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Effect of alkali treatment on physical properties of banana fibre

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Banana fibres have been treated with NaOH at 10, 15 and 20% concentrations and its effect on chemical composition, and physical, morphological, structural and thermal properties are studied. The raw and alkali-treated fibres have been investigated for diameter, density, moisture regain, colour, surface characteristics, functional groups analysis, crystallinity of fibres and thermal behavior. The treated fibres show improvement in cellulose content, lignin removal, tenacity, crystallinity, and thermal resistance till 10% concentration, and after that no improvement is observed beyond 15%. Colour has turned the fibre towards darker side slightly aesthetically unappealing. It is noted that the 15% NaOH concentration is optimum for treating banana fibres used as reinforcement.

Keywords: Alkali treatment, Banana fibre, Single fibre strength, Thermal analysis

1 Introduction

The use of natural lignocellulose fibres in the manufacture of composites is increasing day by day[1]. Natural lignocellulose fibres, such as sisal, hemp, coir, kenaf and jute, have low density, good thermal properties, better specific strength, ecofriendly nature and can be used as replacement for glass fibres during composite manufacturing[2,3]. Banana fibres, due to its high specific strength can be used as replacement of glass fibres as reinforcement in the manufacture of composites. Banana fibre is extracted from the pseudo stem waste of the plant after harvesting the fruits. Like any other ligno cellulosic fibre, the major constituents of these fibres are cellulose, lignin, and hemicellulose[4]. The removal of lignin and hemicellulose substances and roughening of surface is required to improve the interfacial strength of composites[5]. Banana fibres are hydrophilic and have poor adhesion with the matrix materials. To improve adhesion of the natural fibres with matrix materials various treatments, such as alkali, acetylation and benzoylation, are given. Among all these treatments, mercerization or alkali treatment is a versatile one, which brings about changes in dimensions, fine structure, chemical composition, morphology, and crystalline component as well as it improves the wettability, resin pick up of natural fibres like coir, sisal, flax and cotton[6,7]. Alkali treatment improves adhesion between these hydrophilic fibres and the hydrophobic matrix by roughening and exposing more cellulose on the surface. The alkali treatment modifies the chemical constituents which results in changes in mechanical properties, surface roughening, crystallinity and also in the thermal behavior of fibres[8,9].

Available literature reveals that NaOH treatment removes the binding materials, depending on the treatment time, concentration of NaOH used, temperature of treatment and liquor ratio. Study on effect of NaOH treatment on properties of banana fibre has already been performed by few researchers[10-12]. The effect of NaOH concentration on fibre yield (extraction) from the pseudo stem has also been reported[13]. However, the changes in the fibre due to such treatment at various levels are unknown. This is essential for optimum use of alkali and also to avoid degradation of fibres which will happen at higher concentration treatments. Similarly, the effect of NaOH concentration on the other fibre properties is not yet been studied. Hence in this study, the banana fibres are treated with sodium hydroxide (NaOH) at three different concentrations of 10, 15 and 20% and the treated fibres are tested for chemical composition and physical, morphological, structural and thermal properties.

2 Materials and Methods

2.1 Materials

Nendran varieties (*Musa paradisiaca*) stem were fed to banana fibre extraction machine to extract

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banana fibres, which were then dried in shade for 5 days at room temperature. All the chemicals used were of laboratory grade and sourced from Merck, India.

2.2 Methods

2.2.1 Alkali Treatment of Banana Fibres

Banana fibres were treated with alkali at three different concentrations (10, 15 and 20% owm). Treatment was done at 80°C maintaining material-to-liquor ratio of 1:30. The fibres were immersed in the alkali solution for 3 h continuously. The fibres were then washed thrice with hot water and several times with distilled water to remove any NaOH sticking to the fibre surface, till the *p*H was 7. The fibres are then dried at room temperature (25°C) for 24 h.

2.2.2 Determination of Fibre Contents

The α-cellulose and lignin content were determined by TAPPI standard test methods T 212-OM-93 and T 203-I-74. The moisture content was determined as per ASTM D2654-89a Standard Test method for Moisture in Textiles**.**

2.2.3 Determination of Fibre Diameter, Density and Colour

The diameter of the fibres was measured in at least 10 different locations using a polarized microscope with ×100 magnification, interfaced with a computer (Model: Leica DM 750P with DFC 295 Digital Camera). The average of these ten measurements was calculated and used in this study.

