Effect of Surface Treatment on the Physical, Chemical, and Mechanical Properties of Palm Tree Leaf Stalk Fibers

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A new class of leaf stalk fibers of the palm tree were extracted and treated with a 5% NaOH solution for 1 h, 2 h, 6 h, and 12 h. The treated fibers were then characterized by tensile strength testing, chemical analysis, X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and solid state NMR. The tensile strength of the fibers was improved with an alkali treatment, and the 6 h treatment resulted in the maximum fiber strength. The maximum cellulose content was present in the 6 h-treated fibers; cellulose content was reduced with a longer treatment (12 h). Similarly, SEM, FTIR, XRD, and NMR confirmed the removal of hemicelluloses from the raw fiber surface and the formation of new hydrogen bonds between the cellulose fibril chains with respect to the duration of the treatment. The 5% alkali treatment also improved the fiber density from 0.85 gm/cc (raw fiber) to 1.05 gm/cc, 1.13 gm/cc, 1.17 gm/cc, and 1.25 gm/cc after the 1 h, 2 h, 6 h, and 12 h treatments, respectively.

Keywords: Palm fiber; Alkali treatment; Tensile strength; XRD; FTIR; SEM; NMR

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INTRODUCTION

Polymer composites that are reinforced with synthetic fibers and filled with various inorganic fillers have large-scale applications in every field of engineering. However, one problem related to these composites is their disposal, as they contain toxic ingredients that are difficult to recycle. Due to this issue, there is an increasing demand for biodegradable composites (Chand and Fahim 2008), and plant-based fibers are slowly replacing the conventional synthetic fibers in various applications. In addition to being biodegradable, these new fibers are readily available, cost-effective, and have properties comparable to synthetic fibers (Peng *et al.* 2011; Ratna Prasad and Mohana Rao 2011). For better mechanical performance in a natural fiber-reinforced composite, the fiber must be compatible with the matrix, but their interfacial adhesion is generally poor because the fiber is hydrophilic and the matrix is hydrophobic (Westerlind and Berg 1998). Hence, the composite shows poor mechanical properties. The interfacial adhesion can be improved if the matrix, fiber, or both are modified by chemical or physical treatments. Chemically treated natural fibers have potential to be less hydrophilic and have better bonding with the matrix (Chand and Fahim 2008; Hossain *et al.* 2011; Merlini *et al.* 2011; Ramli *et al.* 2011).

Sodium hydroxide (NaOH) has been investigated as a pre-treatment in many different fibers and composites. In Borassus fruit fiber, 5% NaOH treatment removes impurities and improves the tensile strength (Boopathi *et al.* 2012; Reddy *et al.* 2013). Furthermore, alkali treatment completely eliminated amorphous hemicellulose from the fiber surface (Reddy *et al.* 2013). After treating short and randomly oriented palmyra fibers with 5% NaOH, the tensile strength of the resulting composite was greatly influenced by the time of treatment and the fiber volume fraction (Balakrishna *et al.* 2013). An alkali-treated agave fiber-reinforced epoxy composite has shown better tensile, compressive, flexural, and water absorption properties than the untreated composite (Mylsamy and Rajendran 2011). Alkali treatment has also been studied in kenaf fiber-reinforced epoxy composites (Fiore *et al.* 2015). While the impurities were removed by 6% NaOH after 48 h, a longer treatment had a detrimental effect on the fiber surface. Aziz and Ansell (2004) observed that alkalized kenaf polyester had better mechanical properties than alkalized long hemp polyester composites. Alkali pre-treatment is effective in tropical wood polymer composites (Islam *et al.* 2012). Comparing male and female date palm leaves, AlMaadeed *et al.* (2013) found that alkali-treated female leaves had better tensile properties. Anbukarasi and Kalaisevam (2015) investigated the effect of fiber volume, dimension, and alkali treatment on the luffa reinforced epoxy composites. They observed that 40% of the volume fraction of the mat reinforced composite showed better tensile, compressive, and impact strength, and this composite decomposed within a temperature range of 341.40 °C to 387.10 °C. When Bachtiar *et al.* (2008) investigated alkali-treated sugar palm fiber reinforced composite, they observed that the tensile modulus of the treated fiber composite was much higher than that of the untreated fiber composite. Cao *et al.* (2006) prepared a biodegradable composite containing alkali-treated bagasse fiber. The mechanical properties of this composite were superior to those made with untreated fibers.

