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The Effect of Alkaline Treatment onto Physical, Thermal, Mechanical and Chemical Properties of Lemba Leaves Fibres as New Resources of Biomass

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ABSTRACT

The main purpose of this paper is to investigate the effect of alkaline treatment on the physical, thermal, mechanical and chemical properties of pristine lemba leaves fibres (LeLeFs). LeLeFs were treated with 6, 8, and 10 wt% sodium hydroxide (NaOH) solution at room temperature for 24 h. In order to determine the functional group presence after the alkaline treatment, LeLeFs were analyzed using Fourier Transform Infrared (FTIR) Spectroscopy. The density of LeLeFs treated with 10 wt% NaOH solution recorded the highest density with 1.168 g/cm^3. Morphology study showed that the diameter of fibre reduced with the increment of NaOH concentration. The removal of lignin and hemicellulose could be observed in the thermogravimetric analysis (TGA). Alkaline treatment enhanced the tensile properties of fibre and 10 wt% alkaline treated fibre resulted in the highest tensile strength, modulus and elongation of the fibre at 511.10 MPa, 11.76 GPa and 3.69% respectively. Chemical resistance analysis found that the treated fibre had better chemical resistance compared to untreated fibre. Therefore, it is substantiated that alkaline treatment affects the properties of LeLeF.

Keywords: Alkaline treatment, chemical resistance, density, FTIR, morphology, tensile, TGA

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INTRODUCTION

In recent years, the natural fibres have been regarded as great resources in replacing the synthetic polymers that widely used in various applications such as modern apparel, home furnishings, textile, medicine, aeronautics, building, construction and others. This is mainly due to raising awareness of the environment and the ever-depleting trend of petroleum supplies. Natural fibres are renewable, biodegradable and environmentally friendly. Moreover, they provide a lot of advantages over man-made fibres, including low-cost and abundantly available, relatively high specific strength and modulus, light weight, low density, less abrasiveness, and minimal health hazards (Hashim et al., 2017). Due to their abundant availability, natural fibres can be obtained or processed from a wide range of natural resources around the globe.

Malaysia, as a tropical country, has been blessed with an abundance amount of fibrous plants and agricultural resources. One of them is lemba (Figure 1) or scientifically known as *Curculigo latifolia*. Lemba is a member of the Hypoxidaceae family or known as flowering plants (Shaari, 2005). There are about 20 species of genus Curculigo that distributed in the tropical regions of Africa and Asia (Ranjbarfard et al., 2014). *Curculigo capitulata* and *Curculigo latifolia* are the familiar species that can be found in Malaysia and Borneo Island. Lemba grows about one meter tall and the blade elliptical leaf of 30-100 cm × 5-10 cm. It grows well in the hilly area and requires less exposure to sunlight, with abundant water supply (Shaari, 2005).

Figure 1. The lemba plant

Every part of this plant can be utilized to obtain its several advantages. The leaves have been used as wrapping, like banana leaves, by the people in Lahu, Thailand (Brink & Escobin, 2003). In Malaysia and Borneo, lemba leaves fibres (LeLeFs) have been used for making the rope, fishing net and twines (Farzinebrahimi et al., 2016). Meanwhile, in Japan, this plant also known to be a natural sweetener that gives a very sweet taste when its seeds or parts of the plant are chewed (Shaari, 2005). Not only that, high fever also can be treated by using the combination of leaves and flowers, while the concoction of flowers and roots has been used to treat stomachache and frequent urination (Shaari, 2005). The fruit has been recorded to be used to increase the appetite (Farzinebrahimi et al., 2016). It has been reported that lemba rhizome extract could be used to inhibit hepatitis B virus (Wiart & Wong, 2002).

Despite its useful applications, the knowledge of lemba is still restricted and limited to rural people only. There is an investigation on the potential use of LeLeF as a new material for textiles, where it found that LeLeF was much stronger compared to the cotton (Shaari, 2005). However, there is no scientific study in depth that explaining on the chemical and physical properties of LeLeF. Thus, it is worth further investigate the lemba leaves as a new resource of natural fibres in order to fully develop their potential application. Furthermore, newly identified fibres must be analyzed to identify their physical and chemical properties as this knowledge is important to evaluate the properties of the fibres efficiently.

