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Investigations on Alkali Treated Modified Fibers of Desert Plant *Prosopis Juliflora*

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Abstract

The development of natural fiber reinforced composite material is increasing at very fast rate due to their eco-friendly and biodegradable nature. NFCs natural fiber reinforced composites have various properties such as low cost, low density, recyclability, renewability and good physical and mechanical strength. NFCs have wide range of applications such as in automobile, sports, aerospace, marine, home appliances and in building construction. In this paper we used *prosopis juliflora* desert plant fibers as a filler to make biodegradable composites and alkali treatment was done to modification of fiber in order to make high strength composites materials. By using scanning electron microscopy (SEM), water absorption tests, and Fourier transform infrared spectroscopy (FTIR), this paper examines the effects of surface modification on the fibers. By conducting SEM analysis it has been observed that the chemical treatment of fibers can improve adhesion of the composites. Water absorption test concluded that due to the higher porosity and better surface energy of the treated fiber it had a higher rate of water absorption than the untreated fibers. FTIR results concluded that due to more crystalline structure and more ordered structure crystallinity index of the treated fibers increases compared to untreated fibers. FTIR results proves that TCI total crystallinity index and the LOI lateral order index is high for PJ treated fibers as compared to untreated PJ fibers.

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Keywords
ATF (Alkali Treated Fiber);
FTIR (Fourier Transform Infrared Spectroscopy);
NFCs (Natural Fiber Composites);
PJ (*Prosopis Juliflora)*;
SEM (Scanning Electron Microscopy);
UTF (Untreated Fiber).

Introduction

In current scenario, most of the research has been found in material science field. Researcher and scientist are getting a rapid attention towards making biodegradable composite materials reinforced with natural fibers instead of synthetic fibers which causes environment pollution. NFCs can found many applications in the field of aerospace, marine, building construction, home appliances due to the properties like low cost, easily fabrication, high specific strength and good physical properties.[1]

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In automotive industry, natural fiber composites are used for manufacturing of interior and exterior components such as car dashboard, bumper beams and door panels. In medical field, NFCs can be used for manufacturing of artificial bones, implants and other medical devices. NFCs are also used in the sports industry for making of sports equipment like hockey sticks, boxing gloves, golf clubs, cricket bats, tennis rackets, etc. due to its light weight and high strength.[13] NFCs are also used for manufacturing of furniture and packaging material. Apart from the applications mentioned above, the research in the field of material science is being done in order to explore more potential applications of NFCs. Research is being done on developing new processes and techniques to improve the properties of NFCs. Also, research is being done to explore the possibilities of using NFCs in the field of energy storage and generation. Furthermore, research is being done to find out the potential applications of NFCs in the field of defense.[2]

This paper highlights the importance of polymer composites reinforced with Desert plant *prosopis juliflora* natural fibers in order to make biodegradable material. *Prosopis juliflora* plant was chosen as reinforcement for this study due its fast - growth rate and spreading nature in comparison with the other plant. This plant can tolerate harsh environment and wide range of temperature. It can survive in soil quality like sandy, stony, heavy clay etc. Plant fiber consist a complicated ribbon type structure of lumen and cell wall.[9] Cell wall is made up of three layers of middle lamella, primary wall and secondary wall. Secondary wall contains crystalline cellulose. Primary wall consist cellulose, hemicelluloses, pectin, lignin etc. and the middle wall is responsible for mechanical strength.[1,9] Lumen in the fiber structure is responsible for the water transportation.[1]