A variety of other treatments have been investigated. Joseph *et al.* (1996) examined NaOH, isocyanate, permanganate, and peroxide treatments and the tensile properties of short sisal fiber-reinforced polyethylene composites. While a low concentration of permanganate enhanced the mechanical properties, the peroxide treatment resulted in the highest level of interfacial adhesion. Coir pith has also been treated with various chemicals (Narendar and Dasan 2014) and by bleaching, vinyl grafting, and the addition of rubber (Geethamma *et al.* 1998; Rout *et al.* 2001). Liu *et al.* (2014) studied mercerization and a silane treatment in unidirectional abaca fiber epoxy composites; the transverse thermal conductance (TTC) increased as voids in the composite decreased. Mohanty *et al.* (2000) investigated bleaching, dewaxing, alkali treatment, cyanoethylation, and vinyl grafting in a biodegradable jute-fabric polyester amide composite. The tensile and bending strengths of the developed composites were improved by surface modifications, with alkali treatment and cyanoethylation producing the best mechanical properties. Alkali, acetic anhydride, acrylonitrile, $KMnO_4$, diphenylmethane disocyanate, and benzoyl chloride treatments were used on jute fiber mat reinforced PLA composites (Khan *et al.* 2015); the alkali-benzoylated treated composite showed higher tensile, flexural, and impact strength than the composite made from untreated fibers. Similarly, Rahman *et al.* (2015) prepared a photo-catalytic fiber by modifying the surface of jute fiber with a Bi_2O_3/TiO_2 composite. High photo-catalytic activity was observed under visible light irradiation at 420 nm.

In sum, chemical modifications of various natural fibers and the physical, chemical, and mechanical properties of their respective composites have been extensively studied. However, there is no such report available on the study of leaf stalk fibers of the palm tree, which is used in villages for various day-to-day applications. Palm trees are abundant around the world and especially in India. In this study, the prime objective was to investigate the reinforcing potential of palm leaf stalk fiber in order to develop low cost composites. Fibers were extracted from the leaf stalk and chemically treated with 5%

NaOH for different amounts of time. The physical, chemical, morphological, and mechanical properties of the fibers and their resulting composites were studied.

Fig. 1. Extracted raw fibers of leaf stalk

EXPERIMENTAL

Materials

Extraction of fiber

The raw leaf stalks of the palm tree were collected from villages near Bhubaneswar, India, and immersed in tap water for a week. The stalks were then thoroughly washed in tap water. Fibers were extracted from the leaf stalk surface and dried in sunlight for three days. The extracted fibers were then kept in an oven at 105 °C for 2 h to remove all moisture. Finally, the raw fibers were cut with scissors to obtain suitable dimensions of 200 mm in length, 5 mm in width, and 0.4 mm in thickness (Fig. 1).

Alkali treatment of fibers

The dry fibers were treated with a 5% NaOH solution for 1 h, 2 h, 6 h, and 12 h at room temperature and then washed with distilled water to remove excess NaOH. The fibers were then neutralized with a diluted HCl solution at room temperature. The fibers were again washed with distilled water and then dried at room temperature for 24 h.

Physical Properties

Weight loss

The weight loss of the fiber after the alkali treatment was determined using Eq. 1 (Narendar and Dasan 2014),

$$\text{Weight loss (\%)} = \left(\frac{W_2 - W_1}{W_1}\right) \times 100 \qquad (1)$$

where W_1 and W_2 are weight of the fiber before and after treatment, respectively.

Surface morphology

The treated and untreated fibers were examined by a field emission scanning electron microscope (FESEM) (FEI Nova Nano SEM-450, Eindoven, Netherlands).

Fiber density

The Archimedes principle was employed to find the density of the fiber. The weighed quantity of the fiber was completely immersed in water, and the volumetric displacement was observed. The weight-to-volume ratio yielded the density of the fiber.

Chemical Properties

Cellulose content

Dry leaf stalk fiber (1 g) was immersed into a mixture of 1.72% of NaCl and 3 drops of H_2SO_4 in water for 1 h. The excess fluid was removed, and 4 N ammonia was added. The residue was then washed with distilled water, dried at room temperature, and weighed. The percentage of the cellulose was calculated by the ratio of the residue weight to that of the dry sample weight.

Hemicellulose content

Dry leaf stalk fiber (1 g) was immersed in 5% NaOH at room temperature for 0.5 h and then neutralized with 10 N HCl. The fiber was then dried in an oven at 105 °C for 2 h, and the difference in weight was noted as the hemicellulose content.

