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SEM, FTIR study on natural fibers of plant *Prosopis Juliflora* and biodegradable composites reinforced with natural fibers

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Abstract: In current scenario, researcher and scientist are getting rapid attention in research and development in the field of natural fiber composite NFCs due to its better formability, recyclability, low cost and eco-friendly green features. This green technology of making bio-degradable composite material by using reinforcement technique focused scientists and researcher on the utilization of natural fibers as compared to synthetic fibers. Such prepared composite materials can find applications in interior housing; automotive, marine, domestic, and other applications include military, building, packaging, consumer products and construction industries for ceiling paneling, partition boards etc. Further, morphological changes by various physical or chemical treatment methods can also overcome few deficiencies of natural fibers in order to make composite materials. This paper describes the advantages of chemical treatment of natural fiber in order to make composite materials by using green technology. This paper further provides the surface modification of alkali treated prosopis juliflora fiber by using scanning electron microscope and gives the necessity of alkali treatment for further fabrication of composites. This paper further deals the detail classification of chemical bond present in alkali treated and untreated fibers by using Fourier transform infrared spectroscopy.

Keywords **- alkali treatment, Fourier transform infrared spectroscopy FTIR, Natural fiber composites NFCs, prosopis juliflora PJ ,scanning electron microscope SEM**

1. INTRODUCTION

This paper deals with the Natural Fibers and it's Composites NFCs by using the reinforcement technique in polymer matrix; this highlights the important applications of NFCs, due to their renewable resources, low cost, easily fabrication, continuous and easily availability and most essential their ecofriendly properties [1]. Industrial uses of natural fibers increasingly gain attention from various composites is growing rapidly to meet diverse end uses in transportation, low cost building, and other construction industries. Growing environment, temperature, humidity, and soil quality create an impact on the properties and qualities of natural fibers. [2].

There are wide range of varieties of natural fibers such babool(prosopis juliflora), kenaf(Hibiscus cannabinus), sisal(Agave sisalana), hemp(Cannabis sativa), flax(Linum usitatissimum), abaca(Musa textilis), and ramie(Boehmeria nivea) that have been widely used to make natural fiber reinforced composites. Among these natural fibers, babool (prosopis juliflora) fiber was chosen as a reinforcement material in this study because it is planted easily in desert area and can tolerate wide range of temperature , soil quality like sandy, stony, heavy clay etc. and it can also thrive in humid environment . It also has fast growth rate and fast spreading nature in comparison with other plants. The mechanical properties like tensile strength of cheap, lightweight and biodegradable PJ fiber are found good as compared to other natural fibers [3]. NFCs have various physical and mechanical properties including high specific strength and high specific stiffness [4,5].Furthermore, PJ fiber composites are attractive structural materials due to their advantages such asenvironmental friendliness, acceptable biodegradability, low density and low cost [6,7] . Furthermore, PJ fiber composites also have other limitations including incompatibility between the hydrophilic natural fibers and hydrophobic polymers. Among the numerous factors that influence the performance of the polymer composites reinforced with natural fibers is the resulting chemical modification [8]. For improving the compatibility between hydrophobic polymer and hydrophilic cellulose fiber chemical treatment can be a good process to improve the interfacial bonding between fiber-matrix. It has also been proposed that modification of fiber surface is necessary to increase the mechanical and thermal properties of fiber reinforced composites [9,11]. There are several chemical treatments have been suggested by researcher like bleaching, acetylation and alkali treatment for improving interfacial bonding between fiber and matrix [10].

2. MATERIAL AND METHODS

The main disadvantage of natural fibers in reinforcement to composites is the poor compatibility between fiber and matrix due to the hydrophilic nature of fibers and hydrophobic nature of polymer macromolecules. This is very important issue, since the simple addition of natural–organic fibers to a polymer matrix may lead to poor mechanical properties in comparison to the neat polymer. Therefore, natural fiber modification is considered in modifying the fiber surface properties to improve their adhesion with different matrices. The main technique was summarized as follows:

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2.1 PHYSICAL TREATMENT OF NATURAL FIBERS

Physical treatment gives an impact to the surface of natural fibers without changing its. Advantages of these treatments over chemical treatment are enhanced mechanical, physical and thermal properties of treated NFCs. However, physical treatments are costlier than the chemical treatments. Therefore, researchers and scientist are giving importance to chemical treatments over physical treatments [14].

