Tensile and Lignocellulosic Properties of *Prosopis chilensis* Natural Fabric

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ABSTRACT: The uniaxial natural fabric *Prosopis chilensis* was treated with NaOH (alkali), poly (vinyl alcohol) (PVA), and polycarbonate (PC) solutions. The *Prosopis chilensis* fabric belongs to Leguminosae family. The properties of ligno-cellulosic fabric and the effect of sodium hydroxide (NaOH) treatment were evaluated using thermal analysis by means of thermogravimetric and differential scanning calorimetry analysis, Autonated total reflection-fourier transform infrared spectroscopy, X-ray diffraction (XRD), and field emission scanning electron microscopy. Tensile properties of the untreated and fabric treated with NaOH, PVA, and PC were also studied to assess their performance. The fabric has good thermal resistance on alkali treatment. The FTIR method indicates lowering the hemi cellulose and lignin content by alkali treatment. Further, the XRD studies reveal that crystallinity of the fabric increases on alkali treatment. Tensile properties of the fabric were enhanced on treatments with NaOH, PVA, and PC treatments. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 117: 2907–2914, 2010

Key words: natural fabric; thermo mechanical; ligno cellulosic properties; surface treatment and analysis

INTRODUCTION

The renewable natural fibers have attracted many researchers with their properties.[1] They showed many advantageous over synthetic fibers and have been intensively studied in recent years.[2] A nonrenewable and synthetic fiber causes many environmental problems associated with their use and disposal.[3] For this reason, there is an increased interest to find new, renewable, and biodegradable fibers. Research on natural fibers is increasing nowadays;[4] this is because natural fibers offer many advantages, such as low density, enhanced biodegradability, renewable, easy extraction of fiber, and cost-effective.[5] Because of these reasons, natural fiber composites have already been used in fabricating furniture and architectural materials.[6] Recently, they have also gained widespread application in the automobile industry; for example, European renewable fibers, such as flax and hemp, are now used for door panels and car roofs.[7] In certain applications, the materials should require higher strength and stiffness. In this connection, to improve the mechanical properties of composites, surface treatments are used to modify the morphology of the fibers. Reports on various treatments are also available in the literature.[8,9]

In this direction, some ligno-cellulosic fibers have been studied by several researchers recently.[10] Characteristic studies of various ligno-cellulosic fabrics have been reviewed for this investigation.[11,12] To identify new natural fibers and fabrics as reinforcement, their properties have to be studied. In this work, the authors have studied the properties of the ligno-cellulosic fabric from *Prosopis chilensis* (PC). The fabric has been extracted from the sheath of the tree. The photographs of the fabric specimens are shown in the Figure 1(a,b).

Currently, this tree and fabric were used as a fire wood in rural areas, beverage-based food for human being and animal. Particularly, leaves and the seeds of the fruit have been used as feed for sheep and goats. The wood from these trees are used in making furniture and craft items. It also has gained medical importance, leaf extract of this plant reduces the infection of tomato spotted wilt topovirus.[13] So far very limited studies have been reported on the characterization of this fabric. The physical and typical chemical composition of the fabric were reported as follows: Specific gravity 0.82, volume shrinkage 7.0, and 44.8% moisture content.[14] Patel and Safaya[15] reported that the *Prosopis* species contains the following constituents, cellulose 40–45%, hemicelluloses
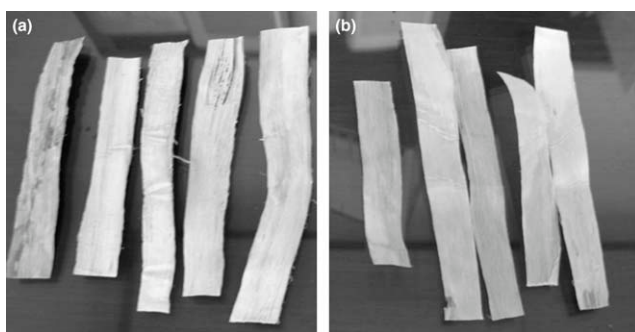
Figure 1 Fabric specimens after extraction (a) Untreated and (b) NaOH treated.

25–30%, lignin content 11–28%, soluble extracts 3–15%, and ash 4.9%. The PC tree comprises of 48% fabric out of the total matter.