Lignin content

Dry leaf stalk fiber (1 g; L_1) was immersed into a mixture of 12.5 mL of H_2SO_4 and 300 mL of water at room temperature and refluxed for 2 h. The solvents were removed, and the residue was weighed (L_2). The lignin percentage was calculated using Eq. 2:

$$\text{Lignin content (\%)} = \left(\frac{L_1 - L_2}{L_1}\right) \times 100 \qquad (2)$$

Wax content

The wax content was measured using a Soxhlet apparatus (Borosil, Mahapatra Scientific Supplier, Bhubaneswar, India). Petroleum benzene was heated to 70 °C, and one gram of the leaf stalk fiber was refluxed in the liquid for 1 h. The samples were then dried and weighed. The difference in weight between the raw and the treated fiber was determined as the wax content (Boopathi *et al.* 2012).

Moisture content

The moisture content of the fiber was determined as described (Narendar and Dasan 2014). A sample (1 g; N_1) was heated in an oven at 105 °C for 2 h. The sample was cooled in a desiccator and weighed every hour by replacing it in the oven until it reached a constant weight (N_2). The moisture content was determined using Eq. 3:

$$\text{Moisture content (\%)} = \left(\frac{N_1 - N_2}{N_1}\right) \times 100 \qquad (3)$$

X-ray diffraction (XRD)

The XRD pattern of fibers was recorded using a Rigaku Dmax Ultima-4 diffractometer (Tokyo, Japan) with Ni-filtered Cu Kα radiation. The diffraction intensities were measured from 10 deg. to 80 deg. (2θ range) at a speed of 20°/min. The crystallinity of the fiber was calculated by using Eq. 4,

$$\text{Crystallinity (\%)} = \left(\frac{I_{crystalline} - I_{amorphous}}{I_{crystalline}} \right) \times 100 \qquad (4)$$

where $I_{crystalline}$ and $I_{amorphous}$ are the intensities of crystalline and amorphous material, respectively.

Fourier transform infrared spectroscopy (FTIR)

The fiber samples were examined with a Thermo Nicolet IS-05 FTIR spectrophotometer (Kolkata, India), with scanning from 4000 to 500 cm^{-1} and a resolution of 2 cm^{-1}.

Tensile testing

Tensile properties were determined with an Instron 3369 machine (Kolkata, India) at room temperature with a gauge length of 50 mm and a crosshead speed of 10 mm/min. The test was repeated five times for each sample, and the average value was recorded.

NMR analysis

The high resolution solid state ^{13}C spectra were obtained through a JEOL Resonance ECX 400 spectrometer (Tokyo, Japan) equipped with a CP MAS probe operating at 400 MHz. The samples were packed into zirconia type rotors of 5 mm, and spun at 8 kHz.

RESULTS AND DISCUSSION

When palm fibers are treated with NaOH, a major modification occurs in the form of removal of hydrogen bonding in the network structure as shown in the following reaction (Reddy *et al.* 2013):

$$\text{Fiber-OH + NaOH} \blacktriangleright \quad \text{Fiber-}O^{-}Na^{+} + H_2O + \text{Surface impurities} \qquad (5)$$

Alkali treatment improves the fiber surface adhesive characteristics by removing surface impurities and thereby producing a rough surface as shown in Fig. 7. Besides, alkali treatment leads to breaking down of the fiber bundles into smaller fiber components in a process referred to as fiber fibrillation. Therefore, alkali treatment reduces fiber diameter and as a result increases the aspect ratio (Joseph *et al.* 1996). The effect of alkali treatment on the palm fiber is discussed in measuring density, weight loss, chemical properties, tensile properties, XRD analysis, FTIR analysis, SEM and NMR study.

Fiber Density

Alkali treatment had a positive impact on fiber density (Table 1); the density increased with increasing treatment time. This result may reflect the densification of cell wall as a result of removal of non cellulosic part after prolonged alkali treatment. Similar observations have been made in Borassus fruit fibers and coir pith (Boopathi *et al.* 2012; Narendar and Dasan 2014).