Density of the untreated and treated banana fibres was measured according to ASTM D 1505 – 03, Standard Test Method for Density of Plastics by the Density-Gradient Technique. Xylene and carbon tetra chloride were used as solvents for the determination of density.

The colour of the fibres was determined using spectrophotometer (Premier Colour Scan Model SS5100A, Mumbai, India). The colour strength of the fibre was obtained using the colour parameters, such as L, a, b, H and ΔE values.

2.2.4 Determination of Tensile Strength of Fibre

Tensile properties of the untreated and treated banana fibres were measured according to the standard test method for tensile properties of single textile fibre (ASTM D 3822 – 01). The test was performed in a tensile tester (model -Zwick Roell, Germany) using 1kN load cell at 5 mm/min crosshead speed and 10mm gauge length. Twenty numbers of fibres were tested for each raw and treated banana fibre.

2.2.5 Surface Characterization of Fibre

The surfaces of raw and treated banana fibres were observed using scanning electron microscope (SEM) model JEOL-JSM-6396. Prior to the test, the samples were coated with a thin layer of gold by a plasma sputtering apparatus. The observation was performed in high vacuum mode with secondary electron detector and accelerating voltage between 5 kV and 10 kV.

2.2.6 Thermal and Crystallinity Analysis of Fibre

Thermo gravimetric analysis (TGA) and DTA of all the banana fibre samples were done using Netzsch STA equipment at a heating rate of 10°C/min, from room temperature (25°C) to 400°C, in nitrogen atmosphere. The crystallinity of untreated, alkali-treated and enzyme treated banana fibres were evaluated by X-ray diffraction (Make: Shimadzu, Model XRD-6000, 2006). The scan range of 2θ was 10 - 45° where θ is the diffraction angle and the scan speed used was 5°/min. The crystallinity percentage of the fibre was calculated according to the Segal empirical method as given below:

$$\text{Xcr}\ (\%) = \frac{(I_{002} - I_{am})}{I_{002}} \times 100$$

The sample crystallinity (Xcr) has been determined by means of equation using the height of the peak, where I_{002} is the maximum intensity of the peak at $2\theta = 22°$ and Iam is the height of the minimum (Iam) between the 002 and the 101 peaks, which is due to amorphous region of the sample.

3 Results and Discussion

3.1 Chemical Composition and Moisture Regain of Banana Fibres

Chemical composition of fibre influences the properties of fibre. Higher cellulosic content gives better strength to the fibre. The alkali treatment removes the non-cellulosic content specifically lignin. The lignin removal improves the cellulose content in a fibre, which is a major factor in determining the strength of the fibre[14]. Table 1 shows the chemical composition of untreated as well as banana fibres treated with NaOH at three different concentrations of 10, 15 and 20%. It is observed that the untreated fibre contains nearly 70% of cellulose content which makes

Table 1 — Chemical composition and other properties of untreated and NaOH treated banana fibres

Chemical composition	Untreated fibre	10 % NaOH	15 % NaOH	20 % NaOH
Lignin, %	17.83	14.19	11.21	12.90
Hemicellulose, %	11.10	8.2	5.05	5.90
Cellulose, %	69.53	75.70	81.95	79.13
Other impurities, %	1.54	1.91	1.79	2.07
Moisture regain, %	13.36	12.05	11.23	14.19
Fibre density, g/cc	1.32	1.42	1.45	1.43
Fibre diameter, μm	377.93	297.40	259.64	216.57
Fibre diameter CV %	25.19	20.29	18.29	14.84

it a suitable raw material to produce composites with adequate strength and durability.

