

# Chemical Modification of Hemp, Sisal, Jute, and Kapok Fibers by Alkalization

LEONARD Y. MWAIKAMBO,<sup>1</sup> MARTIN P. ANSELL

Department of Engineering and Applied Science, University of Bath, Bath BA2 7AY, United Kingdom

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**ABSTRACT:** Plant fibers are rich in cellulose and they are a cheap, easily renewable source of fibers with the potential for polymer reinforcement. The presence of surface impurities and the large amount of hydroxyl groups make plant fibers less attractive for reinforcement of polymeric materials. Hemp, sisal, jute, and kapok fibers were subjected to alkalization by using sodium hydroxide. The thermal characteristics, crystallinity index, reactivity, and surface morphology of untreated and chemically modified fibers have been studied using differential scanning calorimetry (DSC), X-ray diffraction (WAXRD), Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM), respectively. Following alkalization the DSC showed a rapid degradation of the cellulose between 0.8 and 8% NaOH, beyond which degradation was found to be marginal. There was a marginal drop in the crystallinity index of hemp fiber while sisal, jute, and kapok fibers showed a slight increase in crystallinity at caustic soda concentration of 0.8–30%. FTIR showed that kapok fiber was found to be the most reactive followed by jute, sisal, and then hemp fiber. SEM showed a relatively smooth surface for all the untreated fibers; however, after alkalization, all the fibers showed uneven surfaces. These results show that alkalization modifies plant fibers promoting the development of fiber–resin adhesion, which then will result in increased interfacial energy and, hence, improvement in the mechanical and thermal stability of the composites. © 2002 Wiley Periodicals, Inc. *J Appl Polym Sci* 84: 2222–2234, 2002

**Key words:** renewable resources; modification; surfaces

## INTRODUCTION

### Natural Fibers

Natural cellulose-based fibers are increasingly gaining attention as their application is diversified into engineering end uses such as building materials<sup>1</sup> and structural parts for motor vehicles<sup>2,3</sup> where light weight is required. Low cost and less tool wear during processing are among

the known advantages of plant fibers, and ease of recycling makes them environmentally friendly. There are at least 1000 types of plant that bear usable fibers.<sup>4</sup> Apart from wood fibers, the main fibers of interest are listed in Table I.

Agricultural waste is another source of cellulose-based fiber. These include straw residues, coffee husks, bagasse, and many more. Apart from the single cell fibers such as cotton and kapok, retting and decorticating process extract most of the fibers. The steam explosion process (SEP) technique has recently been applied to extract plant fibers. The SEP involves the application of high temperature and pressure on cellulosic materials. The steam explosion process gives

Correspondence to: L. Y. Mwalkambo (msplym@materials.bath.ac.uk).

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**Table I Annual Production of Some of the Commercially Important Fiber Sources and Their Chemical Composition**

Fiber Type	World Annual Production (10 <sup>3</sup> Tonnes)	Cellulose	Hemicellulose	Lignin	Pectin
Cotton	18,450	92	6	—	<1
Jute	2,850	72	13	13	
Flax	830	81	14	3	4
Sisal	378	73	13	11	2
Hemp	214	74	18	4	1
Coir	650	43	<1	45	4
Ramie	170	76	15	1	2
Straw	—	40	28	17	8
Kapok	123	13			

clean fibers with rough surfaces and increased crystallinity index.<sup>5,6</sup>

#### Plant Fibers for Reinforcement of Polymers

The past 20 years have witnessed a high demand for plant fibers for reinforcement of polymeric matrix materials; this is reflected by the increased research and publications.<sup>4,7–9</sup> The polymer matrix materials used are divided into two main groups, namely thermosets and thermoplastics. The most widely used of the thermosets are the unsaturated polyesters, epoxies, and phenols. Polypropylenes, polyethylenes, and elastomers are the most common thermoplastic matrices. The thermosets are mostly polar while thermoplastics are generally nonpolar. Plant fibers, on the other hand, are highly polar due to the presence of the hydroxyl groups, which are readily available for the formation of hydrogen bonds with interacting, resin matrices. However, plant fibers are covered with pectin and waxy substances, thus hindering the hydroxyl groups from reacting with polar matrices, and forming mechanical interlocking adhesion with nonpolar matrices. To produce the reactive hydroxyl groups and the rough surface for adhesion with polymeric materials, plant fibers need to undergo physical and/or chemical treatment to modify the surface and structure.<sup>7,9</sup>

#### Chemical Composition of Plant Fibers

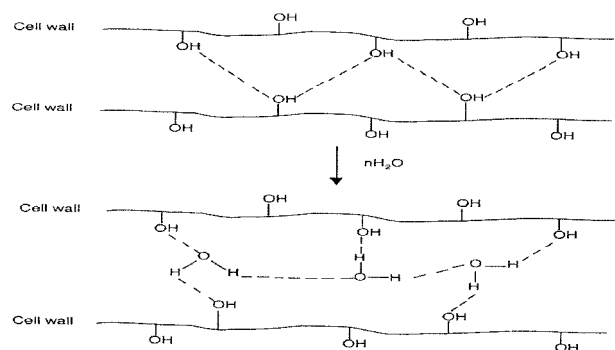
Plant fibers are composed of cellulose, hemicellulose, and lignin. Other components, usually regarded as surface impurities, are the pectin and wax substances shown in Table I. Eventually

chemical composition, including moisture content, can help to determine properties and end uses of the fibers. For instance, lignin facilitates reactivity, thus allowing better response to chemical modifications such as acetylation. Higher cellulose content leads to higher stiffness and, therefore, will be most suitable for resin reinforcement. The most important of the chemical components of plant fibers, especially in the manufacture of composites, is the cellulose.<sup>1,4,7,9</sup> The chemical structure of cellulose contains three hydroxyl groups, which in the macromolecular cellulose structure form hydrogen bonds. Two of these hydroxyl groups form intermolecular bonds, while the third one forms intramolecular hydrogen bonds.<sup>10</sup>

In this article, the literature on alkalization for chemical modification of natural fibers is reviewed. Four characterization techniques, namely DSC, WAXRD, FTIR, and SEM, have been used to elucidate the effect of chemical treatment on hemp, sisal, jute, and kapok fibers.