2.2 CHEMICAL TREATMENT AND FIBER MODIFICATION

Chemical treatment is one of the most used treatments for natural fibers in order to make reinforce thermoplastics and thermosets. This treatment completely removes amount of Lignin, wax and oils which is covering the external surface of the fiber cell wall. The important modification achieved with alkaline treatment is the disruption of the hydrogen bonding in the network structure, thereby increasing the surface roughness and improves the fiber wetting [8,12] . Alkali treatment also reduces the diameter of fiber cell well by removing the impurities of lignin, wax and oil [13].

Furthermore, this alkali treatment leads to develop roughness on the surface of fiber and further increase cellulose content, which results better interfacial bonding between PJ fiber and the polymer and increasing the number of bonds between the fiber surface and the polymer [13]. In this paper this alkali treatment improvement on fiber surface and removal of lignin, wax and oil has proved with SEM and FTIR analysis.

Fig.1 (a) untreated PJ fibersFig.1 (b) PJ fibers after alkali treatment

3. STEM COLLECTION AND FIBER EXTRACTION FROM PLANT *PROSOPIS JULIFLORA*

Firstly, stems were collected of *prosopis juliflora* from different regions of Bikaner district situated in Rajasthan INDIA. After that shade drying of fibers was done for 30 days in laboratory. After drying some of stems were dipped into normal water and some dipped into alkali medium NaOH for 25-30 days. The soaking process loosens the fibers and fibers can be extracted out easily. Than after removing from water and alkali medium stems were dried until all moisture is taken out. After it fiber extraction was done from the each stem. Untreated fibers were washed from normal water and treated fibers were washed from alkali solution. Alkali treatment of PJ fiber was done to remove surface hemicellulose lignin and impurities [13]. Sodium hydroxide (NaOH,) with 6 wt% concentration was used to promote better interaction between the *Prosopis juliflora* fiber and polymer matrix. Fibers were soaked in NaOH solution for 24 h at room temperature. Neutralization of the fibers was done with immersion of fiber into running tap water.[12,13]. Then fibers were dried in an oven at 40 ◦C temperature for 24 h for further using to fabricate composite materials [13]. After this diameter and length of the extracted fibers is measured with the help of screw gauge and scale respectively. The Diameter is ranging from 50 μm – 100 μm and length 2 cm – 10 cm [15].

Scanning electron microscopy (SEM) was used to characterize the properties of treated and untreated PJ fibers.

4. RESULTS

4.1 MORPHOLOGICAL ANALYSIS OF *PROSOPIS JULIFLORA* FIBER

Scanning electron microscope (SEM) was conducted at Malaviya national institute of technology Jaipur(MNIT) by using Nova Nano FE-SEM 450 (FEI) with an accelerated voltage of 15.00kV and an attainable vacuum level of 6.10×10^{-3} Pa with ETD detector. The SEM images of treated PJ and untreated PJ fibers are taken at different resolution up to 100000 magnifications. All the specimens are coated with gold before the analysis.

The SEM images to identify the surface morphology of untreated and treated *prosopis juliflora* fiber are displayed in Figure 2 (a,b) .Comparison images between untreated and treated fibers prove that alkali treatment creates an effect on the surface of the *prosopis juliflora* fiber. As we can clearly see that from SEM images of untreated *prosopis juliflora* fiber are not smooth and have irregular due to the presence of impurities such as pectin, lignin, wax, etc. that is covering the surface of the fiber as shown in Figure 2(a). While in Figure 2(b), the physical appearance shows that treated *prosopis juliflora* fiber has a clear, clean and rough surface after alkaline treatment. This treatment is necessary in order to remove the hemicellulose, lignin and wax from the outer surface of the prosopis juliflora fibers, which improves the interfacial bonding between the fiber and the polymer matrix.

