

Characterization of Musaceae and Saccharum Officinarum Cellulose Fibers for Composite Application

Nasmi Herlina Sari^{1*}, Arif Rahman² and Edi Syafri³

^{1,2}Department of Mechanical Engineering, Faculty of Engineering, Mataram University, NTB, Indonesia.

³Department of Agricultural Technology, Agricultural Polytechnic, Payakumbuh, West Sumatra 26271, Indonesia.

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ABSTRACT

This paper presents experimental results on the effect of alkalis such as Potassium Hydroxide (KOH) and Sodium Hydroxide (NaOH) on morphological changes, physical, heat resistance, chemical, and tensile properties of the Musaceae (PF) and Saccharum Officinarum (BF) rods fibreers. 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 was 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 was obtained after chemical treatment with KOH while the best properties of Saccharum fiber were obtained after alkali treatment with NaOH. The Scanning Electronic Microscopy (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, type of plant and the extraction method. 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, and acetylation have been used to reduce hydrophilic properties and improve physical and the tensile strength 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]. Among 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 a percentage from 0.5% to 8% concentration was able to improve the tensile strength and thermal properties of the

* Corresponding Author: n.herlinasari@unram.ac.id

cornhusk fibers [5,11]. Likewise, NaOH treatment with 5% to 15% concentration was 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 has been used by some studies to improve the 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 carbon from bamboo stem treated with KOH of the epoxy nanocomposite is higher than the activated carbon treated with phosphoric acid [15]. Previous studies have shown that investigations related to the characterization of natural fibers using NaOH have been widely reported. On the contrary, studies related 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; their function as reinforce in composites manufacture need to be improved.

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 NaOH and 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

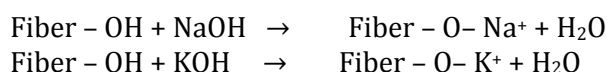
Musaceae (PF) and Saccharum Officinarum (BF) stems were obtained from crops grown in the area of West Lombok, West Nusa Tenggara, Indonesia. The outer surfaces of both types of stems were each cut with length 41.1–43.4 cm. KOH and NaOH solutions were used to modify the fiber surface.

2.2 Fiber Bundles Extraction

Both skins (PF and BF) was immersed in fresh water for fourteen days for microbiological degradation of bacteria [5]. They were washed with fresh water and then combed with a wooden brush to remove residual particles from the surface of cellulose fibers and to retain the same fiber. Fibers rinsed with fresh water and dried in an oven at 105°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 in a chemical solution of KOH and NaOH with each concentration of 8% for 2 hours at 31°C. The reaction scheme is given below [5].



Then, the fibers were cleaned with mineral water to remove the sticky chemical solution on the fiber surface, and dried in an oven for 4 hours at 105°C to remove moisture content. Thus, the fibers were stored in the dry plastic box with humidity of 30%. Musaceae and Saccharum Officinarum plants and the fibers can be seen in Figure 1(a–d).



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

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 |

2.4 Physical Properties of Fibers

2.4.1 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 yields a density value.

2.4.2 Moisture Content

The quantity of weighed fiber was placed in the oven at a temperature range of $104 \pm 2^\circ\text{C}$ 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.

2.5 Fourier Transform Infrared (FTIR) Spectroscopy

The presence of free functional groups in fibers was determined by FTIR. The FTIR used is the Perkin Elmer Spectrum Fourier transforms infrared spectrometer instrument (model Frontier Spectrum) that have a spectral range of $4000\text{--}450\text{ (cm}^{-1}\text{)}$ 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.

2.6 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 constant crosshead speed of 2.5 mm/min and load cell 10 kN under ambient temperature conditions and humidity of 64%.

2.7 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 $10^\circ\text{C}/\text{min}$.

2.8 Scanning Electron Microscope (SEM)

The surface morphologies of raw PF, raw BF, and treated fibers were observed using an FEI model (Inspect-S50-type) SEM operating at 10 kV. The samples were coated with gold sputtered for 5 minutes before their micrographs were recorded.

3. RESULT AND DISCUSSION

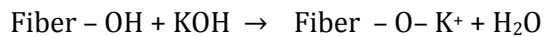
3.1 Chemical Properties of Fibers

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 |

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 reduction 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 the excessive reaction of hemicellulose and lignin. The reaction is given in the following equation:



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 density 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 this fiber in composites.

3.2.2 Moisture Content

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 |

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 the reduction of hemicellulose content (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.

