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Characterization of Leptadenia Pyrotechnica (Khimp) Fibers by FTIR and SEM Analysis

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ABSTRACT

Leptadenia pyrotechnica fibers, an environmentally and ecologically friendly product, were chemically modified and treated with 10 % NaOH and triton 100 solution at 60^0 temperature for 4 hour. In the paper these untreated and treated fibers were characterized and morphologically analyzed by Fourier transform infrared spectroscopy (FTIR) and Scanning electron microscopy (SEM). Leptadenia pyrotechnica fibers were evaluated by FTIR to reveal the functional groups. This research seeks to explore the use of chemical modification with alkali treatment (NaOH & Triton 100 treated) on the fiber surface and fiber surface morphology analyzed by Scanning electron microscopy (SEM).

KEYWORD- Leptadenia pyrotechnica fibers, FTIR, SEM.

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INTRODUCTION

In India, many vegetable fibers, having good potentiality, are not being properly exploited. If these fibers are put to proper use, the rural economy of the country could be improved significantly. One of these is the wildly grown fiber crop (Leptadenia pyrotechnica), belonging to the family 'Asclepiadaceae' and is popularly known as 'Khimp' in Rajasthan, 'Khip' in Gujarat , and 'Kip' in Punjab. Leptadenia pyrotechnica(Khimp) fibers is extensively grown in the northern-west part of India, especially in the states of Rajasthan, Gujarat, Punjab and Haryana. The Chemical constituent of Khimp fiber is given in Table 1.

Table 1- The Chemical constituent of Khimp fiber

Chemical constituent	Percentage (%)
α-cellulose	75.26
Hemicellulose	11.7
Lignin	4.93
Pectin	3.84
Ash	2.77

The characteristics of Khimp fibers are studied in this paper with a view to exploring wider uses for it. The high α-cellulose and low lignin contents of the fiber make it suitable for use in various applications.

MATERIAL AND EXPERIMENTAL TECHNIQUE

Leptadenia pyrotechnica (Khimp) fibers-

Commercially, Khimp plant was collected from the local area of Bikaner district situated in the state of Rajasthan. The fiber was extracted from the green stem of the khimp plant by crushing, followed by retting and combing. These raw fibers were washed with water to remove undesirable materials and dried in an air oven at 80°C for 6 h. After that these fibers were chopped into the desired length ranging from 2 to 15 mm for the characterization of fibers.

Fiber modification (Treated fiber)-

Alkali treatment

The fibers were taken in a stainless steel vessel, a 10% solution of NaOH was added in to the vessel and the fibers were allowed to soak in the solution at 60^0 temperature for 4 hour. The fibers were then washed thoroughly with water to remove the excess of NaOH sticking to the fibers. Final washing carried out with distilled water containing little acetic acid and the fibers were then dried in hot air oven at 70°C for 3 hours. They were then cooled to room temperature and air dried.

Triton 100 treatment

The fibers were taken in a stainless steel vessel. A 10% solution of triton 100 was added into the vessel and stirred well. This was kept for 4 hour at 60^0 temperatures with subsequent stirring. The fibers were then washed thoroughly with water to remove the excess of triton 100 sticking to the fibers. Final washings were carried out with distilled water. After that fibers were heated at 70^0 in an air-circulating oven for 4hrs. They were then cooled to room temperature and air dried.

Fourier Transform Infrared Spectroscopy

Fourier Transform Infrared Spectroscopy (FTIR) is a valuable tool in the determination of functional groups actively interacting within a fiber. The fibers were taken in mixture form for characterization. Mixture form of Khimp fibers was examined by using FTIR measurement which uses a Perkin Elmer Spectrum Version 10.4.00 and the standard KBr pellet technique. Various scans were taken for the sample between 400 cm^{-1} and 4000 cm^{-1}, with a resolution of 2 cm^{-1}. Khimp fibers were ground and mixed with KBr and then pressed into a pellet for FTIR measurement.

Scanning electron microscopy-

The Scanning electron microscopy (SEM) photographs of fiber surfaces of untreated and treated fibers were taken using a scanning electron microscope of model HV 20 KV with 150 Pressure. The surface of the fractured specimens was examined using SEM to study the fracture mechanisms.

