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The Effects of Alkali Treatment on the Mechanical and Chemical Properties of Banana Fibre and Adhesion to Epoxy Resin

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ABSTRACT

The main focus of this study was to obtain the optimum alkaline treatment for banana fibre and the its effect on the mechanical and chemical properties of banana fibre, its surface topography, its heat resistivity, as well as its interfacial bonding with epoxy matrix. Banana fibre was treated with sodium hydroxide (NaOH) under various treatment conditions. The treated fibres were characterised using FTIR spectroscopy. The morphology of a single fibre observed under a Digital Image Analyser indicated slight reduction in fibre diameter with increasing NaOH concentration. The Scanning Electron Microscope (SEM) results showed the deteriorating effect of alkali, which can be seen from the removal of impurities and increment in surface roughness. The mechanical analysis indicates that 6% NaOH treatment with a two-hour immersion time gave the highest tensile strength. The adhesion between single fibre and epoxy resin was analysed through the micro-droplet test. It was found that 6% NaOH treatment with a two-hour immersion yielded the highest interfacial shear stress of 3.96 MPa. The TGA analysis implies that alkaline treatment improved the thermal and heat resistivity of the fibre.

Keywords: Alkaline treatment, banana fibre, biocomposites, interfacial shear stress, natural fibre

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INTRODUCTION

The usage of composite material has been found to increase significantly in the manufacture of aircraft parts, from 5% in the 90s to 50% in 2010 (Gent et al., 2010). The composite parts are mainly made of petroleum derivative products, such as carbon fibre.

However, most of the synthetic fibres are costly and non-biodegradable and they are facing depletion. The rise of global awareness of environmental issues has attracted researchers in various areas to develop renewable materials based on sustainability principles (Mahjoub et al., 2014). A huge shift in the usage of natural fibres to produce composites worldwide has been reported (John et al., 2008). Natural fibre reinforced composites (NFRC) have been used in construction and automotive applications for a while, and are now becoming more substantial as secondary structures for next-generation aircraft (Koronis et al., 2013). The advantages of using natural fibre composites are environmental gain, reduced energy consumption and reduced dependency on petroleum-based materials; in addition the material is light weight and provides improved insulation and sound absorption properties (Joseph et al., 2002). Natural fibres such as jute, sisal, hemp, kenaf, banana and pineapple leaf (PALF) are renewable and non-abrasive and they can be incinerated for energy recovery. They possess good calorific values and present low safety risk during handling. They also exhibit excellent mechanical properties, have low density and are inexpensive (Boopalan et al., 2013).

Numerous research and studies have been carried out on natural fibre reinforced composites (NFRC) in recent years (Alavudeen et al., 2015; John et al., 2008; Sanjay et al., 2015). To develop NFRC, it is vital to understand the chemical composition and the surface adhesive bonding properties of natural fibre. The components of natural fibre include cellulose, hemicellulose, lignin, pectin, waxes and water-soluble substances (Li et al., 2007). Since natural fibre is hydrophilic in nature, chemical modification of the fibre is required in order to improve the interfacial properties between the fibre and any polymer matrix (Asim et al., 2015). Various chemical treatments for natural fibre, namely alkaline, silane, acetylation, benzoylation, acrylation, maleated coupling agent, permanganate and peroxide treatment have been studied (Li et al., 2007). Alkaline treatment is one of the most commonly used chemical treatments for reinforcing thermoplastics or thermosets. Alkaline treatment increases surface roughness and increases the amount of exposed cellulose on the fibre surface by removing a certain amount of the cellulosic content that covers the external surface of the fibre cell wall (Gurunathan et al., 2015) to produce better mechanical interlocking. Atiqah et al. (2014) treated kenaf fibre with 6% sodium hydroxide (NaOH) solution for 3 hours and recorded optimum results for flexural, tensile and impact strengths. Merlini et al. (2011) attempted alkaline treatment of short banana fibre with 10% NaOH solution for 1 hour and Panyasart et al. (2014) treated pineapple leaf fibre (PALF) with 5% NaOH solution and a 5-hour immersion period at room temperature. The results obtained from these alkaline treatments showed superior behaviour in mechanical properties compared to the untreated fibre. The focus of this study was to obtain the optimum alkaline treatment for banana fibre and its effect on the mechanical and chemical properties, surface topography, heat resistivity, as well as interfacial bonding with epoxy matrix.

