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Study of Alkali Treatment and Its Influence on Characteristics of Borassus Palm Fiber

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ABSTRACT
Palmyra fibers are found to be the renewable and sustainable fiber in textile applications. These fibers possess good tensile strength and found to be used as a natural reinforcement material for composites. The main objective of this work is to extract and treat the fibers with varying concentrations of NaOH (2%,3%,4%& 5%) to evaluate the mechanical properties, surface morphology, and fiber orientation. The observations in SEM images denote the removal of impurities like hemi cellulose and lignin after the alkali treatment. 4% NaOH-treated fibers results in higher tensile strength (63.43 MPa) and a further increase in alkali concentration to 5% results in a reduction of the tensile strength (62.65 MPa). For all the treated fibers, elongation at break was found to be higher than the untreated one. The orientation in the fiber structure improves after the alkali treatment up to 4% and a further increase in alkali concentration leads to a decrease in the orientation which was observed through X-ray diffraction studies.

摘要
棕榈纤维被认为是纺织应用中可再生和可持续的纤维. 这些纤维具有良好的拉伸强度, 被发现用作复合材料的天然增强材料. 这项工作的主要目标是用不同浓度的NaOH(2%、3%、4%和5%)提取和处理纤维, 以评估机械性能、表面形态和纤维取向. SEM图像中的观察结果表明, 碱处理后, 半纤维素和木质素等杂质被去除. 4%NaOH处理的纤维具有更高的抗拉强度(63.43 MPa), 碱浓度进一步增加至5%会导致抗拉强度降低(62.65 MPa). 所有处理过的纤维的断裂伸长率都高于未处理过的纤维. 碱处理高达4%后, 纤维结构中的取向得到改善, 碱浓度的进一步增加导致通过X射线衍射研究观察到的取向降低.

KEYWORDS
Borassus palm fiber; sodium hydroxide; morphology; fibrillation; tensile strength; color characteristics

关键词
棕榈纤维; 氢氧化钠; 形态学; 颤动; 抗拉强度; 颜色特征.

Introduction

Palmyra palms (*Borassus flabellifer L.*) are economically useful and widely cultivated, especially in South East Asia. The tree yields many types of products from every part of it. The sapling is cooked as a vegetable or roasted and pounded to make meal. The fruits are eaten roasted or raw. A sugary sap, called toddy, can be obtained from the young inflorescence (both male and female tree). Furthermore, it is fermented to make a beverage called arrack (palm wine) or concentrated to produce a crude sugar called jaggery/palm sugar (Bayton 2007). Apart from the above uses, fully grown fruit papad (jelly) can make mesocarp which is shown in Figure 1. Remaining non-edible fiber content will become waste; these

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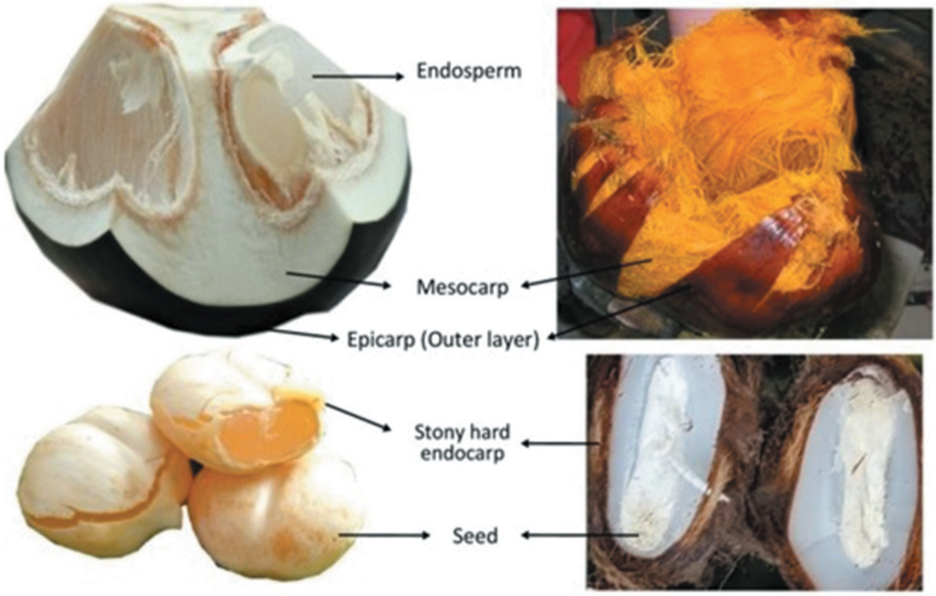

Figure 1. Palm fruit young (left) and ripe (right).

can be used into various applications in the textile sector. The matured palm fruit consists of various segments such as epicarp, mesocarp, endosperm, and seeds. The mesocarp consists good amount of approximately 2.37% of non-dietary fibers along with 51.07% of edible content, 35.99% of seeds, and remaining 10.57% of epicarp and perianth lobes (Davis and Johnson 1987). Extracted fibers cannot be used directly into textile applications, as this consists of cellulose, lignin, hemicellulose, and surface impurities like waxes and pectin etc. Since these impurities are directly relating to fiber properties, so it is necessary to carry out the pre-treatment with alkali (Reddy, Guduri, and Varada Rajulu 2009).