ANALYSIS OF RESULT

Analysis of Fourier Transform Infrared Spectroscopy

Fourier transform Infrared spectrum (FTIR) of Khimp fibers is displayed in Figure 1 to 3. The typical functional groups and the IR signal with the possible sources are listed in Table 2. It could be observed from Table 2 that characteristic feature of lignin, cellulose and Hemi cellulose components indicating ligno-cellulosic nature of Khimp fibers. The absorption band and their assignments in the Infrared spectra of untreated Khimp fibers are compared with those of treated Khimp fibers. The medium intensity absorption band at 1739.47cm^{-1} is assigned to the C=O stretching of carboxyl and acetyl group in the Hemicelluloses content of the untreated Khimp fiber which appear as a peak at 1739.45 cm^{-1} and 1739.10cm^{-1} in case of NaOH and triton 100 treated Khimp fibers. The sharp weak band at 1029.20 cm^{-1} in untreated fiber and 1029.69 cm^{-1} & 1026.51 cm^{-1} in treated fiber is the characteristic of β glycosidic linkage contributed by both Cellulose, hemicelluloses in the fiber while a small peak at 552.87 cm^{-1} in untreated fiber and 549.38 cm^{-1} & 549.92 cm^{-1} in treated fiber is due to the stretching vibration of pyranose ring in the hemicelluloses.

Table 2- Infrared spectrum (FTIR) of untreated and treated Khimp fibers

Group / vibration	Source	Untreated Khimp fiber	NaOH (10%) treated Khimp fiber	Triton 100 (10%) treated Khimp fiber
Hydrogen bonded OH stretching (s)	Cellulose, Hemicelluloses	3345.51 cm^{-1}	3335.13 cm^{-1}	3332.82 cm^{-1}
CH stretch (m)	Cellulose, Hemicelluloses	2907.97 cm^{-1}	2919.2 cm^{-1}	2916.83 cm^{-1}
C=O stretch (m)	Hemicelluloses	1739.47cm^{-1}	1739.45 cm^{-1}	1739.10cm^{-1}
CH_2 symmetrical bending (m)	lignin	Not found	1436.8 cm^{-1}	Not found
CH bending (w)	lignin	1366.43 cm^{-1}	1367.77 cm^{-1}	1366.62 cm^{-1}
Ant symmetrical bridge C-O-C stretch/ C-O stretch (w)	Cellulose, Hemi cellulose	Not found	1218.37 cm^{-1}	1218.51 cm^{-1}
β glycosides linkage (w)	Cellulose, Hemicelluloses	1029.20 cm^{-1}	1029.69 cm^{-1}	1026.51 cm^{-1}
Pyranose ring (w)	Hemicelluloses	552.87 cm^{-1}	549.38 cm^{-1}	549.92 cm^{-1}

