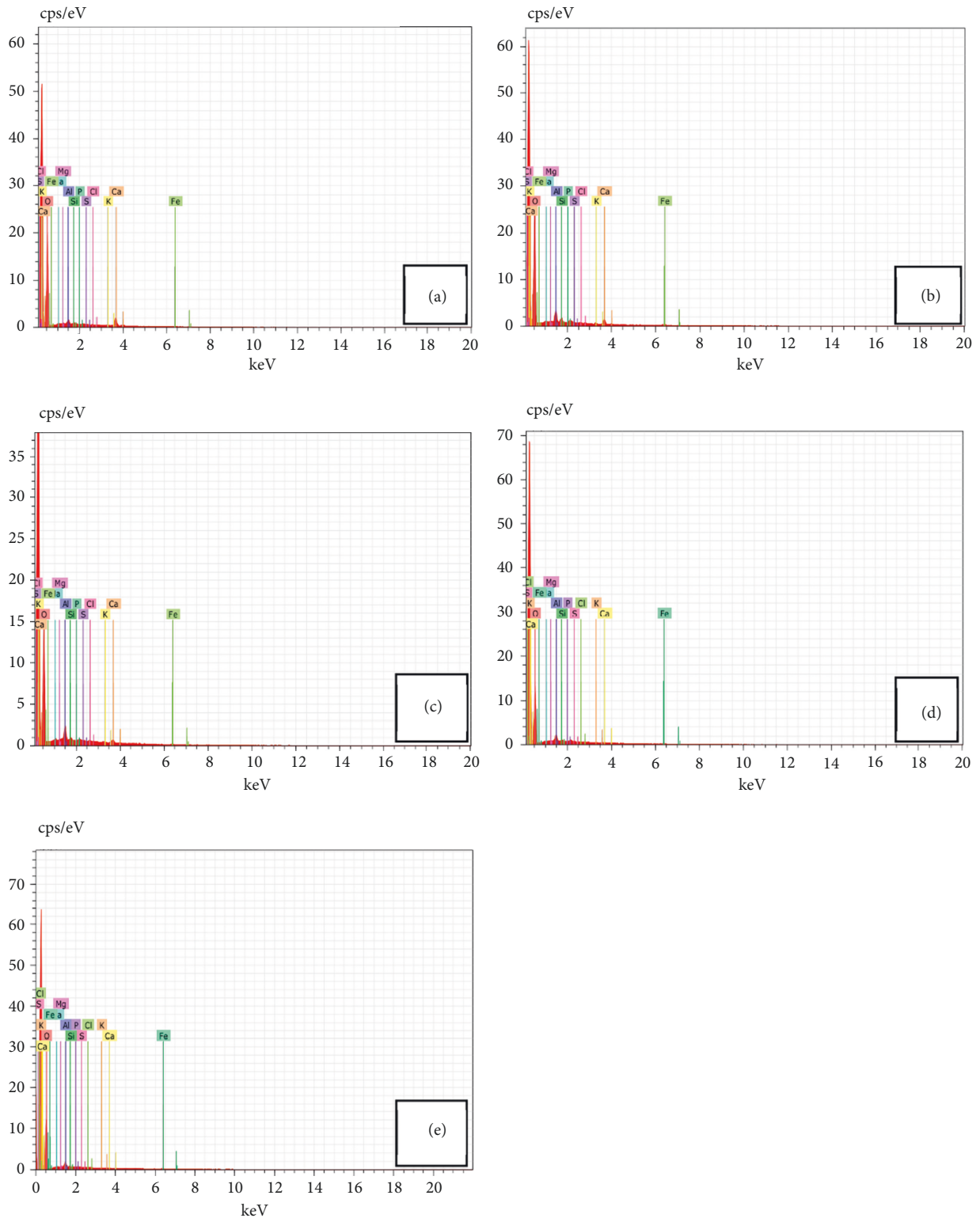


FIGURE 3: KPLF SEM (EDS) test results: (a) TP, (b) P1J, (c) P2J, (d) P3J, and (e) P4J.

the KPLF without immersion appears to have grooves with a large pattern and looks smooth as shown in Figure 4(a). On the other hand, Figure 4(b) shows that the surface of the KPLF appears to have changed in topography, dense grooves, and rough appearance because of the liquid smoke immersion treatment. The liquid smoke immersion

treatment can reduce lignin and hemicellulose due to acetic acid contained in liquid smoke. This is because acetic acid can break the lignin chain bonds and also penetrate the hemicellulose chain bonds so that the fiber surface becomes rough [14, 21, 22], and this is in line with the results of the KPLF SEM as shown in Figure 4(b). The reduced lignin and

TABLE 2: SEM test results (EDS) of chemical elements contained in KPLF.