Main aim of this study is to investigate the potential of using the PC natural fabric as reinforcements in polymeric materials. As there were no reports available on this fabric, we studied some of its properties such as spectral analysis for fiber constituents, thermal properties, crystallinity, and surface morphology. We also evaluated tensile properties of the fabric to assess the mechanical performance. The authors studied all these properties before and after alkali treatment and also studied tensile properties of poly(vinyl alcohol) (PVA) and PC fabric supporting SEM analysis.

EXPERIMENTAL

Materials

Natural fibers were procured from the branches of PC available in local forests of Gunduvaripalli and Kadiri of Anantapur district, Andhra Pradesh, India. Anhydrous sodium hydroxide, PVA (M_w 2000), and poly (carbonate) were purchased from Junsei Chemicals Co., Japan.

Extraction of fabric

Prosopis fibers were extracted in the form of knitted fabric of thickness about 0.21 mm. The fabric extraction is simple, economical, and requires no other process because the gums and other substances from the fabric can be easily removed by dissolving them in water. Fresh and fine branches, and bark of the tree were beaten with thick rod until the sheath layers and fleshy matter are loosened and removed. The resulting layers were kept in water for 20 days. It was observed that the foreign matter was dissolved or separated in water within soaking time. Later, the layers were removed, hand washed thoroughly, and rinsed with clean water to remove greasy matter. Then, the fabric was washed thor-

oughly with distilled water and allowed to dry in the sunlight for about a week.

Fabric surface treatment

Samples were treated with various chemicals and polymers for different time intervals at room temperature (28°C) to assess the influence of chemical modification on the strength and ligno cellulosic properties of the fabric. The following are the chemical modifications of the fabric.

Alkalization (NaOH)

Some of the dried fabric samples were soaked in 3% aqueous alkali solution for about 2 h. Fabrics, thus, obtained were washed several times in distilled water and finally in dilute acetic acid (10% w/w) solution to remove the dissolved substances and allowed to dry at room temperature.

PVA

Some of the fabric samples were treated with 4% PVA solution by dipping the samples in PVA solution for 4 h. The excess PVA solution on the fabric was removed by placing them vertically and then dried at room temperature. The samples, thus, obtained were used for further analysis.

Polycarbonate

The fabric samples were also treated with 10% polycarbonate (PC) solution prepared using dichloromethane as a solvent. The excess of PC solution on the fabric was removed by placing them in vertical position and allowed to dry for 24 h at room temperature.

CHARACTERIZATION METHODS

ATR-FTIR spectral analysis

The fabric specimens were characterized by automated fourier transform infrared spectroscopy (ATR-FTIR) (Perkin Elmer, Spectrum GX model, USA) to study the chemical composition and bonding responsible for the mechanical properties. About 5 mg of the fabric was crushed into tiny particles in the presence of liquid nitrogen to obtain the powder. This powder was then made in contact with detector (ATR) to obtain the spectrum in the range of 4000–560cm^{-1}.

Thermogravimetric analysis

Thermogravimetric analysis (TGA) of the untreated and treated fabric was carried out in N$_2$ atmosphere using Perkin-Elmer TGA 7 USA thermal analyzer.
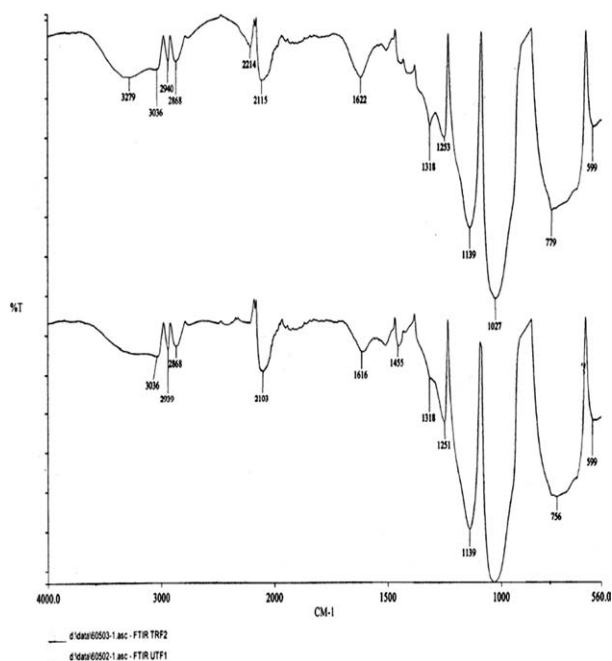
Figure 2 FTIR spectrum of untreated (Lower) and alkali-treated (Upper) *Prosopis chilensis* fabric.