Since, last two decades, tremendous importance was given to use the natural fibers as it is sustainable in nature. These fibers were used in various applications such as thermoplastic and thermosetting fiber composites (Sarasini et al. 2017). Palm fibers are having advantages toward good tensile properties such as breaking strength & elongation and yield approximately 3% on its weight basis. Alkalization of plant-based fibers changes the surface morphology and crystallization rate in the fiber structure (Mwaikambo and Ansell 2002). Reddy et al. (2013) extracted palm fibers by simple water retting and the fibers were treated with 5% sodium hydroxide solution by varying treatment durations from 0–12 h. Furthermore, the fibers evaluated for chemical composition, available functional groups, crystallinity, and tensile properties. They concluded that 8 h treatment found to be best due to improved fiber structure. The effect of chemical treatment on fibers extracted from coconut fiber was studied by using NaOCl, H_2O_2, and its combination. The treated fibers with the combination of both chemicals found to be more effective in the removal of hemicellulose and high cellulose exposition (Brígida et al. 2010).

From the literature, it was observed that the studies focused on treating the palm fiber by chemical means with higher treatment times. The main objective of this exploration is to achieve good properties with reduced treatment time. In the present study, four different alkali concentrations were selected such as 2%, 3%, 4%, and 5% to investigate the properties of extracted fibers. Furthermore, the treated fibers were bleached and dyed with reactive dyes to understand the influence of treatment on absorption of dyes.

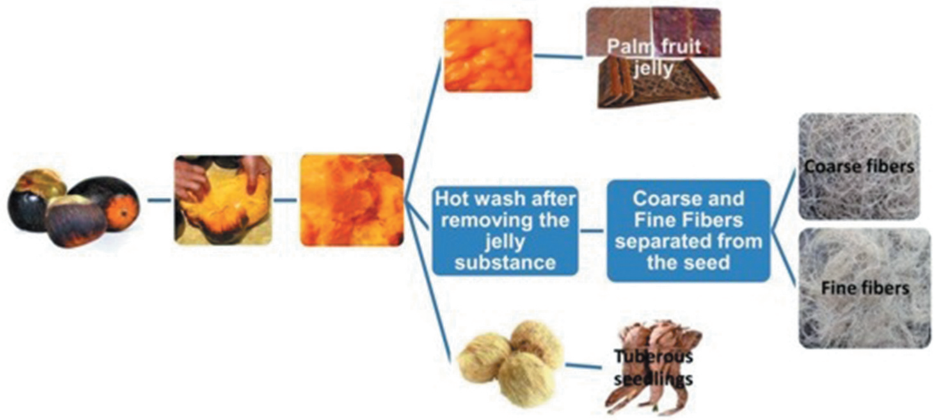

Figure 2. Extraction process of palm fiber.

Materials and methods

Fiber extraction process

Fiber extraction process was started by collecting the ripened palm fruit from the agricultural fields near Guntur, Andhra Pradesh, India. After collecting the required quantity of palm fruit, it is subjected to cleaning process. The outer skin has been removed from the palm fruit to obtain the fibers. Three different products could be extracted as palm fruit jelly, palm seed fibers, and tuberous seedlings. From the palm fruit jelly, the fibers are separated by simple hot wash to remove the remaining jelly substance and the fibers were separated manually and dried. The typical sequence of the process is shown in Figure 2. After drying, the fibers were cut and sorted according to the fineness.

Fibers after the extraction were subjected to scouring and bleaching (pre-treatments) process to remove the non-fibrous content and natural color from the raw palm fibers. Laboratory grade sodium hydroxide (NaOH) and hydrogen peroxide (H_2O_2) were used for the study. The pre-treated palm fibers were also dyed using Corafix Red ME3B (CI reactive Red 195) dyes supplied by Color Tex India Pvt Ltd., Mumbai.

Methodology

A total of four different combinations of alkali selected for this study to understand the effect of alkali on structural and mechanical properties of fiber. Palm fibers were subjected to treat with 2%, 3%, 4%, and 5% NaOH solutions at 90°C for a period of 60 min in an IR beaker dyeing machine. In this process, the material-to-liquor (M:L) ratio of 1:100 and pH at 10 was maintained. After alkali treatment, the treated fibers were washed with cold water and undergo 1% CH_3COOH for neutralization at 40°C for 30 min; furthermore, the fibers were washed with cold water and dried at atmospheric conditions.