KPLF chemical element	Quantity of chemical elements wt %, KPLF with and without liquid smoke immersion treatment				
	TP	P1J	P1J	P3J	P4J
Oxygen (O <sub>2</sub> )	36.79	40.73	40.96	43.98	42.92
Silicon (Si)	4.11	6.17	6.18	6.25	5.78
Aluminum (Al)	11.42	16.90	19.25	20.90	20.06
Sodium (So)	7.25	6.72	6.80	5.57	6.82
Magnesium (Mg)	4.54	4.83	4.96	3.76	3.77
Potassium (Po)	1.12	2.76	2.34	1.84	2.90
Calcium (Ca)	26.81	11.61	7.58	3.29	3.09
Phosphorus (Ph)	1.55	2.42	2.92	4.48	4.65
Sulfur (Su)	1.69	2.62	3.07	3.53	3.16
Iron (Fe)	2.43	3.15	3.06	3.84	3.29
Chlorine (Ch)	2.28	2.08	2.87	2.65	3.56

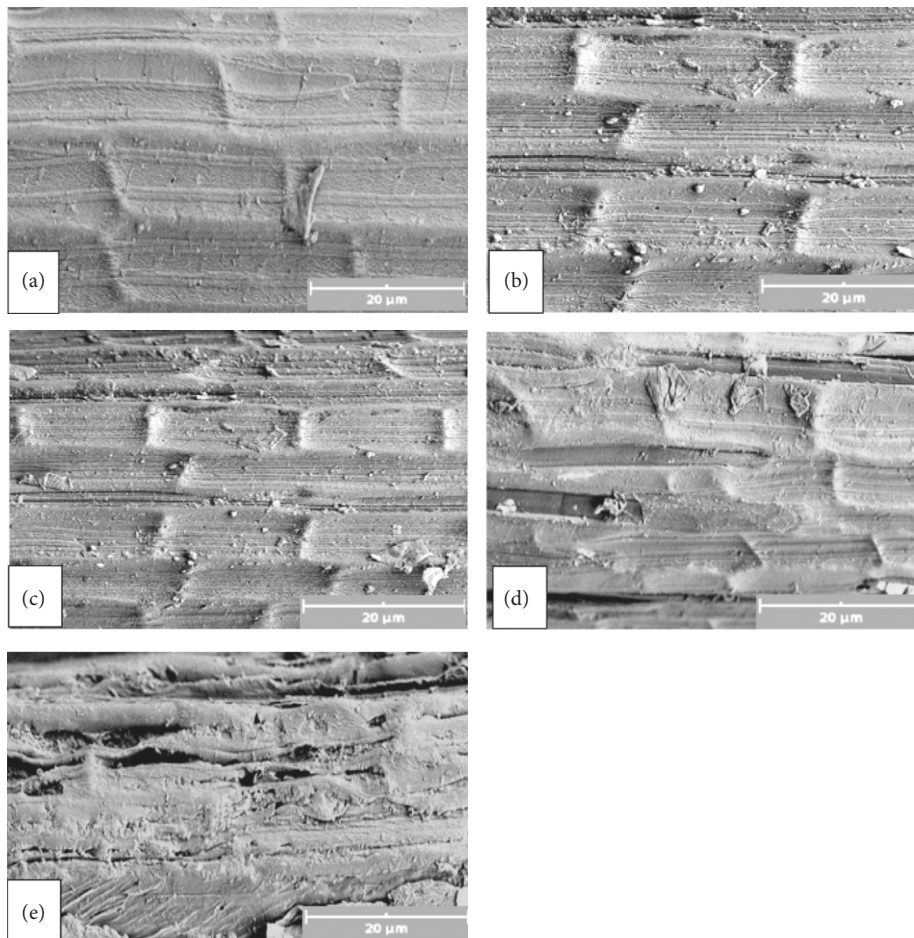


FIGURE 4: The results of the KPLF covering SEM photo: (a) TP, (b) P1J (c) P2J, (d) P3J, and (e) P4J.

hemicellulose elements make the cellulose cellular chain better because the cellulose fibrils become denser and denser so that the diameter of the KPLF shrinks and the impact on the cross-sectional area of the KPLF decreases, resulting in an increase in the mechanical strength of the KPLF [4, 13, 14]. The grooves that are formed and look rough on the KPLF surface for the cases P3J and P4J can facilitate KPLF base ability and matrix adhesion so that compatible adhesion bonds occur because of the strong mechanical bond.