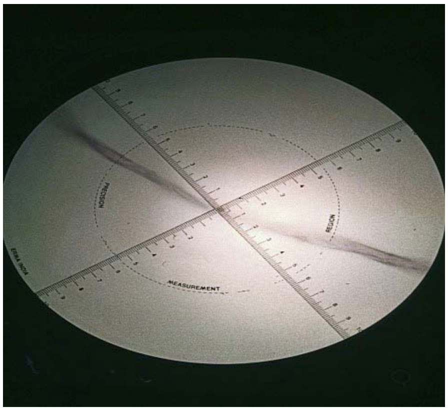

Fig. 1: PJ Plant fiber internal image taken from microscope

The main aims and goals of plant fiber reinforced composites are to create materials with improved mechanical properties, thermal stability, durability and environmental compatibility. Plant fiber reinforced composites have limitations including incompatibility between the fibers and matrix due to the hydrophilic nature of fibers and hydrophobic nature of the matrix. Chemical treatment is required to improve the compatibility between hydrophobic polymer matrix and hydrophilic fibers. Chemical treatment can enhance the interfacial bonding between the fibers and matrix.[3,4] Research on NFCs is developing at an exponential rate as they have the potential to reduce pollution levels, energy consumption and enhance sustainability. Therefore, it is important for researchers to continue to explore and improve the characteristics of NFCs to make them suitable for various applications.[11] NFCs offer an attractive alternative to traditional plastic-based materials because their natural fiber content makes them more durable and recyclable, and they can decompose relatively rapidly when exposed to microbial attack and environmental conditions. Furthermore, they are typically lightweight, biodegrade naturally, and are usually aesthetically attractive. Natural fiber biodegradable composites also have many advantages over synthetic materials, such as a lower carbon footprint and better tensile strength. Research into natural fiber biodegradable composites is ongoing, with recent research involving exploring multiple types of fibers, polymers, and their blends.[11]

Materials

Firstly, stems were collected from plant *prosopis juliflora* in the different desert region of district Bikaner situated in India. After that shade drying of fibers was done for 25 days. Stems were divided into two parts and first part of stem collection was dipped into water and other stem part was dipped into NaOH solution for 15 days. After this step stem were washed with pure water and fibers were easily extracted from each stem. Soaking process loosens the fibers and it can extract easily. NaoH soaked fibers again dipped into Sodium Hydroxide (NaOH) 6% wt Concentration to improve the bonding between *prosopis juliflora* fiber and polymer matrix.

Fig. 2: Untreated fibers

Fig. 3: alkali treatment of fibers

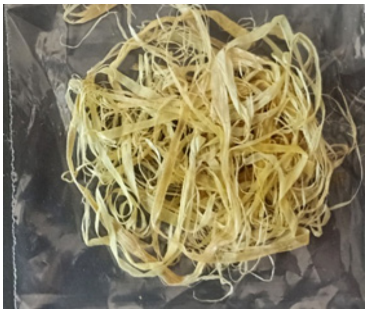

Fig. 4: alkali treated fiber

Fig. 5: Chopped untreated fibers.

Fig. 6: Chopped untreated fibers.

Methods

Scanning Electron Microscopy

Scanning electron microscope (SEM) is a scientific instrument and a powerful tool for seeing Nano-space and microscopic view of any substance on a very fine scale. It uses a very high energetic focused electron beam over a surface to examine the substance. 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 were taken at different resolution up to 100000 magnifications. All the specimens are coated with gold before the analysis.

Water Absorption Test

Water absorption tests indicate the amount of water that a material can absorb, which is an important part of understanding how that material will perform in specific settings. It also can affect the strength, weight, and how easily the material can be formed or shaped. When making composites from natural fibers, it is important to test the water absorption properties of the fiber since it will affect the strength and durability of the composite material.

Water absorption test was conducted at standard atmospheric condition and weight of sample measured by using digital weight balance machine. Graph was plotted for treated and untreated fibers with using origin pro software.