Alkaline treatment is a method that commonly used by scientists and researchers on cellulosic fibres in order to produce high-quality fibres. This method will disperse bulk lignocellulosic materials into lignocellulosic fibres and remove lignin together with hemicellulose. Previously, the alkaline treated napier grass fibres have been studied for their chemical and physical properties. It shows that the alkali treatment enhanced the tensile properties of the fibres compared to those of the untreated fibres (Reddy et al., 2012). Besides, a few researches have been reported on the improvement of mechanical properties of pineapple leaf fibres after alkali treatment (Asim et al., 2016; Motaleb, 2018; Zin et al., 2018).

In this study, experiments were conducted to characterize the properties of pristine and treated LeLeF at different concentrations of alkaline. The characterization conducted includes determining the physical, thermal, mechanical and chemical properties. This investigation is very crucial to gain an established platform on the properties of LeLeF in determining the suitable potential application to be utilized. The removal of amorphous structure which are lignin and hemicellulose is very important as it can enhance the properties of the cellulose thus making it suitable to be used as a filler in polymer matrix and also can be used as a raw material for hydrogel preparation

MATERIALS AND METHODS

Materials

Lemba leaves used in this research were collected in the rural area at Jerantut, Pahang. Sodium hydroxide (NaOH, 98%), sodium carbonate (Na_2CO_3, 99.5%), ammonium hydroxide (NH_4OH,25% NH_3), toluene (99.5%), nitric acid (HNO_3, 69-72%), hydrochloric acid (HCl, 35-37%) and acetic acid (CH_3COOH, 99.8%) were purchased from Sigma Aldrich. All chemicals were analytical reagent grade and were used as received without further purifications.

Sample Preparation

LeLeFs were extracted from the leaves by using the hand-scrapping method. Firstly, lemba leaves were scrapped with a knife. The outer layers of the leaves were removed gently to ensure the fibres did not break and finally, LeLeFs were obtained.

LeLeFs were immersed in 6, 8 and 10 wt% NaOH solution at room temperature for 24 h in order to remove lignin and hemicellulose. After that, LeLeFs were washed several times with distilled water to remove any traces of alkali on the surface of the fibres. Then, they were neutralized with a diluted acetic acid solution and again were washed thoroughly with distilled water. Finally, the treated LeLeFs were air dried for 24 h.

Physical Properties

Determination of Fibre Yield. Ten samples of lemba leaves were taken randomly. The weight of the raw leaves (W_L) and the extracted fibres (W_F) were measured. The yield of fibre extracted from the leaves was calculated by using the following Equation 1:

$$\text{Yield} = \frac{W_F}{W_L} \times 100\% \quad (1)$$

Determination of Fibre Density. The density determination of LeLeF was carried out according to the Archimedes principle. The fibre was placed in a beaker containing 50 ml of water. The fibre was weighed before putting into the beaker. The total mass of water and fibre was recorded then the mass of water displaced by fibre was recorded. The weight to volume ratio yielded the density of fibre. The density of LeLeF before and after alkaline treatment was calculated by dividing the mass of LeLeF with the volume of water displaced.

Morphological Observation. The morphology of pristine and alkaline-treated LeLeF was examined using a scanning electron microscope (SEM) model JEOL JSM-6390LV.

All the surfaces of the solid fibres were sputtered with gold before the testing to avoid any electron charging on the image and poor image resolution.

Thermal Properties

Thermogravimetric Analysis. Thermogravimetric Analysis (TGA) of LeLeFs was conducted using a thermal gravimetric analyzer (TGA-Model: TGA7 Perkin Elmer Pyris). It was used to measure changes in the weight loss (mass) of the sample and determine the degradation of LeLeF. The fibres were chopped into micro size (about 1 mm) for the analysis. The samples were heated up from 25 to 500°C at a rate of 10°C/min in a nitrogen gas (60 ml/min). Each fibre was analyzed separately and overlapped for comparison.

