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Characterization of Musaceaeand Saccharum Officinarum Cellulose Fibers for Composite Application

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Abstract

This paper presents experimental results on the effect of alkalis such as KOH and NaOH on changes in the morphological, physical, heat resistance, chemical, and tensile properties of the Musaceae and Saccharum Officinarum rods fibers. Modified fibers were made using a chemical solution of NaOH and KOH with a concentration of 8% for 2 hours. Physical, chemical, thermal and tensile properties were characterized by density, chemical composition, tensile, and thermogravimetric tests. The surface structure of the fibers has also been analyzed by SEM. The results show that both fibers have superior properties in terms of tensile strength, and thermal resistance after chemical treatment; because hemicellulose and lignin were reduced from the fibers. The superiority of Musaceae fibers is obtained after chemical treatment with KOH; instead, the best properties of Saccharum fiber were obtained after alkali treatment with NaOH. The SEM image also shows that the fiber surface becomes coarse and fibrils. The results show that the effect of alkalis provides a thorough change in terms of properties and morphologies on different fibers.

Keywords: Musaceae fiber, Saccharum Officinarum fiber, FTIR, tensile strength, thermal properties, and SEM.

1. Introduction

Cellulose-based natural fiber products have become a production motto in today's industrial and academic sectors. Strengthening thermoset resins using natural fibers has advantages such as lightness, strength and high modulus of elasticity in materials[1–3] for composite applications. The properties of natural fibers depend on the habitat (where the plant grows), the age of the plant, the type of plant and the method of extraction. Natural fibers can be obtained from stems, leaves, and fruit[4]. The presence of a large number of hydroxyl groups and impurities causes natural fibers to be less desirable for polymer composite.

Modification of natural fiber surfaces such as alkali chemistry, bleaching, acetylation has been used to reduce hydrophilic properties and improve physical and the tensile strength properties of natural fibers[5–9]. Moreover, the use of stirrer, latex, varnish,and paint has been known to reduce the tensile strength of Red Banana, Nendran, Rasthaly, Morris and Poovan fibers[10]. Of the various types of alkali chemicals that have been used in natural fibers, sodium

hydroxide (NaOH) is the most common chemical used to clean the surface of the fiber and change the structure of the original cellulose I to cellulose II. NaOH with percentage from 0.5% to 8% were able to improved the tensile strength and thermal properties of the cornhusk fibers [5,11]. Likewise, NaOH treatment with 5% to 15% concentration were proven to increase ductility and elongation of Borassus fruit fibers[12]. Tensile strength of hemp fiber increased up to 65% after treated with 0,5 % NaOH solution for 30 minutes[13].

Beside NaOH treatment, potassium hydroxide (KOH) treatment had been used by some studies to improve properties of natural fibers. A solution of 5 mol/l NaOH and KOH has been known to increase lyocell fiber fibrillation[14]. The flexural strength of activated carbonfrom bamboo stem treated with KOH of the epoxy nanocomposite is higher than the activated carbon treated with phosphoric acid[15]. These previous studies have shown that investigations related to the characterization of natural fibers using NaOH have been widely reported;on the contrary, studies relating to the characterization of natural fibers using KOH solutions are still very limited. Taking into account the potential of Musaceae, and Saccharum Officinarum fibers that are abundant, inexpensive, and environmentally friendly, it needs an effort to improve the function as a reinforce in composites manufacture.

Therefore, this study aims to provide a more detailed an understanding of the morphological, physical, thermal and mechanical properties of natural fibers of Musaceae and Saccharum officinarum and their modifications. These natural fiber surfaces were modified by using Sodium hydroxide (NaOH) and potassium hydroxide (KOH). Fourier transform infrared (FTIR) spectroscopy, thermogravimetry (TGA), chemistry and tensile tests have been carried out to characterize both types of fibers. In addition, changes in the surface morphology of both fibers have also been analyzed by Scanning electronic microscopy (SEM).

2. Material and methods

2.1 *Materials*

Musaceae (PF) and Saccharum officinarum stems (BF) were obtained from crops grown in the area of West Lombok, West Nusa Tenggara, Indonesia. The outer surfaces of both types of stems were each cut with length 41.1–43.4 cm. Potassium hydroxide (KOH) and Sodium hydroxide (NaOH) solutions were used to modify the fiber surface.