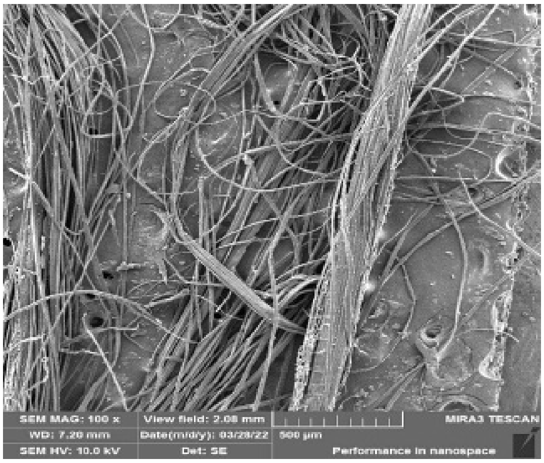

Fig. 7(a): SEM view of bunch of untreated PJ fiber

Fig. 7(b): SEM view of treated PJ fibers

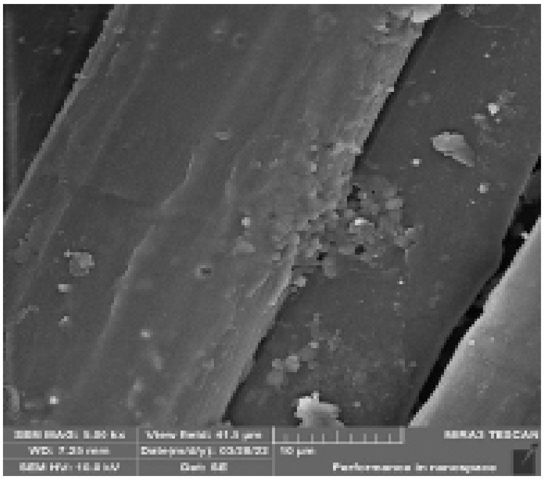

Fig. 8(a): SEM view of surface of untreated PJ fiber

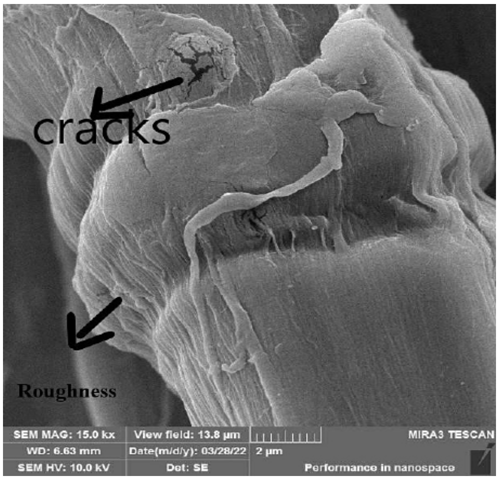

Fig. 8(b): SEM view of surface of treated PJ fiber

Fourier Transform Infrared Spectroscopy

Fourier Transform Infrared Spectroscopy (FTIR) analysis was conducted at Malaviya national institute of technology Jaipur (MNIT). The NaOH-treated and untreated fibers were powdered and tested with FTIR test using FTIR spectrum 2 (Perkin Elmer) with wavelength range 4100-400cm^{-1}. It is used to determine the chemical bonding and % transmittance of any substance. Transmittance is measurement of how much frequency passes through the any compound. Transmittance is ratio of intensity of the incident light to the amount of intensity passes through any material.

Results

Scanning Electron Microscopy of *Prosopis Juliflora* Fiber

Fig.7 (a) shows the bunch of untreated cellulose fibers. Untreated fibers surface consist excessive wax and oil due to this fibers are slicked together but in fig 7(b). After giving alkali treatment the wax, oil and lignin removed and now fibers are not slicked together. As seen in fig. 8(a) surface of untreated fibers are smooth and contains impurities of lignin, wax and pectin while in Fig.8 (b) fiber surface becomes rough in order to enhance interfacial bonding between fibers and matrix. Multiple cracks are also found on surface of alkali treated fibers.[14,15]

The clean, rough and cracked surface of Alkali treated fibers can give the better interfacial bonding between matrix and fiber in order to make the composites. These cracks can fill with resin and increase adhesion which leads to a better performance. In conclusion, the filling of cracks with resin in natural fibers greatly enhances their strength, durability, and performance. This improvement in adhesion and overall properties makes natural fiber-reinforced composites a viable and sustainable alternative to synthetic materials in various industries.[5]

Water Absorption Test for *Prosopis Juliflora* Fibers

Water absorption test was done at standard atmospheric condition to compare the water absorption properties of fibers of plant *prosopis juliflora* for this test 5 grams of Alkali treated and untreated Fibers were taken and kept inside the oven dryer at 50◦C for 6 hours to evaporate any water content and make it completely dry. After this fibers were kept at room temperature for 12 hours for conditioning. Now untreated and treated fibers were soaked into distilled water separately in a container at room temperature and fiber weights were noted with using precise electronic weight machine after every 10 minutes.