#### Plant Fibers Surface and Fine Structure

To understand the physical and chemical properties of materials and ultimately their end use, systematic characterization processes must be established and performed. For instance, the strength of materials is largely dependent on the chemical bond energy such as the C—C bond. In the natural fibers this also depends on the fineness or linear density of the fiber.<sup>11</sup> Similar characteristics such as the compatibility of fiber surface with the interacting chemicals such as resin, depends on the smoothness or roughness of the fiber. Rough surfaces increase the number of an-



**Figure 1** Schematic illustration of the swelling process in cellulose.

chorage points, thus offering a good fiber–resin mechanical interlocking.<sup>12</sup>

The presence of reactive groups, on the other hand, in both fibers and resins, is essential for high interfacial energy. All these parameters are prerequisites for the production of strong plant fiber composites. A compatible fiber–matrix interface results in better adhesion and better mechanical properties of the composites. The performance and stability of fiber-reinforced composite materials depends on the development of coherent interfacial bonding between fiber and matrix. In natural fiber-reinforced composites there is a lack of good interfacial adhesion between the hydrophilic cellulose fibers and the hydrophobic resins due to their inherent incompatibility.<sup>13</sup> Short, cellulose-based fibers will also tend to agglomerate making their use in reinforced composites less attractive. The presence of waxy substances on the fiber surface contributes immensely to ineffective fiber to resin bonding, as earlier stated, and poor surface wetting. Also, the presence of free water and hydroxyl groups, especially in the amorphous regions, worsens the ability of plant fibers to develop adhesive characteristics with most binder materials. High water and moisture absorption of the cellulose fibers causes swelling and plasticizing effects, resulting in dimensional instability and poor mechanical properties. This limits the application of plant fibers as reinforcement for polymeric materials.

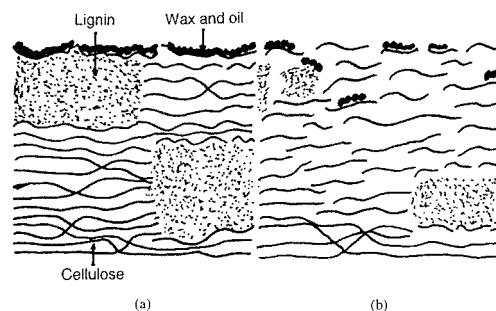
The hydroxyl groups ( $\text{—OH}$ ) in the cellulose, hemicellulose, and lignin build a large amount of hydrogen bonds between the macromolecules in the plant fiber cell wall.<sup>14</sup> Subjecting the plant fibers to humidity causes these bonds to break. The hydroxyl groups then form new hydrogen bonds with water molecules, which induce swelling (Fig. 1). The swelling of the cell wall exerts

very large forces. The theoretical value of these forces is about 165 MPa;<sup>14</sup> however, the actual swelling pressure is reported to be half of the calculated value.<sup>15</sup> Fibers are, therefore, usually subjected to treatment such as alkalization and acetylation at room or elevated temperatures to swell the cell wall to enable large chemical molecules to penetrate the crystalline regions. Acetylation of plant fibers is discussed in another publication.

The fine structure of cellulose materials is composed of crystalline and amorphous regions. The amorphous regions easily absorb chemicals such as dyes and resins, whereas the compactness of the crystalline regions makes it difficult for chemical penetration.<sup>11</sup> The modification of plant fibers may involve the removal of the surface impurities, the swelling of the crystalline region, and removal of the hydrophilic hydroxyl groups. The effective reinforcement of composites with plant fibers is dependent on moisture content, fiber–matrix interfacial adhesion, and crystalline and cellulose content. The alkalization of plant fibers improves their performance as composite reinforcement.

### Alkalization of Plant Fibers

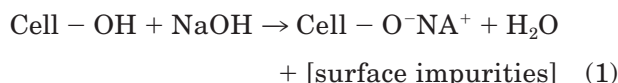
Natural fibers are chemically treated to remove lignin, pectin, waxy substances, and natural oils covering the external surface of the fiber cell wall [Fig. 2(a)]. This reveals the fibrils, and gives a rough surface topography to the fiber [Fig. 2(b)]. Sodium hydroxide ( $\text{NaOH}$ ) is the most commonly used chemical for bleaching and/or cleaning the surface of plant fibers. It also changes the fine structure of the native cellulose I to cellulose II by a process known as alkalization.<sup>16–19</sup> The reaction of sodium hydroxide with cellulose is shown in eq. (1).



**Figure 2** Typical structure of (a) untreated and (b) alkalinized cellulosic fiber.

**Table II First Exothermic Peak Temperature as a Function of Concentration of Alkali Treatment (Second Exothermic Peak Temperature in Brackets) for Hemp, Sisal, Jute, and Kapok Fibers**

NaOH (%)	Hemp	Sisal	Jute	Kapok
	Exotherm Peaks (°C)	Exotherm Peaks (°C)	Exotherm Peaks (°C)	Exotherm Peaks (°C)
0	357.00 (390.35)	365.31 (444.63)	369.48 (417.48)	359.50 (402.87)
0.8	377.03 (389.31)	356.96 (398.70)	359.05 (417.48)	370.53 (434.17)
2	369.50 (386.18)	351.75 (412.26)	354.88 (395.57)	352.79 (402.00)
4	364.27 (389.31)	353.24 (387.04)	346.55 (375.77)	367.00 (406.00)
6	363.22 (382.00)	347.57 (394.52)	349.66 —	341.31 (416.43)
8	362.18 (397.65)	350.74 (393.30)	351.75 (444.60)	353.96 (404.96)
30	359.05 (389.31)	350.74 (396.61)	342.36 (391.39)	343.40 (409.13)
400	363.22 (414.35)	348.23 (374.52)	349.66 —	359.05 (385.13)



It is worth pointing out that alkalization depolymerizes the native cellulose I molecular structure producing short length crystallites [Fig. 2(b)].