Table 1. Density of Raw and NaOH-Treated Palm Fiber

Sample	Alkali Treatment (h)	Density (gm/cc)
1	0 (raw fiber)	0.85
2	1	1.05
3	2	1.13
4	6	1.17
5	12	1.25

Weight Loss

Alkali treatment noticeably reduced the fiber weight, which may have indicated the removal of wax and other non-cellulosic materials. More impurities were removed by longer treatments. After 1 h, alkali-treated fibers showed a 12% weight loss, which increased to 27%, 40%, and 56% for 2, 6, and 12 h, respectively. Similar results were observed for coir pith (Narendar and Dasan 2014).

Chemical Properties of the Fiber

The chemical composition of treated and untreated fibers is presented in Table 2. The 6 h-treated fiber showed the maximum cellulose content of 72.35%, and this value dropped to 61.26% for the 12-h treatment. Hemicellulose was more sensitive to the duration of the alkali treatment, as it was reduced from 13.02% to 0.00% after 2 h in 5% NaOH; wax contents behaved similarly (Gassan and Bledzki 1997). However, the lignin content was reduced from 4.52% (raw fiber) to 2.9% after the 12-h treatment. In sum, alkali treatment enhanced the cellulose content and removed surface impurities from the fiber, which leads to better mechanical properties. Similar observations have been made in Borassus fruit fibers (Boopathi *et al.* 2012; Reddy *et al.* 2013).

Table 2. Chemical Composition of Raw and NaOH-Treated Fiber

NaOH Treatment (h)	Cellulose (wt.%)	Hemicellulose (wt.%)	Lignin (wt.%)	Wax (wt.%)
0	44.10	13.02	4.32	0.20
1	53.75	2.42	4.20	0.11
2	60.18	0.01	3.85	0.06
6	72.35	0.00	3.10	0.00
12	61.26	0.00	2.98	0.00

Tensile Properties of the Fiber

The tensile strength and modulus of the raw fiber increased with increasing length of the treatment, up to 6 h. When the treatment was extended to 12 h, the strength and modulus values were reduced, as shown in Table 3 and in Figs. 2 and 3. This may be due to the rupture of hemicellulose bonds existing between the fiber surfaces, which have made the fiber more homogenous. As a result, the load carrying capacity of the fiber was improved. In case of untreated fibers, presence of hemicelluloses in the inter-fibrillar region separates the cellulose chains from one another and due to this barrier, the molecular chains are in a state of strain. When this barrier (hemicelluloses) is removed, new hydrogen bonds are formed between the cellulose chains and the fibrils are now more capable to carry external loads. However, excess treatment of alkali leads to rupture of the fibril, which reduces the tensile strength of fiber. Similar observations have been found in Borassus fruit fiber (Reddy *et al.* 2013).

Table 3. Tensile Properties of Untreated and Treated Palm Fiber

NaOH Treatment (h)	Tensile Strength (MPa)	Modulus (GPa)
0	74	9.5
1	81	11.4
2	87	16.8
6	95	23.6
12	83	13.2