s- Strong, m- Medium and w- Weak

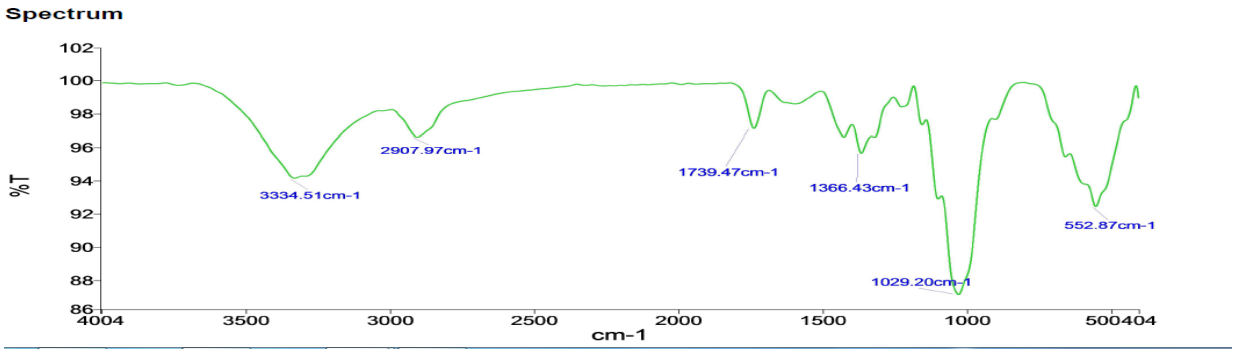

Figure -1 Untreated khimp fiber

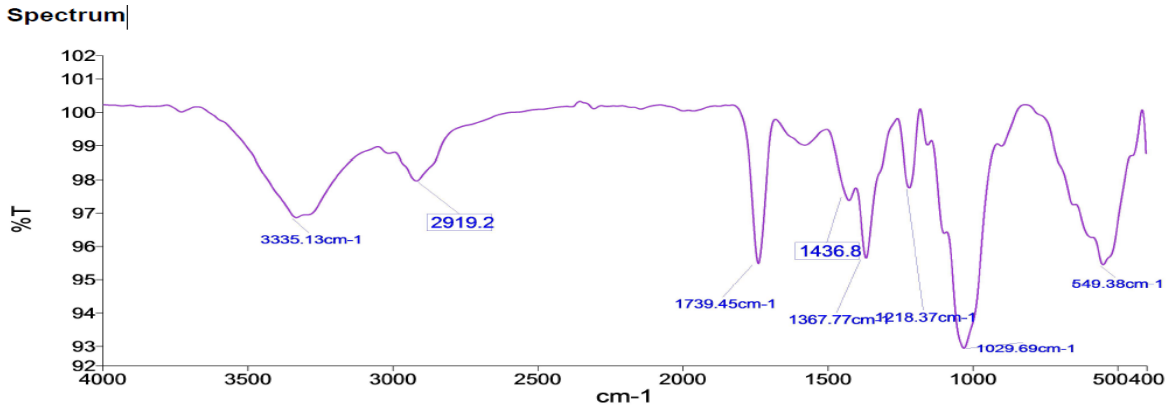

Figure -2 NaOH 10% treated khimp fiber

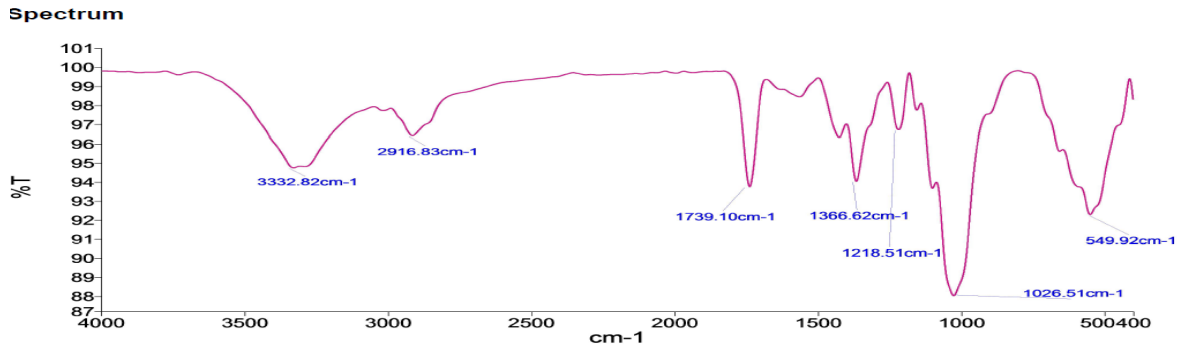

Figure -3 Triton 100 (10%) treated khimp fiber

Analysis of scanning electron microscopy

An analysis of the fiber surface shows the morphological features of khimp fiber. Photomicrography of raw fiber (Figure-4) shows that the fibril is arranged longitudinally. The surface of individually cells is not clearly visible perhaps due to surface debris. The SEM photograph of bleached Khimp fiber (Figure–5) shows that the cells disintegrate from the fiber surface due to partial removal of the cementing material lignin by bleaching, rendering the cell modality**.** Alkali treatment (NaOH treated) could influence the inner cellulosic components of the fiber and the non-cellulosic components such as hemicelluloses, lignin and pectin simultaneously. After alkali treatment, the hemicelluloses, lignin and surface impurities such as waxes and oils were removed from the fiber surface. It is indicated that the hemicelluloses, lignin and pectin of the fibers were dissolved by the alkali solution. The removal of surface impurities such as waxes and oils leads to a cleaner and rougher fiber surface. This rougher surface may be facilitates both mechanical interlocking and bonding reaction due to the exposure of the any thermosetting resin, this effect may increase or decrease the fiber/matrix adhesion.

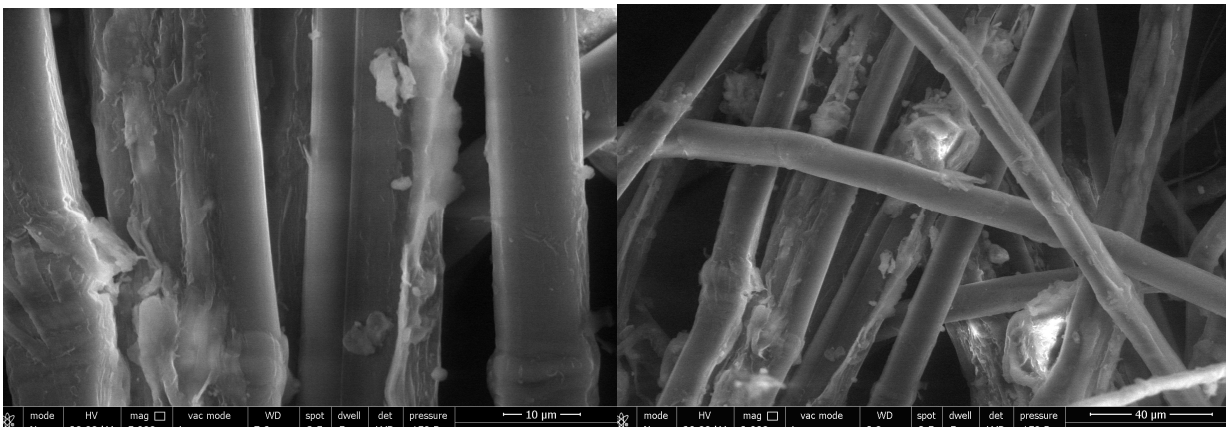

Figure -4 SEM photomicrograph of fibril surface of untreated khimp fiber without bleaching