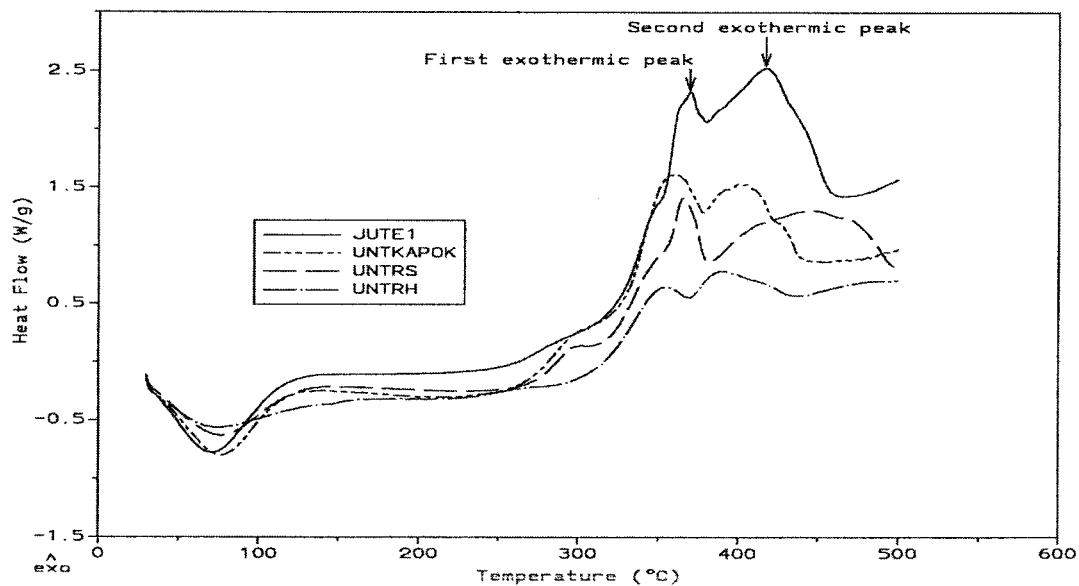
### Aims and Objectives

To properly assess changes at the fiber surface and fine structure due to chemical treatment by alkalization it is necessary to employ appropriate analytical characterization methods. A combination of two or more characterization techniques

allows a much more thorough investigation of the effect of chemical treatment on cellulose-based fibers. In this study, therefore, four characterization methods were employed. Wide-angle X-ray analysis (WAXS), differential scanning calorimetry (DSC), Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM) have been used to analyze the effect of alkalization on the crystallinity and thermal characteristics of four types of natural fibers namely hemp, sisal, jute, and kapok. These fibers are from three fiber origin; hemp and jute are bast fibers, sisal is a leaf fiber, while kapok represents seed fibers.

**Table III WAXRD Counts of Untreated and Alkalized Hemp, Sisal, Jute, and Kapok Fibers Obtained Close to 2θ Angles of 18.5° and 22.5°**

NaOH (%)		0	0.8	2	4	6	8	30	400
Hemp	18.5°	653.7	487.3	670.9	713.1	664.6	534.8	488.7	481.5
	22.5°	5391.7	4348.1	5966.2	5755.3	4936.7	4348.1	4778.5	2579.8
Sisal	18.5°	831.2	729.1	241.9	647.2	136.7	596.0	729.1	512.0
	22.5°	2856.5	3174.6	955.7	2047.5	639.5	2362.9	3243.1	1932.5
Jute	18.5°	607.75	734.2	636.3	717.7	200.2	787.6	278.5	84.1
	22.5°	2822.8	3160.3	2570.1	3231.7	1288.1	4866.4	1444.1	387.9
Kapok	18.5°	282.0	216.5	198.0	334.2	198.0	375.5	221.1	207.0
	22.5°	519.8	403.2	392.1	651.8	380.9	903.0	588.2	447.5



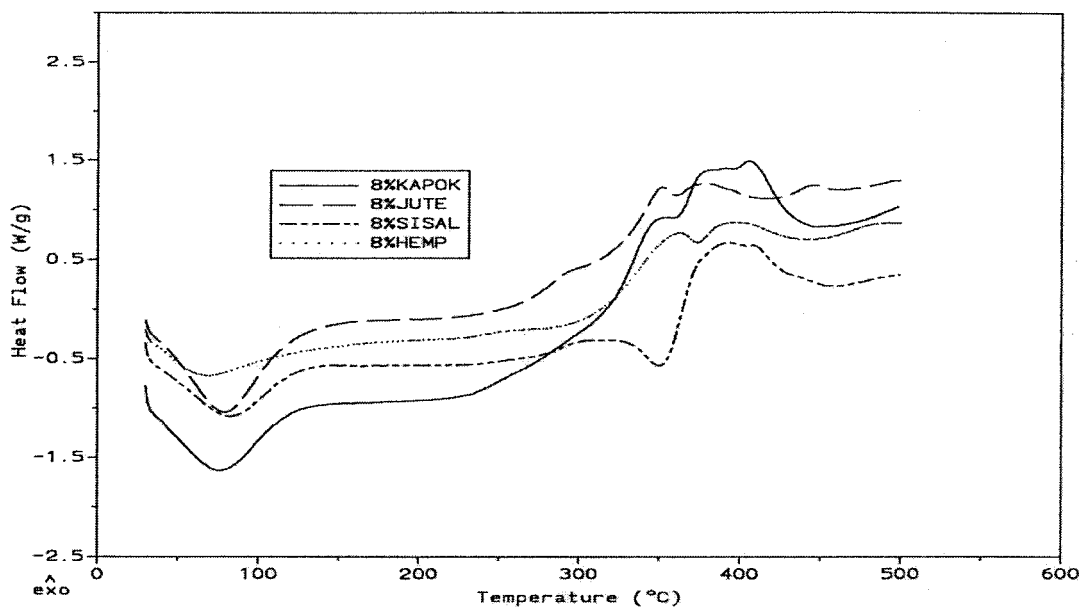
**Figure 3** DSC thermograms of untreated jute (JUTE1), hemp (UNTKAPOK), sisal (UNTRS), and kapok (UNTRH) fibers.