Figure 5 presents that the results of the tensile test of a single fiber at the base of the KPLF and the liquid smoke immersion treatment have an effect on the increase in the KPLF tensile strength. The tensile strengths of the KPLF for the duration of immersion up to three hours have increased and then decreased for the longer immersion as shown in the case P4J.

The tensile strength of KPLF has increased when compared with nonimmersion fibers due to the increase in chemical elements, including elements of silicon (Si) and aluminum (Al) which are used as frames or structures in the

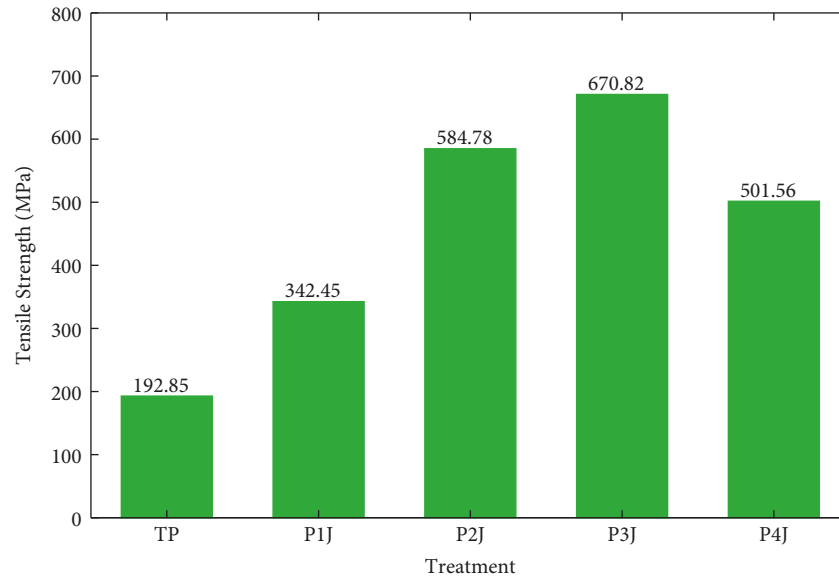


FIGURE 5: KPLF tensile stress at the base vs. immersion time.