The important modification done by NaOH treatment is the disruption of hydrogen bonding, which removes the lignin, hemicellulose and other non-cellulosic contents. These compositions are usually the contents of the fibre cell wall and cover the surface of the fibre. The results indicate that the lignin removal is 20, 37 and 28% for NaOH concentration of 10 15 and 20% respectively. The increment in the concentration of NaOH decreases the lignin content up to the 15% concentration, and for 20% NaOH, the lignin removal percentage is comparatively less than that at 10% NaoH treatment. The hemicellulose removal is observed as 26, 54.5 and 44.1% for NaOH concentration of 10, 15, 20% respectively. Due to the removal of lignin as well as the other non-cellulosic impurities, the cellulose % of all the treated fibres is found higher as compared to untreated fibre. The cellulose % increase is found 10, 18 and 14% for NaOH concentration of 10, 15 and 20% respectively. The treatment with 15% NaOH provides highest cellulose content (82%). The reduction in cellulose content at 20% NaOH concentration might be due to the cellulose degradation at higher concentration of NaOH.

Table 1 shows the moisture regain of untreated and NaOH treated banana fibres. There is a decrease in the moisture regain from 13.36% for untreated to 12.05% for the 15% NaOH treated fibres, which are due to the removal of amorphous lignin and hemicellulose. At 20% NaOH concentration, there is an increase in moisture regain to 14.19%, which may be due to the degradation of cellulose. The increase in hydrophobicity of the fibre till 15% alkali treatment will aid better wetting by a hydrophobic resin during the development of composites[15].

3.2 Effect of NaOH Treatment on Density and Diameter

Table 1 also shows that the fibre density and diameter change after the NaOH treatment. The results show that the fibre diameter value decreases steeply from 377 micron to 216 micron from untreated to 20% NaOH treated fibres, this size reduction might be attributed to the removal of lignin and other non-cellulosic material from the fibres[16]. It can also be noted that the CV% of diameter of the fibres is decreased as the treatment concentration increases. This is because, fibrillation takes place between fibres during the alkali treatment and the fibres are individualized from each other. As the treatment concentration increases, fibre individualization improves and therefore variation in fibre diameter gets reduced.

Density of the treated fibre is increased from 1.32g/cc for untreated fibre to 1.45g/cc for 15% alkali treatment. The increase in density is due to the removal of low dense non-cellulosic materials, and for 20% alkali treatment, density decreases slightly to 1.43g/cc, which may be due to degradation of cellulose. The findings of the density analysis results support the chemical composition analysis.

3.3 Effect of NaOH Treatment on Surface Morphology of Banana Fibre

The SEM images of the untreated and NaOH treated banana fibres are shown in Fig. 1. They reveal that the surfaces of the untreated fibres are covered with a layer of substances which may include lignin, hemicellulose and other non-cellulosic contents. Ten percent NaOH treated fibres show few ridges between fibres and roughness on the surface, confirming partial removal of non-cellulosic contents. Again, 15% NaOH treated fibres show ridges between the cellulosic fibril due to further removal of the non-cellulosic material. Twenty per cent NaOH treatment indicates the clear ridges and clean rupture of the cellulosic substances on the surface, which coincides with the chemical composition and density results of degradation of cellulose. The removal of the non-cellulosic content and the improvement in the surface roughness of the fibres will facilitate better interlocking with resin and the matrix[17].

3.4 Effect of NaOH Treatment on Single Fibre Strength of Banana fibre

Table 2 shows the results of tensile strength of the alkali-treated and untreated fibres. The alkali treatment shows increase in tenacity by 7 and 15% for 10 and 15% alkali treatment respectively due to the