## EXPERIMENTAL METHODS

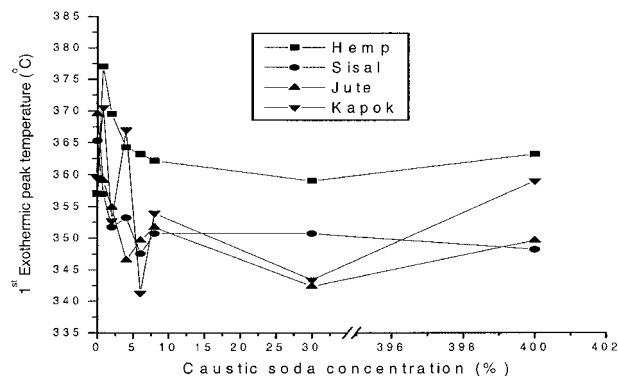
### Alkalization of Fibers

Kapok, sisal, jute, and hemp were soaked in beakers containing caustic soda concentrations as shown in Table II and placed in a water bath

controlled at  $20 \pm 2^\circ\text{C}$  for 48 h. The fibers were then removed, washed, with distilled water containing 1% acetic acid, to neutralize excess sodium hydroxide, and then thoroughly rinsed with distilled water. The fibers were then dried to remove free water and placed in a glass container in a conditioning chamber.



**Figure 4** DSC thermograms of alkalized Kapok, jute, sisal, and hemp at a selected caustic soda concentration of 8%.



**Figure 5** The effect of caustic soda concentration on the temperature of the first exothermic peak for hemp, sisal, jute, and kapok as observed by the DSC method.

### Differential Scanning Calorimetry of Fibers

A Du-Pont DSC 2910 equipped with TA modules was operated in a dynamic mode with a heating scheme of 30 to 500°C and heating rate of 10°C/min. in a nitrogen environment purged at 25 mL/min. The DSC elucidates the effect of alkali treatment on hemp, sisal, jute, and kapok fibers. The first exothermic peak is identified, and the temperature of this peak is used as a measure of the extent of fiber damage to the cellulose structure. The peak temperature is plotted vs. caustic soda concentration.

### Wide-Angle X-ray Diffraction (WAXRD)

Untreated and treated fibers were mixed with a very small amount of an adhesive material Tragacanth BP, soaked in a drop of distilled water, and compressed into thin sheets and dried. A wide-angle diffractometer equipped with a scin-

tillation counter and a linear amplifier was used. The diffraction intensities were recorded between 5 and 60° (2θ-angle range). The crystallinity index ( $I_c$ ) was determined by using eq. (2) below, where  $I_{(002)}$  is the counter reading at peak intensity at a 2θ angle close to 22° representing crystalline material and  $I_{(am)}$  is the counter reading at peak intensity at a 2θ angle close to 18° representing amorphous material in cellulosic fibers.

$$I_c = \frac{(I_{(002)} - I_{(am)})}{I_{(002)}} \times 100 \quad (2)$$

The use of WAXRD counts offers a simple and quick method of determining the crystallinity index and the minimum between 101 and 002 peaks (Fig. 6, Table III) is an indication of the reflection intensity of the amorphous material ( $I_{am}$ ).

### Infrared Spectroscopy of Natural Fibers

Infrared spectra were obtained using a Perkin-Elmer FTIR Spectrometer model PARAGON 1000. About 2 mg of fiber was crushed into small particles in liquid nitrogen. The fiber particles were then mixed with KBr and pressed into a small disc about 1-mm thick.

### Scanning Electron Microscopy (SEM)

SEM micrographs of fiber surface and cross sections of untreated and treated fibers were taken using a scanning electron microscope Model JEOL 6310. Prior to SEM evaluation, the samples were coated with gold by means of a plasma sputtering apparatus.

**Table IV** The Crystallinity Index of Untreated and Alkalized Hemp, Sisal, Jute, and Kapok Fibers obtained from Table IV

NaOH Concentration (%)	Hemp	Sisal	Jute	Kapok
	Crystallinity Index (%)	Crystallinity Index (%)	Crystallinity Index (%)	Crystallinity Index (%)
0	87.87	70.90	71.39	45.75
0.8	88.79	77.03	76.77	46.31
2	88.75	74.68	75.24	49.50
4	87.61	68.39	77.79	48.73
6	86.54	78.62	84.46	48.02
8	87.70	74.78	83.82	58.42
30	89.77	77.52	80.15	62.41
400	81.34	73.51	78.32	53.74

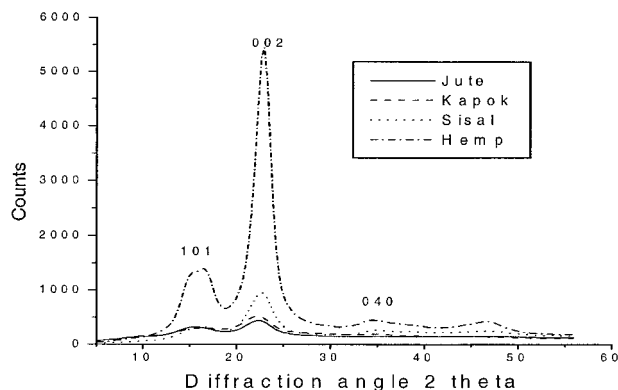


## RESULTS AND DISCUSSION

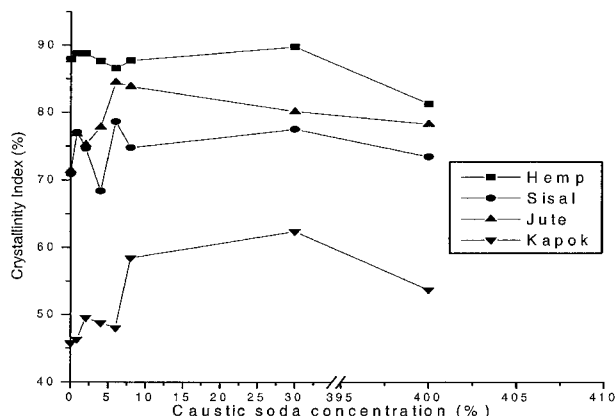
### Differential Scanning Calorimetry

The DSC technique is used to determine the drop in crystallinity and decomposition of plant fiber cellulose.<sup>19,20</sup> The results obtained in this work using the first exothermic DSC peak (decomposition) (Figs. 3 and 4, Table II) correspond well with the results obtained using the second and stronger endothermic peak (reduction in the crystallinity) found at around 350°C to assess the thermal degradation of crystallites in plant fibers.<sup>16,18–20</sup> To avoid repetitions only Figure 3 has been used to show the first and second exothermic peaks.