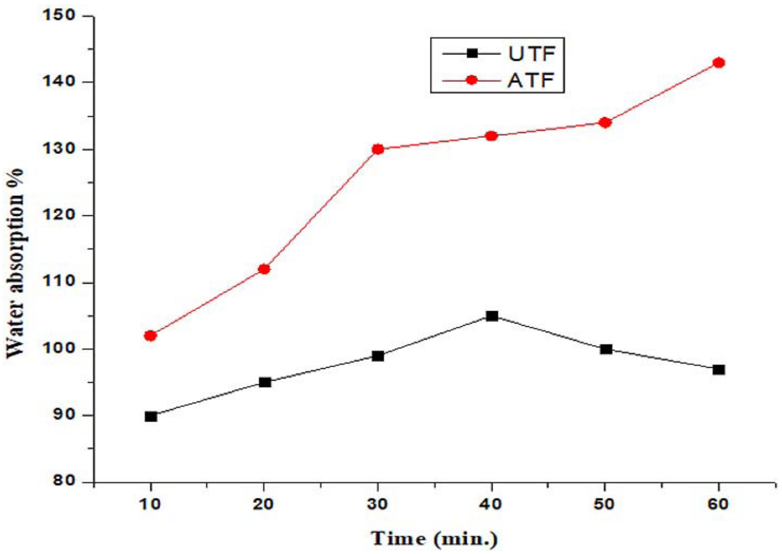

Fig. 9: water absorption test for PJ untreated and treated fibers

From fig.9 it has been found that alkali treated fibers absorb more water than untreated fibers. Plant fiber contains cellulose and hemicelluloses which are hydrophilic in nature but wax, pectin and oil are hydrophobic so alkali treatment removes lignin, wax and oil from the surface of fiber and enhances its water absorbance capacity. Also after treatment cracks and pores increases on the surface of fiber so it can hold more water into it.[6] The use of resin helps to fill the cracks and increase the strength of the composite by providing a more uniform surface area, which reduces the amount of stress concentrations. When the resin fills the cracks, it can reduce water absorption. Additionally, the resin can help to stiffen the composite material, thereby increasing the strength and stiffness.[7]

Table 1: Cross-sectional area of PJ untreated and treated fibers

Fiber	Cross-section area(mm^2)
Untreated fiber A1	0.070098
Untreated fiber A2	0.040056
Treated fiber A1	0.013644
Treated fiber A2	0.008026

The cross- sectional area of treated and untreated *prosopis juliflora* is shown in table 1. Cross- sectional area of fibers was measured with using Image J software. Two random fibers were taken from both treated and untreated fibers (A1, A2) and area is measured. It is clear from results that after giving alkali treatment cross-sectional area reduces which further increases its aspect ratio (length/ diameter). This made the larger surface area of fiber for good adhesion.[10]

Fourier Transform Infrared Spectroscopy of *Prosopis juliflora* fiber

The transmittance vs. wavelength FTIR spectra of alkali treated and untreated PJ fiber is shown in figure 10. As we can see from the fig. that the 1.61% transmittance of the alkali treated PJ fiber decreases with respect to the untreated PJ fiber it means absorption of Alkali treated PJ fiber is more than Untreated PJ fiber. If transmittance decreases absorbance increases further output intensity of any material decreases. There is a sharp and weak absorption peak of OH stretching is found at 3334 cm^{-1} in untreated and 3323 cm^{-1} in treated which shows the presence of cellulose in fibers. This proves that the treated fibers are more processed so the cellulose content is decreased compared to untreated fibers. Next peak 2913 cm^{-1} is the CH stretching peak which shows the presence of methyl and methylene groups found in both curve and C=O stretch peak is found at 1729 cm^{-1} in untreated PJ fiber while in treated PJ fiber it was absent which indicates the acetyl groups of hemicelluloses and lignin. Similarly, the peak for Anti symmetrical bridge C-O-C stretch is found at 1241 cm^{-1} in untreated PJ fibers and in the treated PJ fibers it was absent Which again shows which removal of hemicelluloses. It shows that after giving alkali treatment it eliminates hemicelluloses and lignin from the surface of fiber. This conclusion also supports the SEM results of this study.

