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Structure and properties of new natural cellulose fabrics from *Cordia dichotoma*

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ABSTRACT

Natural cellulose fabrics were newly identified from the branches of the *Cordia dichotoma*. The structure of the fabrics was analysed by FTIR and X-ray diffraction. The net-like morphologies of the untreated, bleached and 5% NaOH (alkali) treated lignocellulose fabrics were observed by SEM and POM. The increase in thermal stability of alkali treated fabrics when compared to the untreated and bleached fabrics was confirmed by the TGA analyses. The effect of bleaching and alkali treatment on the properties of the fabrics was also studied. The alkali treated fabrics exhibited the highest tensile strength of 36.2 MPa, elongation at break of 2% and Young's modulus of 3699.2 MPa when compared to the untreated and bleached fabrics. The structure and properties of the fabrics indicated that they could be suitable for blending and processing with biodegradable polymers to make green composites for various types of applications.

1. Introduction

Over the past decade, polymer based hybrid materials reinforced with natural fibers have received ever-increasing attention, from academic and industrial point of view. The main advantages of the renewable natural fibers are their low cost, low density, non-toxicity, biodegradability and environmentally friendly nature. The conventional glass fibers are not biodegradable (Bledzki & Gassan, 1999; Bledzki, Reihmane, & Gassan, 1996; Corbiere-Nicollier et al., 2001; Saheb & Jog, 1999; Spinacé, Lambert, Fermoselli, & Paoli, 2009). In order to overcome this problem, many different natural fibers such as jute, coir, flax, kenaf, hemp and sisal are widely used as reinforcements in making the green composites (Bullions, Hoffman, Gillespie, Price-O'Brien, & Loos, 2006; Dhakal, Zhang, & Richardson, 2007; Joffe, Andersons, & Wallström, 2003; John & Thomas, 2008; Ouajai & Shanks, 2005; Rao & Rao, 2007) with several types of biodegradable/non-biodegradable polymers.

However, the plants bearing the natural fibers grow only in certain areas based on climatic conditions. It is necessary to identify new variety of fibers/fabrics with properties suitable for usage as natural reinforcement for the development of green composites. Recently, Varada Rajulu et al., found new natural fabrics named as *Hildegardia populifolia* and studied the structure and properties of polymers coated fabrics. They also suggested that this fabric is suitable for making the green composites (Guduri, Rajulu, & Luyt, 2006; Li, Meng, Wang, Rajulu, & Tjong, 2004; Varada Rajulu, Babu Rao, Ganga Devi, Li, & Meng, 2004; Varada Rajulu et al., 2002).

In this direction, we identified natural fabrics *Cordia dichotoma* is a small to moderate-sized deciduous tree with a short bole and spreading crown. The stem bark is greyish brown or longitudinally wrinkled. In the present study, we have chosen *C. dichotoma* fabrics because it is available mostly in all the countries, tropical, subtropical regions, variety of forests ranging from the dry deciduous forests of Rajasthan to the moist deciduous forests of western Ghats and tidal forests in Myanmar compared to our previously identified fabrics. In the present paper, we collected *C. dichotoma* locally in Nallamala forest area located near Sresailam and Atmakur Taluk, Kurnool district's of Andhra Pradesh, India. During this quest, some trees bearing uniaxial fabrics were identified. The local tribal people use these natural fabrics to tie small bundles of firewood to carry from one place to another. They also construct their huts by using these natural fabrics as ropes. It has also been observed that these fabrics are thin and have fibers arranged in uniaxial directions. As uniaxial fabrics are rare in occurrence, the authors planned to utilize these unique fabrics as reinforcement for the preparation of green composites. A photograph of the tree is shown in Fig. 1.

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Fig. 1. Photograph of the *Cordia dichotoma* tree.

The plant *C. dichotoma* from which the fabrics was extracted from the branches belongs to the *Cordiaceae* family and exists in large quantity in the local area.

In the present study, the properties of the fabrics of *C. dichotoma* are reported. The effects of bleaching and alkali treatment on the structure and properties for the *C. dichotoma* fabrics have been studied. The characterizations such as chemical analysis, FTIR, SEM, POM, TGA and X-ray diffraction for *C. dichotoma* fabrics have been studied. The effects of bleaching and alkali treatment on the tensile properties for the *C. dichotoma* fabrics have been investigated.