The sharp decrease in the decomposition temperature of the alkali-treated plant fibers between 0.8 and 8% NaOH concentration (Fig. 5) is an indication of the increase in amorphous cellulose, known to have poor thermal resistance, and the decrease in cellulose crystallite length. Several researchers share this view.<sup>7,19,21,22</sup> It is observed that more than 8% caustic soda renders the converted cellulose structure less stable to thermal degradation. Alkali-treated hemp fiber is more crystalline than sisal, jute, and kapok (Table IV), and was found to be more stable to thermal degradation as measured by the DSC. Some researchers report that alkali-treated plant fibers used as reinforcement in the manufacture of composites improve mechanical properties in comparison with fibers that have not been alkali-treated.<sup>6,7</sup> The application of 5% sodium hydroxide to cotton/kapok fabric for reinforcement of unsaturated polyester resin has been reported to increase tensile strength but decrease modulus of elasticity and impact strength.<sup>22</sup> The tenacity of alkali-treated Pima S-5, Giza 76, and Giza 77 cotton species



**Figure 6** WAXRD spectra of untreated jute, kapok, sisal, and hemp fibers.

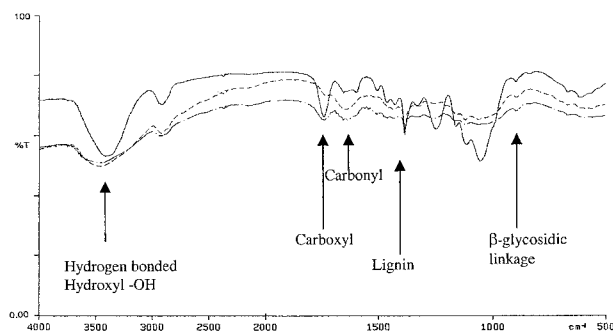


**Figure 7** Crystallinity index vs. caustic soda concentration for untreated and treated hemp, sisal, jute, and kapok fibers measured by WAXRD.

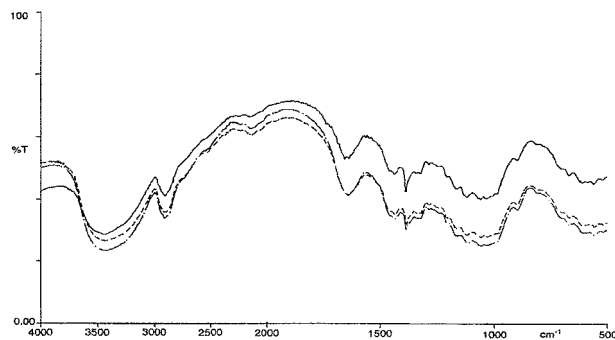
have been found to decrease while alkali-treated Giza 75, Giza 80 Dendara, Deltapine Smooth Leaf produced higher tenacity values.<sup>20</sup> This implies that the alkali-treatment of plant fibers can have different effects on the mechanical properties of fibers and also composite materials reinforced with these fibers.

### Wide-Angle X-ray Diffraction (WAXRD)

WAXRD results for alkali-treated fibers (Fig. 7), which show an overall initial increase in the crystallinity index of hemp, sisal, jute, and kapok, indicate improvement in the order of the crystallites as the cell wall thickens upon alkali treatment. Alkali treatment is reported to reduce the proportion of crystalline material present in plant fibers, as observed by several others.<sup>7,9,11,23</sup> The crystallinity index initially increases but then declines at high alkali concentrations when damage to the



**Figure 8** FTIR spectra of (—) untreated hemp fiber, (— · — · —) untreated sisal fiber, and (---) untreated kapok fiber.



**Figure 9** Infrared spectra of hemp fiber: (—) 0.8% NaOH, (— · —) 8% NaOH, and (— — —) 30% NaOH.

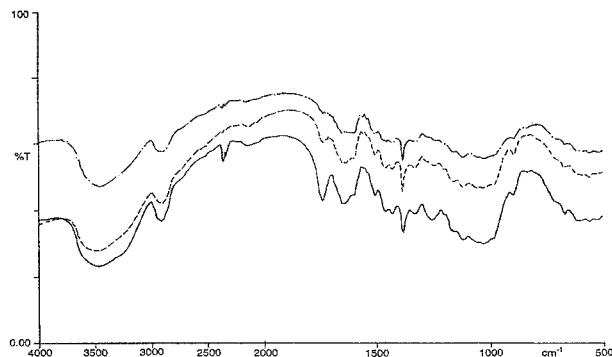
cell wall occurs. However, because alkalinization with and without tension increases the crystallite packing order; it is, therefore, logical to deduce that the crystallinity index measures the order of the crystallites rather than the crystallinity of the crystallites.

#### Fourier Transform-Infrared Analysis of Alkalinized Fibers

The absorbance peaks of interest in this study have been identified and shown in Figure 8. Alkali treatment reduces hydrogen bonding due to removal of the hydroxyl groups by reacting with sodium hydroxide. This results in the increase of the —OH concentration, evident from the increased intensity of the peak between 1000 and 1500  $\text{cm}^{-1}$  bands compared to the untreated fiber (Figs. 8–12). Absorbance between these ranges is indicative of the hemicelluloses. The hydroxyl groups are also involved in hydrogen bonding with the carboxyl groups, perhaps of the fatty



**Figure 10** Infrared spectra of sisal fiber (— — —) 2% NaOH, (— · — · —) 4% NaOH, and (—) 6% NaOH.

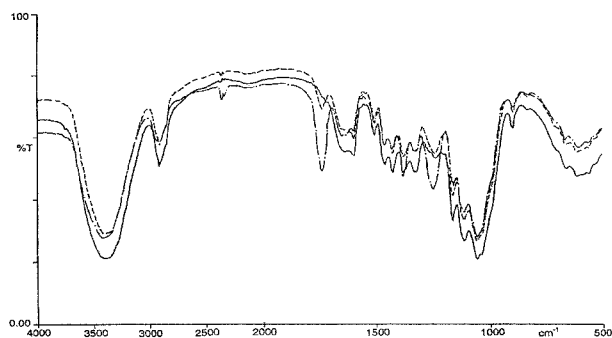


**Figure 11** Infrared spectra of jute fiber: (—) untreated, (— — —) 2% NaOH, and (— · — · —) 4% NaOH.

acids, available on the fiber surface of natural fibers. This is indicated by the reduction of the peaks between 3200–3600  $\text{cm}^{-1}$  (Figs. 9–12).