Analysis was carried out at a heating rate of 10°C/min and the fabric powder was heated from 50 to 700°C. TGA was used to investigate the weight loss behavior and the critical temperature at which the fabric material starts degradation. It was also used to investigate the stability of the fabric against temperature.

DSC measurements

Glass transition temperature of the dried fabric powder was investigated using differential scanning calorimeter (DSC, USA Perkin Elmer PYR; S1 Instruments). The fabric powder was heated from 50 to 400°C at a heating rate of 10°C/min in nitrogen atmosphere.

X-ray analysis

The degree of crystallization of the untreated and treated fabrics was examined with an X pert MPD Philips X-ray diffractometer with Cu Kα radiation of wave length of 1.5406 A° at 40 kV, 30 mA. The scanning experiment was carried out at the equator, over a 2θ range from 5° to 80° at a scan speed of 4°/min. The degree of crystallinity of the fabric was calculated from X-ray diffractograms of the samples, which were analyzed in the powder form and using the computer program.

Field emission scanning electron microscopy analysis

The fabric surface microstructure was probed by a field emission scanning electron microscopy (FE-SEM, JSM 6700 F microscope, JEOL). The scanning electron micrograms of the fabric specimens before and after treatments were also recorded and studied.

Tensile testing

The tensile strength, modulus, and elongation at break of the fabric specimens, with and without treatments were determined using SHIMADZU (AGS-10kNG model) Universal testing machine. The fabric specimens with dimensions 100 mm × 15 mm with a gauge length of 20 mm were used for the analysis and the tests were conducted at a crosshead speed of 1 mm/min with 10 kN load. The temperature and humidity for these tests were maintained at 22°C and 50%, respectively. The tensile stress was applied along the fiber axis (longitudinal axis). Ten specimens were tested in each case and the average value was reported.

RESULTS AND DISCUSSION

ATR-FTIR spectral analysis

The spectrum of the fabric before and after alkali treatment is shown in Figure 2. It shows many absorption bands. The peak positions and corresponding assignment groups are presented in Table I. In case of alkali-treated fabric, some changes are observed. The peak observed at 3038 cm^{-1} in untreated fabric indicates the presence of hydrogen bonding and tends to shift to higher absorbency values in alkali treated fabric, e.g., 3279 cm^{-1}. The strong and weak absorption bands between 3038 and 3280 cm^{-1} in the spectrum is due to the presence of OH groups of the fabric constituents.

Peak around 1600–1620 cm^{-1} is probably due to the absorbed water in crystalline cellulose. Similar results were also reported by Marianne et al.[9] The peak at around 1500 cm^{-1} is associated to the

TABLE I
Peak Positions and Assignments of Chemical Groups in the Untreated and Treated *Prosopis* Fabric

Wave number (cm^{-1})	Assignments
3279–3038	OH—Stretching
2940	CH—Stretching
2868	C—H stretch
1622–1616	Absorbed water
1450	CH—Bending
1250–1253	C—O Stretching
1140	C—O—O Asymmetry vibration
1032–1027	Symmetric CO Stretching of lignin
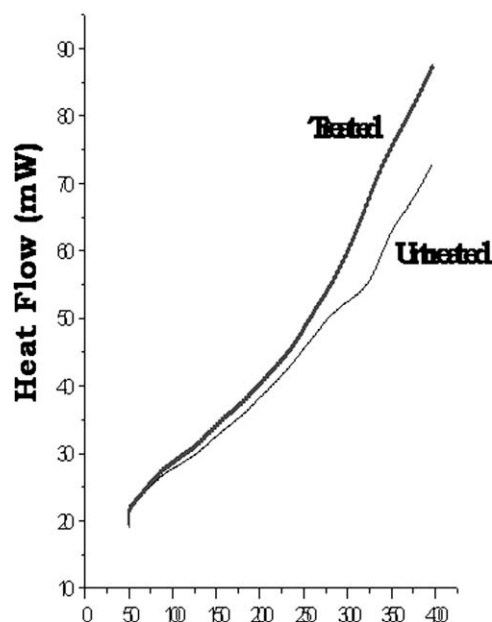
Figure 3 DSC analysis of untreated and alkali-treated prosopis fabric in N_2 atmosphere.