Mechanical Properties

Evaluation of Tensile Properties. The tensile properties of LeLeF were performed using a Texture Analyzer according to ASTM D3822. The gauge length was kept constant at 50 mm and test speed 0.50 mm/s. Fibres length was maintained 50 mm for all samples. In each case, five specimens were tested and the average value was tabulated. Tensile strength (TS), Young's modulus (YM) and elongation at break (EB) were determined from the graphs plotted. The fundamental relationships applied to determine these properties are as shown in Equation 2, 3 and 4:

$$TS = \frac{F}{A_F} \tag{2}$$

$$EB = \frac{L_f - L_i}{L_i} \times 100\% \tag{3}$$

$$YM = \frac{S_S}{S_T} \tag{4}$$

Where F is force, A_F is average fibre area, L_i is initial length, L_F is final length, S_s is stress and S_T is strain

Chemical Properties

Evaluation of Chemical Resistance. Chemical resistance test was performed according to the method used by Gupta and Kumar (2012). The effect of some solvents such as toluene, the effect of some acids i.e.nitric acid, hydrochloric acid and the effect of some alkalies such as NaOH, Na_2CO_3, and NH_4OH were studied. For each case, five pre-weighed samples were dipped for 24 h in the respective chemical reagents. Then, the samples were

removed and washed with distilled water. They were dried at room temperature by using filter paper. The samples were then weighed and the percentage weight loss/gain (WL) was determined using the following Equation 5:

$$WL = \frac{W_f - W_i}{W_i} \times 100\% \quad (5)$$

where W_i is the initial weight of the fibre and W_f is the weight of the fibre after dipped in chemical reagents.

Functional Group Analysis. Fourier Transform Infrared (FTIR) spectra were acquired using IR Tracer-100 FTIR Spectrophotometer SHIMADZU. The LeLeFs before and after treatment were chopped into micro size (about 1 mm) and placed on the ATR crystal surface. The ATR-FTIR spectra were obtained with an accumulation of 40 scans and with a wavelength from 4000 to 800 cm^{-1}.

RESULTS AND DISCUSSION

Physical Properties

Determination of Fibre Yield. Initially, the fibre content in LeLeF based on fibre yield was analysed by using the hand-scraping method. Based on the data collection, it was found that the fibre yield was in a range of 6.73 to 13.21% with an average of 9.59%. The previous study on pineapple leaves stated that conventional methods like retting and scraping provide fibre yield of 1.8 % and 1.4% respectively (Kengkhetkit & Amornsakchai, 2012). As compared to the conventional method, the fibre content in the lemba leaves was higher than the pineapple leaves.

The appearance of the obtained LeLeF before and after alkaline treatment was then observed as shown in Figure 2. As seen in Figure 2, the pristine LeLeFs were naturally dark brownish colour. After alkaline treatment, the colour of LeLeFs appeared to be relatively lighter compared to the pristine one. This difference can be attributed to an increase in removal of lignin and hemicellulose content with the increasing of NaOH concentration. The result is in agreement with the FTIR result where the peaks at which associated to the lignin and hemicellulose decreased as the NaOH concentration increased. Hence, this shows that the changes in the colour of the fibres in Figure 2 were due to lignin and hemicellulose removal. Besides, previous study by Wunna et al. (2017) on the pre-treatment of sugarcane bagasse also proved that the amount of lignin removal increased as the concentration of NaOH increased. Fareez et al. (2018) reported that the colour of pineapple leaf fibre changed from brown to white after undergoing alkali treatment and bleaching.

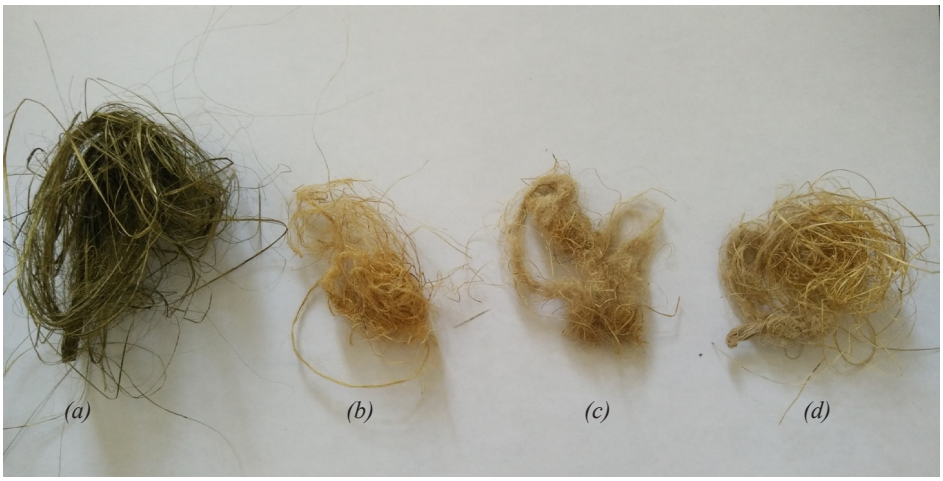

Figure 2. Pictures of a) The pristine LeLeF and LeLeF treated with b) 6 wt% NaOH, c) 8 wt% NaOH and d) 10 wt% NaOH