The peak between 1736 and 1740  $\text{cm}^{-1}$  seen in untreated fibers (Fig. 8) disappears upon alkali treatment. This is due to the removal of the carboxylic group by alkali treatment by a process called deesterification.<sup>24</sup> The carboxylic group may also be present in the fiber as traces of fatty acids present in oils. The reduction in the peak intensity found at around 1654  $\text{cm}^{-1}$  in alkali-treated hemp and kapok fibers indicates the partial reaction of the C=O bonds of hemicelluloses, whereas in sisal and jute it is completely removed, indicating that the hemicellulose component in these fibers is easily removed by alkalinization and that the hemicellulose in hemp and kapok fiber is less affected by caustic soda.

The FTIR spectra of untreated kapok fiber indicate that it contains more fatty acids than the other fibers studied due to the intensity of the 1596  $\text{cm}^{-1}$  peak (C=C stretching) followed by hemp, jute, and sisal. However, the peak at 1596



**Figure 12** Infrared spectra of kapok fiber: (— · — · —) untreated, (— — —) 6% NaOH, and (—) 30% NaOH.



**Table V Infrared Transmittance Peaks (cm<sup>-1</sup>) of Untreated Hemp, Sisal, Jute, and Kapok Fibers**

Bond type	Hemp (cm <sup>-1</sup> )	Sisal (cm <sup>-1</sup> )	Jute (cm <sup>-1</sup> )	Kapok (cm <sup>-1</sup> )
—OH stretching	3448	3447.2	3447.9	3419.7
C—H vibration	2920.5	2924.2	2918.8	2918.1
C=O stretching	—	1736.5	1737.2	1741.1
C=C stretching	1654	1653.9	1653.8	1596.1
C—H bending	1384.1	1384.1	1384.1	1383.6
C—H bending	—	1259.9	1255.6	1245.5
C—C stretching	1000–1162	1000–1162	1000–1162	1000–1162
C—H stretching	—	—	—	897.9
—OH	668.9	—	668.9	668.5

cm<sup>-1</sup> (C=C) disappears in alkali-treated kapok fiber (Fig. 12). This may be due to the removal of unsaturation present in the traces of oils. The observed peak at 1384 cm<sup>-1</sup> and between 1245–1259 cm<sup>-1</sup> (Fig. 8, Tables V and VI) indicates the presence of lignin and hemicellulose, respectively.<sup>9,24</sup> The peak at 1384 cm<sup>-1</sup> shows diminishing intensity as the fibers are subjected to higher concentration of caustic soda (Figs. 9–12).

The disappearance of the peak between 1245–1259 cm<sup>-1</sup> after alkalization indicates the complete removal of hemicellulose materials rather than lignin. This implies that hemicelluloses are easily removed by alkalization compared to lignin. The peak observed at 898–cm<sup>-1</sup> indicates the presence of the  $\beta$ -glycosidic linkages between the monosaccharides. The C—OH bending peak is observed between 610–668 cm<sup>-1</sup>. From these results it is clear that several reactions take place during alkalization.

### Scanning Electron Microscopy

Following alkalization the surface topography of hemp, sisal, and jute fibers is rougher than before

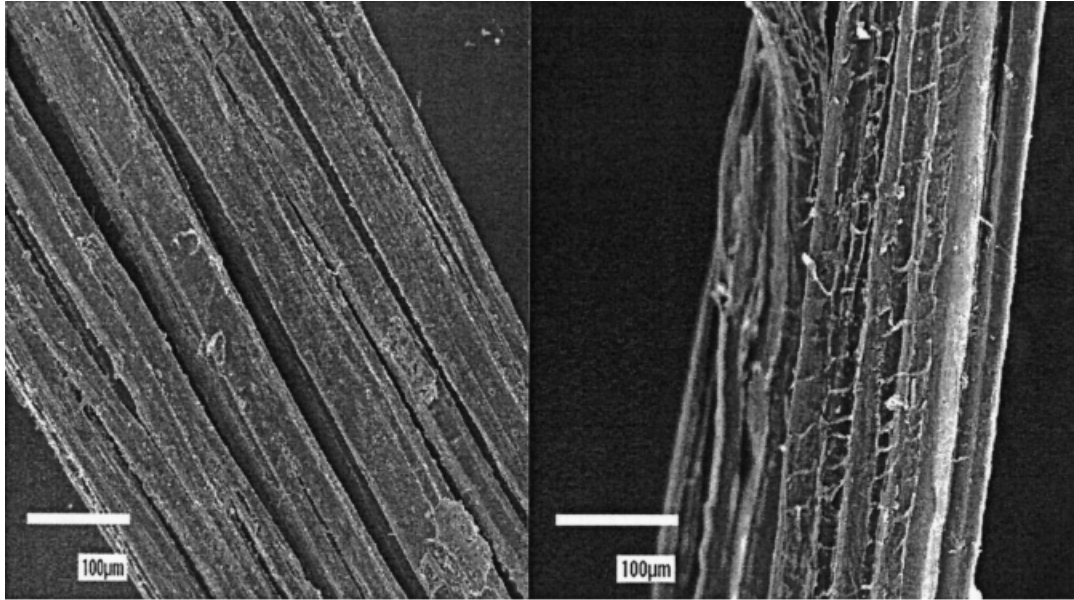
treatment (Figs. 13–16). SEM spectrographs of hemp, sisal, and jute shown in Figures 13–15, respectively, comprise bundles of individual cells that have been bound together by lignin-rich, weak intermolecular bonds. The individual cells of sisal fibers are discontinuous, comprising short lengths joined together end to end, whereas hemp and jute fibers have continuous cells. Individual cellular elements can be seen on the surface of clean jute fiber after alkali treatment. Hemp fiber shows partly separated individual cells before alkalization. Alkali-treated hemp fiber looks cleaner, and fiber bundles are more separated, with a highly serrated surface. The surface of kapok fibers appears to be unaffected by alkalization [Fig. 16(a) and (b)] except at the highest concentrations [Fig. 16(c)].