2.2 *Fiber bundles extraction*

Both the skin (PF and BF) was immersed in fresh water for fourteen days for microbiological degradation of bacteria[5]. Theywere washed with fresh waterand then combed with a wooden brush to remove residual particles from the surface of cellulose fibers and retained the same fiber. Fibers rinsed with fresh water and dried in an oven at 105 ^{o}C for 4 hours. It is known that the lengths of the PF and BF fibers were 41.1–42.6 cm and 18.04 – 18.8 cm, respectively.

2.3 *Alkaline treatment of fibers*

Both types of fibers were ready to be immersed into a chemical solution of KOH and NaOH with each concentration of 8% for 2 hours at 31 ^{o}C. The reaction scheme is given below[5].

$$\text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{Fiber} - \text{O}- \text{Na}^{+} + \text{H}_2\text{O}$$

$$\text{Fiber} - \text{OH} + \text{KOH} \rightarrow \text{Fiber} - \text{O}- \text{K}^{+} + \text{H}_2\text{O}$$

Then, the fiberswere cleaned with mineral water to remove the sticky chemical solution on the fiber surface, and dried in an ovenfor 4 hours at 105^{o}C to remove moisture content. Thus, thefibers were stored in the dry plastic box with humidity of 30%. Musaceae and Saccharum officinarum plants and thefibers can be seen in **Figs. 1(a–d).**

Cellulose, hemicellulose, and lignin content of fiber samples were determined by the standard TAPPI method. Cellulose and lignin content of the fibers were carried out according to TAPPI T203–om93 and T222 om88 respectively[16,5].

Nomenclature	
BF–Raw	Saccharum officinarum L. Stem raw fibers
BF–NaOH	Saccharum officinarum L Stem fibers treated NaOH
BF–KOH	Saccharum officinarum L fibers treated KOH
PF–Raw	Musaceae L Stem raw fibers
PF–NaOH	Musaceae L Stem fibers treated NaOH
PF–KOH	Musaceae L. Stem fibers treated KOH

Figure 1. Photographic images of fibers (a) Musaceae stem (b) Saccharum officinarum stem.

2.5. Physical properties of fibers

2.5.1 *Density*

The method of water movement was used to find raw and treated fiber density. The amount of weighed fiber was completely immersed in water and volumetric displacement was observed. The weight to volume ratio yielded a density value.

2.5.2. *Moisture content*

The quantity of weighed fiber was placed in the oven at a temperature range of 104 ± 2 °C for 4 hours. The fiber weight taken from the oven was measured and the difference in weight takes into account the moisture content present in the fiber.

2.6 *Fourier transform infrared (FTIR) spectroscopy*

The presence of free functional groups in fibers was determined by FTIR. Perkin Elmer Spectrum Fourier transforms infrared spectrometer instrument (model Frontier Spectrum) in a spectral range of 4000–450 (cm^{-1}) with 4 cm^{-1} resolution and a scan rate of 32 scans per minute. The chopped fiber samples were grounded and then mixed with KBr powder and pelletized.

2.7 *Tensile strength properties*

Elongation and tensile strength were determined according to ASTM D–3379–7 [5]. Each sample of fibers was tested using INSTRON 1390 at the constantcrosshead speed of 2.5 mm/min and load cell 10 kN under ambient temperature conditions and humidity of 64%.

2.8 *Thermogravimetric*

The thermogravimetric analyses (TGA) of raw and chemical treated fibers samples were carried out using TGA Q500, TA instrument was under a nitrogen atmosphere. The samples were scanned from 25 °C to 600 °C, at a heating rate of 10 °C/min.

2.9 *Scanning electron microscope (SEM)*

The surface morphologies of PF and BF raws and treated fibers were observed by using an FEI model (Inspect–S50–type) scanning electron microscopy operating at 10 kV. The samples were coated with gold sputtered for 5 min before their micrographs were recorded.

3. Result and discussion

3.1. *Chemical Properties of fibers*

The chemical composition of various fibers before and after alkali treatment is shown in **Table 1**. Under alkaline chemical treatment, the cellulose content in the PF–NaOH,and PF–KOH samples increases, whereas, the hemicellulose and lignin content in the fiber decreases. Similar results were also found in BF–NaOH samples. The loss of non–cellulose material in fibers leads to an increase in mechanical properties and interfacial bonds between fiber/resin in composite applications[5].

For the BF–KOH sample, in addition to reduced hemicellulose and lignin in the fiber, the cellulose content in the fiber is also reduced; this is because the concentration of 8% of KOH used in fiber is very high; consequently, the structure of the BF–KOH fiber becomes damaged due to excessive reaction of hemicellulose and lignin. The reaction is given:

$$\text{Fiber} - OH + KOH \longrightarrow \text{Fiber} - O - K^{+} + H_2O$$

Table 1 Amount of fiber constituents (weight %) exhibits in the different treated fibers.