The scoured palm fibers after drying were subjected to bleaching process with 3% hydrogen peroxide, 2% sodium silicate, and 1% sodium carbonate solution at a M:L ratio of 1:100 for a period of 60 min at 80°C. Residual peroxide was removed from the bleached fibers by using 0.1% peroxide killer RS for a period of 20 min at 40°C. After that, the bleached fibers were dyed in an IR beaker dyeing machine with 3% dye, 70 g per liter (g/L) sodium chloride, and 20 g/L sodium carbonate, M:L ratio of 1:100 at a temperature of 60°C about 60 min at pH of 10. Figure 3 shows the different stages of fiber dyeing.

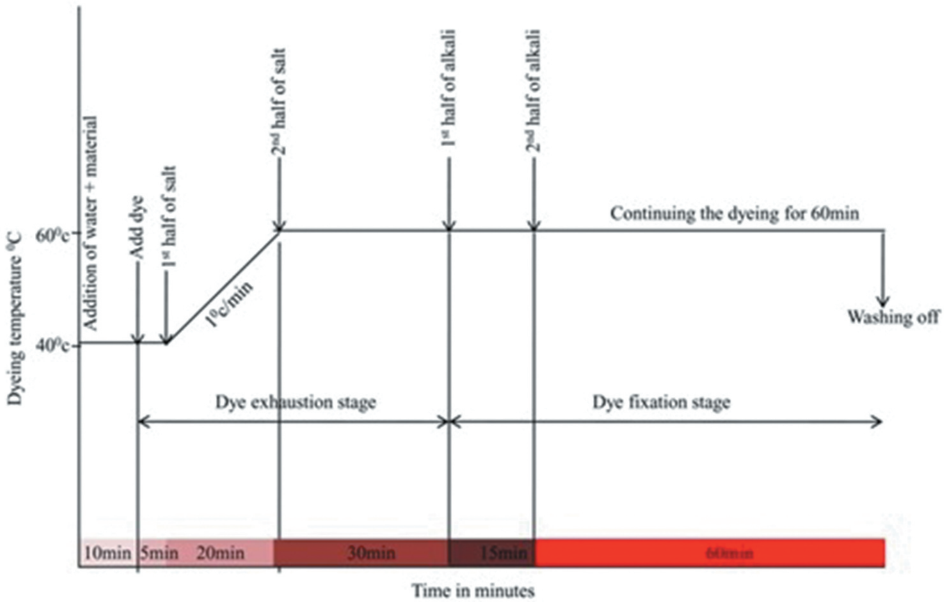

Figure 3. Dyeing curve of palm fibers.

Evaluation

Tensile characteristics

The mechanical properties like single fiber strength and percent breaking elongation of palm fibers were evaluated as per ASTM-D3822. The typical sample holder is shown in Figure 4. All the fibers prior to measurement were conditioned at standard atmospheric conditions, i.e. temperature of 27 ± 2°C and 65 ± 2% RH% for 24 h. The testing was conducted at the span length of 50 mm and traverse speed of 1 mm/s.

Scanning electron microscope (SEM)

Tescanvega3 SEM was used to study the morphological properties of fibers at various stages of treatments. Palm fiber surface was sputter coated with a thin layer of gold to make the fiber conductive in a vacuum chamber before starting the measurement. The SEM images taken at required magnification range to observe the surface changes clearly at 10.0 KV electron voltage.

Thermogravimetric analysis (*TGA)*

To understand the thermal behavior of palm fibers, TGA was carried out by using Thermal Analyzer STA 7200. The experiments carried out in the presence of nitrogen gas with furnace temperature of 40–800°C (Stevulova, Hospodarova, and Estokova 2016).

X-ray diffraction (XRD)

An X-ray diffractometer (Model: Rigaku Miniflex 300/600), which is a multipurpose powder diffractometer, is used to estimate the structural characteristics of raw and scoured palm fibers. The samples measured with 40 KV X-ray, 15 mA at an incident beam wavelength CuKa/1.54 A, scanned in the axis of 2θ range varying from 3° to 90° at a speed of 10° per minute. The crystallinity index and percentage of crystallinity were estimated to know the structural changes before and after the treatment (Balasundar et al. 2017).