### CONCLUSIONS

Alkalization of plant fibers changes the surface topography of the fibers and their crystallographic structure. The removal of surface impu-

**Table VI Infrared Transmittance Peaks (cm<sup>-1</sup>) of Alkalized Hemp, Sisal, Jute, and Kapok Fibers**

Bond type	Hemp	Sisal	Jute	Kapok
—OH stretching	3447–3449	3447–3448	3448–3474	3407–3421
C—H vibration	2893–2901	2930	2915–2998	2916–2918
C=O stretching	Nil	Nil	Nil	1740 to nil
C=C stretching	1638–1654	1636	1636–1654	nil
C—H bending	1382–1384	1384	1384	1383 to 1381
C—H bending	1248–1250	Nil	Nil	diminished
C—C stretching	1000–1162	1000–1162	1000–1162	1000–1162
C—H stretching	892–897	898	895	897
—OH	667–670	—	668	609–668



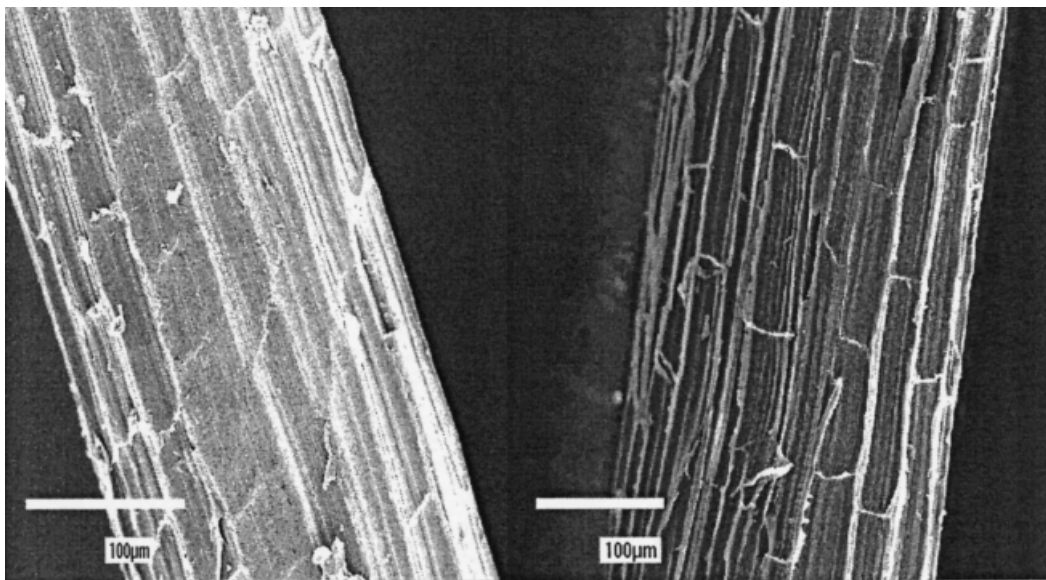
(a)

(b)

**Figure 13** SEM of hemp fiber (a) untreated and (b) 8% NaOH treated.

rities on plant fibers is advantageous in fiber-matrix adhesion, as it facilitates both mechanical interlocking and the bonding reaction due to the exposure of the hydroxyl groups to chemicals such

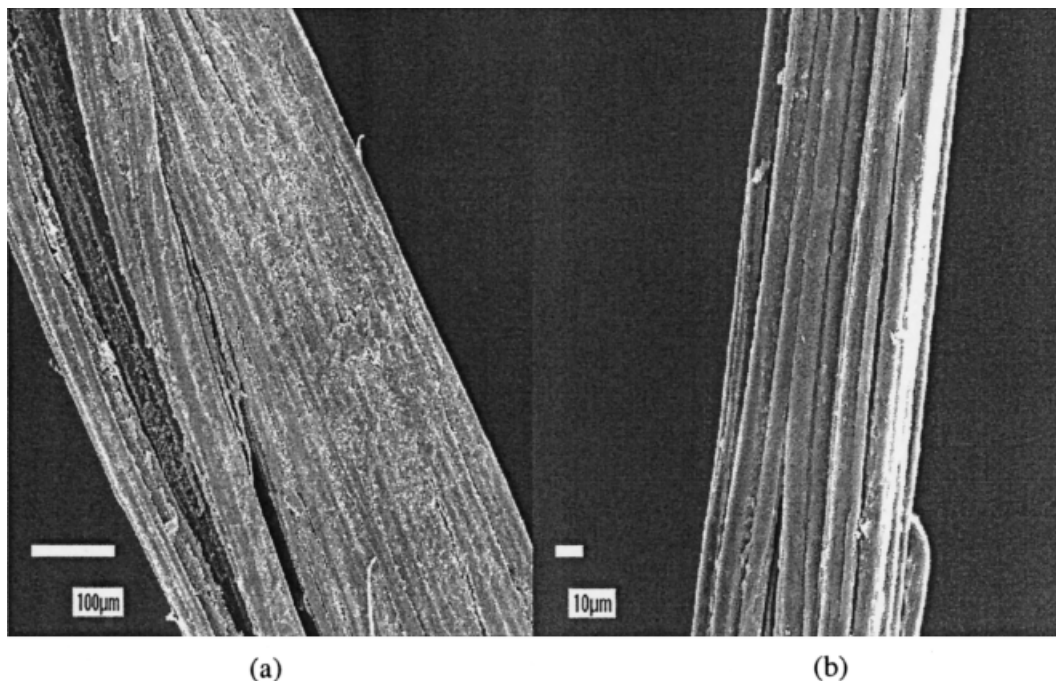
as resins and dyes. However, care must be exercised in selecting the concentration of caustic soda for alkalinization as results show that some fibers at certain NaOH concentration have reduced