Figure 4 TGA curves of untreated and alkali-treated prosopis fabric in N_2 atmosphere. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com]

bending of the aromatic C—H (ring) bond, mostly in lignin based materials.[9] The large peak observed at 1032 cm^{-1} in untreated fabric would be due to the presence of lignin, whereas in alkali-treated fabric, the peak observed at 1027 cm^{-1} shows diminishing intensity; this may be due to the removal of lignin on alkali treatment. The other peaks in the range of 1800–560 cm^{-1} is typical of cellulose structural units, which are similar to those reported earlier for other lignocellulosic fibers such as sisal, hemp, jute, hilde guardia, and Kapok.[16] No significant changes are observed in the intensity or position of the other peaks in the spectra.

Thermal studies

Thermal behavior of the fabric was characterized by means of differential scanning calorimetry and thermogravimetric analysis. Figure 3 shows the DSC curves of untreated and treated fabric. Untreated fabric shows higher glass transition temperature (T_g 341°C) than alkali treated one (321.3°C). This indicates that the decrease in T_g is due to the removal of hemicellulose and lignin in treated fabric.

TGA of untreated and alkali-treated *Prosopis* fabric samples was also carried out in nitrogen atmosphere; the results are shown in Figure 4, and the values are presented in Table II. In each case, initial, inflection point and final degradation has been observed. In the initial stage, although 3% alkali-treated fabric shows degradation at 234°C, weight loss is not much difference in each case. In the second and final stage, the alkali-treated samples degrade at a higher temperature range; weight loss in
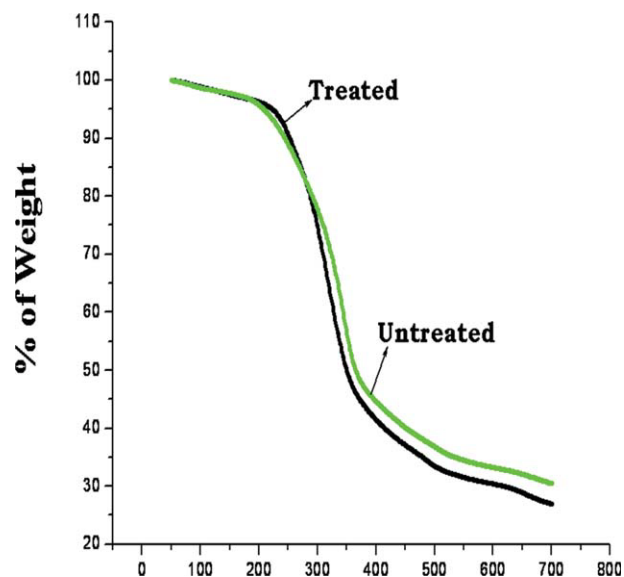
untreated fabric is more, compared to that of the alkali-treated fabric.

From the above observations, we can say that overall thermal stability of the *Prosopis* fabric increases on alkali treatment. This is understandable that the fabric on NaOH treatment increases its crystallinity, tending to increase the thermal stability. This is due to the fact that amorphous cellulose is more easily degraded on alkali treatment than that of crystalline form. Thus, more crystalline and heat resistant cellulose molecules are left over after alkali treatment. Similar results were also reported by Debasish et al.[17]

X-ray diffraction studies

The X-ray diffraction patterns of untreated and alkali-treated *Prosopis* fabric specimens are shown in Figure 5. It shows the patterns of *Prosopis* specimens exhibit four well-defined peaks at 15.2, 23.2, 25.1, and 38.4 2θ. The major diffraction peaks for 2θ ranging between 23θ and 25θ, corresponding to the crystallographic planes of cellulose I. When the cellulose content is high (treated fabric), at around 23θ one

TABLE II
Thermal Degradation Parameters of Untreated and Treated *Prosopis* Fabric in N_2 Atmosphere