EXPERIMENTAL DETAILS

Material

For this research investigation, loose pseudo stem banana fibre was obtained from local farmers in Kuala Langat, Malaysia. The common mechanical properties of banana fibre are shown in Table 1.

Table 1
Mechanical properties of banana fibre (Li et al., 2007; Merlini et al., 2011; Venkateshwaran et al., 2012)

Properties	Value
Density (g/cm^3)	1.35
Tensile Strength (MPa)	161.8 ± 11.8
Young's Modulus (GPa)	8.5 ± 0.9
Elongation at break (%)	2.0 ± 0.4
Diameter (μm)	120 ± 5.8

For the resin system, epoxy resin DM15 made of Bisphenol A was chosen as it is widely used in the automotive and aerospace industries and has moderate viscosity, low molecular weight and high mechanical strength. The resin consists of part A (base) and part B (hardener). The formulation of the resin is as shown below:

$$\text{DM15 (Part A)}: \text{DM15 (Part B)} = 5:1 \qquad \text{Equation 1}$$

Chemical Composition of Banana Fibre

Figure 1 shows strands of untreated banana fibre in loose form. The strands of fibre were cut into 400 mm length and separated into single filaments. The type of banana used cannot be revealed due to the confidential nature of the information.

Figure 1. Untreated banana fibre in loose form

However, the common chemical composition of banana fibre is cellulose, hemi-cellulose, lignin and other components as shown in Table 2.

Table 2
Chemical composition of banana fibre (Arthanarieswaran et al., 2014)

Fibre Type	Cellulose %	Hemi Cellulose %	Lignin %	Moisture Content %
Banana	62	18	5	11

Chemical Treatment

The strands of banana fibre were treated with sodium hydroxide (NaOH) of different combinations of concentration and duration, as presented in Table 3. In order to fill the gap in the findings of a previous study by Venkateshwaran et al. (2013) and to determine the optimum treatment from a smaller range that meets with industrial requirement, 4% (w/v) to 8% NaOH (w/v) concentration with a 2- and 4-hour immersion time was selected. In addition, this study used purely single strand pseudo stem banana fibre unlike previous research, which used banana/epoxy fibre. The NaOH concentration and immersion period are shown in Table 3.

Table 3
Banana fibre treatment using NaOH solution

Sample	NaOH %	Duration (Hours)
Untreated	0	0
4% 2h	4	2
4% 4h	4	4
6% 2h	6	2
6% 4h	6	4
8% 2h	8	2
8% 4h	8	4

The alkaline treatment method was adopted from a previous study by Mahjoub et al. (2014). In short, sodium hydroxide pellets were weighed according to the designated concentration; 40g NaOH was dissolved in 1 litre of distilled water to make 4% NaOH solution. Strands of banana fibre were cut into approximately 400-mm length and immersed in alkaline solution according to the period specified for each designated concentration. Each series was labelled with the group name and the type of treatment condition. Once the immersion time ended, the fibres were washed thoroughly with distilled water and pH paper was used to check the alkalinity of the treated strands of fibre. The pH paper was immersed in the solution in which the treated fibre strands were soaked and rinsed with distilled water. The fibres were washed until the neutral value of pH 7 was obtained. Finally, the strands were dried in an oven at 60°C for 24 hours.

Physical Analysis

The diameter of a single strand of banana fibre was measured using an Olympus SZX 12-CCD Digital Image Analyser at 20-times magnification. For each fibre strand, the diameter was

measured at five different points, and the average value was taken. For each combination of NaOH treatment, an average of 10 single fibre samples were obtained for recording.

Morphology Analysis

The surface microstructure of untreated and treated fibre strands was observed using a Hitachi S-3400N Scanning Electron Microscope (SEM) set at 5.0 kV and a magnification between 300 and 800 times.

Functional Chemical Group Analysis

Fourier Transformed Infrared Spectroscopy (FTIR) was performed on a Perkin Elmer Spectrum 100 instrument with a resolution setting of 4 cm^{-1} to identify the functional groups in untreated and treated fibres.