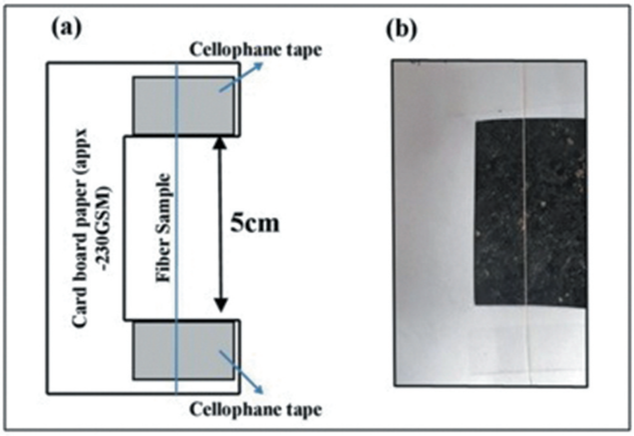

Figure 4. Sample holder for single fiber tensile strength: (a) sample holder and (b) fiber placed in the sample holder.

Result and discussions

Diameter

Diameter of fibers was measured under the optical microscope with 10× magnification, and the images are shown in Figure 5. Microscopic observation on the fibers leads to categorized into two types according to the diameter, namely, coarse and fine. The average diameters of both coarse and fine fibers were observed throughout the length at various places. For each fiber, 50 readings were taken and likewise 10 fibers were measured. The results are given in Table 1. If the diameter ranges from 0.1330 mm to 0.1826 mm (±0.029 mm) as considered to be fine fiber and the diameter varies from 0.2738 mm to 0.3865 mm (±0.056 mm) deemed to be coarse.

It was observed that coarse and fine fibers found in the range of 5–10% and 90–95%, respectively. The increase in concentration of alkali has significant effect on the mean fiber diameter. The mean diameter of raw fiber (RF), scoured, bleached, and dyed fibers were tabulated in Table 2. It was observed that the mean diameter of RF was observed as 0.1561 mm, whereas after treating with varying concentrations of alkali, the mean diameter was contracted and found as 0.1545, 0.1503, 0.1414, and 0.1304 mm.

Tensile properties

Tensile properties of palm fruit fibers have been assessed by various authors shown in Table 3. The effect of alkali treatment for various concentrations on tensile properties of palm fiber was shown in Table 4. It is obvious that the alkali treatment made a large variation in properties of natural cellulosic fibers, approximately the CV% in the range of 35–50% (Govardhana Rao, Venkatesh, and Sai Chandana 2019; Hearle and Morton 2008). The average tensile strength (MPa) was calculated by considering fiber diameter and cross section, which results in the CV about 25–45%, as shown in Table 3. Raw fine palm fibers show the least tensile strength when compared alkaline treated, bleached, and dyed palm fibers. It was also evident from the result that the raw fine palm fibers show the tensile strength of 54.08 MPa, whereas subjecting the palm fibers to 2% alkali at 90°C for 60 min not have much influence on tensile properties of palm fiber and it was shown to be 54 MPa. The tensile strength has increased with increasing concentration from 3 to 4%. (i.e. 57.23 MPa and 63.43 MPa, respectively). Further increase in alkali concentration to 5% leads to reduction in tensile strength, i.e 62.65MPa. The same pattern of tensile strength was found in the work of Oushabi et al. (2017) and Chen et al. (2017). This increase in the tensile strength was due to the effective removal of

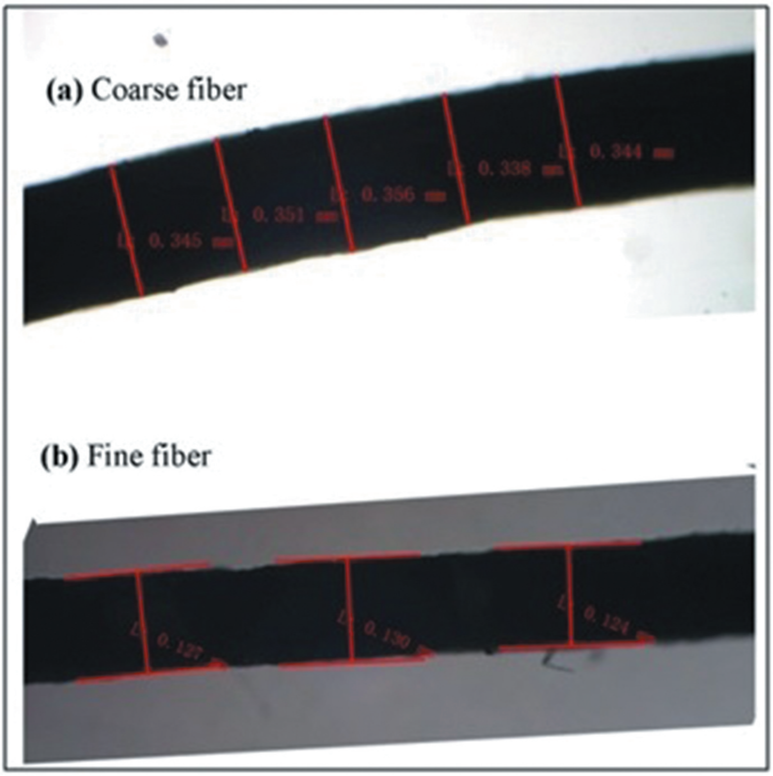

Figure 5. Microscopic images of coarse and finer fibers.