Fiber types	Cellulose (%)	Hemicellulose (%)	Lignin (%)
BF–Raw	57,3	0.7	31.1
BF–NaOH	54.5	0.57	28.3
BF–KOH	53.7	0.55	27.7
PF–Raw	63.2	10.3	5.3

PF–NaOH	66.6	8.6	4.5
PF–KOH	67.52	8.4	4.1

3.2. *Physical properties of fibers*

3.2.1. *Density*

Alkaline chemical treatment increases the density values of PF and BF fibers. The results are shown in **Table 2**. It can be seen that the density values of PF–raw and BF–raw samples are lower than those of the fibers treated with KOH and NaOH. The low fibers densities exhibit that the PF dan BF fibers had hollow structures(5), as confirmed by the SEM images in the next section. Hence, raw and treated fibers can be used as a reinforcement in making lightweight composite structures. In addition, biodegradability is an additional feature for the use of this fiber in composites.

3.2.2. *Moisture content*

Table 2 shows the water content of both types of fiber before and after treated with NaOH and KOH. These results indicate that after alkali treatment, the moisture content of the two types of fibers is reduced; due to reduced hemicellulose content of fiber (see **Table 1**). Furthermore, the low moisture content of the chemically treated fibers can provide the interface strength of the polymer matrix when fibers are used as reinforcement in polymer composites.

Table 2 Physical properties of raw and treated fibers.

Fiber types	Diameter (mm)	Moisture Content (%)	Density (g/cm^3)
BF–Raw	0.24 ± 0.02	12.96	0.147
BF–NaOH	0.18 ± 0.02	11.60	0.236
BF–KOH	0.17 ± 0.02	11.78	0.225
PF–Raw	0.16 ± 0.04	10.4	1.423
PF–NaOH	0.09 ± 0.03	10.3	1.544
PF–KOH	0.08 ± 0.02	9.87	1.565

3.3. *Tensile strength properties*

Figure 2a shows that, after alkaline treatment, the tensile strength of the fiber is higher than that of raw fiber. It has been found that the average tensile strength of PF–KOH, PF–raw and PF–NaOH fibers were 889,557 MPa, 691.1447 MPa, and 593,287 MPa, respectively. The average tensile strength of PF–KOH fiber samples is higher than other fiber samples studied. The increased tensile strength of PF–KOH sample is estimated to occur because the fiber diameter is smaller than the PF–NaOH, and PF–raw samples (see **Table 2**), so that the aspect ratio of the fiber becomes high with a rough fiber surface; so that the tensile strength of the fiber becomes high.

In contrast, BF-raw gives the highest tensile strength (263.40 MPa) compared to BF-NaOH (255.39 MPa) and BF-KOH (187.77 MPa) (**Fig. 2c**), it is due to after NaOH and KOH treatment, the cell wall in the fibers to be damaged due to excessive extraction of hemicellulose and lignin so that the tensile strength of BF-KOH and PF-NaOH samples becomes decrease.