2. Materials and methods

2.1. Materials

C. dichotoma natural fabrics with length in the range of 300–500 cm, breadth of between 50 and 70 cm, and thickness of 0.19 mm, were obtained from the branches of the tree. NaOH pellets and bleaching powder (Ca(OCl)Cl) were purchased from Merck Ltd., Mumbai, India. In the present study, benzene, sodium chlorite, acetic acid, sodium bisulphite and ethanol (S.D. Fine Chemicals Ltd., Mumbai, India) were used as the chemicals.

2.2. Extraction and treatment of the fabrics

In order to protect the trees, we have chosen only the branches of the tree for extraction of the fabrics. Since the fabrics are found to adhere to the skin more than the stem, the skin was slit opened with a sharp knife. Additionally, we observed that the extracted portion of the tree filled up within two months. The skin with multilayer fabrics was kept in water for two weeks in order to separate the fabrics spontaneously. The fabrics separated from the skin were washed thoroughly with water and dried under the sun until the layers of the fabrics become easily separated.

Some fabric specimens were treated with 5% NaOH solution (w/v) in an ambient temperature for a holding time of 45 min. After the treatment, the fabrics were thoroughly washed with distilled water and then dried in the vacuum oven at 80 °C for 24 h. Some of the fabrics were bleached with (Ca(OCl)Cl) for 45 min. Bleached fabrics were washed with distilled water and then dried at same conditions used for the NaOH-treated samples. The samples were also re-dried under vacuum oven at 80 °C for 24 h before use for the experimental analyses.

2.3. Chemical analysis

The untreated, bleached and alkali-treated fabrics of *C. dichotoma* were preconditioned before cellulose extraction took place. The fabrics were thoroughly washed with distilled water for several times and dried under vacuum oven at 80 °C for 24 h until the weight of the fabrics became constant. Then preconditioned fabrics were chopped to an approximate length of between 5 and 10 mm. After that, a de-waxing step was carried out by boiling the chopped fabrics in a mixture of toluene/ethanol (2:1, v/v) in a soxhlet apparatus for 6 h at 60 °C and followed by filtered, washed with ethanol for 30 min and dried at 80 °C under vacuum oven for 24 h.

To remove the lignin from the fabrics and to calculate the contents of α-cellulose and hemicellulose, the de-waxed and pre-weighed samples were boiled for 2 h with 0.7% (w/v) sodium chlorite solution adjusted to a pH 4 by the addition of CH_3COOH–CH_3COONa buffer and maintaining a fabric to liquor ratio of 1:50. After that, sodium chlorite treated fabrics were placed with 5% (w/v) sodium bisulphite solution for 45 min. In this way, the lignin was removed so that the lignin and holocellulose contents could be calculated. To determine the contents of α-cellulose and hemicellulose, the isolated holocellulose was treated with 17.5% (w/v) aq. NaOH solution. The insoluble α-cellulose was filtered, washed thoroughly with distilled water and dried under vacuum oven at 60 °C to get a constant weight. In this way, the components of the fabrics were estimated. This procedure was adopted from the earlier works (Chattopadhyay & Sarkar, 1946; Sarkar, Mazumdar, & Pal, 1948). In each case, five samples were analysed and the average values are reported in this article.

2.4. Characterization of the fabrics

A JOEL JSM-820 microscope (Akishima, Japan) was used to obtain the micrographs of the untreated, bleached and alkali treated fabrics with gold coated before recording the micrographs. The optical micrographs were also recorded using an Olympus Bx 50 polarizing optical microscope. For the FTIR analyses cryogenically cooled and powdered samples were mixed with KBr and made pellets by employing a hydraulic press. A Perkin Elmer 16PC FT-IR instrument was used to obtain the FTIR spectra of the untreated, bleached and alkali-treated samples in the 4000–500 cm^{-1} region with 32 scans at a resolution of 4 cm^{-1} in each case. A Perkin Elmer TGA-7 instrument was used to record the thermograms of the untreated and treated fabrics in N_2 atmosphere and at a heating rate of 10 °C/min. A Rigaku Dmax 2500 diffractometer (Tokyo, Japan) was employed to record the diffractograms of the untreated, bleached and the alkali-treated fabrics. The system has a rotating anode generator operated at 40 kV and 150 mA with a copper target and a wide-angle powder goniometer using Cu Kα radiation with a wavelength λ = 1.5406 Å. Young's modulus, maximum stress and % elongation at break (tensile properties) of untreated and treated fabrics were determined using an INSTRON 3369 Universal Testing Machine (Norwood, Massachusetts, USA) at a crosshead speed of 3 mm/min and maintaining a gauge length of 50 mm. Ten samples