Determination of Fibre Density. Table 1 shows the densities of pristine LeLef and alkaline treated LeLef. From Table 1, it is clearly evident that the density values of LeLeF were increased gradually with an increase of the NaOH concentration. This is attributed to the densification of the cell wall as a result of removing non-cellulosic parts of fibre which were lignin, hemicellulose, pectin and others after increasing the concentration of sodium hydroxide (Sawpan et al., 2011). The density values mainly depended on the environment in which the plant was grown, extraction procedure of fibres, soil condition, and age of the plant (Hulle et al., 2015). The extraction process with higher NaOH concentration increased the density of the fibre as more of the amorphous part of the plant which contributed to low density was removed. Non-treated fibre consists of cellulosic and non-cellulosic part and the density of the fibre was the average density of all the components. Meanwhile, fibre treated with higher NaOH concentration resulted in higher purity of cellulose. High purity of cellulose indicates high crystallinity where the crystalline structure was packed in an orderly manner thus increase the density of the fibre. Our finding shows that the density of LeLeF was in the range of other natural fibres obtained from leaves as shown in Table 2.

Table 1

Density Properties of LeLeF

Condition of LeLeF	Pristine	6 wt% NaOH	8 wt% NaOH	10 wt% NaOH
Density (g/cm^3)	0.809±0.006	0.899±0.008	1.021±0.011	1.168±0.020

Table 2

Density of natural fibres

Natural Fibre	Density (g/cm^3)	Reference
Sisal	1.3	(Li et al., 2007)
Curaua	1.1	(Spinacé et al., 2009)
Pineapple Leaf	1.07	(Zin et al., 2018)

Morphological Observation. The morphologies of pristine, 6, 8, and 10 wt% treated LeLeFs were studied by using SEM (Figure 3). Analogous to other lignocellulosic reinforcing plants, LeLeF is a multicellular composite fibre. Each unit cell of fibre composed of cellulose microfibrils with different fibrillary orientations surrounded and cemented together with lignin and hemicellulose. Due to alkali treatment, NaOH disrupted the hydrogen bonding in the network structure of the fibre thus removing a certain amount of lignin, wax and oils, covering the external surface of the fibre cell wall and also depolymerized the cellulose (Li et al., 2007).

The effect of different concentrations of alkaline treatment on the LeLeFs was further investigated via SEM analysis at 300x magnification, as seen in Figure 3 (a-d). As expected, the morphological images of surfaces and cross-sectional of pristine LeLeF and treated

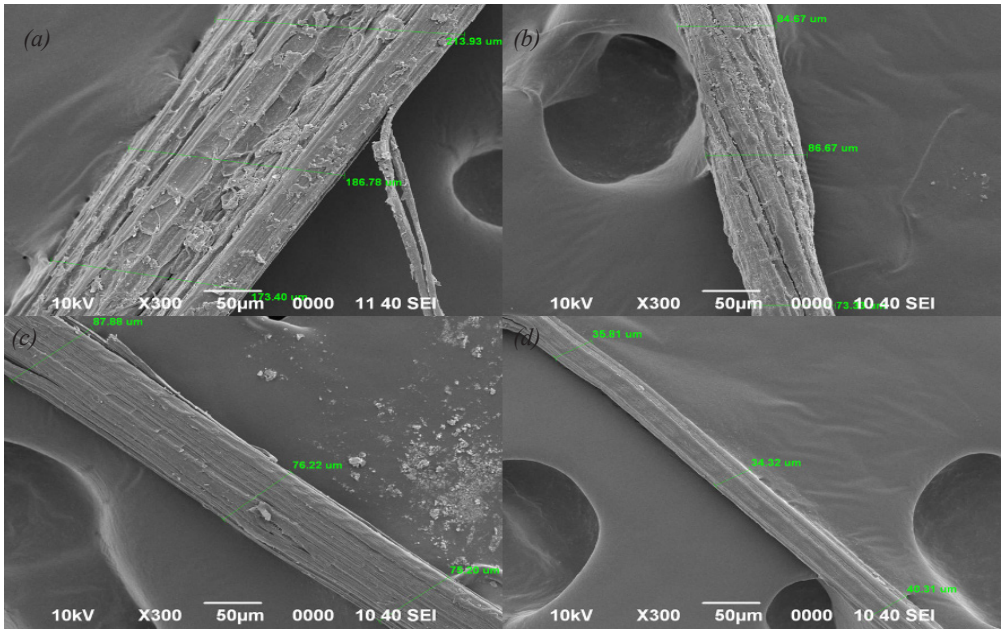

Figure 3. Scanning electron micrographs surface of a) pristine LeLeF and LeLeF treated with b) 6 wt% NaOH, c) 8 wt% NaOH and d) 10 wt% NaOH at magnification x300