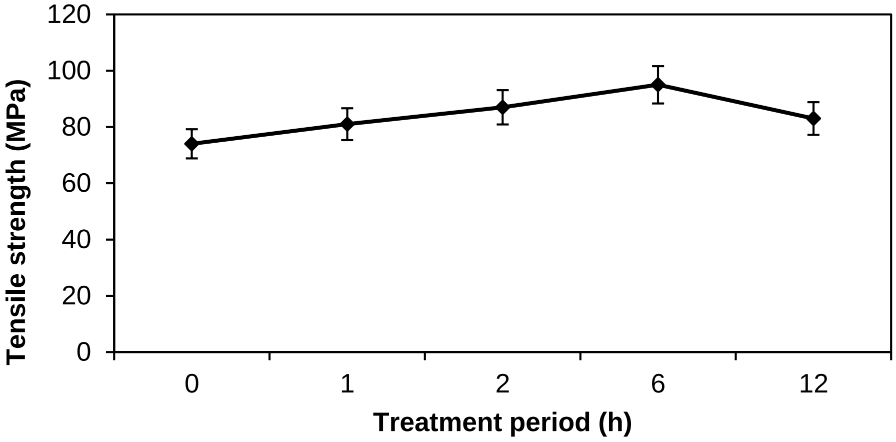

Fig. 2. Tensile strength of untreated and treated palm fiber

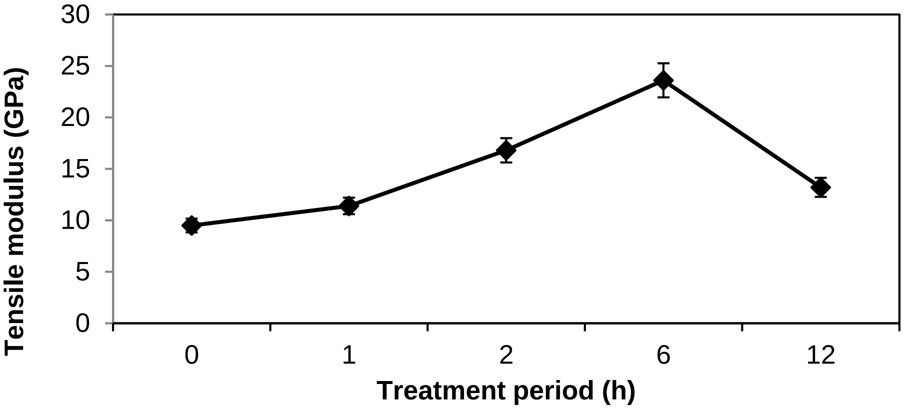

Fig. 3. Tensile modulus of untreated and treated palm fiber

X-ray Diffraction Analysis

XRD analysis of the raw fiber and alkali-treated fibers showed two major reflections corresponding to the 2θ values of 17 deg. and 22 deg. (Fig. 4). The sharp and intense reflection at 22 deg. signified the presence of crystalline cellulose. The low angle of reflection (17 deg.) represented amorphous hemicelluloses. The crystallinity index of each sample was calculated using Eq. 4. The crystallinity index of the raw fiber was 46.37. This value increased to 57.55 after the 6-h treatment but decreased to 48.9 for the 12-h treatment. This result was attributed to the removal of amorphous hemicelluloses and the exposure of crystalline cellulose on the fiber surface. Similar observations were made in Borassus fruit fiber and coir pith (Reddy *et al.* 2013; Narendar and Dasan 2014).

FTIR Analysis

The FTIR of raw fiber contained an O-H stretching band at 3431 cm^{-1} (Fig. 5). The band at 2925 cm^{-1} indicated the C-H modes of methyl and methylene groups (Boopathi *et al.* 2012). Similarly, the peak at 1741 cm^{-1} indicated the presence of C=O stretching in acetyl groups of hemicelluloses. The peak at 1254 cm^{-1} was due to C-O stretching in hemicelluloses (Reddy *et al.* 2013). There was no peak at 1741cm^{-1} for the alkali-treated fibers, which indicated the removal of hemicelluloses, as previously noted (Reddy *et al.* 2013). Similarly, the peak at 1254 cm^{-1} was not visible, confirming the lack of C-O stretching from hemicelluloses. The band at 1037 cm^{-1} was gradually reduced with extended alkali treatment, which indicated the reduction of lignin content (Boopathi *et al.* 2012).