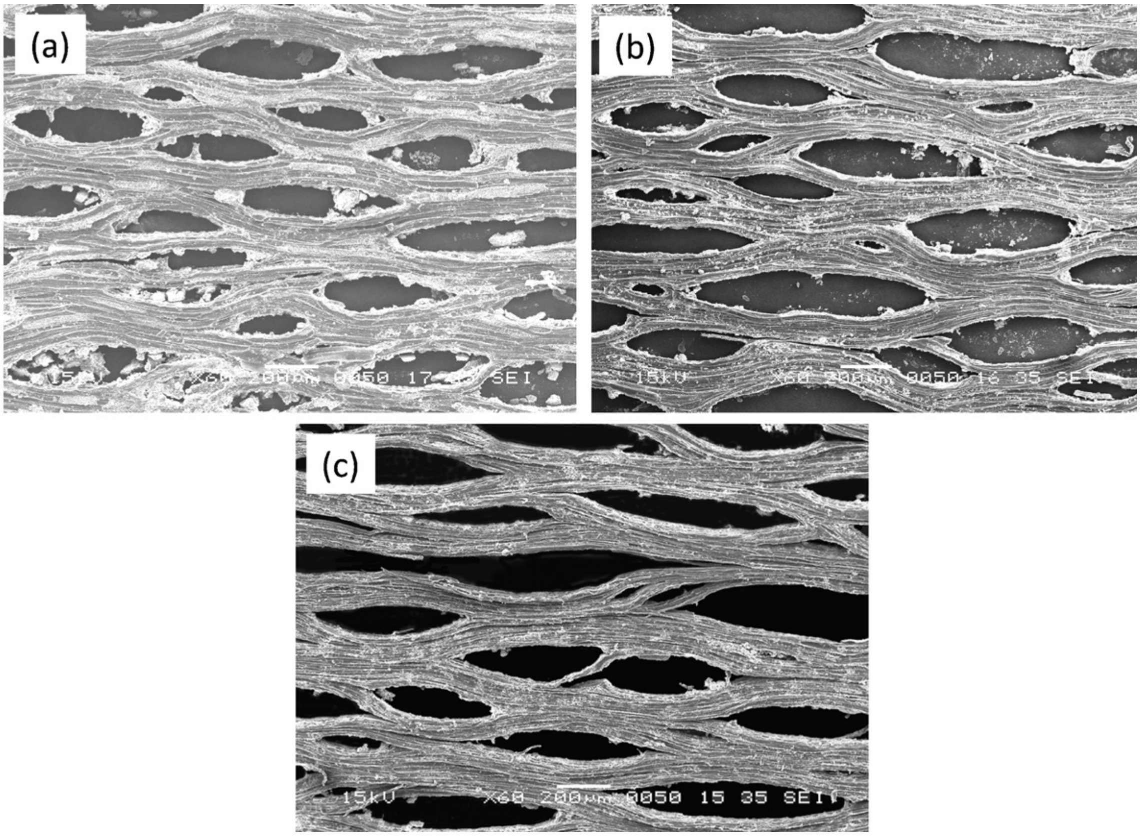

Fig. 2. Scanning electron micrographs of surface of *Cordia dichotoma* fabrics (a) untreated, (b) bleached and (c) 5% NaOH treated fabrics at same magnification.

were tested for each set of experiment and their average value are reported in this article.

3. Results and discussion

3.1. Morphology

The scanning electron images of the untreated, bleached and alkali treated fabrics are presented in Fig. 2(a)–(c), respectively. From Fig. 2(a) and (b), it can be seen that the gummy polysaccharides of lignin, pectin and hemicellulose are localized as white layer on the surfaces of untreated and bleached fabrics. Such observation has also been reported in the case of other natural fabrics such as *Hildegardia populifolia*, *Polyalthia cerasoides*, *Sterculia urens* and *Grewia tilifolia* (Jayaramudu, Guduri, & Rajulu, 2009; Jayaramudu, Guduri, & Varada Rajulu, 2010; Jayaramudu, Jeevan, Guduri, & Rajulu, 2009; Varada Rajulu et al., 2003). It is clearly indicated that the white layer is not evenly distributed along the fabrics surface, but its thickness varies from point to point. As seen in Fig. 2(c), the alkali treated fabrics have highly reduced thickness and they appear to have clean but rough surface with large number of etched striations. These micrographs also reveal the uniaxial orientation of fabrics in parallel direction and the fabrics also have many void regions. The rough and clean surface morphologies of the alkali treated fabrics are expected to assist in the direct interaction of the fabrics with polymer matrix which can be used for the preparation of green composites.