LeLeF demonstrated significant different images in the terms of their level of smoothness, fibre binding and diameter (Li et al., 2007). As a benchmark, the pristine LeLeF fibres appeared to have many uplifted filaments that were randomly embedded and stacked together on the surface, as indicated in Figure 3a. Owing to the weak binding of lignin and hemicellulose (Ramadevi et al., 2012), they can be easily separated from the fibre surface by an external force (Mwaikambo et al., 2007).

After treatment, these filaments disappeared and the fibre surface became cleaner as well as a reduction in the fibre diameter from 191.37 μm to 36.81 μm. This suggests that alkaline treatment caused the removal of the surface impurities such as wax, hemicellulose, and other inorganic impurities, as seen from Figure 4c-d (George et al., 1997; Zannen et al., 2014).

Thermal Properties

Thermogravimetric Analysis. TGA was carried out to study the weight loss occurring on fibres at different temperatures (Figure 4). In general, the weight loss steps of natural fibres consists of three phases which are; i) moisture evaporation, ii) decomposition of components of hemicelluloses with lignin and iii) decomposition of cellulose. From the curve in Figure 4, the first stage of weight loss started from 37-100°C which is 12% for pristine and 5%, 4% and 3% for 6, 8 and 10 wt% NaOH respectively. This low-temperature weight loss occured due to the loss of moisture or water vaporization. The weight loss decreased for higher NaOH concentration treated LeLeF. This is in agreement with the FTIR result where the NaOH concentration increased, the broadness band of the hydroxyl group decreased which showed decreasing of OH group that can absorb water. This resulted in the decrease of the water absorption of LeLeF which means that the moisture was lost through alkali treatment thus decreasing the weight. Some other lignocellulosic fibres showed similar results for this low-temperature loss of weight like piassava fibre (5.18%) (D'Almeida et al., 2006), jute fibre (10.52%) (Das et al., 2000), flax (6.3%) and wheat straw (7.3%) (Hornsby et al., 1997).

Weight loss temperature ranged at about 250-420°C, indicating the degradation of cellulose. The cellulose degradation temperature of other lignocellulosic fibres like sugarcane was 350°C (Hoi & Martincigh, 2013), amazon piassava (361°C) (Rebelo et al., 2019), pineapple's crown, rice husks and cotton (257-390°C) (Prado & Spinacé, 2015). From this figure, it can be observed that the pristine LeLeF started to decompose at 286 °C while for 6, 8 and 10 wt% NaOH treated LeLeF, they started to decompose at 287°C, 291°C, and 293°C respectively. The treated fibres showed higher initial temperature of thermal decomposition which related to the partial removal of lignin and hemicellulose (Santos et al., 2013). The decomposition temperature increased as the concentration for the NaOH treatment increased. The thermal stability of fibres increased with increasing of

NaOH concentration thus requires higher temperature to decompose. Due to the complex structure of lignin, the fibre decomposition occured slowly within the whole temperature range (Rosa et al., 2010).

