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THE EFFECT OF FUMIGATION TREATMENT TOWARDS AGAVE CANTALA ROXB FIBRE STRENGTH AND MORPHOLOGY

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Abstract

The objective of this study is to reveal the morphology, physical properties and strength of the king pineapple leaf fibre (Agave Cantala Roxb) after fumigation treatment. The king pineapple leaf fibres (KPLF) before and after the fumigation treatment are then separated into groups. The fumigation treatment on KPLF is given in different durations, and the smoke comes from burning coconut shells. Before and after fumigation, the surface morphology, chemical content, and functional group character of KPLF were observed by SEM, XRD, and FTIR, respectively. While the physical characteristics were identified by measuring fibre density, moisture content and fibre strength were tested by a single fibre tensile strength test. The results show that chemical contents of KPLF were cellulose, hemicellulose and lignin, accounting for as much as 55.8%, 21.27%, and 7.66%, respectively. After fumigation, the KPLF surface morphology becomes rough and grooved, the fibre density increased, and the single fibre tensile strength increased notably at the base of the king pineapple leaf. With the tensile strength increase and a rough and grooved KPLF surface morphology due to fumigation, fumigated KPLF would have the potential to be used as a strengthened composite.

Keywords: King pineapple leaf fibres, Fumigation, Morphology, Chemical properties, Physical.

1. Introduction

The king pineapple plant (Agave Cantala Roxb) is easily cultivated on land that is not too wet and rocky. It has a stem height of approximately 2 m and a leaf length of about 1 m. It has a strong leaf fibre. Traditionally, the KPLF has been used by the people in Tana Toraja, South Sulawesi Province, Indonesia as straps, and is also woven as corpse wrapping and could last for hundreds of years when it is fumigated

Nomenclatures

m_a	Fibre mass in air, gram
m_o	Fibre mass in methanol, gram
M_a	Sample test initial weight, gram
Mb	Fibre final weight, gram
M_f	Fibre moisture content level, %
Mp	King pineapple leaf middle part
Pj	King pineapple leaf base part
Pt	King pineapple leaf end part

Greek Symbols

ρ_0	Methanol solution density, g/cm^3
ρ_f	Fibre density, g/cm^3
σu_{Mp}	Leaf middle part fibre tensile strength, Pa
σu_{Pj}	Leaf base part fibre tensile strength, Pa
σu_{Pt}	Leaf end part fibre tensile strength, Pa

Abbreviations

EDX	Energy Dispersive X-Ray
FTIR	Fourier Transform Infra Red Spectroscopy
KPLF	King Pineapple Leaf Fibre
SEM	Scanning Electron Microscope

Given the enormous potential of the king pineapple plant, people are seeking to enhance its role not only as a traditional material, but also to improve the function of the raw fibre composites. The advantage of natural fibre composites is that they are cheap when with synthetic fibre reinforcement such as glass fibre and carbon, renewable and environmentally friendly. The main content of natural fibres is lingocellulose, namely, cellulose, hemicellulose and lignin [1, 2].

Cellulose and hemicellulose are polysaccharide compounds, while lignin compounds are polyphenols macromolecular compounds [3]. Various methods have been used to improve the compatibility of natural fibres that are hydrophilic [3, 4] to improve the fibre strength or ductility both physically and chemically.

Chemical treatment is one of the solutions to improve the fibre's mechanical properties and surface morphology, remove dirt, degrade hemicellulose and lignin compounds, and improve the interaction between the fibre and matrix [5-7].

The fibre surface morphology should be treated early to improve the fibre wettability so that mechanical strength increases [1, 3, 4]. The fibre surface treatment is expected to increase fibre surface wettability properties and matrix adhesion bonding. Fibre bonding degradation is the binding infiltration process of getting into the fibre pores and does not damage the surface, which could reduce the mechanical strength of the fibre [6-8].

The natural fibre mechanical property is still far below that of fibre glass [2-4]. In other words, composites reinforced with natural fibres have a much lower mechanical strength compared with composites reinforced with fibreglass [5, 9]. Composite mechanical properties are determined by the fibre interaction strength with the matrix, in which the interaction is influenced by the cellulose fibre molecules' quality and epoxy is used [9-11].

This study uses a smoke stream from burning coconut shells. Coconut shell smoke is comprised of elements from carbonyl, phenol and acidity. Carbonyl has the greatest effect in the formation of colour on the product being smoke. Phenol also contributes to colour formation, although its intensity was not as great as carbonyl an acidity as an antioxidant so that the product been smoke being durable [12, 22, 26]. Based on these explanations, it is necessary to study natural fibres, especially cellulose fibres of king pineapple leaves (Agave Cantala Roxb) that has been fumigated to reveal the physical properties, chemical content, fibre surface morphology, and single fibre tensile strength.