3.2. Polarized optical micrographs

The polarized optical photographs (POMs) of the untreated, bleached and alkali treated fabrics are shown in Fig. 3(a)–(c), respectively. The POM images of the untreated and bleached fabrics were found to be diffused. The sharpness of the images depends on the crystalline content i.e. α-cellulose in the material. Hence, the diffuse images seen in Fig. 3(a) and (b) indicate the presence of amorphous components, i.e. hemicellulose and lignin on the surface of the fabrics. Fig. 3(c) shows that the alkali treated fabrics pattern becomes bright and well defined when compared to untreated and bleached fabrics, indicating an increase in crystallinity due to the removal of major amounts of hemicellulose and lignin. This feature also confirmed by the XRD analyses (see XRD section) and also observed by Varada Rajulu et al. (2003) in the case of *Hildegardia* fabrics. This indicates the elimination of hemicellulose and lignin to a large extent upon alkali treatment. However, the extent of removal of hemicellulose and lignin upon alkali treatment can better be determined via chemical analysis.

3.3. Chemical composition

The chemical analyses of the untreated, bleached and alkali treated *C. dichotoma* fabrics are presented in Table 1. The untreated fabrics contain ~59.7% cellulose, ~23.6% hemicellulose and ~14.7% lignin. From Table 1, it is clearly established that upon bleaching and alkali-treatment, the content of hemicellulose and lignin has decreased and as a result, the % α-cellulose increased. In the present study, the α-cellulose content of 77.8% upon alkali treatment is relatively a higher quantity when compared to other recently identified fabrics such as: *Hildegardia populifolia* (~69%), *Grewia tilifolia*(~67.9%), and *polyalthia cerasoides* (~70.5%) and lower than *Sterculia urens* (~81.5%) as shown in Table 2. It is also observed that the α-cellulose contained in the *C. dichotoma* fabrics is relatively higher when compared to other fibers such as abaca, alfa, bagasse, bamboo, banana, coir, jute and kenaf (Maya & Rajesh, 2008).