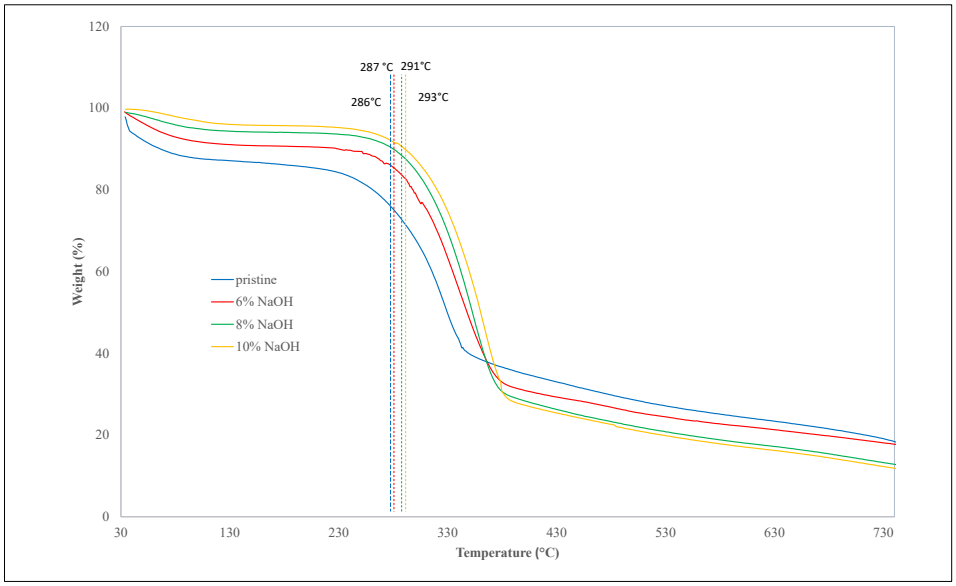

Figure 4. TGA analysis for pristine and treated LeLef at different concentrations

Mechanical Properties

Evaluation of Tensile Properties. The result in Table 3 shows that the maximum tensile strength of the LeLeF was achieved after 10 wt% NaOH treatment at 429.5 MPa. The tensile strength of LeLeF was significantly increased from pristine LeLeF, 6 wt% NaOH treated and 8 wt% NaOH treated which were recorded at about 14.5 MPa, 80.8 MPa and 121.7 MPa, respectively. This result proves that the treated fibres show higher tensile strength compared to pristine fibre. Changes of cellulose crystallinity during alkaline treatment causes increased in the tensile strength of LeLeF from pristine to 10 wt% NaOH treated. This result is in agreement with the density data where the remaining structure left was high crystallinity cellulose. The crystalline structure of cellulose was packed in an orderly manner and more compact thus increasing the density resulting in an increment of tensile strength as the brittle amorphous structure had been removed during the alkaline treatment. Ridzuan et al. (2015) also reported that the removal of the weak amorphous component and retaining only the crystalline component enhanced the strength of the fibre.

Table 3

Tensile properties of pristine LeLeF and different concentration of NaOH treated LeLeF

Fibre	Tensile Properties		
	Maximum stress (MPa)	*Young's modulus (GPa)*	*Elongation at break (%)*
Pristine	14.45	0.83	2.06
6 wt% NaOH	80.79	4.16	2.44
8 wt% NaOH	130.12	4.19	2.78
10 wt% NaOH	511.10	11.76	3.69

The result for the elongation at break in Table 3 shows a similar increasing trend as the tensile strength. However, the increment was insignificant as it was only about 2.4 to 3.7% for pristine LeLeF to 10 wt% NaOH treated LeLeF. As for the modulus of elasticity, the result in Table 3 shows that the highest modulus of elasticity was achieved with 10 wt% NaOH treatment at 11.8 GPa. Modulus of elasticity increases from pristine LeLeF and 6 wt% NaOH treated LeLeF at 0.83 GPa and 4.16 GPa, respectively. The modulus of elasticity for 8 wt% NaOH treated LeLeF was found to be the same as the modulus of elasticity for 6 wt% NaOH treated LeLeF. The results obtained for the tensile strength, elongation at break and modulus of elasticity exhibited a similar trend with research done on napier grass fibres which showed an increment from pristine napier grass fibre to 5% NaOH treated napier grass fibre for tensile strength, elongation at break and modulus of elasticity (Reddy et al., 2012). The dispersion of hemicellulose and lignin in the interfibrillar region of pristine fibres separated the cellulose chain from one another thus making it always in a state of constraint. After alkali treatment, the removal of hemicellulose eased the fibrils to rearrange themselves in a compact manner, resulting a closer packing of the cellulose chains and thus contributing to improve the strength and tensile properties of the fibres (Reddy et al., 2012).