2. Materials and Methods

The material used is the KPLF with an average age of 11 months, approximately 1 meter in length. The KPLF was firstly washed with distilled water and then dried at a room temperature of 31°C. The KPLF was then treated by fumigation with time variations. To find out which parts of KPLF are strongest, the KPLF was cut into three parts, the base (*Pj*), centre (*Mp*) and end part (P_t) as shown in Fig. 1(b).

KPLF fumigation was carried out in a fumigation box where smoke is obtained by burning coconut shells. The smoke produced in this box was then sent into another container for the fumigation process. The smoke was continuously sent into the fumigation box. The KPLF fumigation process is performed for 5, 10, 15, and 20 hours. During fumigation, the fumigation box room temperature was maintained at approximately 45 °C, as shown in Fig. 2.

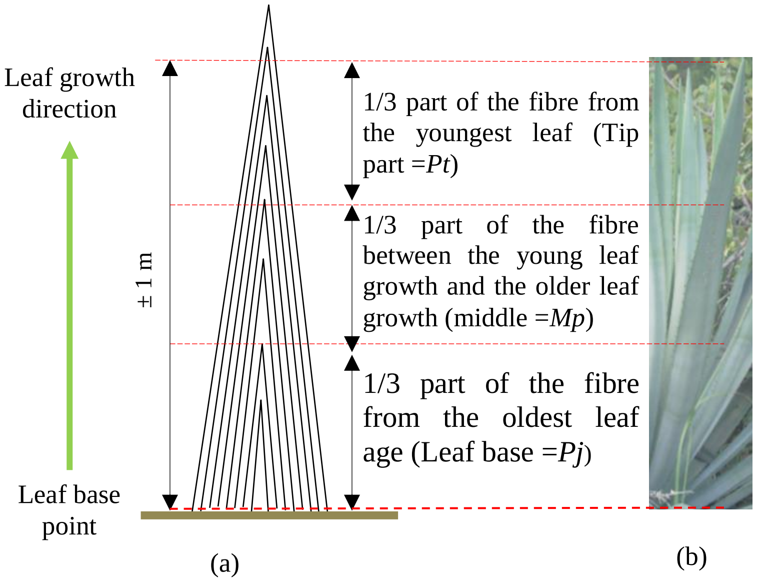
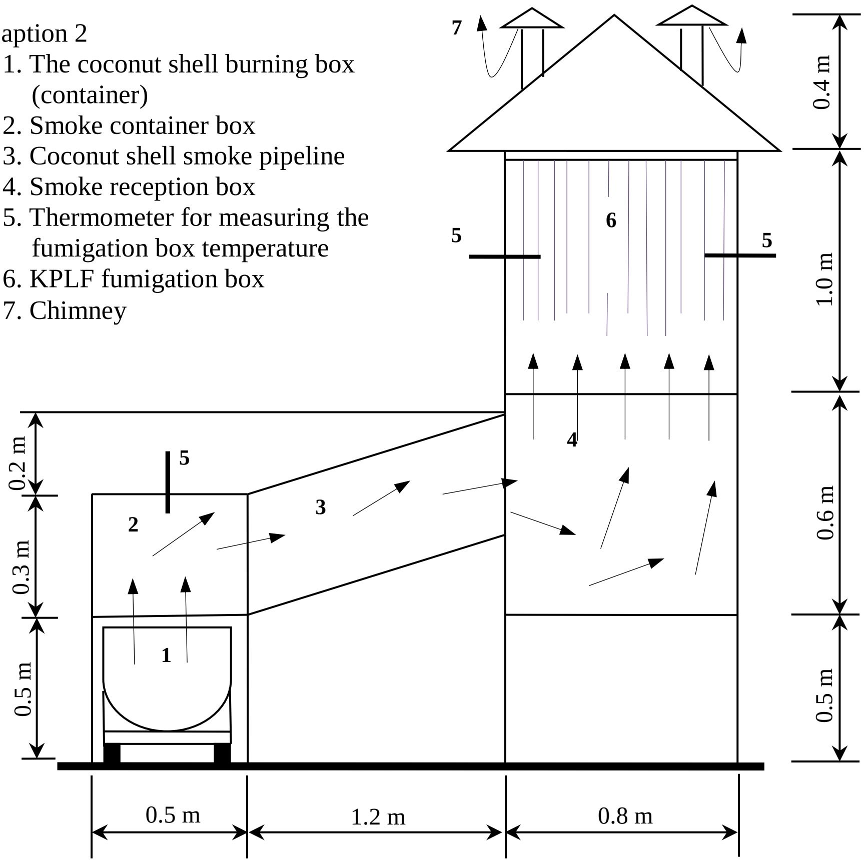


Fig. 1. (a) Plant growth illustration, (b) King pineapple leaf.