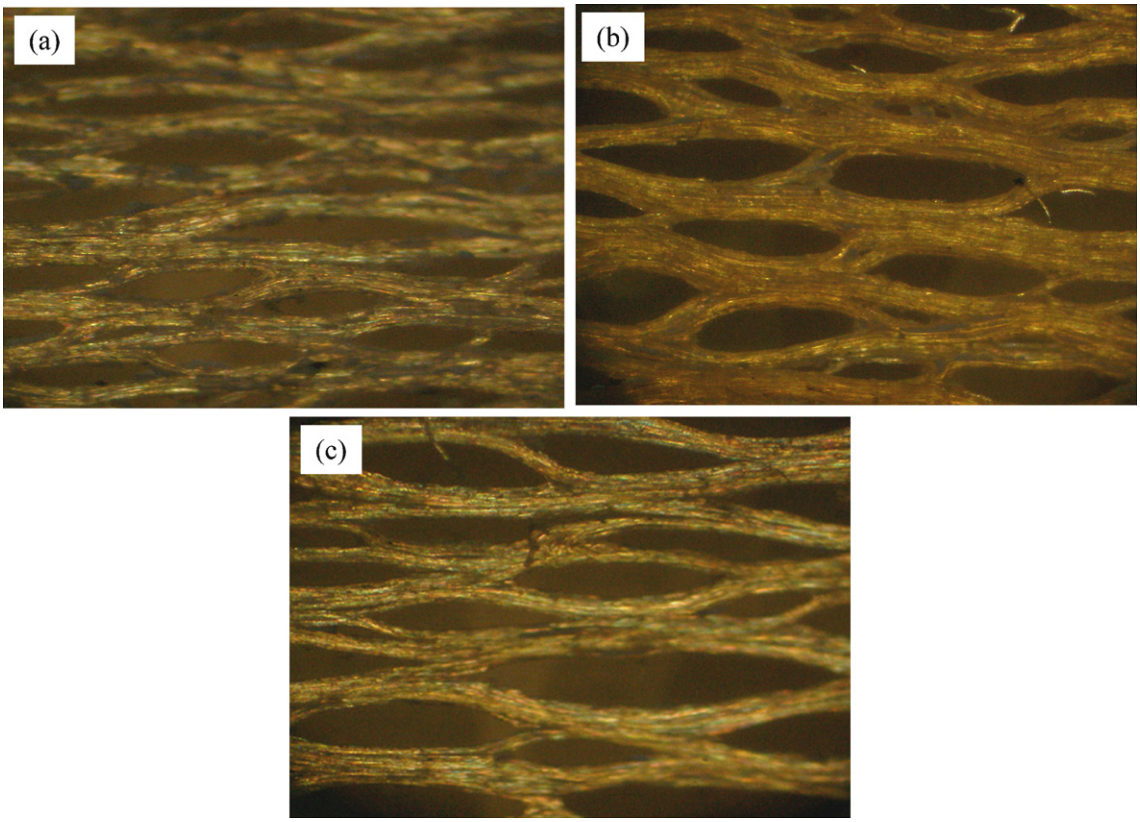

Fig. 3. Polarized optical photographs of *Cordia dichotoma* fabrics (a) untreated, (b) bleached and (c) 5% NaOH treated fabrics at same magnification.

Table 1
Chemical composition of untreated, bleached and 5% NaOH treated *Cordia dichotoma* fabrics.

Component (%)	Untreated	Bleached	5% NaOH treated
α-Cellulose	59.7	67.1	77.8
Hemicellulose	23.6	19.3	7.38
Lignin	14.7	12.7	9.30

3.4. Functional group analysis

Fig. 4 presents the FTIR spectra of the untreated, bleached and alkali treated *C. dichotoma* fabrics. It can be observed from Fig. 4 that the untreated and treated fabrics contain α-cellulose, hemicellulose and lignin due to the presence of their well defined peaks at 3431 cm^{-1} for O–H stretching of α-cellulose, 2924 cm^{-1} for alkyl C–H stretching of α-cellulose, 1730 cm^{-1} for C═O stretching of hemicellulose, 1623 cm^{-1} for C═O stretching of lignin, 1320 cm^{-1} for the asymmetric C–O–C stretching of lignin, and 780 cm^{-1} for aromatic C–H stretching of lignin (Bismarck et al., 2001; Moran, Alvarez, Cyras, & Vazquez, 2008; Pandey, 1999; Yang, Yan, Chen, Dong Ho, & Zheng, 2007). It is interesting to note that the intensities of the peaks at 1730 cm^{-1}, 1623 cm^{-1}, 1320 cm^{-1}, and 780 cm^{-1} for the alkali treated fabrics significantly, decreased when compared to the untreated fabrics (Liu, Han, Huang, & Zhang, 2009; Lojewska, Miskowiec, Lojewski, & Pronienwicz, 2005; Pandey, 1999; Ray & Sarkar, 2001). These features indicate the reduction of hemicellulose and lignin content in the alkali treated fabrics. The results obtained are in good agreement with the chemical composition values.

3.5. X-ray analysis

The position of crystalline peaks and the relative amounts of crystallinity and amorphous materials in the fabrics can be visualized from Fig. 5. It can be observed from Fig. 5 that the fabrics contain two peaks at $2\theta = 15.69^\circ$ (broad) and 22.29° (sharp), crystallographic planes. The broadening of the peak at $2\theta = 15.69^\circ$ is mostly due to the presence of non-cellulosic materials like hemicellulose and lignin in the fabrics, while the sharp and intense peak at $2\theta = 22.29^\circ$ for the content of α-cellulose in the fabrics. In contrast, higher α-cellulose content fibers, like cotton, linen, hemp, or

Table 2
Chemical composition and moisture content of *Cordia dichotoma* natural fabrics compared with other fabrics identified by our laboratory.

Fabrics	α-Cellulose (wt.%)	Hemi celluloses (wt.%)	Lignin (wt.%)	Moisture content (%)	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)
Hildegardia populifolia	69.0	17.2	14.0	9.2	80.1	2.7	3.52
Grewia tilifolia	67.9	17.0	15.0	2.3	75.3	5.0	2.0
Sterculia urens	81.5	7.5	10.8	5.0	18.92	2.0	2.47
Polyalthia cerasoides	70.5	18.5	10.7	6.4	66.3	5.7	2.7
Cordia dichotoma[a]	77.8	7.38	9.30	5.52	36.2	3.6	2.0

[a] Present work.