Mechanical Analysis

A single-fibre tensile test was conducted using an Instron 3365 Dual Column Table Top Universal Testing Systems with a 5 kN maximum load at a rate of 2 mm/min per ASTM D3039. A single strand of banana fibre was attached to a paper holder, as shown in Figure 2.

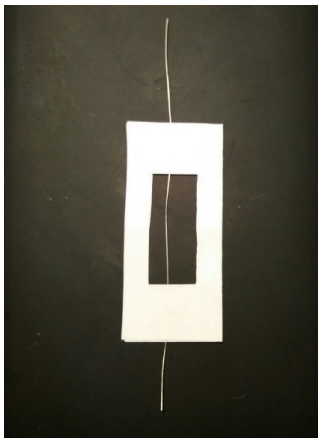

Figure 2. Banana fibre filament attached to a paper holder

The sample was then attached to the tensile machine gripper for testing. For each combination of NaOH treatment, the average based on 10 single fibre samples was recorded.

Adhesion Analysis

The micro-droplet test was conducted to evaluate the interfacial shear strength between the epoxy resin and the banana fibre using a technique adopted from a previous researcher, Dai et al. (2011). The resin was mixed according to a 5:1 ratio and then stirred for a few minutes. The resin was wetted onto the single banana fibre strand that had been fixed to a paper holder

to form a micro-droplet surrounding the fibre diameter due to surface tension. The embedded length of the micro-droplet was measured using a digital image analyser. The force required to de-bond the solid resin droplet from the fibre while the loading blade held the droplet was recorded. The schematic of the micro-droplet test is illustrated in Figure 3.

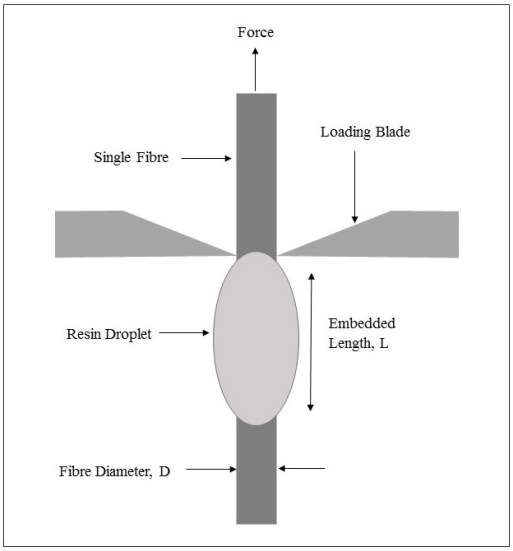

Figure 3. Schematic of the micro-droplet test

The figure shows vertical arrangement of the micro-droplet test. The top part of the fibre was clamped and pulled upwards while the bottom part of fibre strand was left unclamped. While the fibre was pulled upwards, the resin droplet was held by the loading blade, resisting the clamping force direction. The resisting force was recorded until the fibre breakage point. The interfacial shear strength (IFSS) was calculated based on:

$$\tau = \frac{F}{\pi DL} \qquad \text{Equation 2}$$

where F is the maximum load, D is the fibre diameter and L is the embedded length. The method was adopted from previous work by Dai et al. (2011). For each treatment condition, 10 samples were tested and recorded. The tensile test was carried out on the droplet samples using the Instron 3365 Dual Column Table Top Universal Testing Systems with 5 kN maximum load at a rate of 0.1 mm/min.

Thermal Stability Analysis

A thermogravimetric analysis (TGA) was performed to evaluate the thermal properties of banana fibre using the TA Instrument TGA Q500 with a temperature setting between 30°C

and 600°C at a rate of 10°C/min. Thermogravimetric analysis measures the amount and the changing rate of material weight as a function of time or temperature in a controlled atmosphere (Boopalan et al., 2013). TGA can also be used to characterise the effect of decomposition, oxidation and dehydration on the material's weight loss or gain.

RESULTS AND DISCUSSION

Physical Analysis

The average diameter (10 samples for each combination) for all treatment conditions is shown in Table 4.