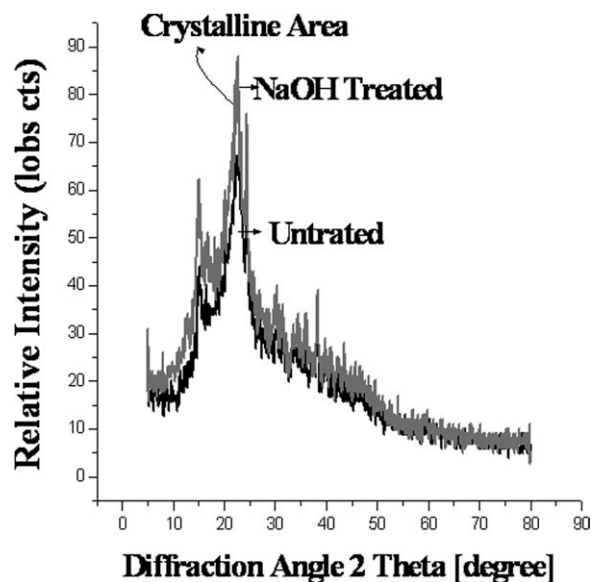| Degradation parameter	Untreated (°C)	Alkali treated (°C)
Initial degradation Temperature	232	234
Inflection point	318	344
Final degradation Temperature	616	637

Figure 5 XRD of untreated and alkali-treated prosopis fabric.

may observe two peaks, but when the fabric contains high amount of amorphous materials such as lignin, hemicellulose and amorphous cellulose (untreated), as in the untreated fabric, those two peaks are tarnished and appearing as one broad peak (Figure 5).

From the Figure 5, it is clearly evident that the crystallinity of the fabric increased on alkali treatment. From this, it is understandable that the crystalline cellulose content is increased, whereas the amorphous hemicellulose and lignin content decreased on alkali treatment. These results are also supported by FTIR results. The effect of alkali treat-
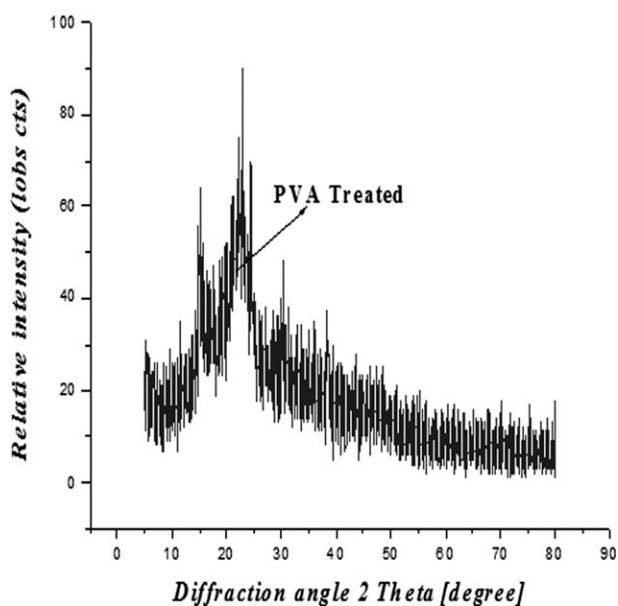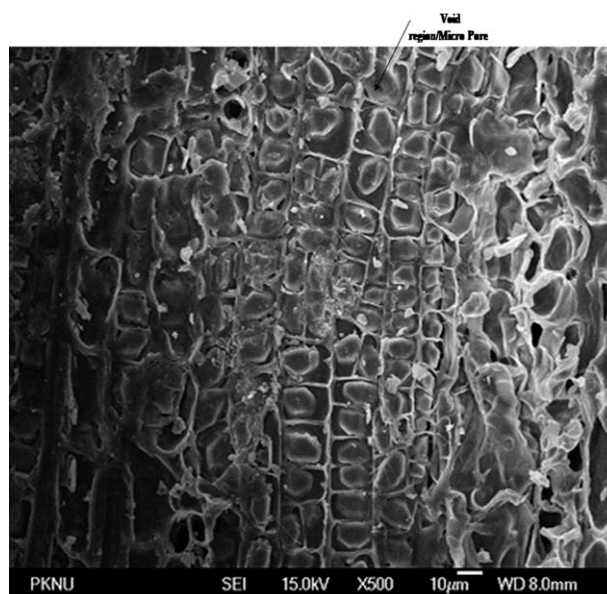
ment on the crystallinity of cellulosic materials has been investigated by various researchers. Marianne Le et al.[17] reported that alkali treatment hydrolyses the amorphous parts of cellulose present in fibers and treated material contains more crystalline cellulose. This is due to formation of Cell O-Na$^+$ structure after the treatment and Na$^+$ ions come to fit in the unit cell of cellulose.[18] Increase in the crystallinity of alkali-treated fabric is due to the removal of interfibrillar cementing materials, which leads to better packing of cellulose chains.[19] In XRD pattern of PVA-treated fabric (Figure 6), it was observed that the fabric has high crystallinity than untreated, whereas the PC-treated fabric shows the amorphous peaks rather than crystalline.
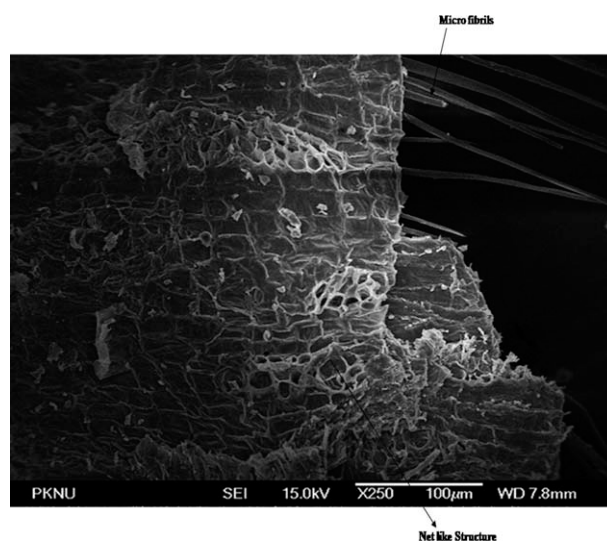