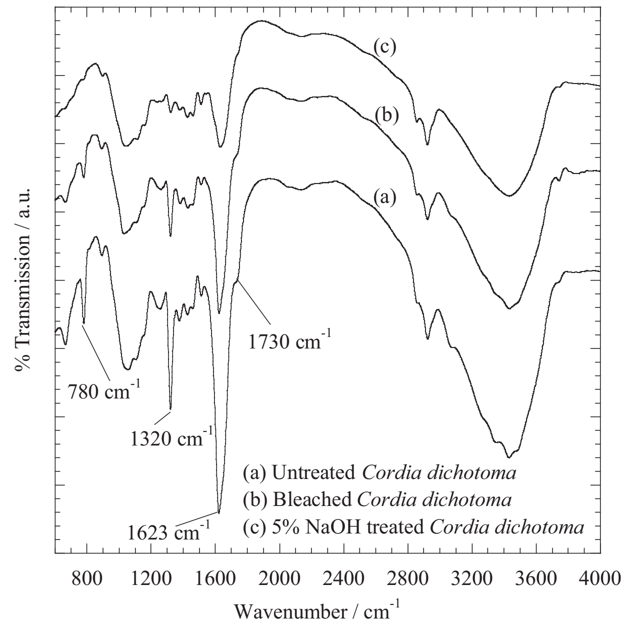

Fig. 4. FTIR spectra of (a) untreated, (b) bleached and (c) 5% NaOH treated *Cordia dichotoma* fabrics.

flax (Ouajai, Hodzic, & Shanks, 2004; Reddy & Yang, 2007) have multiple peaks at around $2\theta = 15.69°$, but for *C. dichotoma* fabrics, only one broad peak is observed due to the presence of amorphous materials.

The crystallinity index of the fabrics has been determined by using the following Eq. (1) (Mwaikambo & Ansell, 2002)

$$I_c = \frac{I_{(0\,02)} - I_{(am)}}{I_{(002)}} \times 100 \qquad (1)$$

where $I_{(002)}$ ($2\theta = 22.29°$) represents the intensity of crystalline peak, while $I_{(am)}$ ($2\theta = 15.69°$) denotes the intensity of the amorphous peak in the crystallographic planes. The crystallinity indices obtained from X-ray diffractograms for untreated, bleached, and

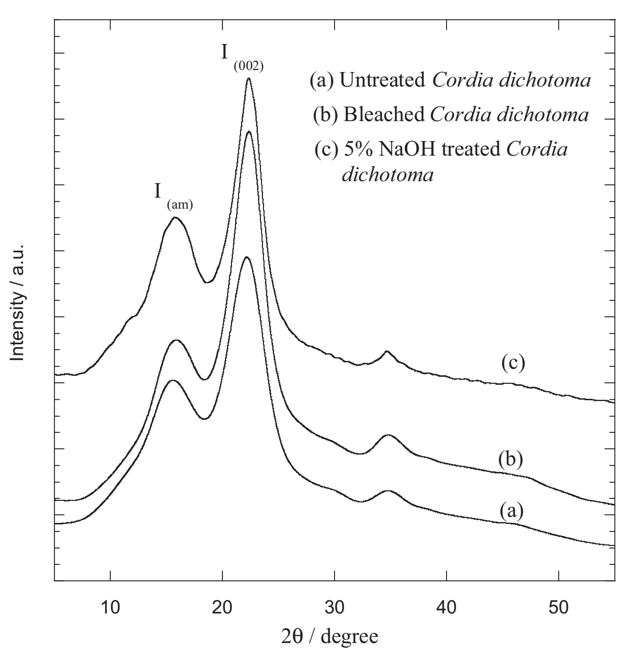

Fig. 5. X-ray diffractograms of (a) untreated, (b) bleached and (c) 5% NaOH treated *Cordia dichotoma* fabrics.