Table 4
Average diameter of untreated and NaOH treated banana fibre strands

Sample	Average Diameter (mm)
Untreated	0.11 ± 0.01
4% 2h	0.10 ± 0.01
4% 4h	0.10 ± 0.006
6% 2h	0.11 ± 0.007
6% 4h	0.11 ± 0.008
8% 2h	0.09 ± 0.006
8% 4h	0.08 ± 0.005

In general, the average diameter showed slight variation in value across different concentrations of NaOH. The diameter of the untreated single fibre was 0.11 mm, which was close to the finding reported by another researcher, Idicula et al. (2005). The fibre diameter started showing reduction beyond 6% NaOH concentration. At the highest NaOH concentration of 8% and with a 4-hour immersion period, the diameter became reduced by 33% compared to the untreated fibre diameter. This could be due to the fact that higher alkaline concentration increases the delignification of banana fibre, thus reducing the diameter of the fibre, as reported by Gurunathan et al. (2015). The fibre delignification is illustrated in the FTIR Spectrography shown in this paper.

Morphology Analysis

The effect of alkaline treatment can be explained by considering SEM micrographs presented in Figures 4a, 4b and 4c.

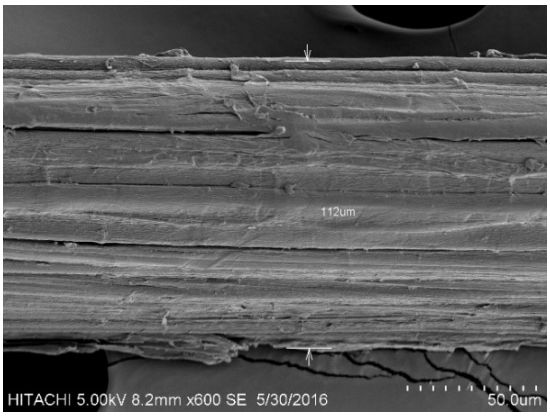

Figure 4a. SEM image of untreated banana fibre

Figure 4b. SEM image of banana fibre treated with 6% NaOH for 2 hours

Figure 4a shows considerable deposits of impurities and natural components on the surface of the untreated fibre. The deposits led to poor fibre-matrix interfacial adhesion by reducing the interfacial area of contact between the matrix and the fibre. This result was in agreement with previous work by Asumani et al. (2012). Figure 4b shows a rougher surface of treated banana fibre, as well as the removed impurities. Damage to the fibre is noticeable at a few locations. Removed impurities, as well as increased surface contact area, contribute to better surface interlocking between the fibre and epoxy resin, as also found by Mahjoub et al. (2014).

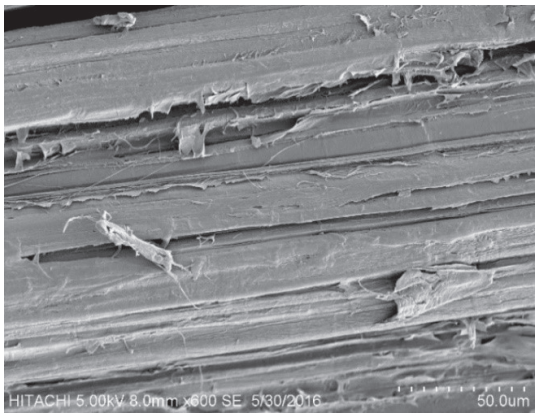

Figure 4c. SEM image of banana fibre treated with 8% NaOH 4 hours

However, as shown in Figure 4c, as the NaOH concentration reached 8%, the fibre damage was more severe, which in turn, reduced the strength of the banana fibre. This was in line with the tensile testing results as explained earlier.

Functional Chemical Group Analysis

The FTIR spectrum of untreated and treated banana fibres, which reflects the lignocellulosic components of the fibres, is illustrated in Figure 5.