Chemical Properties

Evaluation of Chemical Resistance. The resistance of the fibres to water and certain chemicals was studied (Figure 5). The acids (HCl and CH_3COOH), alkaline (NaOH) were used as a polar inorganic solvent while toluene was used as a non-polar organic solvent. The weight gain for all pristine fibres, 6, 8 and 10 wt% NaOH treated fibres with different chemicals are shown in Figure 5. From the figure, it is seen that the weight gained was observed in all cases. Therefore, it did not seem as if any erosion of fibres occurred as the fibres did not lose weight (Jayaramudu et al., 2015). The percentage of weight gain for the pristine fibres in all chemicals was higher compared to treated fibres. According to Reddy

et al. (2018), the weight gain of the fibre corresponded to a better interaction with the fluid which indicated poor chemical resistance while less weight gain corresponded to the less absorption of solvent thus indicated better chemical resistance. This shows that fibres treated at highest NaOH concentration had the highest chemical resistance. Higher NaOH removed more of the non-cellulosic part of fibres thus increased the crystallinity structure of the cellulose. The crystalline structure of cellulose was built up of denser packing structure thus possesing higher resistance to chemical as indicated by the percentage of weight gained.

The percentage weight gain of all the polar solvents were almost the same while for the non-polar solvent the percentage weight gain was low compare to polar solvent. This indicates that cellulose was more susceptible to polar solvents compared to non-polar solvents because of OH interaction.

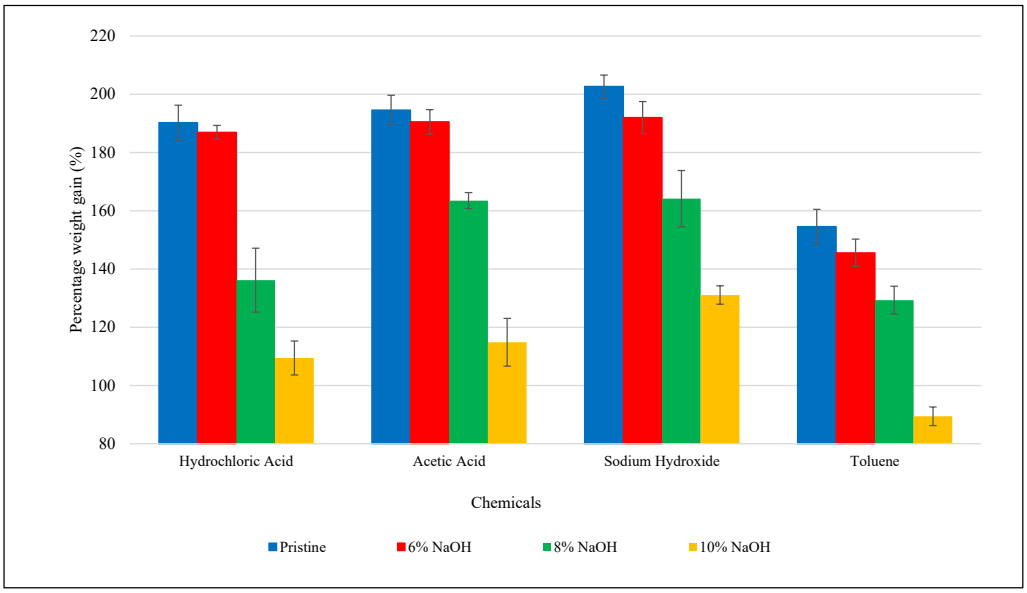

Figure 5. Chemical resistance properties of pristine, 6, 8, and 10 wt% alkaline treated LeLeF

Functional Group Analysis. The FTIR analysis was employed to investigate remarkable differences in the functional groups of pristine LeLeF and LeLeF after the NaOH treatments (Figure 6). Figure 6 shows that the spectra obtained have a similar shape to those of other types of plant fibres (Guimarães et al., 2009; Hoi & Martincigh, 2013; Santos et al., 2013). The appearance of a broad absorption band was observed in the region of 3117–3480 cm^{-1}, with a peak at 3334 cm^{-1} which corresponded to the hydroxyl groups of cellulose (Garside & Wyeth, 2006; Neto et al., 2013). The peaksat 2918 cm^{-1} and 2848 cm^{-1} were attributed

to the asymmetric stretching of CH and CH_2 in cellulose and hemicellulose (Alvarez & Vázquez, 2006; Fan et al., 2012; Rosa et al., 2010). Meanwhile, the peak at 1713 cm^{-1} was associated with the C=O stretching vibration linkage of the ester group in hemicellulose (Guimarães et al., 2009). The peak at 1578 cm^{-1} was corresponded to the C=C stretching of the aromatic ring, characteristic of the lignin (Guimarães et al., 2009) while the peak at 1240 cm^{-1} which only present in the spectra of pristine LeLeF was represented C-O stretch of acetyl group of lignin (Sgriccia et al., 2008).