3.3. Tensile Strength Properties

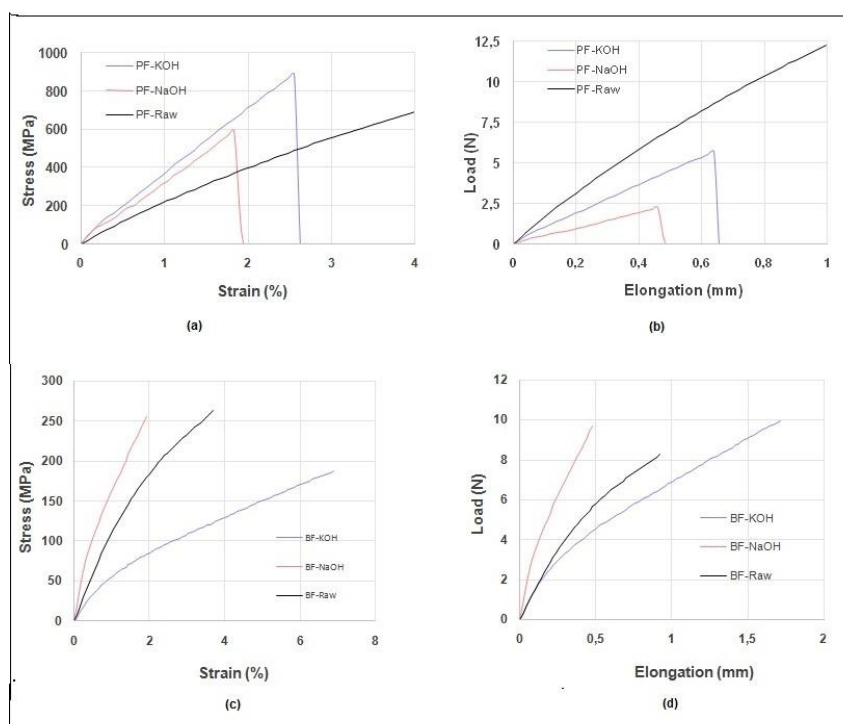


Figure 2. (a) Stress vs strain of Musaceae fiber, (b) Load vs elongation of Musaceae fiber, (c) Stress vs strain of Saccharum Officinarum fiber and (d) Load vs elongation of Saccharum Officinarum fiber.

Figure 2(a) 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). The aspect ratio of the fiber becomes high with a rough fiber surface. In addition, the tensile strength of the fiber also 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) (Figure 2c) because, after NaOH and KOH treatment, the cell wall in the fibers is damaged due to excessive extraction of hemicellulose and lignin. Hence, the tensile strength of BF-KOH and PF-NaOH samples decreases.

It is known that (see Figure 2b and 2d) 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, a different pattern of Figure 2(a) and Figure 2(c) indicate that fiber types and alkali treatment at the surface are also different. The largest differences can be observed through surface morphology and chemical composition. The same reasons also explain the different pattern in Figure 2(b) and 2(d). These results confirmed the results from chemical composition and SEM.