Table 1. King pineapple leaf fibre sample group name

Fibre sample group	Information
KPLF WF	King pineapple leaf fibre without fumigation
KPLF F 5H	King pineapple leaf fibre with fumigation 5 hours
KPLF F10H	King pineapple leaf fibre with fumigation 10 hours
KPLF F15H	King pineapple leaf fibre with fumigation 15 hours
KPLF F20H	King pineapple leaf fibre with fumigation 20 hours



Caption 2
1. The coconut shell burning box (container)
2. Smoke container box
3. Coconut shell smoke pipeline
4. Smoke reception box
5. Thermometer for measuring the fumigation box temperature
6. KPLF fumigation box
7. Chimney

Fig. 2. KPLF fumigation box.

The KPLF that was not fumigated and KPLF that had been fumigated were compared to determine the cellulose composition, hemicellulose and lignin. Fibres from the functional groups were observed with Energy Dispersive X-Ray (EDX), and the chemical properties were observed with FTIR spectroscopy. The fibre surface morphology was tested using a Scanning Electron Microscope (SEM).

Fibre surface roughness was tested using SJ-301equipment under a JIS-B0601-2001 standard. Fibre surface roughness was obtained by placing the test sample under the stellus-preparat, which was set at a certain position so that it could easier to detect the fibre surface. The stylus tool (sensor surface roughness) was shifted from the start end to the end point of the corresponding end of the fibre surface detected. When the stylus was shifted, the screen automatically displayed the graph in numbers.

2.1. Moisture content

Moisture content is the quantity of water contained in a material. Moisture content is used in a wide range of scientific and technical areas, and is expressed as a ratio, which can range from 0 (completely dry) to the value of the materials porosity at saturation. It can be given on a volumetric or mass (gravimetric) basis.

The fibre moisture level was determined by D 629 Standard ASTM Quantitative Analysis of Textiles Materials. The sample test was weighed with a digital scale with an initial weight (M_a). The fibre sample was then heated in an oven for about 4 hours until it reacheda temperature of 110° C, after which the sample was cooled in a desiccator for 30 minutes. The next process was weighing the fibre sample, and the result was the final weight (M_b). The moisture content level (M_f) was calculated by Eq. (1).

$$M_f = \frac{M_a - M_b}{M_a} \times 100\ \% \tag{1}$$

2.2. Specific gravity

Specific gravity is the ratio of the density of a substance to the density of a reference substance; equivalently, it is the ratio of the mass of a substance to the mass of a reference substance for the same given volume.

The KPLF specific gravity was tested before and after fumigation by an Archimedes method using methanol solution as the soaking liquid. The specific gravity was the comparison between the sample mass in air and the sample soaked in a methanol solution (ρ_o= 0.791 g/cm^3). The fibre sample mass was measured with a digital scale. The fibres' mass in air was (m_a), and the fibre mass in methanol was (m_o). The fibre density (ρ_f) was calculated using Eq.(2)

$$\rho_f = \frac{m_a \times \rho_0}{m_a - m_0} \tag{2}$$

2.3. Tensile testing

Tensile testing is a fundamental materials science test in which a sample is subjected to a controlled tension until failure. The results from the test are commonly used to select a material for an application, for quality control, and to predict how a material will react under other types of forces. Properties that are directly measured via a tensile test are ultimate tensile strength, maximum elongation and reduction in area.

Single fibre tensile test was carried out using a tensile testing machine LR10K plus 10 kN Universal Materials Testing Machine, under an ASTM 3379-02 standard. The fibre sample length was 30 mm. The fibre diameter was measured through an optical microscope. Each set consisted of 5 specimens. The tensile strength of each specimen was automatically recorded on the monitor screen [3].

3. Result and Discussions

Figure 3 shows the morphology of the surface KPLF without fumigation. The figure shows the regular rectangular pattern on the surface of the KPLF, which is smooth, as indicated by the arrow. However, after being treated with fumigation for 5, 10, 15 and 20 hours, as shown in Figs. 4, 5, 6, and 7 respectively. The surface of the rectangular pattern is rougher compared to the KPLF without fumigation. The roughness depends on the duration of the fumigation treatment.

Figure 4 shows the morphology surface KPLF with a 5 hour fumigation time. The KPLF surface changed as shown by the arrow direction, which indicates a rectangular pattern with bumps that looks a bit rough. In addition, there are longitudinal grooves such as trenches in the rectangular pattern.