Figure 7 FE-SEM of the untreated fabric surface structure.

Figure 6 XRD of PVA-treated prosopis fabric

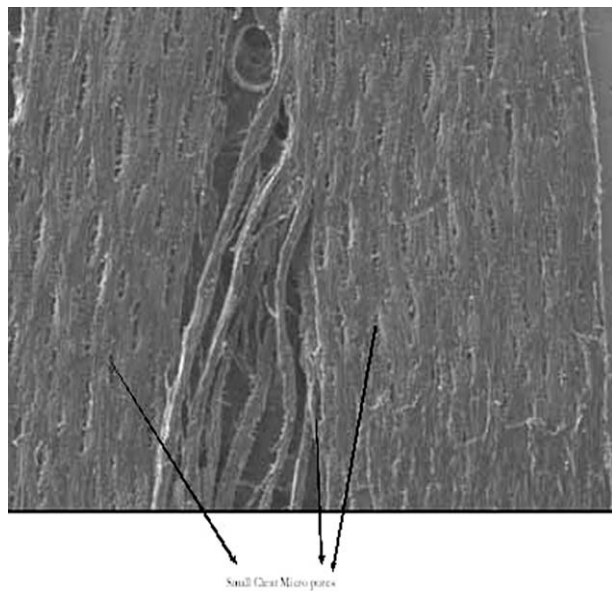**Figure 8** FE-SEM of the untreated fabric cross surface.

Figure 9 FE-SEM of the alkali-treated fabric longitudinal surface.