3.4. FTIR Analysis

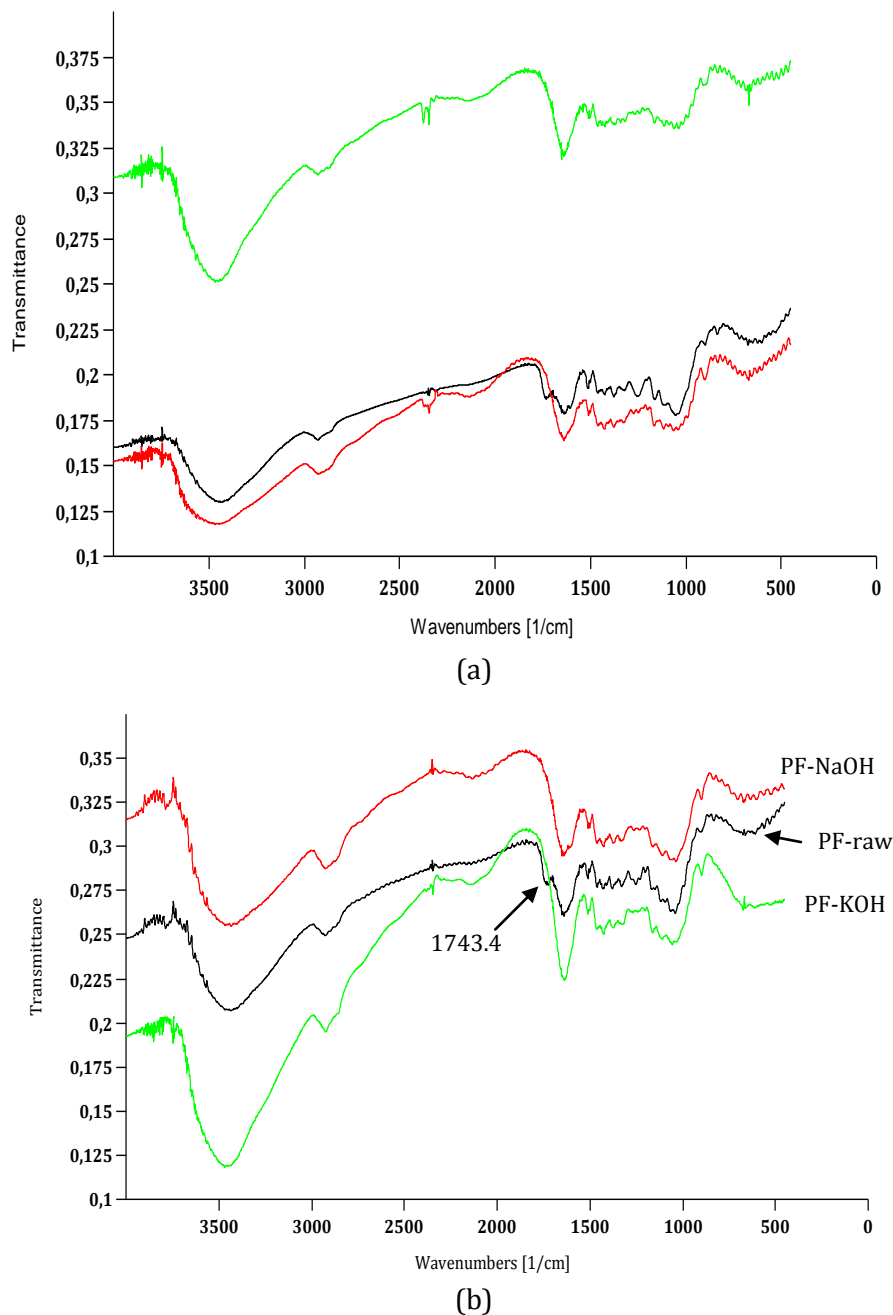


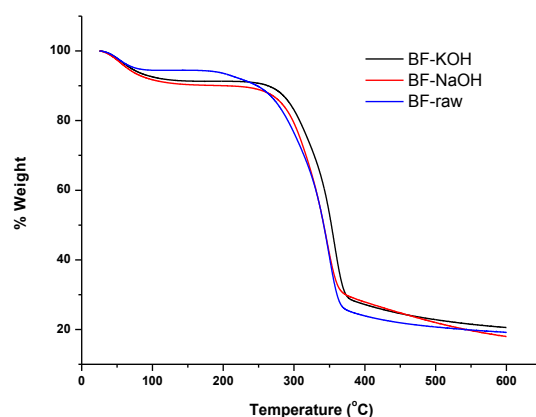
Figure 3. FTIR, (a). *Saccharum officinarum* fiber, (b). *Musaceae* fiber.

FTIR fiber analysis results are shown in Figure 3(a) and 3(b). The FTIR spectra exhibited eight peaks of fiber. For BF-raw and PF-raw samples in Figure 3(a) and 3(b) shown another band at the peaks 1736.8 cm^{-1} and 1743 cm^{-1} that correspond to hemicellulose respectively [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 below, bands near 3400 cm^{-1} 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 (Figure 3a and 3b), indicating α -cellulose decreased.

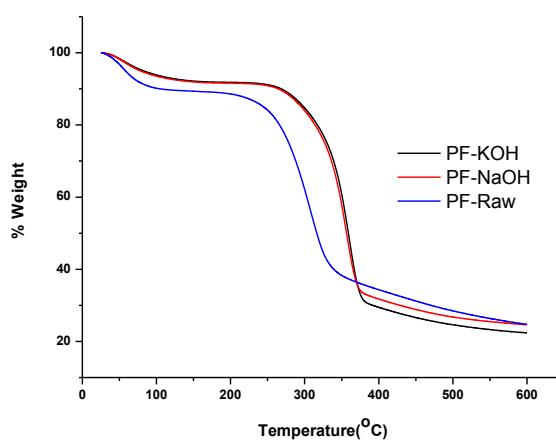
Table 4 Infrared transmittance peaks in the raw and treated fibers.

| 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 Thermogravimetric analysis



(a)



(b)

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

Figure 4(a) and 4(b) shows the TGA thermographs of raw and chemically alkalinized fiber samples. The alkalinized NaOH and KOH fibers showed higher decomposition temperatures compared to the raw fibers. Compared to NaOH treated fibers, the thermal resistance of sample PF-KOH is higher than PF-NaOH. Instead, sample BF-NaOH better than KOH treated fibers. For the main fiber decomposition region 250–310°C, the alkalinized fibers had less weight 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.