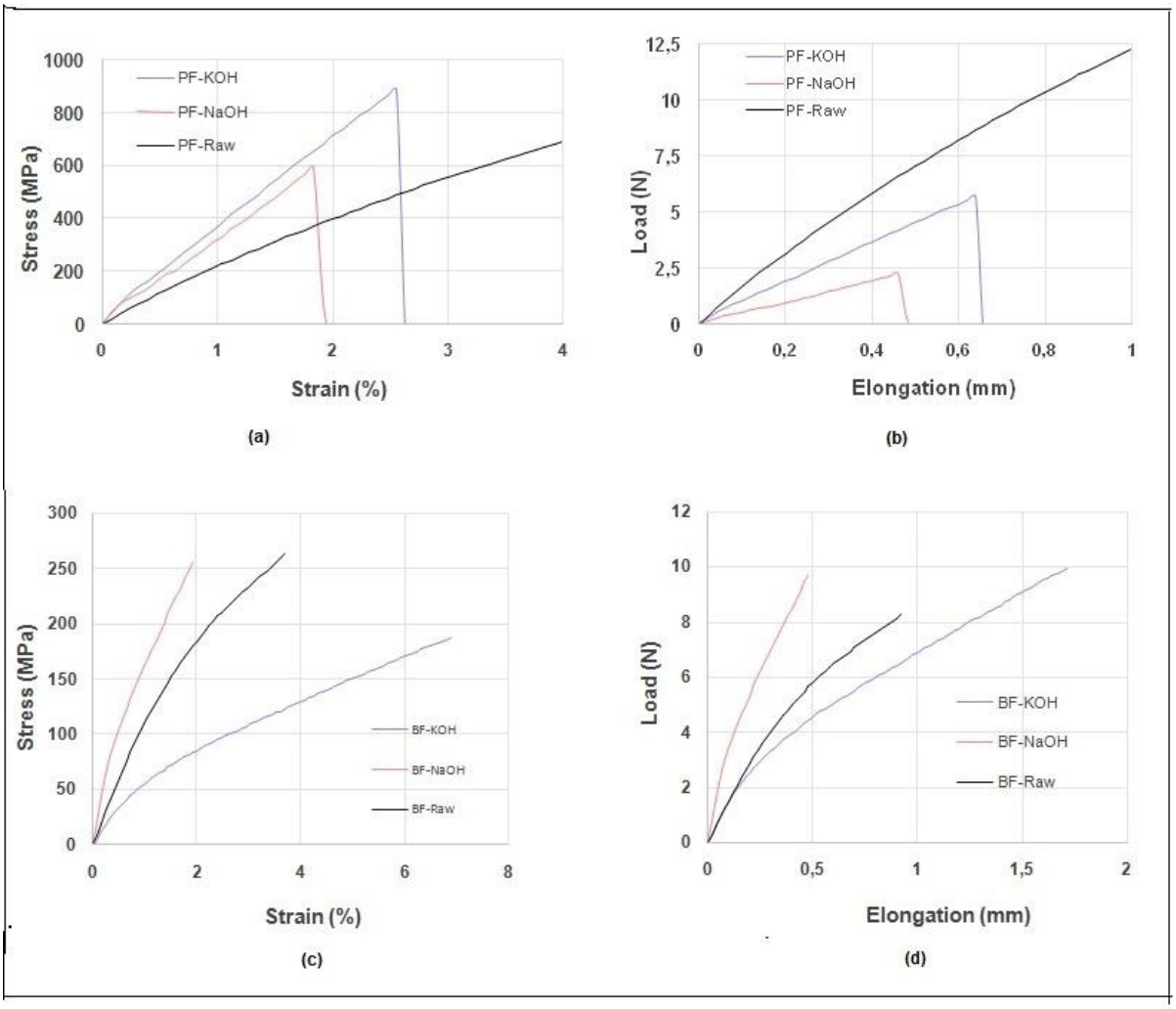


Figure 2 a. Stress vs strain of Musaceae fiber, b. Load vs elongationof Musaceae fiber, c. Stress vs strain of Saccharum Officinarum fiber and d. Load vs elongation of Saccharum Officinarum fiber.

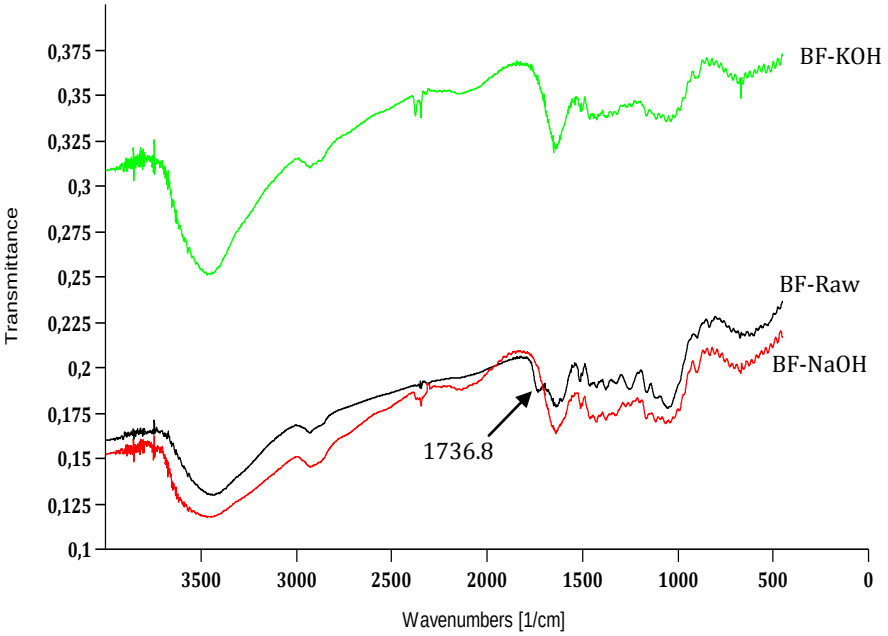
It is known that (see **Figs.2b** and 2**d**) the maximum load of PF–raw, PF–KOH, and PF–NaOH samples are 0.99 mm, 0.64 mm, and 0.46 mm, respectively and BF–KOH, BF–raw, and BF–NaOH are 1.72mm, 0.92 mm, and 0.48 mm, respectively.