This is supported by the research results of Masitah et al. who concluded that no impurities found at the surface of alkali treated fibers and this alkali solution increased the interfacial bonding between the fiber and the polymer matrix.

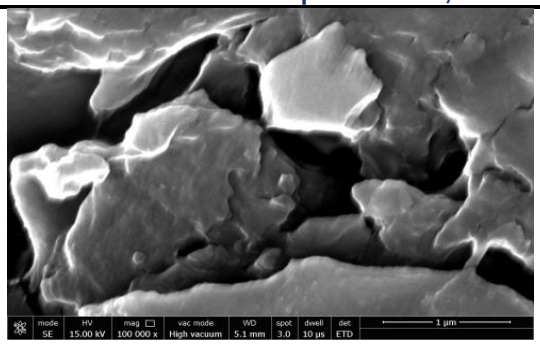

Fig. 2(a). SEM image of untreated fiber of *prosopis juliflora*

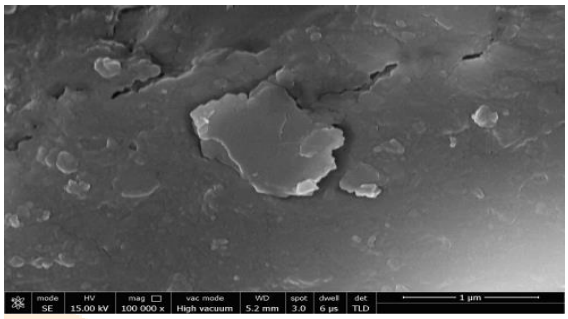

Fig.2(b). SEM image of Treated fiber of *prosopis juliflora*

4.2. FUNCTIONAL GROUP ANALYSIS OF *PROSOPIS JULIFLORA* FIBER

Fourier Transform Infrared Spectroscopy (FTIR) analysis was conducted at Malaviya national institute of technology Jaipur(MNIT). The NaOH-treated and untreated fibres were powdered and tested with FTIR test using FTIR spectrum 2 (Perkin Elmer) with range 4100-400cm^{-1} spectrum version 10.4.00 instrument.