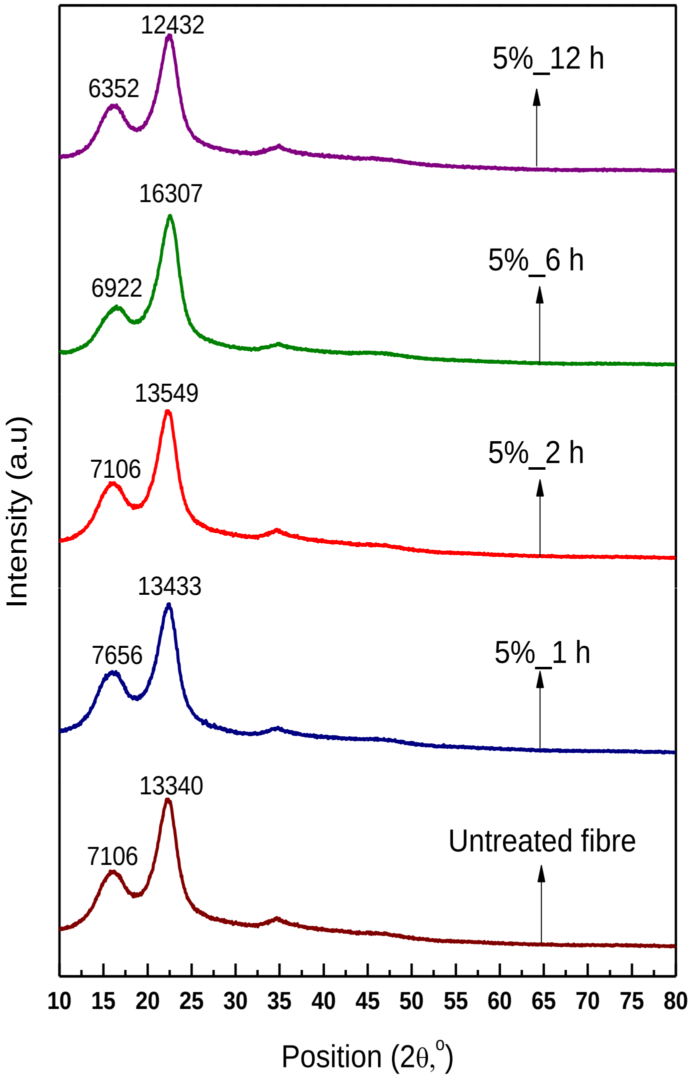

Fig. 4. XRD patterns of untreated palm fiber and fiber treated with 5% NaOH for 1, 2, 6, or 12 h

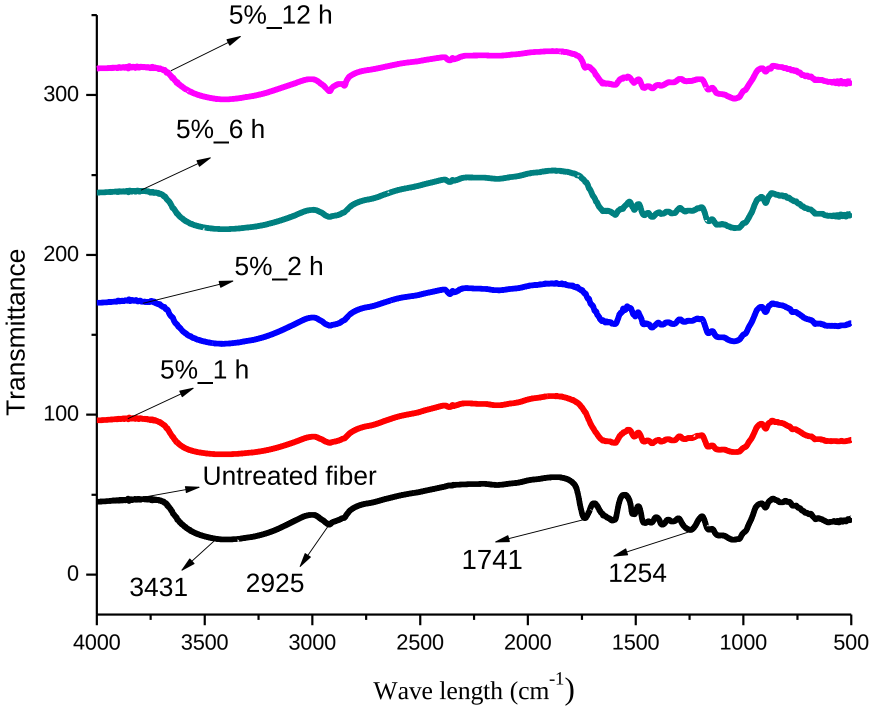

Fig. 5. FTIR spectra of raw and alkali-treated fiber

Surface Morphology of the Fiber

Fiber morphology was studied by SEM (Figs. 6, 7, and 8). The raw fiber surface was compact, smooth, and contained a white layer of visible impurities. In the micrograph of fiber treated for 6 h with 5% NaOH, there were many pores visible on the fiber surface. Most surface impurities including hemicellulose, wax, and lignin were removed. When the fibers were treated for a longer duration, the rough and degraded surfaces reflected the breakage or partial removal of crystalline cellulose materials (Fig. 7). Also, fiber weight loss was observed, which may signify the removal of impurities. Similar observations were made in Borassus fruit fiber (Boopathi *et al.* 2012; Reddy *et al.* 2013). The rough surface of the fiber increases the wettability with the polymer, and consequently, the mechanical properties of the developed composites. However, the longer alkali treatment was detrimental to fiber strength due to the partial removal of the aligned grain structured cellulose content (Fig. 8). Therefore, the treatment should be judiciously selected for developing laminated composites reinforced with these fibers.