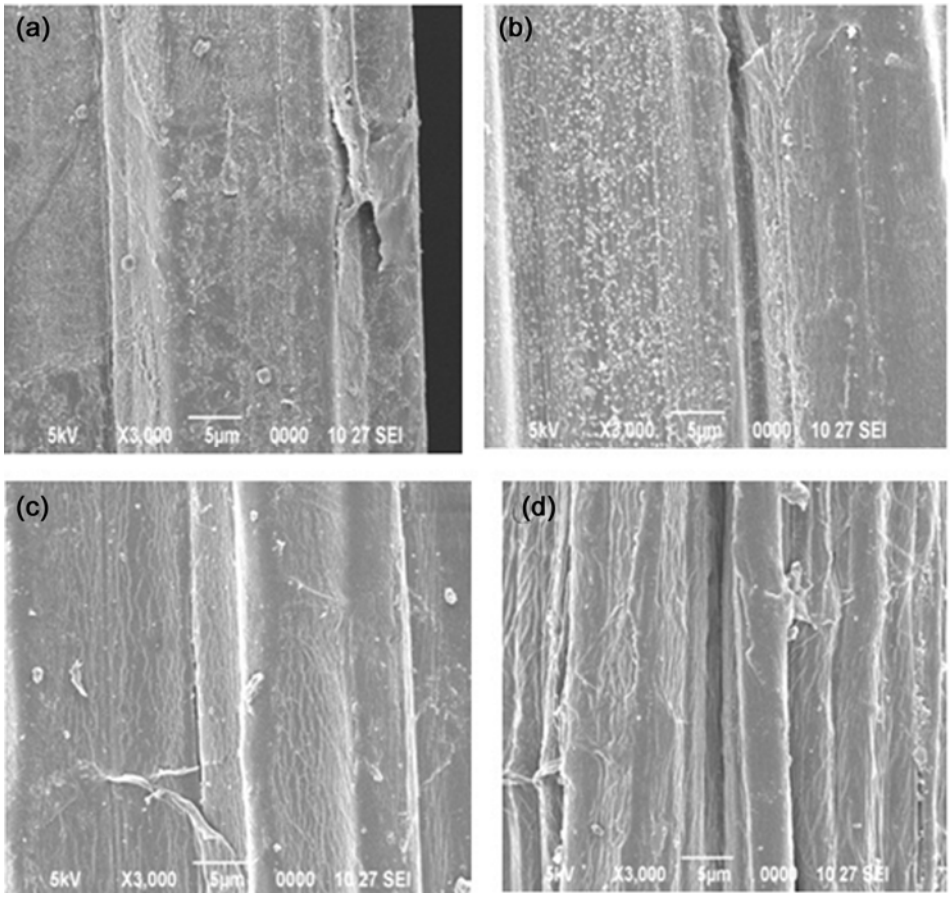

Fig. 1 — SEM images of (a) untreated, and (b) 10%, (c) 15% and (d) 20% NaOH treated banana fibres

Table 2 — Tensile Properties of untreated and treated banana fibres

Property	Untreated	10 % NaOH	15 % NaOH	20 % NaOH
Breaking tenacity, g/tex (average)	30.26	32.51	35.02	33.37
Breaking tenacity, CV%	11.81	10.7	10.1	11.1
Elongation, %	2.51	1.99	1.51	1.71
Elongation, CV%	8.24	7.33	7.8	7.1
Tensile strength, MPa	412.48	452.72	501.41	467.97
Young's modulus, GPa	16.43	22.75	33.21	27.37

removal of hemi-cellulose, lignin and other non-cellulosic materials, which are amorphous in nature and have low strength[18]. The removal of hemicelluloses and non-cellulosic material allows the cellulose chains to align in the direction of loading and thus increases the fineness and strength of the fibre[19]. The increase in Young's modulus is 38% and 102% for 10% and 15% NaOH treatment respectively. There is a slight decrease in the elongation (< 2%) for all the treated fibres. At 20% alkali treatment due to the degradation of cellulose, there is a reduction in tenacity by 10% and Young's modulus by 66%, when compared with 15% NaOH treated fibres.

3.5 Effect of NaOH Treatment on Colour of Banana Fibre

The colour values of untreated and NaOH treated banana fibres are shown in Table 3. There is a significant change in colour, which is evident with higher ΔE values for 10, 15 as well as 20% NaOH treated fibres. The decrease in L value of the treated fibre indicates that the fibres have become darker, as also evident from the visual observation. The fibres are towards redder side as the values of 'a' are positive and the redness has also gradually increased due to increase in NaOH concentration from 10% to 20%. The increase in 'b' value shows that the fibre is in the yellower side and the yellowness has increased gradually from 10% to 20% NaOH concentration. Also, there is an increase in the hue value. It shows that the fibres become aesthetically poor as the NaOH treatment concentration increases.