Table 1. Morphological parameters of untreated coarse and fine fibers.

	Untreated fiber morphological parameters	
Particulars	Coarse fiber	Fine fiber
Mean fiber diameter (mm)	0.3346	0.1561
SD	0.0330	0.0144
CV%	9.7621	9.5086
Minimum	0.2738	0.1330
Maximum	0.3865	0.1826

Table 2. Mean diameters of raw (fine), scoured, bleached, and dyed palm fibers.

Particulars/fiber type and combinations		Mean diameter (mm)	SD	CV%	Minimum	Maximum
Raw fiber (fine)		0.1561	0.0144	9.5086	0.1330	0.1826
Scoured fiber	S2	0.1545	0.0172	11.1327	0.1095	0.1648
	S3	0.1503	0.0189	12.5749	0.0998	0.1587
	S4	0.1414	0.0176	12.447	0.0887	0.1492
	S5	0.1304	0.0156	11.9632	0.0974	0.1537
Bleached fiber	S2B	0.1448	0.0184	12.9299	0.1075	0.1708
	S3B	0.1421	0.0195	12.8510	0.0775	0.1705
	S4B	0.1249	0.0162	12.9417	0.0896	0.1452
	S5B	0.1134	0.0140	12.7845	0.0918	0.1520
Dyed fiber	S2D	0.1618	0.0134	8.5222	0.1370	0.1830
	S3D	0.1456	0.0163	11.2733	0.1192	0.1718
	S4D	0.1412	0.0157	11.119	0.1101	0.1754
	S5D	0.1393	0.0173	12.4671	0.1114	0.1708

Note: RF – Raw Fine palm fibers; S2 – Scoured 2%; S2B – Scoured with 2% then bleached; S2D – Scoured with 2% then bleached followed by reactive dyeing.

Table 3. Tensile properties of palm fruit fiber by various authors.

Sl. no.	Particulars		Tensile strength	% Breaking Elongation	Reference
1	Coarse fiber	Untreated	50.9 MPa	41.2	(Reddy, Guduri, and Rajulu 2009)
		Alkali treated	53.5 MPa	41.9	
	Fine fiber	Untreated	65.2 MPa	47.2	
		Alkali treated	90.7 MPa	58.5	
2	Alkali treatment time	0 hr	70.8 MPa	34.8	(Obi Reddy et al. 2013)
		1 h	106.3 MPa	42.7	
		4 h	117.5 MPa	51.1	
		8 h	121.3 MPa	58.1	
		12 h	101.8 MPa	35.7	
3	Palm fiber		4.2 g/den	21	(Muthuvelammai 2017)

Table 4. Tensile properties of raw fine palm, scoured, bleached, and dyed fibers.

Sample code	Tensile strength (MPa)	Tensile strength (CV%)	Breaking elongation (%)
RF	54.08	29.87	30.23
S2	54.00	25	32.15
S3	57.23	32	35.76
S4	63.43	35	38.35
S5	62.65	24	40.24
S2B	55.16	20	32.02
S3B	61.15	25	35.82
S4B	64.80	45	38.23
S5B	63.59	22	40.03
S2D	55.12	30	32.53
S3D	62.52	39	35.89
S4D	65.36	36	38.85
S5D	63.51	31	41.38

Note: RF – Raw Fine palm fibers; S2 – Scoured 2%; S2B – Scoured with 2% then bleached; S2D – Scoured with 2% then bleached followed by reactive dyeing.

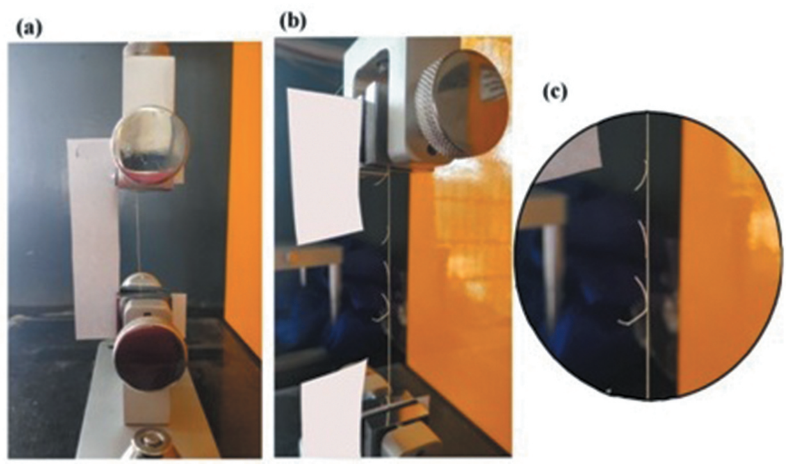

Figure 6. Tensile measurement of palm fibers: (a) position of fiber, (b) fracture of fiber leading to fibrillation, and (c) close view of fibrillation.