(a)

(b)

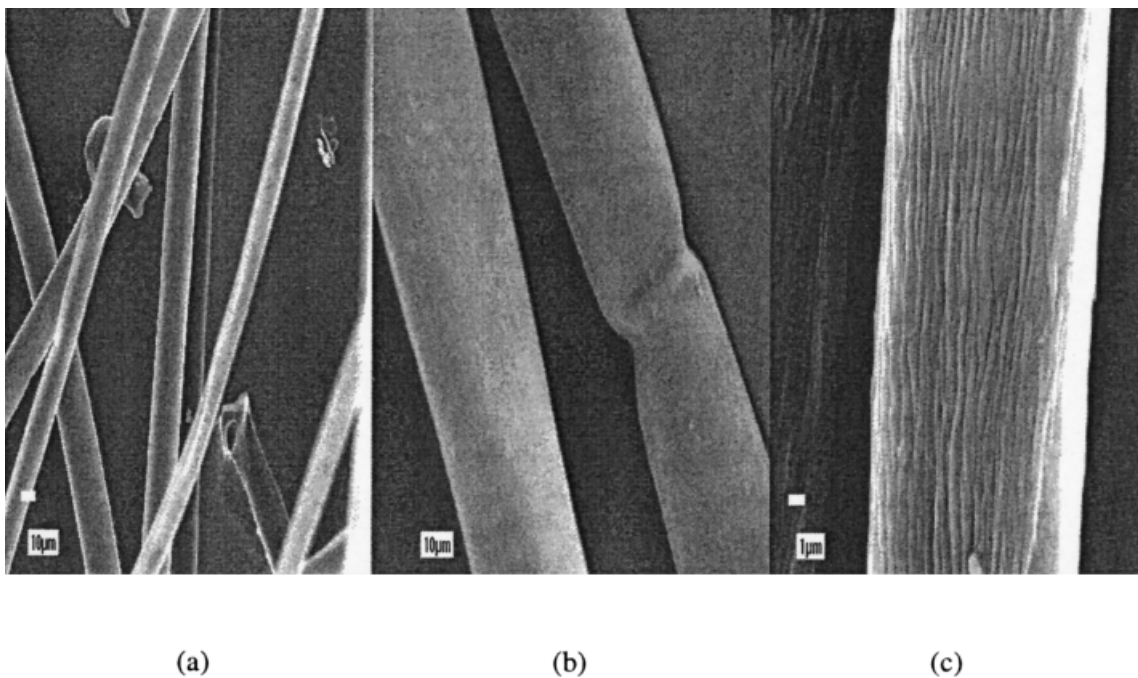
**Figure 14** SEM of sisal fiber (a) untreated (b) 8% NaOH treated.



**Figure 15** SEM of jute fiber (a) untreated and (b) 8% NaOH treated.

thermal resistance as elucidated by the DSC method. The application of the DSC technique as well as the WAXRD method gives a better analysis of the fine structure of the plant fibers than

the WAXRD method alone. It is believed that the increase in the crystallinity index obtained by WAXRD is, in fact, an increase in the order of the crystallite packing rather than an increase in the



**Figure 16** SEM of kapok fiber (a) untreated, (b) 6% NaOH, and (c) 40% NaOH treated.



**Table VII Property Ranking of Hemp, Sisal, Jute, and Kapok Fibers**

Technique	Treatment	Hemp	Sisal	Jute	Kapok
Crystallinity index (CI) (WAXRD)	Alkalization	Highest index			Lowest index
First exothermic peak (DSC)	Alkalization	4	2	3	1
Reaction to chemicals (FT-IR)	Alkalization	Strongest peak	Irregular	Irregular	Irregular
SEM	Alkalization	Least reactive			Most reactive
		1	2	3	4
		4	4	4	1

intrinsic crystallinity. A high crystallinity index is likely to result in stiff, strong fibers of interest in the formation of plant fiber composites. It is essential, therefore, to use several complementary techniques when studying the fine structure of natural fibers to confirm trends.

Fourier Transform Infrared (FTIR) spectroscopy has provided additional information on the reactivity of fibers following treatment by alkalization. This technique has also enabled to identify and confirm the effective removal of chemical groups upon alkalization. The high reactivity of kapok fiber to alkalization is due to the high content of amorphous materials such as hemicellulose and lignin and the presence of the reactive hydroxyl groups on the fiber surface and in the amorphous region. Fibers with high crystallinity index, hence low amorphous regions, such as hemp, exhibit least reactivity because they are highly crystalline, thus rendering fewer hydroxyl groups available for reactions with interacting chemicals. This also explains why the first exothermic peak temperature (DSC) is lower in fibers with the highest amount of amorphous cellulose (kapok fiber) than those with the least amount of amorphous cellulose such as hemp.

Scanning electron microscopy (SEM) results (Figs. 13–16) indicate that after alkalization at low concentrations all the fibers, except kapok, possess rougher surfaces, which will enhance mechanical interlocking with resins. However, high concentration of caustic soda result in a grooved serrated surface for kapok. Chemical treatment will produce composites with improved mechanical properties. Table VII shows a summary of the property ranking of fibers when subjected to chemical treatment. The fiber with the highest ranking is ranked 4, and the lowest is ranked 1.

However, although Table VII does not give any general trend, hemp appears to have the highest crystallinity index, and thermal stability following alkalization kapok fiber has the highest reaction affinity to chemicals followed by jute, sisal, and hemp fibers. Therefore, fibers with highest ranking in thermal stability and crystallinity index will offer better thermal-mechanical properties either alone or in a combination with other fibers when applied as reinforcements. For instance, a blend of fiber rank 4 with a lower ranked fiber will generate specific properties not attainable by one fiber alone.

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