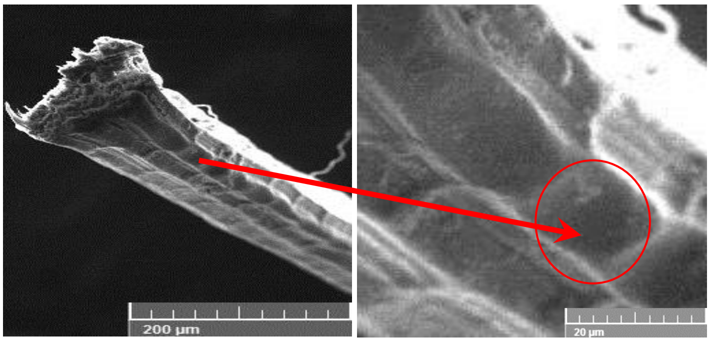


Fig. 3. Fibre surface SEM picture without fumigation.

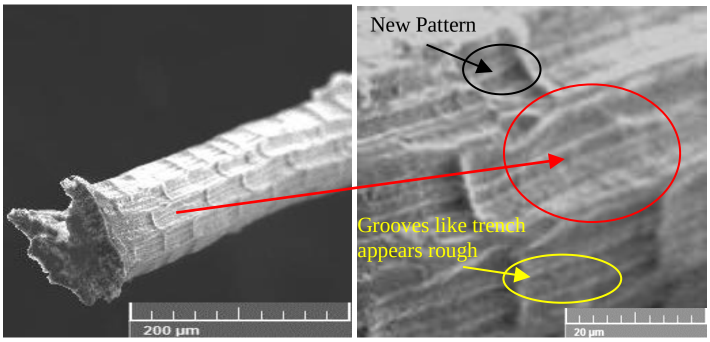


Fig. 4. Fibre surface SEM picture with 5 hours fumigation time.

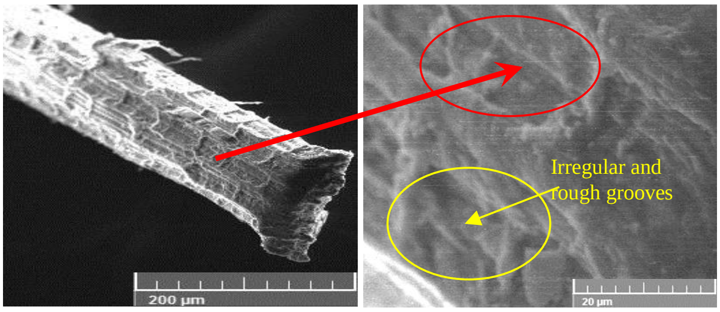


Fig. 5. Fibre surface SEM picture with 10 hours fumigation time.

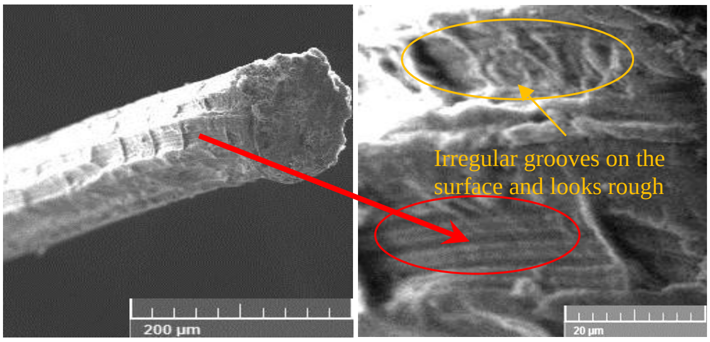


Fig. 6. Fibre surface SEM picture with 15 hours fumigation time.

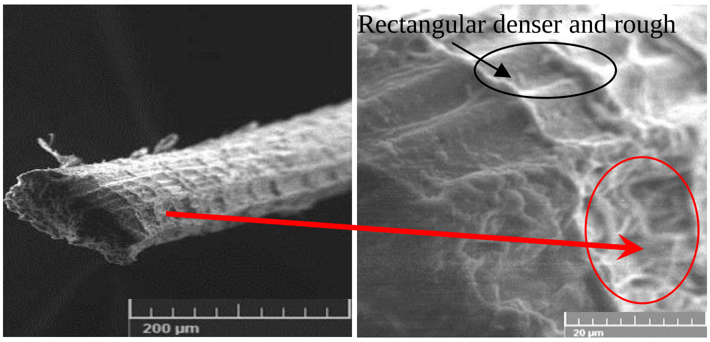


Fig. 7. Fibre surface SEM picture with 20 hours fumigation time.

The rectangular pattern that emerged on the surface of fibre after it was fumigated for 5 hours is still the same pattern found on the surface of fibre that was not fumigated. However, after being fumigated for 10 hours, the rectangular pattern become irregular, and the groove direction in the pattern is not longitudinal anymore, as shown in Fig. 5 and indicated by the arrow.