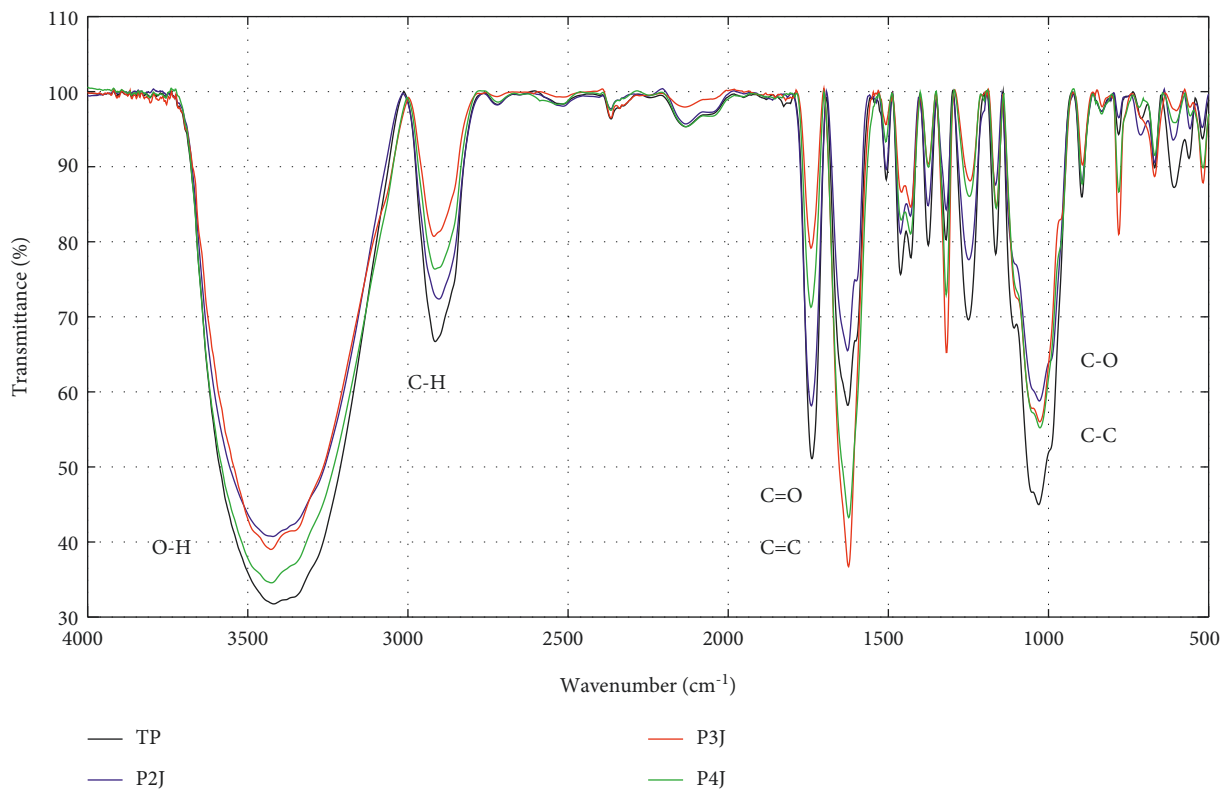


FIGURE 6: FT-IR KPLF test results.

fiber. However, calcium (Ca) as well as hemicellulose and lignin elements as shown in Table 2 has decreased. As a result, the KPLF strength increased [4, 12, 14, 23–25]. By reducing the hemicellulose and lignin elements, the fiber diameter decreases [4, 14], and the mechanical properties of the KPLF can increase [13, 23, 25]. In this study, what was observed was the immersion time of the liquid smoke which caused a change in the tensile strength of the fiber. The results of the

tensile test in the middle of the KPLF obtained the highest single fiber tensile strength at P3J of 670.82 MPa as shown in Figure 5. Then, the strength decreased after 3 hours of immersion. This can occur due to damage to the fiber structure as a result of too long soaking treatment so that the fiber becomes brittle [13, 14, 20]. Hence, based on the tensile test results, we found that the highest tensile strength of KPLF was 670.82 MPa, with an elongation of 0.68 mm.

Figure 6 depicts the result of the FT-IR KPLF test which shows the effect of immersion in liquid smoke. As can be seen from the figure, KPLF without immersion in liquid smoke (TP) is clearly different from the KPLF which is treated with immersion in liquid smoke. For simplicity, only three treated cases were presented, namely, one hour, two hours, and four hours immersion time.

The difference in functional groups in fibers without treatment and with treatment from the ends of the FT-IR test revealed that the intensity absorption of the O-H group in the range of  $3500\text{--}3000\text{ cm}^{-1}$  was seen in the very high intensity without treatment, but in the treated fiber there was a decrease in intensity because the treatment with liquid smoke can erode lignin where lignin has an O-H functional group. While the absorption intensity of the C-H group in the range of  $3000\text{--}2500\text{ cm}^{-1}$  was seen in the high without treatment but with the treatment, there was a decrease in intensity. The absorption of C=O and C=C groups in the  $2000\text{--}1500\text{ cm}^{-1}$  range and C-O and C-C in the  $1500\text{--}500\text{ cm}^{-1}$  range also showed differences in intensity between the untreated and treated fibers wherein the fiber with the treatment decreased in intensity; This is because the changes in the functional groups C-H, C=O, and C=C decrease with the treatment process, where the lignin compound in the hydroxyl group decreased. From the SEM test, it can also be seen that the longer the immersion period, the rougher the surface of the fiber. It is also confirmed that when the immersion time is longer, the tensile strength increases. However, after reaching a certain point, the strength decreases.

With the increase in the mechanical properties of KPLF after undergoing the immersion treatment process, the material has the potential and opportunity to be used as a reinforcement for composite materials. The strength of fiber-reinforced composite materials is generally determined by the mechanical properties of the fiber, so it is important to know the fiber strength before combining it with the matrix to understand how the final composite product behaves.

#### 4. Conclusions

We have investigated the influence of time immersion of the pineapple king leaf fiber on the increasing of tensile strength. Based on our experiments, there are two points that can be concluded. First is the morphology of the KPLF surface. The results without immersion show grooves with a large pattern and looks smooth. The treated surface of the KPLF appears to experience topographic changes, namely, dense grooves, due to acetic acid, which can break the lignin chain bonds and penetrate the hemicellulose chain bonds. Consequently, the morphology of the fiber surface of the king pineapple leaves becomes rougher and more grooved. Another point is that the tensile strength of a single fiber has increased when compared with the fiber without immersion of 192.85 MPa. The tensile strength of the single fiber at the base of the highest KPLF was obtained at 670.82 MPa when the material was immersed for three hours. Moreover, when combined with the matrix, the interfacial bond or compatibility became stronger due to

mechanical bonding. Moreover, its elongation is about 0.68 mm. We believe that these results can show another alternative source good quality fiber.

#### Data Availability

The data used in this work are available from the authors upon request.

#### Conflicts of Interest

The authors declare that there are no conflicts of interest.

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