Furthermore, different pattern of **Fig. 2a** and **2c** indicate that fiber types and alkali treatment at the surface also different. The largest differences can be observed through surface morphology, and chemical composition. The same reasons also to answer why **Fig. 2b** and **2d** have different pattern. These results have been confirmed from the results of chemical composition and SEM.

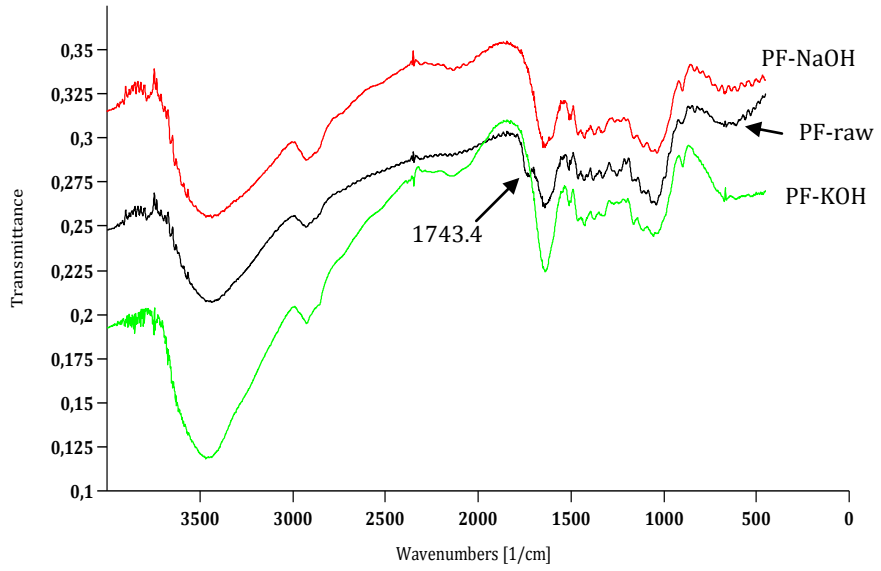
3.4. *FTIR analysis*

FTIR fiber analysis results are shown in **Figs 3a** and **3b**. The FTIR spectra exhibited eight peaks offiber. For BF-raw and PF-raw samples (**Figs 3a** and **3b**) were found another band at the peaks 1736.8 cm^{-1} and 1743 cm^{-1} respectively corresponding to hemicellulose[17]. The intensity of the hemicelluloses band decreased after NaOH and KOH treatment, indicating a

reduction of hemicelluloses substance. Possible band positions and tasks are given in **Table 4**. As shown in **Table 4**, bands near 3400 and 2930 cm^{-1} correspond to α–cellulose, while the remaining bands attributed to lignin. The chemical treatments cause the intensities of the bands corresponding to α–cellulose increased. For BF-NaOH and PF-KOH samples were found have higher intensity values than the other samples studied (**Figs 3a** and **3b**), indicating α–cellulose decreased.



(a)



(b)

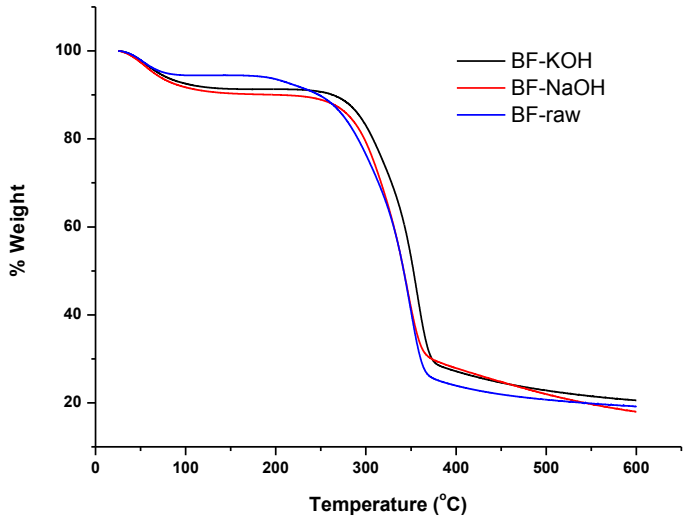
Figure 3. FTIR, (a). Saccharum Officinarum fiber, (b). Musaceae fiber.