The surface morphology of the fibre fumigated for 15 hours is almost the same as the morphology of the fibre fumigated for 10 hours. The longitudinal grooves are still observable in some rectangular patterns together with a non-longitudinal pattern as shown in Fig. 6. In this figure, the non-longitudinal groove pattern is rougher compared to those patterns observed on the fibre fumigated for 5 hours. The black arrow indicates the rougher non-longitudinal groove pattern. Although the rectangular pattern is still observable after the fibres were fumigated for 20 hours, the longitudinal grooves are not observable anymore. In this case, the grooves become irregular and rougher. The irregular groove pattern was expected because it facilitated a matrix to fill that pattern so it could improve the engagement between the fibre and matrix when the fibres are used to reinforce a composite.

Figure 7 shows the KPLF surface morphology after 20 hours of fumigation. A significant change on the KPLF surface increases compared with KPLF that was not fumigated. The arrow indicates that where pores appears, grooves appear uneven and rough. In addition, on the rectangular pattern edge, there are some irregular protrusions that are denser and look rough due to fumigation. The changes of pores and grooves and the increase of surface roughness are expected to facilitate the matrix to fill the pores and grooves so it can improve the fibre matrix bonding [15].

Because engagement between the fibre and matrix affect composite strength, and engagement is affected by surface morphology and roughness [3, 16, 17], in the present study, it is important to determine the influence of the duration of fumigation on the surface of the fibre.

Figure 8 shows the effect of the duration of fumigation on surface roughness of the fibre. The figure shows that the longer duration may increase the roughness. The increasing surface roughness together with irregular grooves in the rectangular pattern may increase engagement between fibres and matrix, and a stronger composite is expected. How the condition of the surface fibre is affected by fumigation in association with composite strength will be investigated in the very near future.

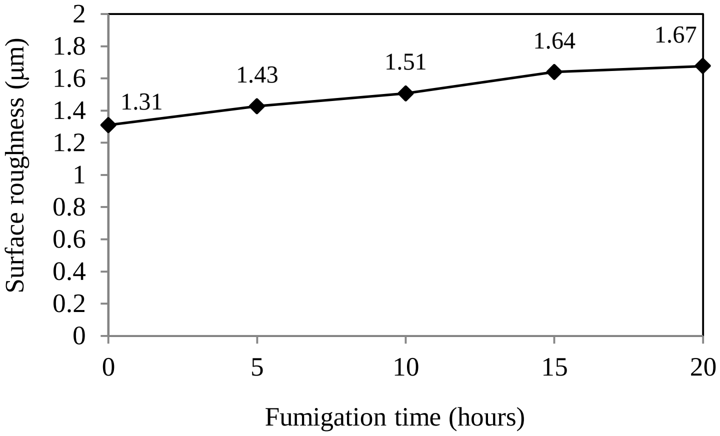


Fig. 8. Surface roughness vs. fumigation time.

As shown in the previous figures, fumigation changed the morphology of the fibre surface, and the extent of change depended on the duration of the fumigation process. The longer the duration, the rougher the surface would be, as shown in Fig. 8. This was caused by the removal of a layer of substances on the surface of the fibre such as lignin and hemicelluloses. In Fig. 3 and Fig. 8, the surface of the un-fumigated fibre is relatively smooth compared to the fumigated fibres. The smooth surface indicates that the un-fumigated fibre is still covered with lignin and hemicellulose [27, 29, 30]. However, fumigation removed the lignin and hemicellulose, as indicated by the decreasing content of both substances as shown in Fig. 9. Figure 9 also shows, that the content of the cellulose is relatively stable. Because the duration of the fumigation process lowers lignin and hemicellulose, the fibre's surface is rougher.

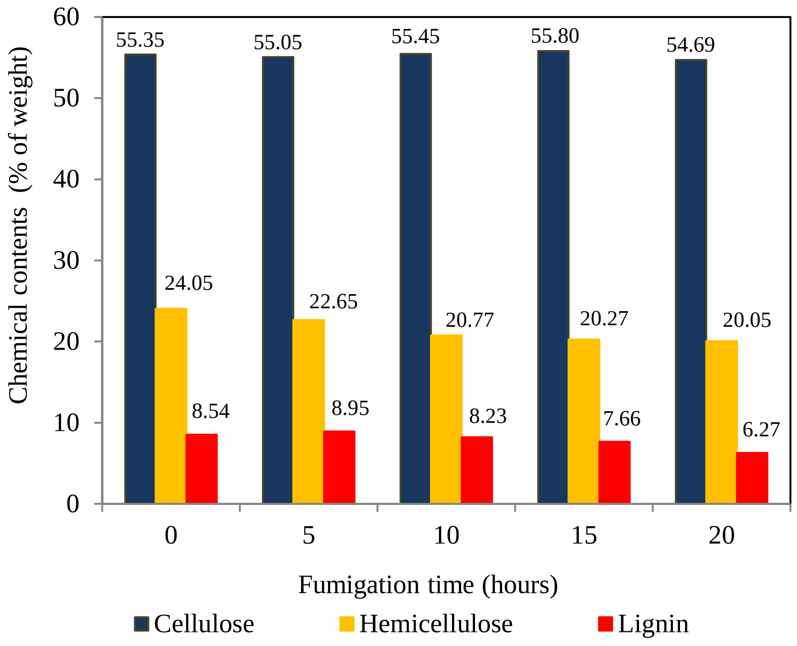


Fig. 9. Chemical contents vs. fumigation time.