3.6 Fiber Morphology Analysis

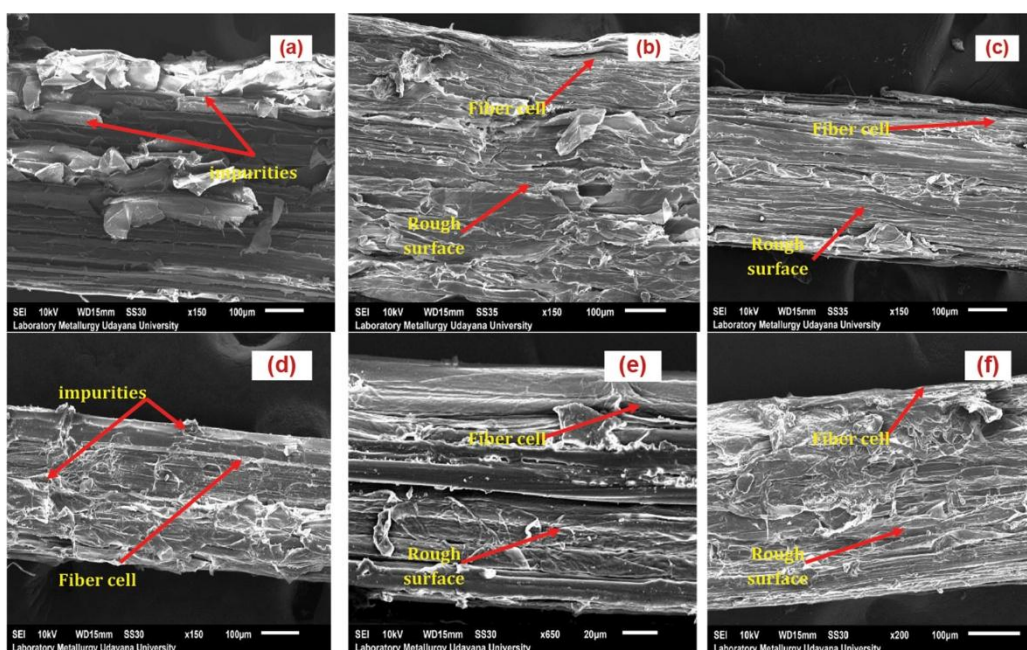


Figure 5. SEM of the surfaces of fibers: (a) BF-Raw, (b) BF- NaOH, (c) BF- KOH, (d) PF-Raw, (e) PF- NaOH, (f)PF-KOH.

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

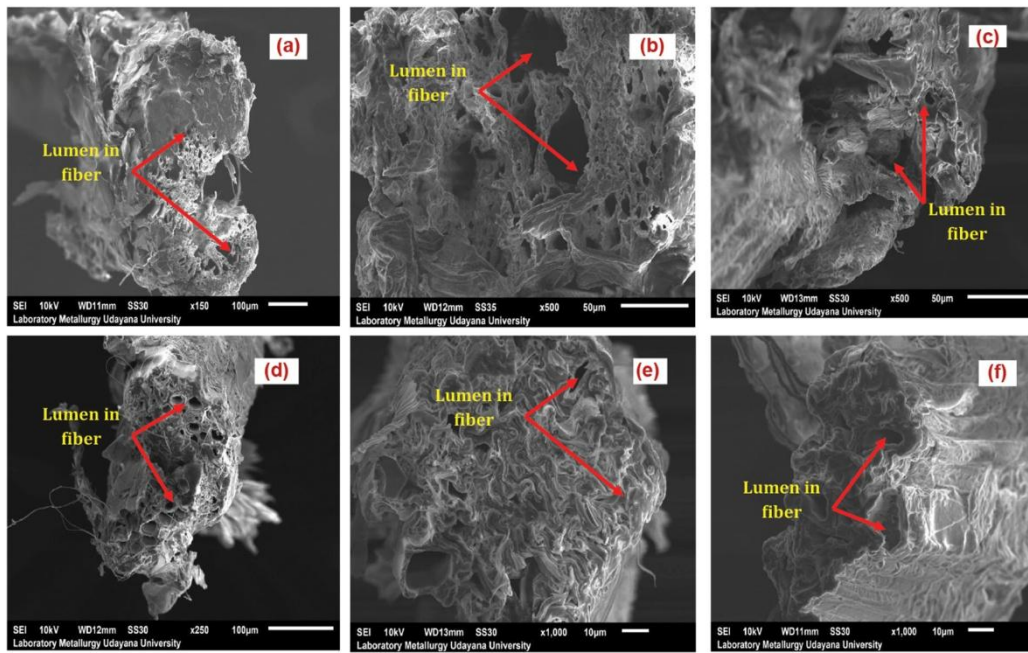


Figure 6. SEM of the cross-sections surfaces of fibers: (a) BF-Raw, (b) BF- NaOH, (c) BF- KOH, (d) PF-Raw, (e) PF-NaOH, (f) PF-KOH.

4. CONCLUSION

Both types of Musaceae and Saccharum fibers and their modifications have been characterized and compared. Experimental results and analysis showed 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.

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