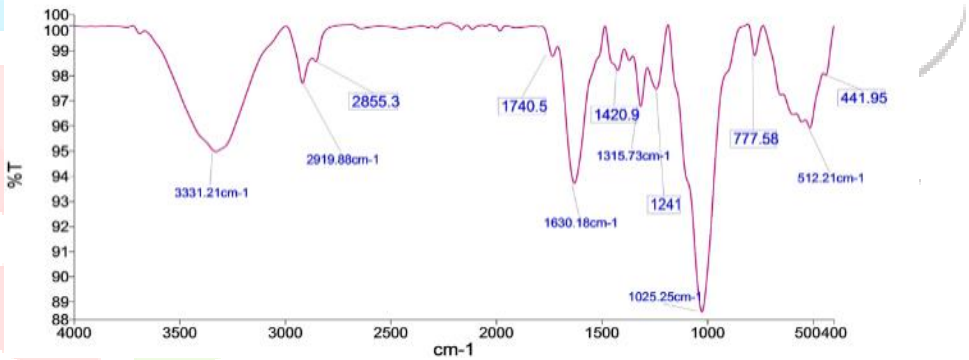

Fig. 3(a) FTIR Spectrum of untreated PJ fibers

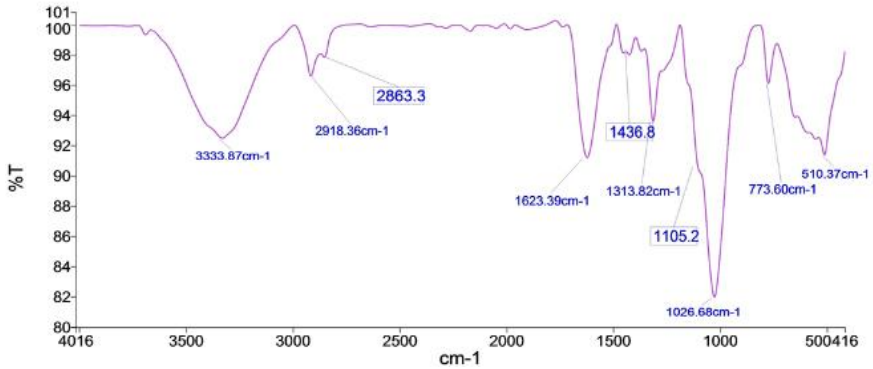

Fig. 3(b) FTIR Spectrum of treated PJ fibers

Fig.3(a) and Fig.3(b) gives the infrared spectrum of PJ untreated and treated fibers respectively. there is an absorption peak is sharp and weak due to OH stretching which is found in untreated PJ fiber at 3331.21 cm^{-1} while in treated fiber it is found at 3333.87 cm^{-1} which shows the presence of cellulose in plant. CH stretching peak is found at 2919.88 cm^{-1} in untreated while in treated it is found at 2918.36 cm^{-1} which shows the presence of methyl and methylene groups and C=O stretch peak is found at 1740.5cm^{-1} in untreated PJ fiber while in treated PJ fiber it was absent which indicates the acetyl groups of hemicelluloses. CH_2 symmetrical bending peak is found at 1420.9 cm^{-1} in untreated PJ fibers while in treated PJ fibers it is found at 1436.8 cm^{-1}. CH

bending peak is found at 1315.73 cm^{-1} In the untreated PJ fibers while in treated PJ it found at 1313.82 cm^{-1}. Similarly, the peak for Anti symmetrical bridge C-O-C stretch is found at 1214 cm^{-1} in untreated PJ fibers and in the treated PJ fibers it was absent Which again shows which removal of hemicellulose. β- Glycosides linkage peak is found 1025.25 cm^{-1} in the untreated PJ and in treated it was at 1026.68 cm^{-1}. And pyranose ring peak was at 512.21 cm^{-1} in untreated PJ fibers and 510.37 cm^{-1} in treated found at 510.37 cm^{-1}

Table 1. Assignment of IR absorption peaks for untreated and treated PJ fiber [16,17,18]

Chemical bond	Vibration mode	Untreated PJ fiber Wavenumber (cm^{1})	Treated PJ fiber Wavenumber (cm^{1})	Reference
O-H	stretching	3331.21 cm^{-1}	3333.87 cm^{-}	Rout,Arun Kumar, et al.(2016)
C-H	stretching	2919.88 cm^{-1}	2918.36 cm^{-1}	Boopathi et. al. (2012)
C=O	stretching	1740.5cm^{-1}	Absent	Rout,Arun Kumar,et.al.(2016)
CH_2	Bending	1420.9 cm^{-1}	1436.8 cm^{-1}	Poljansek, Ida, et. al. (2005)
CH	Bending	1315.73 cm^{-1}	1313.82 cm^{-1}	Latif, Rashid,et al. .
pyranose ring	Stretching	512.21 cm^{-1}	510.37 cm^{-1}	Latif, Rashid,et al.

5. CONCLUSION AND FUTURE SCOPE

- Alkali treatment increases the free voids of surface of fiber which can improve the bonding between fiber and the matrix.
- By SEM analysis It has also been found that by giving an alkali treatment to the fiber it increases the diameter of pores as well as the number of pores on the surface of fiber
- The removal of hemicelluloses, lignin, and other surface impurities was confirmed with SEM and FTIR analysis. Peak for C=O and C-O-C was absent in alkali treated PJ fiber this can be due to removal of lignin and hemicellulose.
- Mercerization process may leads to an increase in the amount of amorphous cellulose at the cost of crystalline cellulose and to remove the hydrogen bonding in the network structure i.e. increases the fiber surface adhesion characteristics by removing natural and artificial impurities, thereby it produces rough surface.
- PJplanthasnocommercialvalueespecially inurbanareaandgrowsveryeasilyatunwantedpublicplacessoinsertionoffiberintopolymermatrix will createnon-toxicand Easilybio-degradablematerials and can reduce environment pollution.
- By using Sodium hydroxide (NaOH,) having 6 wt% concentrations can be used to give better interaction between the fiber and polymer matrix.

CONFLICTS OF INTEREST

The authors declare no conflict of interest.

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