The bio-composites' strength base on the fibre is not only affected by the interface condition between the fibre surface and the matrix, but is also influenced by the single fibre tensile strength itself [3, 17, 28]. In this study, the fumigation process time is observed to determine which fumigation treatment would cause changes in the fibre tensile strength.

Figure 10 shows that fumigation process time can increase the single fibre tensile strength at each KPLF section part. The tensile strength of the end part (σu_{Pt}) is 523.18 MPa. It is 658.98 MPa for the middle part (σu_{Mp}). Single fibre tensile strength is highest at the fibre base (σu_{Pj}) at 738.61 MPa, after been fumigated for 15 hours. It is very interesting that the single KPLF tensile strength is proportional to low carbon steel tensile strength. Therefore, fumigated KPLF with would be a very good potential reinforcement in bio-composites.

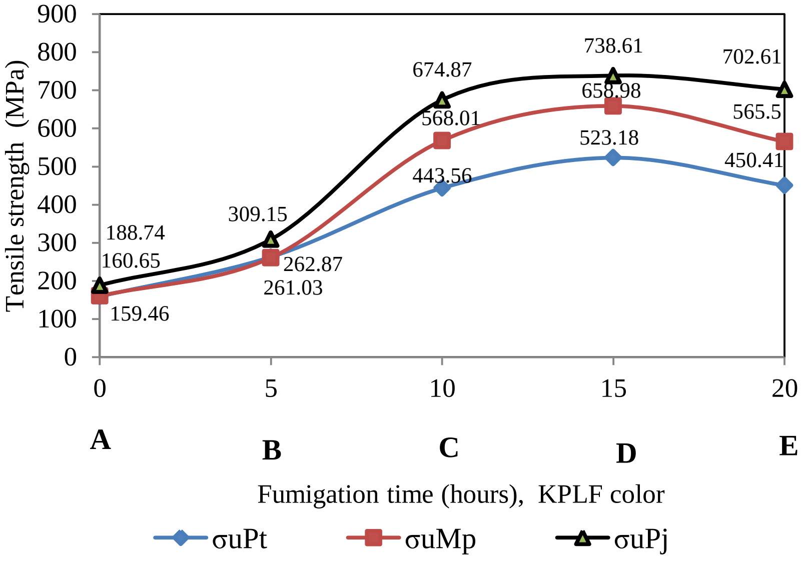


Fig. 10. Tensile strength vs. fumigation time and KPLF color.

Fig. 11. KPLF (A) WF,(B) F5H, (C) F10H, (D) F15H, (E) F20H.

Figure 11 shows that the un-fumigated fibre surface is whiter, while fumigated fibre is darker. The longer the fumigation time, the darker the fibre surface becomes (dark brown) due to the effects of coconut shell smoke, which contains carbonyl elements [20, 21, 26]. The colour change had an impact on the changes in the fibre tensile strength compared to un-fumigated fibres, as shown in Fig. 10.

Fumigation also increases the fibre tensile strength, depending on the duration of the fumigation process as shown in Fig. 10. The KPLF tensile strength increased compared to un-fumigated fibres because the cellulose content remained constant while the hemicellulose and lignin levels decreased [12, 27, 28]. With the hemicellulose and lignin levels reduced, the fibre diameter is reduced as shown in Fig. 12, which results in a tighter bond [21], causing the KPLF's mechanical properties and density to increase [23-25, 27, 29].