3.6 FTIR Analyis of NaOH Treated Banana Fibre

The FTIR results of untreated and NaOH treated banana fibres are given in Fig. 2. The C-H stretching of the glycosidic linkages are indicated by the peaks at 893 cm^{-1}, and there is reduction in the peaks at 20% NaOH treatment, which is a similar to the findings of Oh *et al*[20]. The C-O stretching of alcohol present in cellulose, lignin and hemicelluloses are indicated by the peak at 1055 cm^{-1}, and its intensity reduction shows the removal of cellulose, lignin and hemicelluloses from the fibre[21]. The lignin removal by the action of NaOH is also observed by reduction in intensity of peaks at 1250 cm^{-1} due to reduction in aryl group C-O, which is a similar case for hemp treated with NaOH[22]. The peak at 1429.25 cm^{-1} is due to the C-H bond in aromatic ring of lignin[23], which have shown lowered intensities, indicating the removal of lignin and hemicellulose contents in the fibres.

The C=C stretching of alkenes present in lignin is indicated by the peak at 1643.35 cm^{-1}, and the reduction in its intensity shows the removal of lignin, as in case of NaOH treated bamboo fibre[21]. The reduction in the peak intensity at 2922 cm^{-1} is due to the C-H stretching vibration in cellulose and hemicelluloses. The reduction in the peak intensity shows hemicelluloses removal. The reduction of OH groups that are present in the cellulose, hemicellulose and lignin is indicated by the reductions in intensity of the peaks at 3373 cm^{-1}, as already reported [24,25]. Here removal of hemicellulose and lignin has resulted in the decreased intensity of these peaks. Thus FTIR analysis confirms the lignin and hemicellulose removal.

Table 3 — Colour values of untreated and NaOH treated banana fibre

NaOH concentration, %	L	a	b	c	H	ΔE
Nil (Untreated)	69.393	4.401	17.747	18.285	76.042	-
10	57.359	5.308	18.18	18.939	73.694	12.0759
15	56.385	4.877	21.371	21.92	77.114	13.51177
20	48.981	6.254	22.875	23.715	74.679	21.1277

Table 4 — Percentage of crystallinity for untreated and NaOH treated banana fibres

NaOH concentration, %	I_{22} (at 2θ scale)	I_{18} (at 2θ scale)	Crystallinity %
Nil (Untreated)	270	184	59
10	294	160	65
15	386	172	69
20	296	165	64

3.7 X-Ray Diffraction Analysis of Untreated and NaOH-treated Banana Fibres

The X-ray diffraction for 2θ values from 10° to 80° for untreated and NaOH treated fibres are shown in Fig. 3. The intensity at 2θ = 22° (I_{22}) and 18° (I_{22}) for the crystalline and amorphous peaks respectively as well as crystallinity index are given in Table 4. All the NaOH treated fibres show a higher crystallinity percentage. The removal of the amorphous hemicellulose and lignin content by alkali treatment should have improved the crystallinty as also observed in previous studies[26]. At higher alkali concentration (20%), there is a reduction in crystallinity % from 69 (15% NaOH) to 64. This might be attributed to the cellulose degradation of the banana fibre.

3.8 Thermal Analysis of NaOH Treated Banana Fibres

TGA analysis is also carried out to find out the effect of various treatments on the weight loss in the fibres at temperature range 0 - 400°C, where a normal