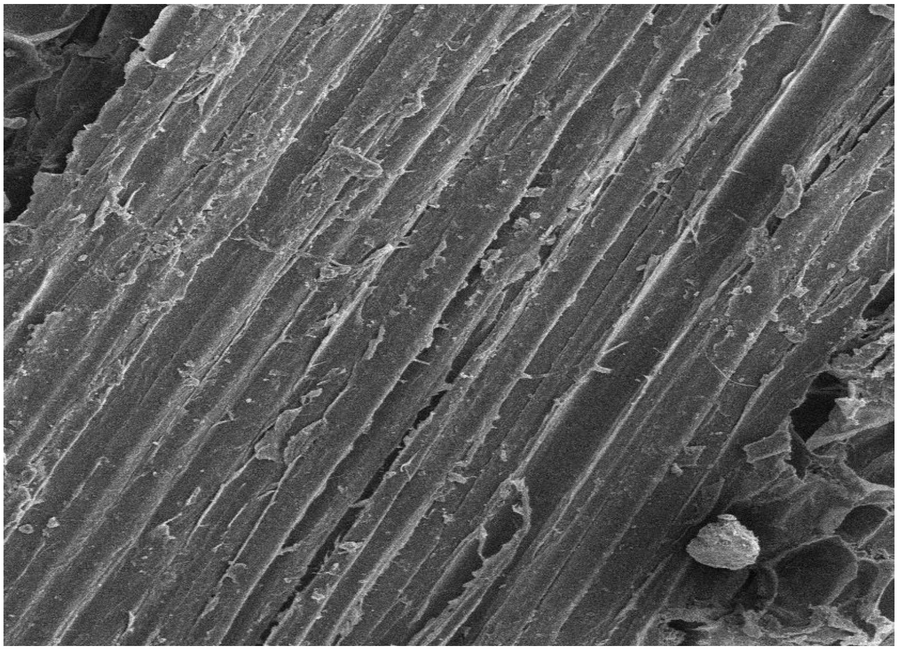

Fig. 6. SEM of raw palm fiber (500x magnification)

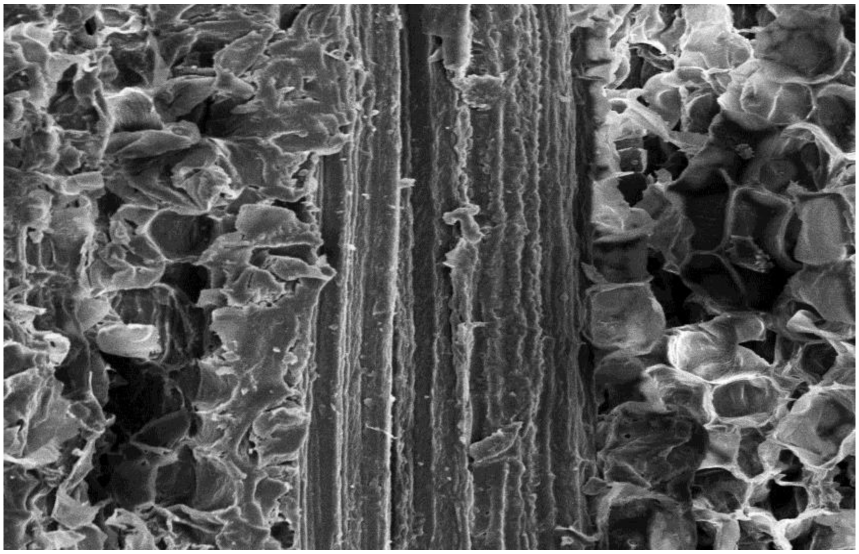

Fig. 7. SEM of palm fiber treated with 5% NaOH for 6 h (1000x magnification)

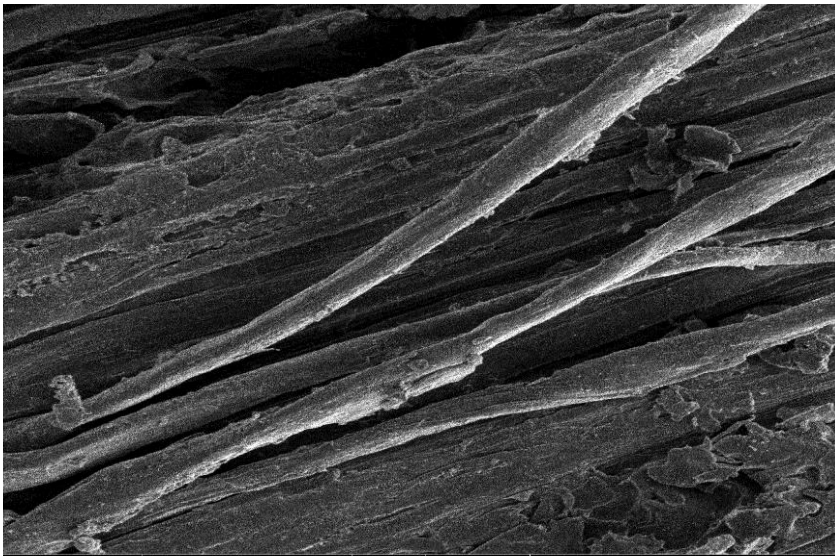

Fig. 8. SEM of palm fiber treated with 5% NaOH for 12 h (1000x magnification)