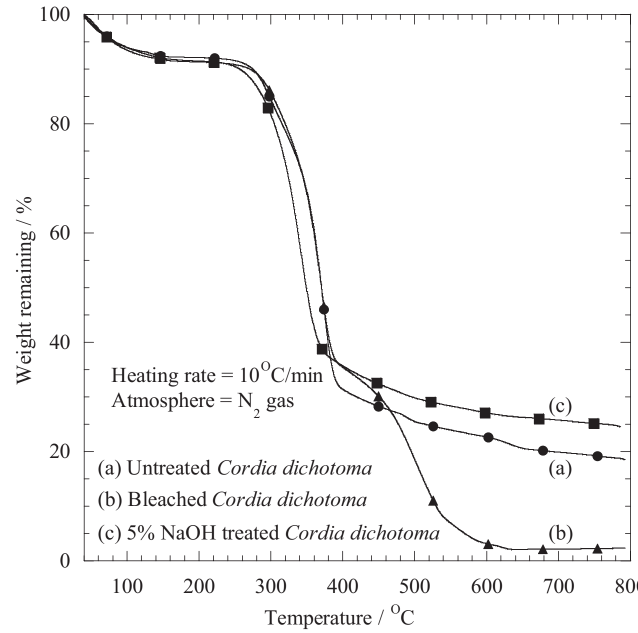

Fig. 6. TGA curves of (a) untreated, (b) bleached and (c) 5% NaOH treated *Cordia dichotoma* fabrics.

alkali treated fabrics show variations as a function of surface treatment. The crystallinity index as shown in Table 3, of the bleached and alkali treated fabrics increased from 12.14% for the untreated fabrics to 39.3% and 50.20% respectively. A similar observation has been made for hemp and flax fibers when treated with alkali (Ouajai et al., 2004). By the removal of some of amorphous constituents of the fibers there is a possibility for rearrangement of crystalline regions as a result the degree of crystallinity increases (Ouajai et al., 2004).

3.6. Thermo gravimetric analysis

The primary thermograms of the untreated, bleached and alkali treated fabrics are presented in Fig. 6. Using these thermograms, the initial degradation temperature, the final degradation temperature, inflection point (where the degradation rate is maximum), refractoriness (T^*), and integral procedural degradation temperature (IPDT) were calculated using the Doyle method (Doyle, 1985). These values are presented in Table 4. From this Table 4, it is clearly evident that the thermal stability of the fabrics has increased slightly upon alkali treatment. This is due to the removal of considerable amount of amorphous hemicellulose and lignin from the fabrics upon alkali treatment. Using the thermograms, the % moisture and char contents were also calculated and these values are also presented in Table 4. There is minimum variation in the moisture content of the fabrics under all conditions. However, the % char content is significantly decreased in the case of alkali treated fabrics because of the less content of amorphous materials. The TGA analyses indicate that the untreated, bleached, and alkali treated *C. dichotoma* fabrics are thermally stable up to 350–400 °C (Raveendran, Ganesh, & Khilar, 1996; Yang et al., 2007).

3.7. Tensile properties

Generally, natural fibers/fabrics have a characteristic high tensile strength and lower elongation at break. The tensile properties of the fabrics in terms of maximum stress, % elongation and Young's modulus are presented in Table 3 and their corresponding bars are shown in Fig. 7. While computing the tensile strength, the

Table 3
Crystallinity index and tensile properties of *Cordia dichotoma* natural fabrics.

Cordia dichotoma fabrics	Maximum stress (MPa)	Young's modulus (MPa)	Elongation at break (%)	Crystallinity index (%)
Untreated (S.D.)	16.9 (1.3)	2222.7 (135.1)	1.4 (0.1)	12.14
Bleached (S.D.)	24.2 (1.4)	3274.4 (153.6)	1.6 (0.3)	39.30
Alkali treated (S.D.)	36.2 (1.6)	3699.2 (178.3)	2.0 (0.4)	50.20

S.D., standard deviation.

Table 4
Thermal degradation parameters and moisture content of *Cordia dichotoma* fabrics.