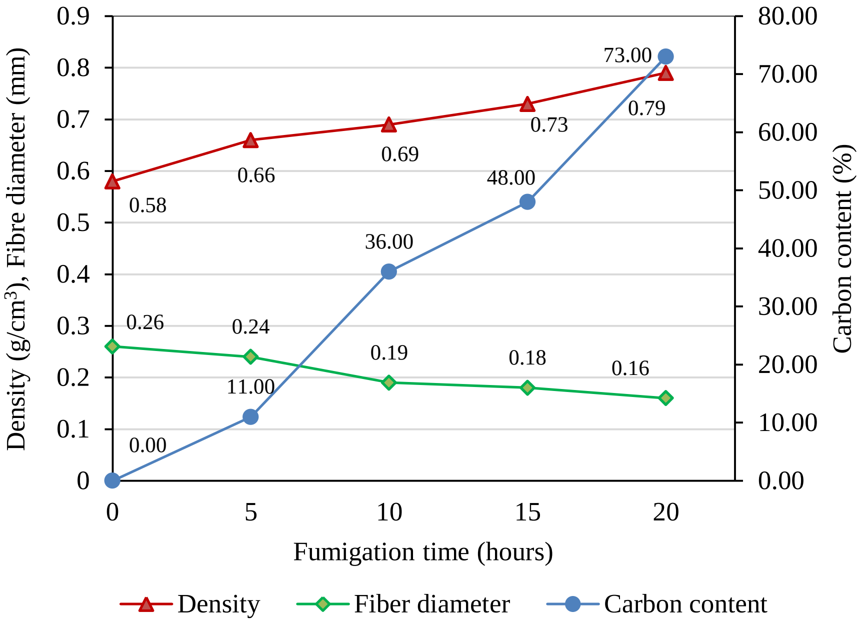


Fig. 12. Density, fibre diameter, carbon contents vs. fumigation time.

Fig. 12 shows that the KPLF density increases, probably because the fibre's moisture content decreases with the length of fumigation time [21].

The KPLF colour change has to do with carbonyl, which is contained in the coconut shell smoke [20-22]. Carbonyl creates a brown colour on the surface of fumigated products [11, 14, 26]. This is supported by EDX test results, showing that KPLF without fumigation does not contain carbon and the carbon level increases with the length of fumigation time as shown in Fig. 12, so the mechanical properties of KPLF also would be changed.

Looking at the moisture content level test results, the KPLF tensile strength should increase in line with the increase in fumigation time, but after 20 hours of fumigation, the KPLF tensile strength decreased due to too high carbon content (see Fig. 12). This also happens because of a decrease in moisture content (see Fig. 13).

The moisture content dropped too sharply after 20 hours of fumigation, resulting in dry and brittle fibres [19], which in turn resulted in decreased tensile strength.

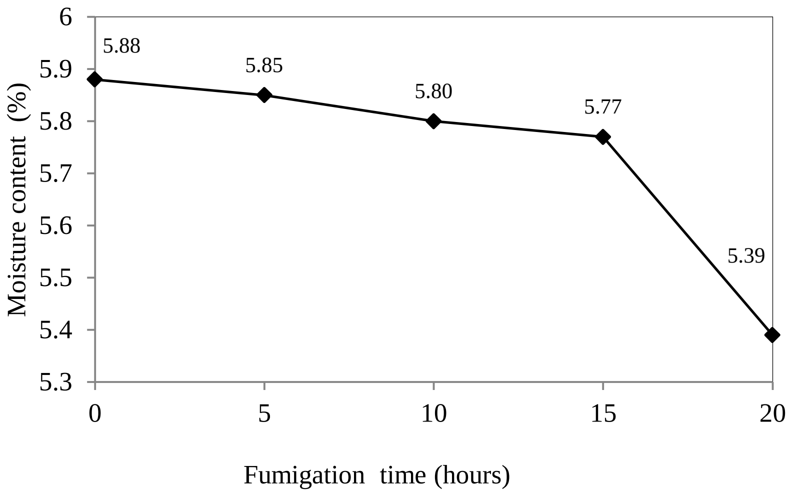


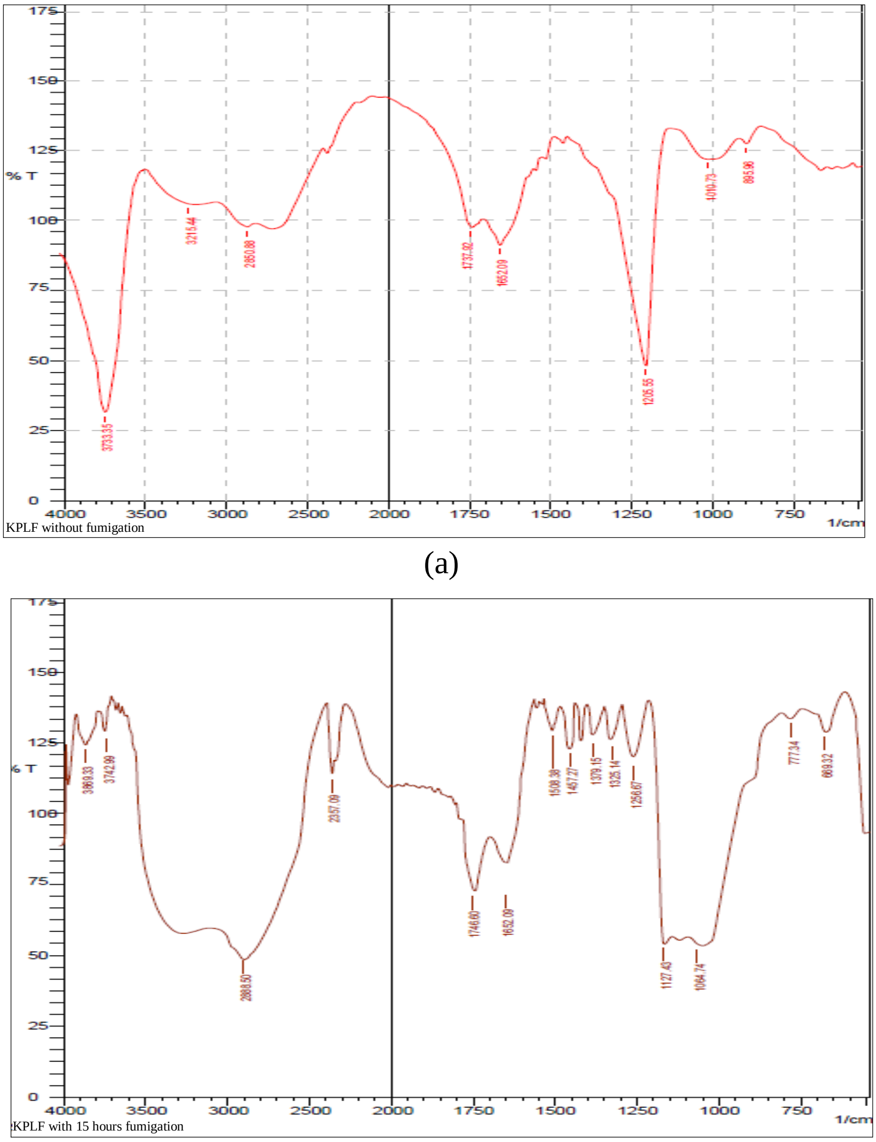
Fig. 13. Moisture contents vs. fumigation time.