Table 4. Infrared transmittance peaks in the raw and treated fibers.

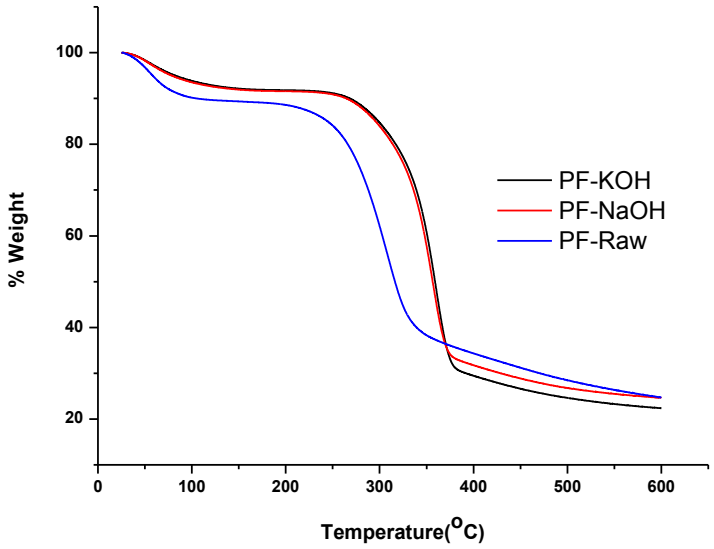
Wave number (cm^{-1})						Assignments
BF–Raw	BF–NaOH	BF–KOH	PF–Raw	PF–NaOH	PF–KOH	
3440	3444	3411	3450	3437	3453	O–H stretching vibrations of α–cellulose
2927	2927	2930	2921	2930	2917	Alkyl C–H stretching
2345	2348	2352	2338	2342	2345	C ≡C alkynes group
1736	–	–	1743	–	–	CO stretching of Hemicellulose
1641	1638	1641	1641	1634	1634	CO stretching of lignin C= C aromatic stretching with strong conjugated C–C bond
1457	1457	1457	1430	1424	1430	C–H bending
1049	1046	1052	1046	1032	1032	Symmetric CO stretching of lignin
667	671	664	891	904	888	Saline content

3.5. *Thermogravimetricanalysis*

Figs. 4a and **4b** show the TGA thermographs of raw and chemically alkalizedfiber samples. The alkalized NaOH and KOH fibers showed higher decomposition temperatures compared to the raw fibers. Comparing with NaOH treated fibers, the thermal resistance of sample PF–KOH higher than PF–NaOH. Instead, for sample BF–NaOH better than KOH treated fibers. For themain fiber decomposition region 250–310°C, the alkalized fibers had lessweight loss than the raw fibers. These results indicate that alkali treatments removed portions of hemicellulose and lignin constituents from the fiber. The fiber decomposition region 200°C to 500°C corresponds to the degradation of lignin[18]. Due to this, the decomposition process mainly occurred on the cellulose which in turn increased the overall degradation temperature of the treated fibers.



(a)



(b)

Fig. 4. TGA, (a). Saccharum Officinarum (BF) fiber, (b). Musaceae (PF) fiber.

3.6. *Fiber morphology analysis*

The SEM images of the surface of the raw and the alkali–treated fibers are shown in **Fig. 5**. The microscopic image showed that the surface of both of fibers (BF–raw and PF–raw) were lots of shallow grooves, fiber cell and other impurities (see in F**igs. 5a** and **5d**) and a number of lumen in the fiber bundles (see in **Figs. 6a** and **6d**). From these micrographs reveal a white layer on the raw fibers, which may be due to the hemicellulose. On alkali treatment (**Figs. 5**(**b, c, e,** and **f**), the white layer is found to decrease and the surface of the fibers sample is found become rougher and fibril; due to the loss of hemicellulose in the fiber surface. However, SEM images also exhibited that for BF-KOH (**Fig. 5c**) and PF-NaOH (**Fig. 5e**) a number of the cell walls in the fiber were damaged after chemical treatment, it is resulting in the strength of fiber todecreased. Further, the large number of lumen diameter in the KOH treated fibers and NaOH treated fibers become smaller after alkali treatment (**Figs 6**(**b, c, e,**and **f**)); due to to the reduction in the hemicellulose.