hemicellulose, lignin from the fiber, and also rearrangement of fiber structure causes close packing of cellulose molecules in the polymeric structure. The elongation at break for all treated palm fibers was found to be higher than the untreated one. As NaOH concentration increases to 3%, the elongation at break starts to increase significantly. Once the concentration increases to 4 and 5%, the elongation at break increase by 27 and 33%, respectively.

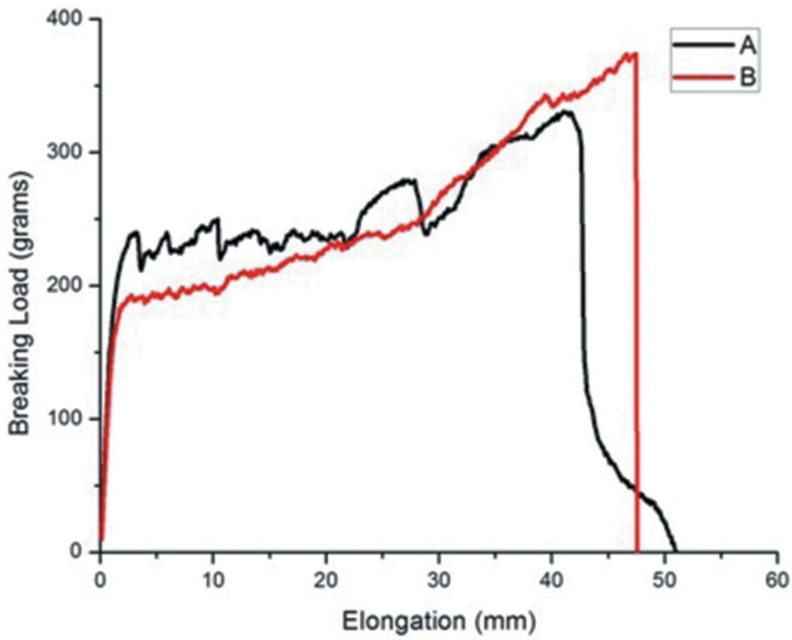

Figure 7. Tensile behavior of single palm fibers: (a) fiber breaking with stick slip effect and (b) fiber with regular breaking.

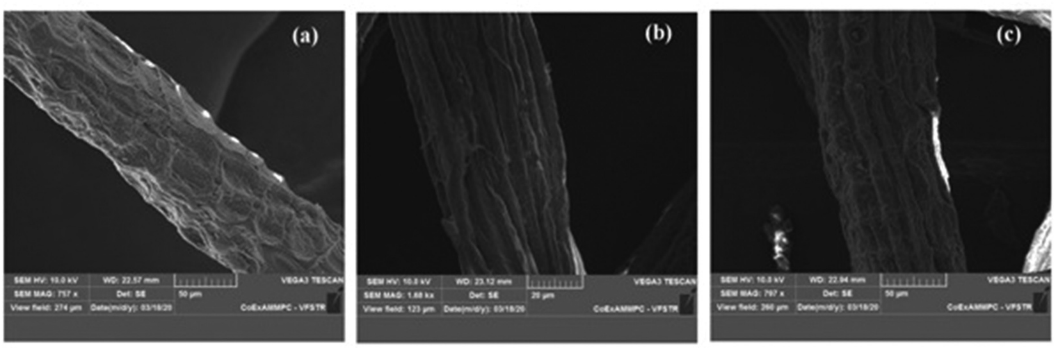

Figure 8. SEM images of palm fibers: pristine fiber (a), bleached (b), and dyed fiber (c).

Further increase in the alkali concentration leads to decrease in the tensile strength, which has also been evident in the other research work (Mwaikambo and Ansell 2002). The same pattern was observed in case of alkaline pre-treated palm fibers after bleaching and dyeing. The mean breaking elongation % is found to be in increasing order from 30.23% (RF) to 40.24% (4% alkali treated) fibers. This is due to the removal of impurities which induces structural changes that make the fiber higher in elongation at break and the same phenomenon was reported by other author (Moghaddam and Mortazavi 2016). Figure 6a shows the mounting of fiber in the instrument and its breaking phenomena i.e. fiber fracture with the increment of load. During the tensile measurement apart from the fiber breaking, fibrillations were also noticed which is clearly shown in the Figure 6b and the close view of the fiber fracture is shown in the Figure 6c.