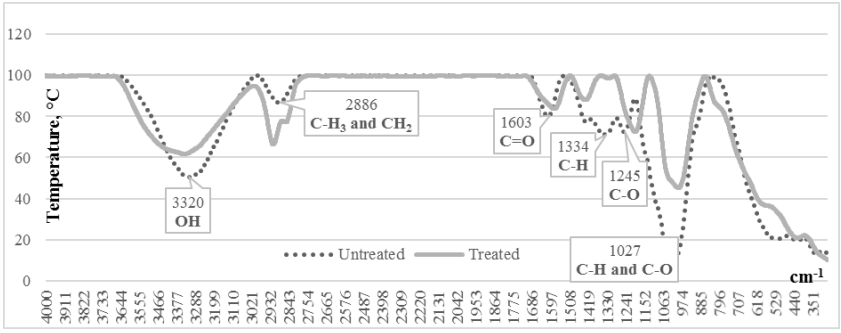

Figure 5. FTIR elements of treated and untreated fibres

The wide band at 3320 cm^{-1} indicates vibrational stretching of the hydroxyl group (OH) for natural fibre, as stated by Merlini et al. (2011). The treated fibre showed reduced hydroxyl stretching, which is responsible for the hydrophilic character of the fibre as also mentioned by Benítez et al. (2013) in their study. The sharp band at 2886 cm^{-1} demonstrates C-H stretching of methyl and methylene (alkene) groups. A similar pattern was reported by Corrales et al. (2007). The stretch at band 1603 cm^{-1} implies the presence of associated carbonyls (C=O) in the lignin, which was apparent in untreated banana fibre. This trend was also found by Asim et al. (2015).

The absorption reduced with alkaline treatment. The presence of C-H group with the frequency of vibration interval between 1535 cm^{-1} and 1330 cm^{-1} indicates the characteristic of methoxy radical in lignin, as also reported by Benítez et al. (2013). The reduced stretching within the region demonstrates that alkali treatment removed hemicelluloses and lignin from the surface of the natural fibre, as also reported for other vegetable fibre, as confirmed by Sgriccia et al. (2008). Meanwhile, the absorption at band 1245 cm^{-1} and 1027 cm^{-1} indicated C-O stretching of the acetyl group in hemicellulose, which reduced with alkaline treatment of NaOH. This result confirmed the finding by a previous team of researchers, Guimares et al. (2009).

Mechanical Analysis

Figure 4a presents the average ultimate tensile strength (UTS) and the tensile strain of the alkaline treated and untreated single banana fibre strands.

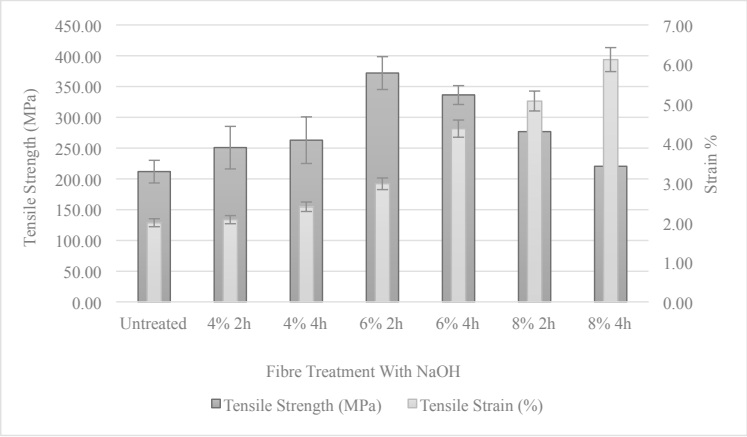

Figure 6a. Tensile strength and strain of untreated and treated single banana fibres

Based on the histogram, the tensile strength of the untreated fibre was 212 MPa, which is 30% higher compared to that reported in a previous study by Merlini et al. (2011). This could be due to various factors such as species, variety, type of soil used, plant age and weather factors, as mentioned by Rowell et al. (2000). The trend shows improved tensile properties for alkaline-treated banana fibre up to a certain point, followed by deteriorating tensile properties with increasing NaOH concentration and immersion time. The improvement in tensile properties was most noticeable for the fibre treated with 6% NaOH for 2 and 4 hours. The highest tensile strength recorded for a single strand of banana fibre was 371 MPa when treated with 6% NaOH with a 2-hour immersion time, which is equivalent to a 75% increment compared to that of untreated fibre. From the result, the tensile strength decreased with higher NaOH concentrations and longer immersion periods. This is because excessive delignification of natural fibre occurs in higher alkaline concentrations, resulting in weaker or damaged fibre. The result is in agreement with previous work by Asumani et al. (2012). This can be proven from the morphology as illustrated in Figure 4c.

Increased tensile strain was noticed for alkaline-treated samples. The increasing trend implies that alkaline treatment improved the fibre's ductility due to the removal of impurities, such as lignin and pectin, as found by a previous researcher, Gu (2009), hence resulting in more flexible fibre.