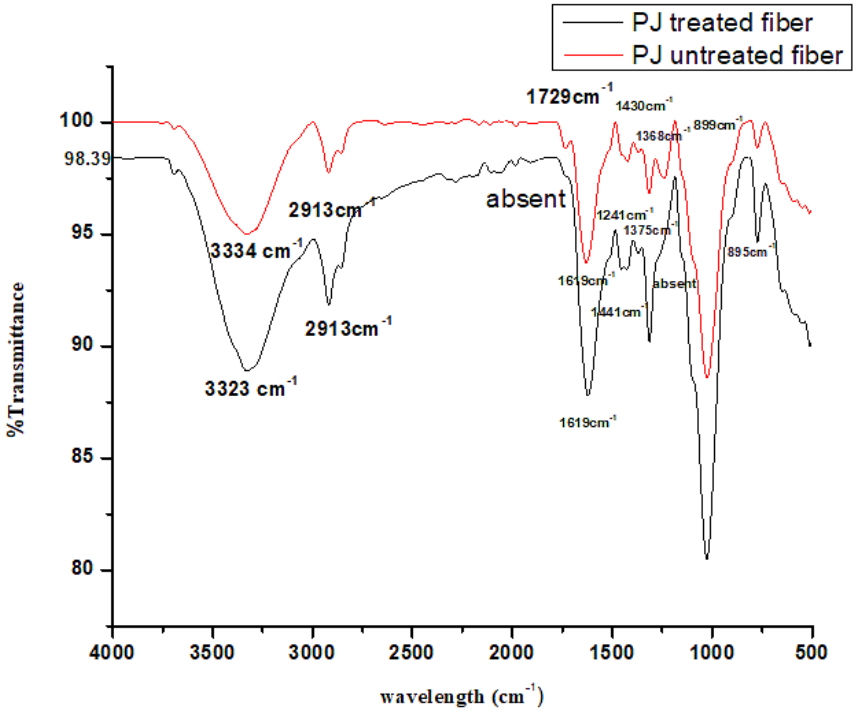

Fig. 10: FTIR spectroscopy of PJ untreated and treated fibers

Plant fibers consist of cellulose and cellulose has both crystalline and amorphous structure. From the figure 10 the band associated with amount of crystalline structure is found at 1430 cm^{-1} in untreated at whereas in treated it is found at 1441 cm^{-1} and the band at 899 cm^{-1} in untreated and at 895 cm^{-1} in treated is due to amorphous region in cellulose and Ratio of these two bands defines the lateral order index (LOI) or empirical crystallinity index (ECI). the ratio between the bands 1369 cm^{-1} and 2913 cm^{-1} is called total crystalline index in untreated fiber whereas in treated this ratio is found in between 1375 cm^{-1} and 2913 cm^{-1}. The combination of TCI and LOI is called infrared crystallinity ratio (IR). The crystalline region is found to increase in treated sample (1441cm^{-1}) as compared to untreated sample (1430cm^{-1}). The increase in crystalline region is due to the removal of amorphous region by the treatment process. Thus, the results clearly demonstrate that the PJ fibers are pretreated by chemical treatment to remove the lignin and hemicelluloses from them.[12]

Table 2: Infrared crystallinity ratio of PJ untreated and treated fibers

Fiber	TCI	LOI
Untreated fiber	0.46	1.59
Treated fiber	0.47	1.61

TCI directly depends on the cellulose crystallinity degree whereas LOI is related to the overall degree of cellulose in fiber. From table 2 we can see that TCI and LOI comparatively high after treatment.[8]

Conclusions and Future Scope

The study concluded that Natural fibers from desert plant species could be successfully used as reinforcement for biodegradable materials. Thus these natural fibres from desert species could be a viable alternative to synthetic fibres commonly used for reinforcement in biodegradable polymers. *Prosopis Juliflora* plant has no commercial value especially in urban area and grows very easily at unwanted public places so insertion of fiber into polymer matrix will create non-toxic and easily bio-degradable materials and will reduce environment pollution.

- SEM results it has been proved that the chemically treated PJ fibers are better than the untreated fibers in order to make further polymer composites.
- Alkali treatment increases the surface roughness and cracked has been found on treated surface of fibers which can promote the better interlocking between fiber-matrix.
- By SEM images it has been also proved the removal of lignin, pectin and wax from treated fiber surface which can increase bonding of fibers with matrix.
- By water absorption results it has been found that treated fibers absorb more mater due to removal of oil and wax this is due to the higher porosity and better surface energy of the treated fiber.
- FTIR results proves that TCI total crystallinity index and the LOI lateral order index is high for PJ treated fibers which shows treated fibers have more crystalline structure and more ordered structure than untreated fibers.

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Conflicts of Interest

The authors declare no conflict of interest.

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