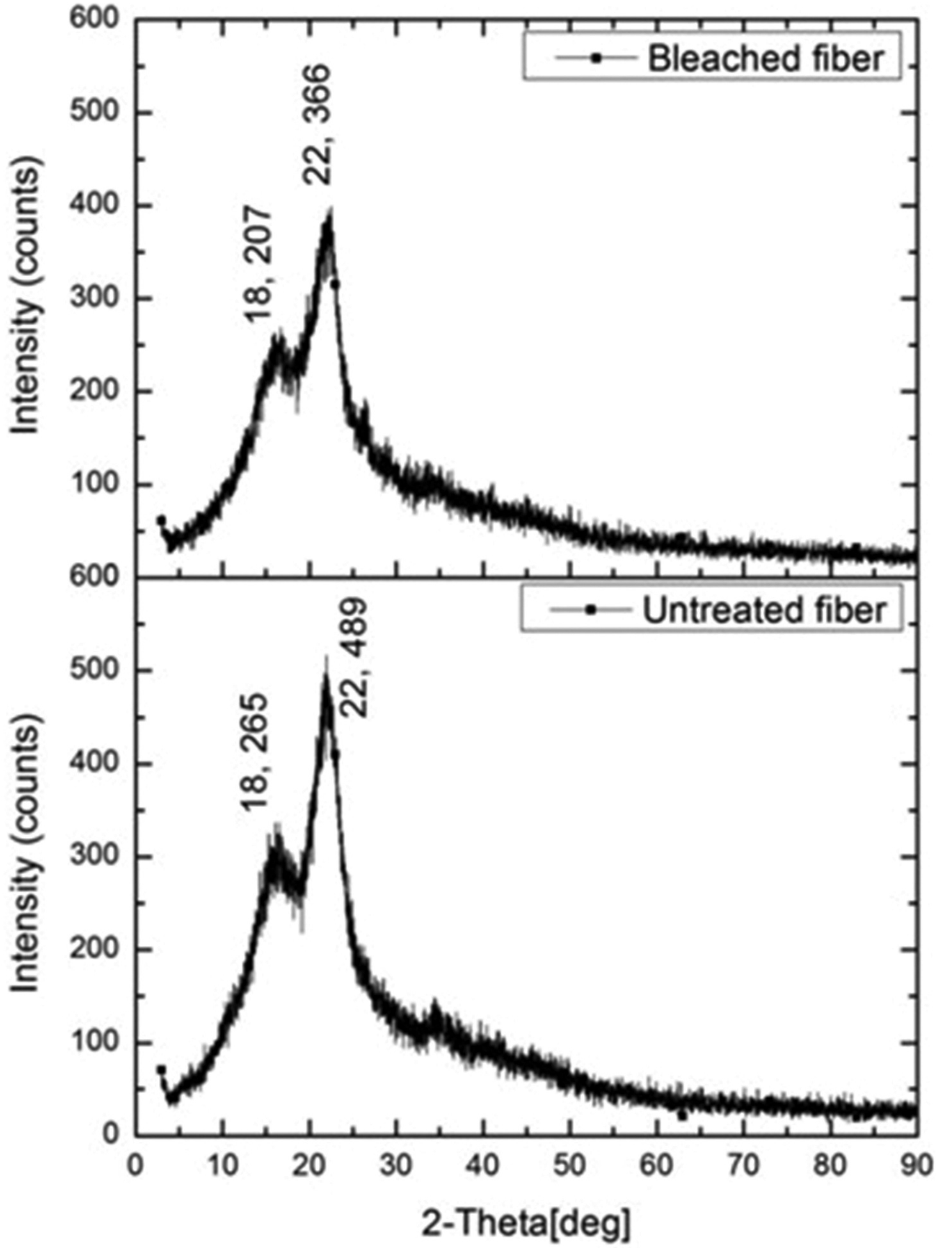

Figure 9. X-ray diffractograms of untreated and 5% scoured/bleached palm fibers.

The nature of fibrillation in the fiber during its fraction leads (Periyasamy 2020) to stick slip effect which is given in Figure 7. This phenomenon was not noticed with all the fibers and few fibers observed with the forming the fibrillations during the fiber rupture. The load vs elongation curves of such fibers A and B were taken and plotted a graph (Figure 7). In the case of fiber 'A' with the increment of load, the fiber elongates in normal way and it gets breaks. But in case of fiber 'B', it was notices that with the increase in load, the fiber extends and it slips which propagates up to certain