4. Conclusion

Both types of Musaceae and Saccharum fibers and their modifications have been characterized and compared. Experimental results and analysis show that different natural fibers have different properties. Alkaline treatments with different chemical solutions cause changes in different properties of the fibers. After being chemically treated, samples of PF–KOH and BF–NaOH show the best properties in terms of tensile strength and heat resistance; because hemicellulose and lignin from fiber were reduced. Compared to Saccharum fiber, Musaceae fibers exhibit superior properties. Overall, the properties of these two types of fibers have enormous potential to be developed again as fillers of polymer composites.

Conflict of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

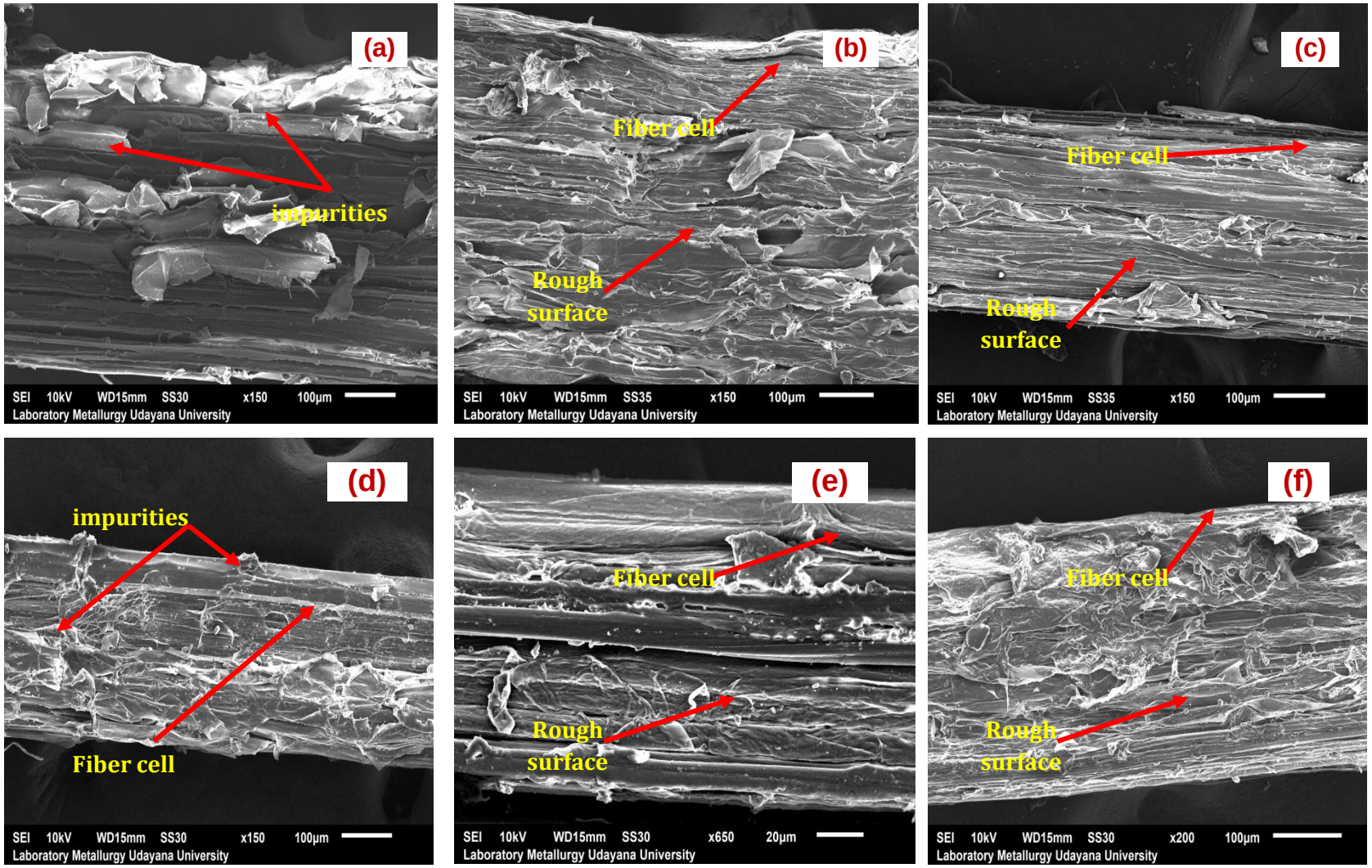


Fig. 5. SEM of the surfaces of fibers: (a) BF–Raw, (b) BF– NaOH, (c) BF– KOH, (d) PF–Raw, (e) PF–NaOH, (f)PF–KOH.