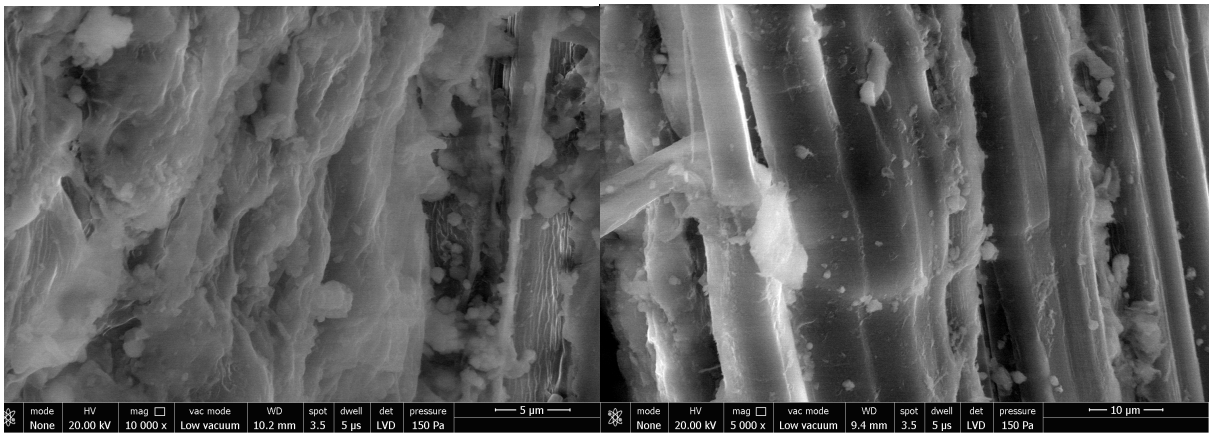

Figure -5 SEM photomicrograph of fibril surface of untreated khimp fiber with bleaching

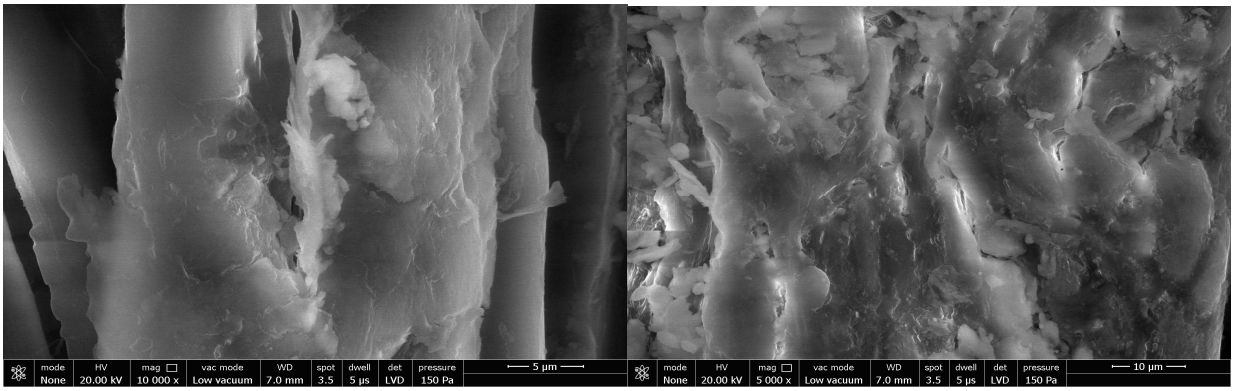

Figure -5 SEM photomicrograph of fibril surface of NaOH 10 %Treated khimp fiber

CONCLUSION

A systematic and comprehensive study on the characteristic and behavior of Khimp fibers presented in the paper concluded that-Treated khimp fiber may be considered an alternative for the manufacturing of the polymeric composites. FTIR analysis shows a slight difference between in untreated and treated Khimp fiber. The SEM photomicrograph of the longitudinal surface of untreated fiber bundles in (Figure-4) shows the presence of wax, oil, and surface impurities. Waxes and oils provide a protective layer to the surface of the fibers. The longitudinal view of the 10% NaOH-treated Khimp fibers in (Figure-5) shows a very clean surface. Its surface topography shows the absence of surface impurities, which were present in the untreated fiber. This rougher surface may be facilitates both mechanical interlocking and bonding reaction due to the exposure of the any thermosetting resin, this effect may increase or decrease the fiber/matrix adhesion.

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