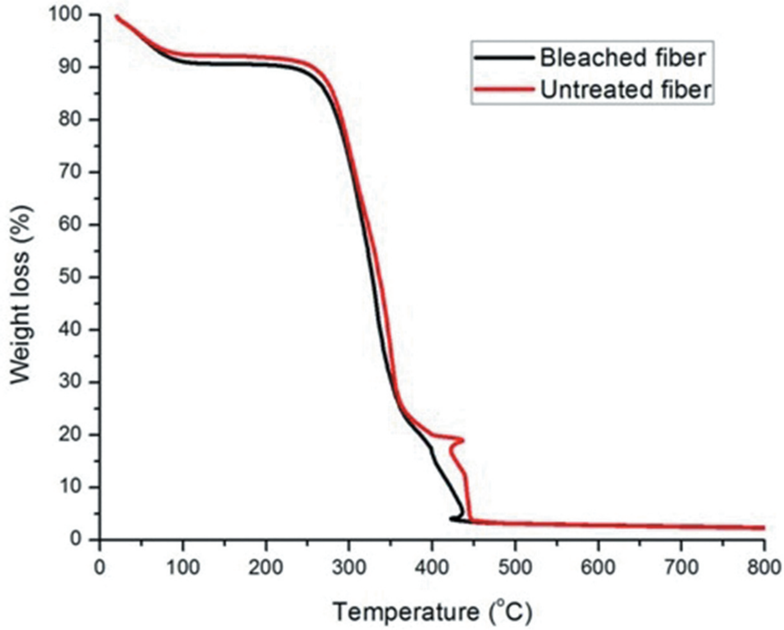

Figure 10. Effect of change in temperature on weight loss of raw and bleached palm fibers.

distance and then fiber extends normally. This happened frequently due to the formation of fibrils at the point of fiber fracture and the fiber break occurs compared to fiber 'A.' This is also one of the reasons for higher CV% in the tensile results which is given in Table 3.

SEM analysis

The SEM images of raw, bleached, and dyed fibers were shown in Figure 8. The raw fibers are not uniform and it is due to the presence of impurities such as fatty acids and waxes. Additionally, the fiber surface was also observed the rough surface due to the presence of lignin which is clearly seen in Figure 8a.The alkali treated and subsequent bleaching process improves the fiber surface morphology by removing the impurities and some extent lignin, due to that the surface roughness was reduced (Figure 8b). Figure 8c shows the dyed fiber surface.

XRD

The X-ray diffractograms of raw and 5% scoured/bleached palm fibers are shown in Figure 9. It was observed from the figure that there are two main peaks at 18° and 22°. Out of these two reflections, the lower angle (18°) is broad and corresponds to the amorphous region of the fiber. The higher angle (22°) is sharp and intense and attributed to the crystalline region of the fiber. There is an increased pattern of crystalline % was observed till 4% scoured and bleached samples. Similar pattern was observed for other plant based fibers (Rajeshkumar, Hariharan, and Scalici 2016; Suryanto et al. 2014). But in case of 5% bleached palm fibers, the crystallinity % was found 63.8% which is slightly less, but not significant compared to raw palm fibers, i.e. 64.8%. This may be due to the removal of amorphous content in the fiber and rearrangement of fiber structure. The difference was found to be smaller and does not have greater significance in the tensile properties of fibers.

Thermogravimetric analysis (TGA)

The extracted palm fibers were subjected to TGA before and after the pre-treatment, weight loss % of fibers toward the temperature is given in Figure 10. It was evident from the figure that the fiber decomposition was observed at different stages. The moisture loss was found at 80–100°C, cellulose decomposed between 240–350°C, hemicelluloses at 200–260°C, and lignin at 250–460°C (Adeniyi et al. 2019; Obi Reddy et al. 2013). In addition, there is a difference of 1.5% in the rate of decomposition from raw to bleached palm fibers, i.e. compared to raw fiber, the bleached fiber decomposed quickly due to the removal of impurities from the fiber.

Conclusion

In this investigation, the Borassus palm fine fibers were extracted and treated with 2, 3, 4, and 5% of alkali for 60 min at 90°C. Alkali treatment improves the tensile strength from 54.08 MPa to 63.43 MPa, and the elongation of individual fibers were increased from 30.23% to 38.35% through removal of impurities on surface and improving the fiber texture, at the 4% concentration. Increase in concentration of alkali from 4% to 5% leads to decrease in tensile strength from 63.43 MPa to 62.65 MPa. It is also concluded that slight improvement in the crystallinity at 4% alkaline-treated fibers. But increasing the alkali concentration to 5% leads to reduction of the crystallinity to 63.8% compared with 64.8% for raw palm fiber. Some fibers exhibit fibrillation which was not reported till now and the alkali treatment shows little or no significant effect on TGA and shows good strength, which shows that the fibers are not being damaged. The fibrillation is being observed in the few fibers, but not uniform with all the fibers. It is because of the structure and maturity of fibers is not uniform. These fibers can be spun into yarns easily after alkali treatment and in the present study which is optimized at 4% alkali.

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