The highest single fibre tensile strength obtained at the KPLF base part was 738.61 MPa. KPLF tensile strength decreased after 15 hours of fumigation due to a drastic reduction of moisture levels. This decrease of moisture damages the fibre's structure. as a result of too long a fumigation treatment [21]. This long fumigation time also results in a brittle fibre [19], causing the KPLF strength to decrease as shown in Fig. 10.

As shown in Fig. 14, the fibre's chemical functional group changes are caused by fumigation. It can be analysed using FTIR spectroscopy; either on un-fumigated KPLF or on fumigated fibre treated with varying fumigation times. Fig. 14 shows that the untreated fibre pattern's infrared ray’s transmission is different from the fumigated fibre's transmission pattern, which affects the fibre's chemical structure. Fumigation also affects the wave crest that is formed as a peak or KPLF ribbon vibration peak, in which the wave number or frequency was changed compared to un fumigated KPLF.

The FTIR spectrum shows that the KPLF constituent molecules consist of an O-H group with a wave range of 3000-4000 cm^{-1}. The hydrogen bonding groups (O-H) on hemicellulose, cellulose and lignin were changed after fumigation. The wave numbers of 2850-3000 cm^{-1} are related to the C-H bonds often observed in the alkane group. The peak intensity of the cluster, C=O in ketones and carbonyl group was indicated at the peak in a range of 1715-1765 cm^{-1} and the peak range of 2000 to 2500 cm^{-1}.

The C=O uptake start appears in the 5 hour fumigation sample and C=C for the further fumigation time. Thus, fumigation causes carbon to increase so that the KPLF colour changes as shown in Fig. 12, this could also improve the KPLF's mechanical properties (see Fig. 10). The top range of 1450-1650 cm^{-1} refers to the alcohol compounds and lignin aromatic structure in the KPLF, which are the C=C bonds.



(a)

(b)

Fig. 14. FTIR: (a) KPLF without fumigation, (b) KPLF with fumigation.

Peaks at the 1000-1250 cm^{-1} wave numbers are seen as C-H of the hemicellulose bond, the cellulose bond and alcohol group. Peaks at wave numbers 500-900 cm^{-1} are associated with the lignin C=O, and its intensity decreases after being subjected to fumigation. This indicates that the lignin and hemicellulose content in the KPLF is reduced after fumigation (see Fig. 9). The peak is in the wave number range of 890-900 cm^{-1} and is characteristic of β-glycoside links on fibre cellulose and hemicelluloses, [3, 16, 27].

4. Conclusions

Based on the previous description about the chemical composition and morphology of the KPLF surface due to fumigation, the following conclusions can be drawn.

- The fumigation treatment could reduce the hemicellulose and lignin elements. While the cellulose is relatively constant, causing the KPLF surface morphology to change its colour and become groovy and rough, so if it is used as a reinforced composite, the bonding effect will be better.
- Fumigation treatment could increase the KPLF carbon level and KPLF density so that the single fibre KPLF strength, on the leaf base part and with less than 15 hours of fumigation time, has a tensile strength of 738.61 MPa, and the KPLF single fibre tensile strength decreases as the result of a drastic decrease of moisture content after 20 hours of fumigation.

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