In comparison to the pristine LeLeF, a reduction in hydroxyl (-OH) stretching intensity could be observed as LeLeF was treated with a higher concentration of NaOH. This is probably due to the free hydroxyl group participated in the chemical reaction (Samal & Ray, 1997). The intensity of peak at 2918 cm^{-1}, 2848 cm^{-1} and 1713 cm^{-1} decreased as LeLeF was treated with a higher concentration of alkali solution. This clearly indicates that the amount of hemicellulose on the LeLeF was successfully reduced by the treatment with NaOH. In placing more emphasis, Sgriccia et al. (2008) reported that the peak 1730 cm^{-1} of hemp fibre that attributed to the C=O stretching of the acetyl group of hemicellulose was not present in the alkali treated samples as the removal of the hemicellulose caused this peak to disappear. After the alkaline treatment, the reducing peak occurred at 1578 $cm^{-1}$and no distinct peak could be observed at the peak 1240 cm^{-1}, indicating that the removal of lignin was achieved.

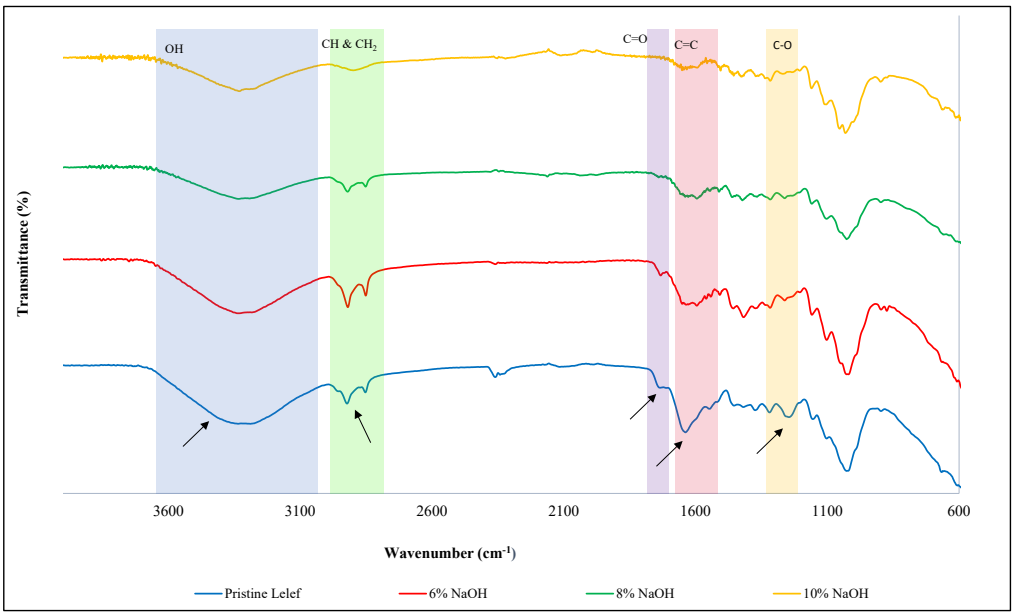

Figure 6. FTIR spectra of pristine and treated LeLeF with different concentration of NaOH

CONCLUSIONS

FTIR analysis showed that the higher the NaOH concentration, the more lignin and hemicellulose removed from the fibre as the intensity of peak associated with hemicellulose and lignin were reduced upon increase of NaOH. The density of LeLeF increased with the increment of the NaOH concentration. Higher NaOH concentration removes more surface impurities such as wax, hemicellulose and lignin resulted in the decreasing of fibre diameter from 191.37 μm to 36.81 μm. This study shows that as the NaOH concentration increased the tensile properties of LeLeF also increased up to 3400% (from 14.45 to 511.10 MPa). The highest tensile strength, elongation and modulus were achieved at 10 wt% NaOH treated LeLeF. TGA results showed that the decomposition temperature of fibre increased from 286 °C to 293 °C as the fibre was treated with higher NaOH concentration. Thus, alkaline treatment enhanced the thermal stability of the fibre. Chemical resistance analysis showed that alkaline treated LeLeF had better chemical resistance compared to pristine LeLeF where the percentage weight gain of 10 wt% NaOH treated for all polar solvent were less than 130% while the percentage weight gain of pristine LeLeF for all polar solvents were more than 180%.

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