Solid State NMR Analysis

Solid state NMR spectra of raw and alkali treated palm fiber are shown in Fig. 8.

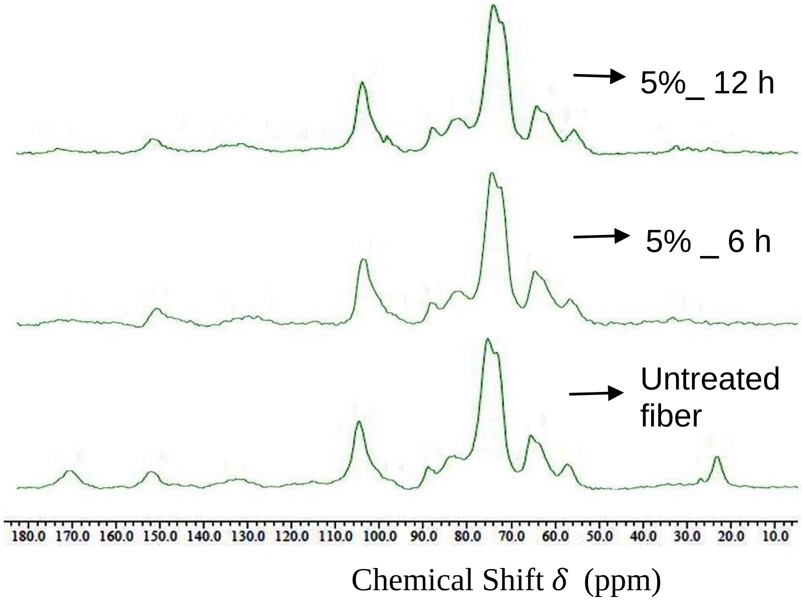

Fig. 9. CP/MAS ^{13}C NMR spectra of raw and alkali treated fiber

In the untreated fiber, it can be observed that the most intense signals were from cellulose carbons that appear between 60 to 110 ppm. The signals from 60 to 70 ppm are assigned to C6, from 70 to 80 ppm to C2, C3, and C5, from 80 to 90 ppm to C4, and from 97 to 108 ppm to C1. The signal of methyl and carboxylic carbons of acetyl groups attached to hemicelluloses indicated at 22 and 170 ppm respectively. The less intense signals resonated at 56 ppm, from 130 to 136 ppm and from 154 to 158 ppm were assigned to methoxyl and aromatic groups of lignin. For the alkali treated fibers, it can be seen (Fig. 9) that the signals at 22 and 170 ppm due to acetyl groups of hemicelluloses were not found, which confirmed the removal of hemicelluloses from the surface. Similarly, the signals in the lignin aromatic region, that is, from 130 to 136 ppm and from 154 to 158 ppm were gradually reduced, which indicated the partial removal of lignin content from the treated fiber surface. These observations are in agreement with the previous works (Martins *et al.* 2006; Wikberg and Mannu 2004; Stewart *et al.* 1997).

CONCLUSIONS

1. Alkali treatment improved the density of the raw fiber from 0.85 gm/cc to 1.05, 1.13, 1.17, and 1.25 gm/cc after 1, 2, 6, or 12 h of treatment, respectively.
2. Alkali treatment removed hemicelluloses, lignin, and wax from the fiber surface, making it rough. The removal of hemicelluloses, lignin, and other surface impurities was confirmed with SEM, FTIR, XRD and NMR analyses. The treatment led to a better fiber-matrix interface, fiber wetting characteristics, and improved mechanical properties.
3. The tensile strength value of the fiber treated for 6 h with 5% NaOH was higher than other samples, which reflected the highest cellulose content in these fibers. A longer treatment (12 h) reduced the tensile strength.
4. The 6 h-treated fibers showed the best mechanical properties. Hence, fibers treated for 6 h with 5% NaOH can be used as reinforcements in composites for various applications.

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