	IDT °C	FDT °C	Inflection point	IPDT	Refractoriness (T^*)	% Char content	% Moisture content
Untreated	295.3	384.9	371.5	199.4	265.6	1.08	5.07
Bleached	289.41	366.7	369.3	193.9	254.2	1.49	5.41
5% alkali treated	318.2	435.3	341.9	161.6	194.7	0.12	5.52

IDT °C, initial degradation temperature °C; FDT °C, final degradation temperature °C; IPDT, integral procedural degradation temperature.

area occupied by the void regions was also taken into account whereas the actual area occupied by the fibers in the fabrics may be far less. Hence, the actual tensile properties may be higher than reported in Table 3. From Table 3 and Fig. 7, it is evident that the tensile properties of the fabrics have improved with bleaching and the alkali treatment. The tensile strength of treated fabrics has increased from 16.9 MPa to 24.2 MPa (bleached) and to 36.2 MPa (alkali). Similarly, Young's modulus increased from 2222.7 MPa to 3274.4 MPa (bleached) and to 3699.2 MPa (alkali) respectively, when compared to untreated fabrics. This is due to the fact that the maximum removal of amorphous materials from the alkali treated fabrics when compared to the untreated and bleached fabrics. It is also clearly shown that the % elongation at break has slightly increased in the case of bleached and alkali treated fabrics. The elongation at break for the fabrics under consideration is 2% which is comparable with that of other fabrics listed in Table 2. The tensile strength of the fabrics is relatively low for the recently identified fabrics except *Sterculia urens*. This may be due to the presence of more void regions in the fabrics. Because of the presence of more void regions in the fabrics, it should be a suitable candidate as reinforcement in the development of green composites with various types of biodegradable polymers (Jayaramudu, Guduri, & Rajulu, 2009; Varada Rajulu et al., 2003).

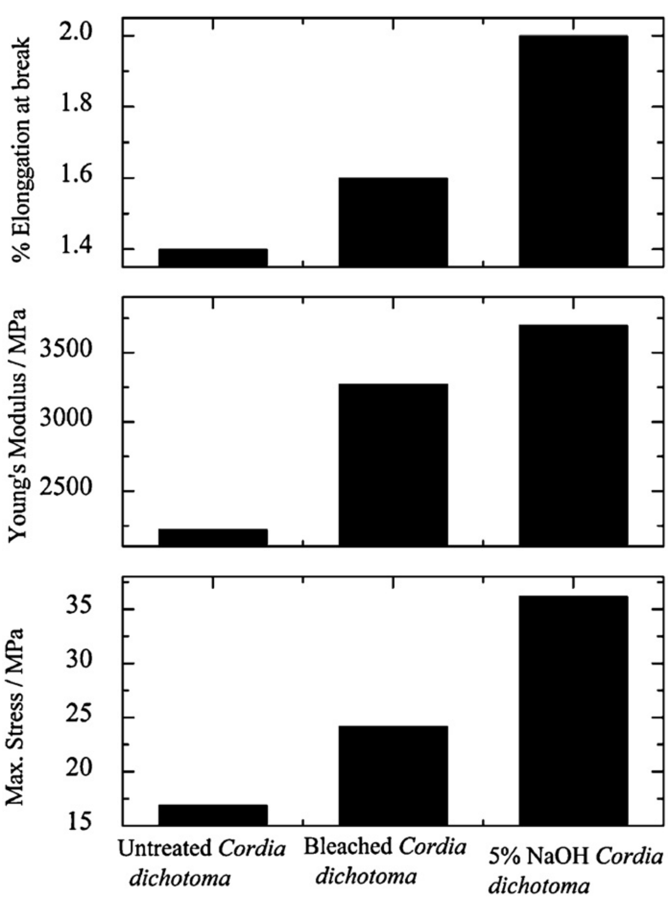

Fig. 7. Tensile properties of untreated, bleached and 5%NaOH treated *Cordia dichotoma* fabrics.

4. Conclusions

The structure and properties of the *C. dichotoma* fabrics were studied. The SEM analysis showed the morphology of the fabrics as less thickness, rough surface with large number of etched striations and uniaxial orientation of the fabrics in parallel direction for bleached and 5% NaOH treated due to the removal of amorphous on the surface of the fabrics. The FTIR and chemical analysis showed removal of lignin and hemicellulose content for bleached and 5% NaOH treatment. The sharpness, brightness and increase in crystallinity obtained when untreated fabrics treated with Ca(OCl)Cl and 5% NaOH revealed by XRD and POM analysis. The thermal stability and tensile properties of this fabric increased on bleached and 5% NaOH treatment. The tensile properties of the 5% NaOH treated are increased when compared to bleached and untreated. Due to its higher modulus for alkali treated natural fabrics can be utilized as reinforcement in the green composites and high-significance fabrics applications. In addition, we are extracting the fabrics from the skin of the tree without affecting the environmental ecosystem. Our future work mainly focused on packaging and textile application using this fabric.

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