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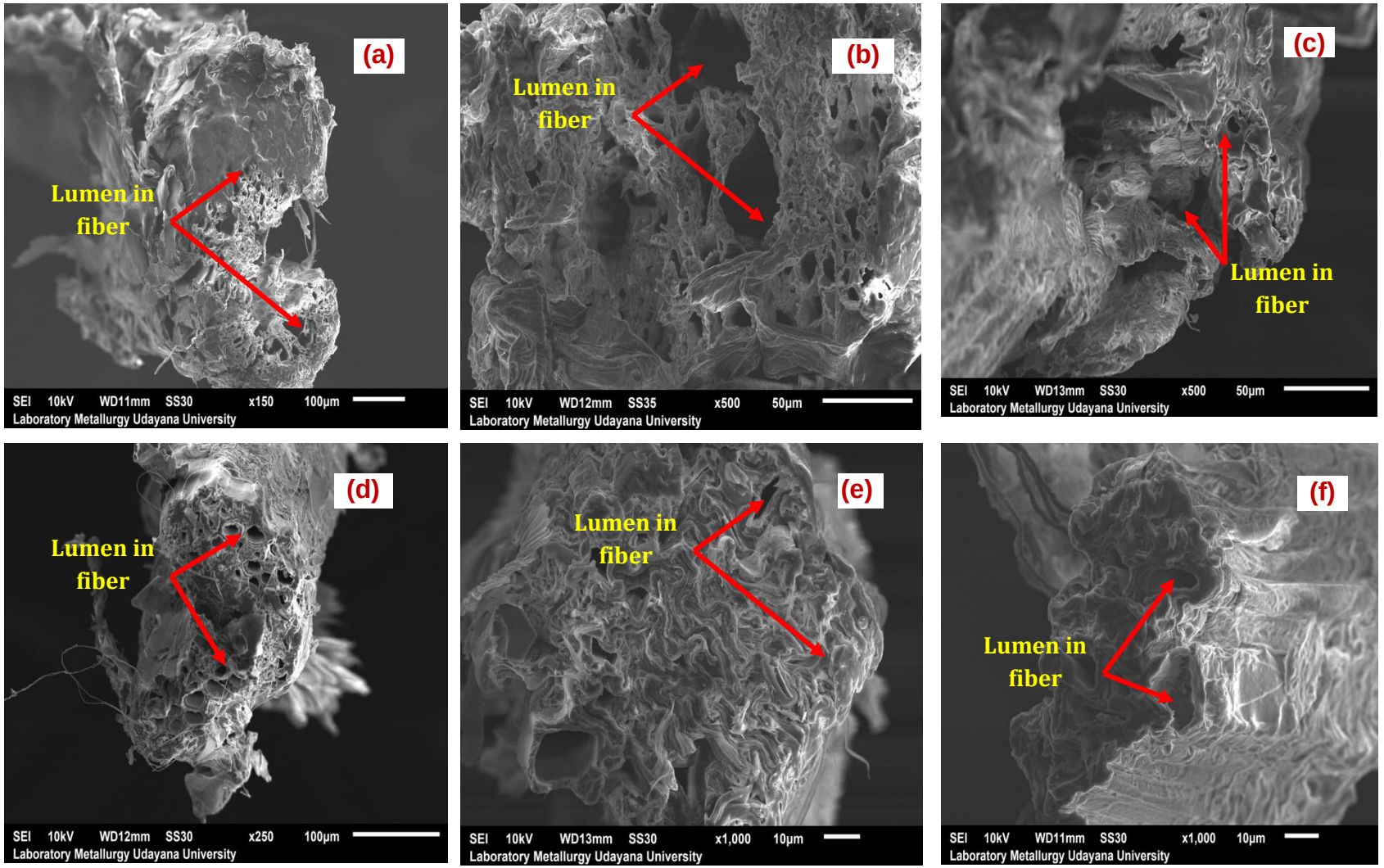

Figure 6. SEM of thecross–sectionsurfaces of fibers: (a) BF–Raw, (b)BF– NaOH, (c) BF– KOH, (d)PF–Raw, (e)PF–NaOH, (f)PF–KOH.

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