Figure 6b presents the tensile modulus of a single strand of banana fibre subjected to various alkaline treatments.

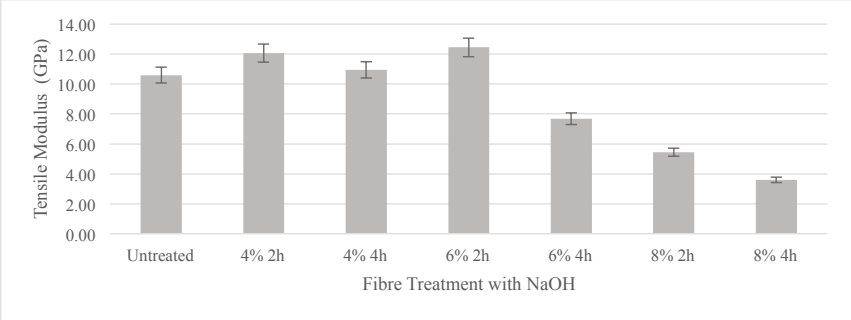

Figure 6b. Tensile modulus of untreated and treated single banana fibres

The variation in tensile modulus with alkaline concentration is similar to that of tensile strength, as presented in Figure 6a. In general, it can be observed that alkaline treatment improved the tensile modulus property of a single strand of banana fibre compared to that of untreated fibre. This was most noticeable for the fibre treated with 4% and 6% NaOH. The highest tensile modulus was 12.49 GPa, which was treated with 6% NaOH and given a 2-hour immersion time. The value was 53% higher than that of untreated fibre, with 8.09 GPa. Alkaline treatment may enhance fibre stiffness and expedite rearrangement of fibrils along the direction of the tension force after eliminating binding materials, as reported by Lopattananon et al. (2008). However, at 8% NaOH treatment, the tensile modulus reduced significantly, indicating decreased tensile strength due to the degradation of banana fibre, as also mentioned by Meon et al. (2012).

Adhesion Analysis

The interfacial shear strength is shown in Table 5.

Table 5
Interfacial shear strength (IFSS) for treated and untreated fibre

Sample	IFSS (MPa)
Untreated	1.12 ± 0.03
4% 2h	2.87 ± 0.02
4% 4h	3.74 ± 0.03
6% 2h	3.96 ± 0.03
6% 4h	3.85 ± 0.02
8% 2h	3.24 ± 0.02
8% 4h	2.51 ± 0.03

Looking at the trend, the interfacial shear stress increased with alkaline treatment to a certain value, then decreased with higher NaOH concentration. The interfacial shear strength of untreated fibre was 1.12 MPa, while 6% NaOH and a 2-hour immersion period treatment yielded the highest interfacial shear strength for treated fibre at 3.96 MPa. The same treatment condition also recorded the highest tensile strength for the treated single fibre strand, as illustrated in Figure 6a, which could be due to increased surface roughness, leading to better interlocking adhesion and greater amount of exposed cellulose on the fibre surface. The results obtained are in agreement with those obtained by previous researchers, Mostafa et al. (2015). This condition allowed better fibre wetting through increased number of possible reaction sites. Alkaline treatment also improved the surface adhesion properties of the fibre through the removal of natural and artificial impurities, and also created rougher surface topography. A similar trend was seen in Anuar et al. (2008). In this study, this was shown in the surface morphology result in Figure 4b and the FTIR spectroscopy results in Figure 5. Further treatment containing higher concentration of NaOH reduced the tensile strength due to excessive delignification, causing damaged and weaker fibre strands. The outcome was in accordance with that reported by Mishra et al. (2003).

Thermal Stability Analysis

Figure 7a illustrates the thermogravimetric analysis (TGA) of combustion residue for treated and untreated single banana fibre strands.