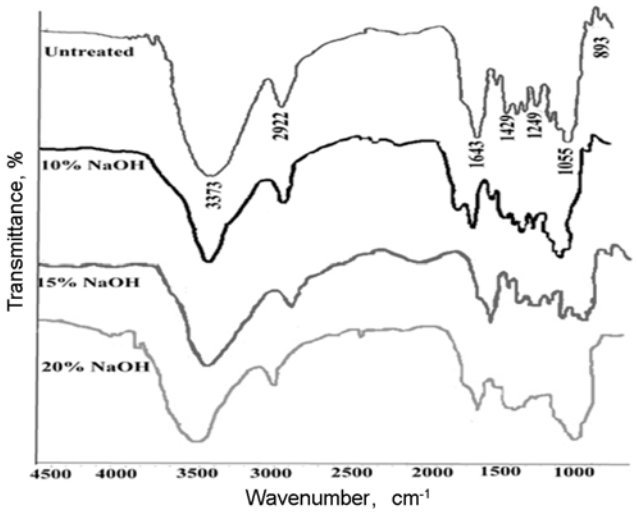

Fig. 2 — FTIR of untreated and NaOH treated banana fibres

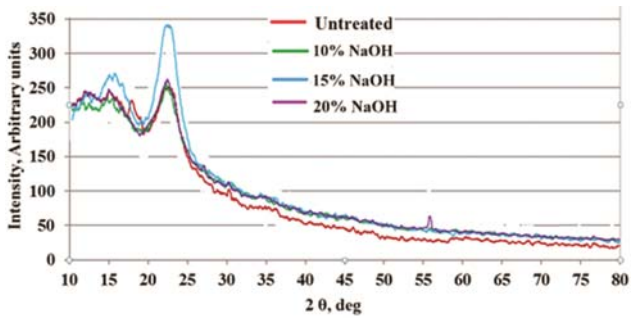

Fig. 3 — X-Ray diffraction pictures for untreated and NaOH treated banana fibres

Table 5 — Thermal analysis of untreated and NaOH treated banana fibre

NaOH concentration, %	ΔT, °C	Mass loss %	Residual mass at (400 °C), %
Nil (Untreated)	29 – 103	12.5	67.7
	103 – 222	1.5	
	222 – 397	53.7	
10	29 – 103	6	64
	103 – 226	6	
	226 – 397	52	
15	29 – 103	6.8	62
	103 – 252	6.2	
	252 – 397	49	
20	31 - 100	8	63
	100 – 222	3.3	
	222 – 397	51.7	

thermoplastic material is usually subjected. As shown in Fig. 4 (a), the thermal degradation can be divided into three distinct regions, which are given in Table 5. The first region is between 29°C and 103°C; where there is a weight loss in fibres due to the moisture evaporation from both the untreated and NaOH treated fibres. The first region shows that the hydrophobicity of the fibre has increased due to the removal of noncellulosic materials like lignin, as the weight loss is lower for the treated fibres as compared to that of the untreated fibres. All the NaOH treated banana fibres show better thermal stability in the second region, which is evident from the lower weight loss, as compared to that of the untreated fibre. Previous thermal studies, done for the NaOH treated hemp[27] and jute fibre[28], also show similar results. The third and the final region are between 290 °C and 400°C, where the weight loss is due to degradation of cellulose as well as the hemicelluloses. The final part in the third region is above 350°C, where the degradation occurs due to cellulose as well as the lignin degradation.

The degradation temperature is understood from the DTA graph [Fig. 4 (b)]. The degradation temperature for untreated banana fibre is 293°C, whereas for the 10% NaOH treated fibre and 15% NaOH treated fibres it has raised to 310°C and 329°C respectively, showing higher thermal stability. The 20% NaOH treated fibres show a lower degradation temperature of 298°C, showing lower thermal stability. The thermal analysis reveals that all the NaOH treated fibres have better thermal stability than the untreated fibres, and out of the NaOH treated fibres, the 15% NaOH treatment shows better thermal stability than 10% and 20% NaOH treatment.

4 Conclusion

It is found that the hemicellulose and lignin removal occur till 15% NaOH treatment concentration, and after that there is no much removal occurred. In line with those finding, the density of the fibre also shows an increment up to 15% NaOH and further increment in alkali percentage reduces the density. The moisture regain of the banana fibre also shows the same trend as that of density. At 20% NaOH treatment, the moisture regains are found higher than at 15% NaOH treatment. Increment in the NaOH concentration also increases the lignin and hemicelluloses removal percentage, which is confirmed by the scanning electron microscope analysis. The breaking strength and the tenacity values of the banana fibre increase with NaOH concentration up to 15%. Further increment in alkali concentration degrades the fibre. The removal of lignin and hemicellulose contents from the banana fibre is confirmed by the FTIR and crystallinity analyses. The thermal stability analysis also confirms that the alkali treatment improvs the thermal stability value up to 15% NaOH concentration. The colour assessment studies show that the appearance of banana fibre becomes darker and red in colour due to the alkali treatment.

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