Morphological studies of the fabric

The surface and cross-section of fabric were examined by FE-SEM technique. Figures 7–12 demonstrates FE-SEM micrograms of untreated, NaOH, PVA, and PC treated of fabric surfaces at different magnifications (15 and 20 KV), respectively. The micrograms of the untreated and treated fabric surfaces reveal the changes in the porous structure of the fabric. Figures 7 and 8 show that the untreated fabrics are covered with a layer, whose composition is probably waxy substances, lignin, and hemi cellulose. It was observed that the layer is
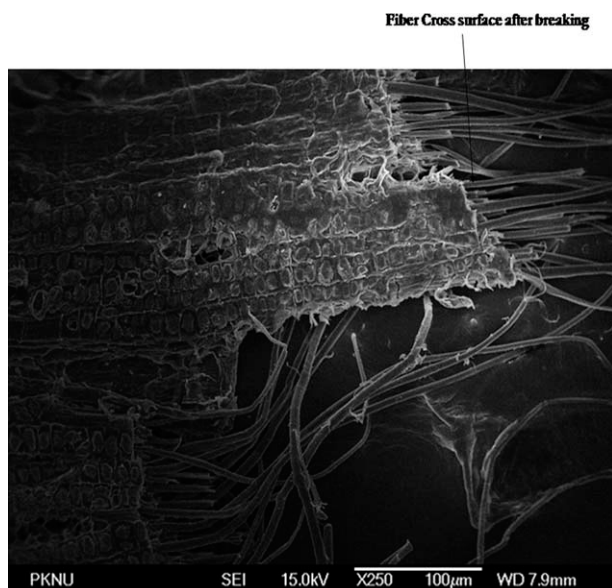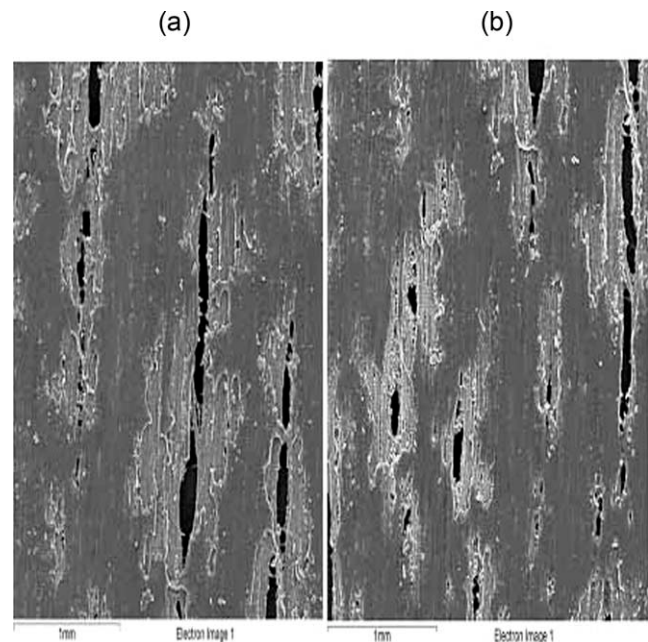

Figure 11 FE-SEM of the PVA-treated fabric surface at (a) 15 KV and (b) 20 KV.

not evenly distributed along the fabric surface, but its thickness varies from point to point. From the micrograms, it is clearly evident that the fabric has several void regions/micropores (ring and semi ring porous) in between fibers. This structure could be possible for the penetration of resin into void regions, which is favorable for making fibrous composites and for obtaining good bonding between the matrix and the fabric. The fabric actually consists of
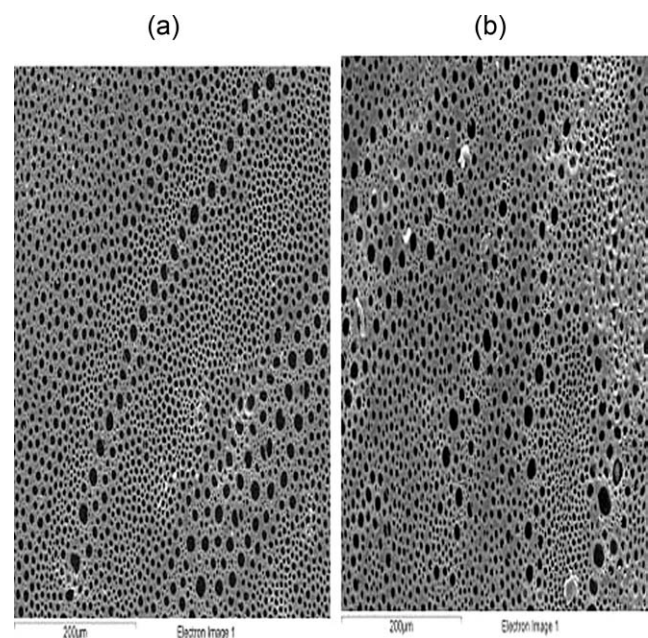

Figure 10 FE-SEM of the alkali-treated fabric side cross surface.

Figure 12 FE-SEM of polycarbonate-treated fabric surface at (a) 12 KV and (b) 15 KV.

TABLE III
Tensile Properties of *Prosopis chilensis* Fabric
With and Without Treatment

Prosopis chilensis fabric	Tensile strength (MPa)	Tensile modulus (MPa)	Elongation at break (%)
Untreated	148.3	6475.4	1–3
Alkali treated	156.7	7446.8	2–4
PVA treated	159.4	7653.6	2–3
PC treated	161.5	7918.9	1–2

individual fibers having many cells with small micro pores, which have been surrounded together by rich lignin. Each individual fiber consists of micro fibrils made of cellulose chains in amorphous matrix of lignin and hemi cellulose.