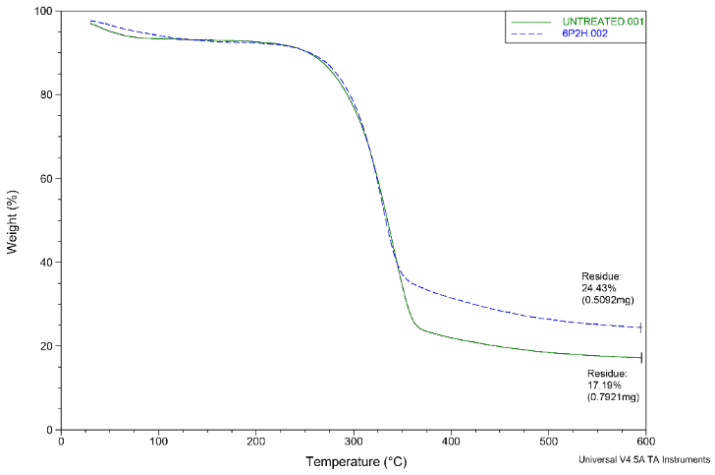

Figure 7a. TGA combustion residue for treated and untreated fibres

An initial weight loss of about 9.5% was recorded at a low temperature of 130°C due to the removal of moisture from untreated fibre. A similar pattern was found by Shih et al. (2014). The greatest weight loss occurred after 290°C with 75.61% weight reduction in untreated banana fibre, corresponding to component degradation and decomposition such as cellulose,

hemicellulose and lignin content in the fibre. The result is in line with the work of Zainudin et al. (2009). The starting temperature of weight loss for treated fibre shifted to a temperature beyond 290°C. This indicates that alkaline treatment resulted in higher thermal stability for the fibre, as also reported by Nopparut et al. (2016). For treated banana fibre, moisture was released from the fibre between 40°C and 175°C, and further degradation of cellulosic substances occurred between 175°C and 375°C. Nevertheless, subsequent decomposition requires elevated temperature as residues form and accumulate during degradation. The final residue degradation was recorded at 600°C. The higher percentage of residue for treated fibre (24.43%) showed that less fibre was burnt due to higher heat resistivity. This result is in line with the findings by Benítez et al. (2013).

The combustion temperature for treated and untreated fibre is shown in Figure 7b.

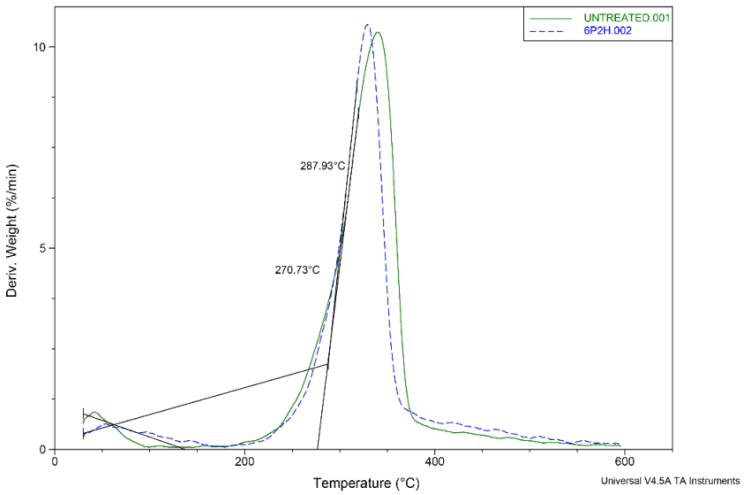

Figure 7b. TGA combustion temperature for treated and untreated fibres

The result showed that the decomposition temperature for untreated fibre at different weight-loss levels was lower than those of the alkaline-treated fibre. The burning of treated fibre occurred at 287.93°C, which was about 17°C higher than that required for untreated fibre. This indicated that NaOH treatment had slightly increased the fibre's heat resistivity due to the presence of inorganic materials such as sodium (Na) in the fibre, which requires a higher decomposition temperature, as also reported by Parker (2000).

CONCLUSION

This study showed that the optimum alkaline treatment for banana fibre is 6% NaOH concentration with a 2-hour immersion period, which resulted in 371 MPa tensile strength, 12.45 GPa tensile modulus and 3.96 MPa interfacial shear strength. The tensile strain increases with higher NaOH concentration. As the concentration increases beyond 6%, the mechanical properties of banana fibre deteriorate significantly. The removal of impurities and natural

deposits was evident through FTIR spectroscopy. The SEM micrographs showed that alkali treatment increased the surface roughness of the fibre, while the TGA results showed that the treatment enhanced the heat resistivity of the fibre.

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