FE-SEM of alkali-treated fabric at different magnifications is shown in Figures 9 and 10. From the figures, it is clearly evident that the fabric has a rough cellular structure and clear micro pores; this structure may be due to the removal of lignin. Similar observations were also reported in the case of some ligno-cellulosic fibers.[1,2,4,10] On NaOH treatment, a smoother surface morphology of the fabric appears (Figures 9 and 10) in comparison with untreated fabric (Figures 7 and 8). The fibrils are well exposed in treated fabric. This treatment cleans the fibers by leaching out the amorphous waxy cuticle layer and partial removal of lignin and hemicellulose, which are the cementing materials in vegetable fibers.[20–22] Figure 11(a,b) reveals the SEM of the PVA-treated fabric surface at (a) 15 KV and (b) 20 KV magnifications, respectively. It shows that PVA has been formed on the fabric surface as a layer. Immersion of the fabric samples in 4% PVA solution do not lead to any notable modifications of fabric. Figure 12 demonstrates the SEM micrograms of the poly (carbonate)-treated fabric surface at (a) 12 KV and (b) 15 KV, respectively. From the figure, it was observed that the PC film has been formed on the fabric surface, filling the void regions in it and thus making the fabric a continuous one with looking as porous film.

Tensile properties

Tensile strength, modulus, and elongation at break of the treated and untreated fabric specimens are shown in Table III. Tensile properties were performed 10 times in each case, and the average values have been reported in Table III. From Table III, it is seen that the tensile strength of poly carbonate-treated fabric are the strongest, followed by alkali-treated fabric. It was also observed that untreated fabric specimens show the lowest tensile strength. Varada rajulu et al.[23] reported that the tensile

strength of the Hilde guardia fabrics increased on alkali treatment. The tensile strength of PC treated fabric in the present case is 161.5 MPa, the highest strength when compared with other fibers, such as Kapok, Hilde guardia, and Ridge guard. Therefore, we conclude that this fabric is a useful for fabrication of composites.

Table III shows the tensile strength, tensile modulus, and elongation at break of untreated and NaOH-, PVA-, PC-treated fabrics. From Table III, maximum tensile strength was observed for PC and PVA treated fabrics. This may be due to formation of PC and PVA films on the fabric, filling the void regions in it. Such a filling is expected to make the fabric continuous and brings cohesion between the fibers of the fabric, leading to uniform stress transfer between the fabrics. On the other hand, small changes occurred in % of elongation at break. Increment in % of elongation at break for PC- and PVA-treated fabric is 9 and 7.4%, respectively, when compared with untreated. From Table III, it is clearly evident that the fabric having higher strength shows lower % elongation at break and vice-versa. This may be attributed due to the modifications that occurred at the cellulose regions.

The % of increase in tensile modulus of treated fabric is 15% than that of the untreated fabric, while the increment in modulus for PVA- and PC-treated fabrics are 10.4 and 22%, respectively. Tensile strength and tensile modulus of the fabrics increases with surface modification by polymer treatment. This is understandable that polymer treatment cleans off the week bonding material on the fabric, then the fabric becomes rough and strong. The fabric shows better elongation at break. Based on the above results, we can conclude that the fabric on surface treatments will produce composites with improved mechanical properties. For that reason, we can recommend this fabric in developing well-oriented fibrous green composites.

CONCLUSIONS

Based on the above studies, the following conclusions are drawn:

FTIR studies revealed the structural features such as stretching of O—H, C—O, C—H and bending of CH bond characteristics of chemical constituents of the fabric, such as cellulose, hemi cellulose and lignin in the fabric as well as presence of some acids. Fabric on treatment with NaOH leads to the modification of its thermal stability and was found to increase on alkali treatment. Treatment with 3% NaOH cleans the fabric by removing the amorphous compounds and increases the crystallinity, which was confirmed by XRD analysis. FE-SEM reveals that the surface morphology of the fabric was better

after the treatment and is favorable for making fiber reinforced composites. Scanning electron micrograms indicates that the formation and filling of the void regions of the fabric on PVA and PC treatments. Tensile properties of the fabric samples were also determined and found to be enhanced on PVA and PC treatments. Because of its high tensile strength and modulus of this fabric, one can prepare the composites using *Prosopis* fabric as reinforcement for improved properties where the high strength and thermal resistance is required.

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