Regular Article

PHYSICAL CHEMISTRY RESEARCH
Published by the
Iranian Chemical Society
www.physchemres.org
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Phys. Chem. Res., Vol. 12, No. 2, 349-359, June 2024
DOI: 10.22036/pcr.2023.408365.2381

Investigating the Influence of Alkali over Morphology, Functional Groups, and Physical and Mechanical Properties of Fiber obtained from Cordia Dichotoma (Gum Berry)

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(Received 23 July 2023, Accepted 13 August 2023)

Natural fiber is typically derived from plants that exhibit low density, lightweight, and abundant availability, along with the added advantage of being biodegradable. The Cardia Dichotoma plant's branches serve as a prominent source of natural cellulose fiber, offering a vast quantity for extraction. However, despite its hydrophilic nature, natural fiber exhibits poor compatibility with both fiber and matrix during the mixing process. To enhance its adhesive properties, the fiber undergoes a series of treatments, including Alkalization, Benzoylation, and Acetylation. In this research, the influence of alkali (untreated, 5%, 10%, and 15% NaOH) was investigated over morphology, functional groups, and physical and mechanical properties of fiber obtained from Cardia Dichotoma. SEM indicated bigger pore size, reduced thickness, and rough surface with etched striations in alkali-treated samples. FTIR peaks inferred that the use of alkali-receded hemicellulose, cellulose, and lignin. Tensile Force & Elongation at intervals showed that the mechanical properties of the increasing amount of Alkali reduced the mechanical properties of the fiber by 5 percent. Conclusively, 5% NaOH was found to be the optimum ratio that effectively removed hemicellulose, cellulose, and lignin, however, the higher percentage decreased tensile strength and elongation at break of the respective fiber.

Keywords: Cordia dichotoma, Fiber, Tensile strength, NaOH, Mechanical properties

INTRODUCTION

Natural fibers are taken out from wood, plants, feathers, and also from human hair. Extraction and processing techniques Natural fiber are cheap, easy than synthetic fiber. Nowadays engineering industries are trying to manufacture environmentally friendly materials. Natural fibers have advantages over synthetic fibers like carbon fiber and glass fiber high in processing costs, and properties like high strength and biodegradability properties of natural fibers [1]. In thermoplastic matrix composites, natural fibers have proven to be an efficient reinforcement [2]. Nowadays researchers are interested in man-made composite because the monolithic (conventional) material is high in weight, corrosiveness, none biodegradability, though conventional materials wide range of applications. Due to high in weight, corrosiveness and none biodegradability use of conventional materials drastically decreasing to overcome researcher are focusing on manmade materials from such as fiberglass and carbon fiber reinforced to produce thermosetting and thermoplastics. Manmade fibers have many drawbacks like creating environmental pollution by creating solid waste because they are non-bio biodegradable in nature. To get beyond these obstacles, the thinking of researchers has diverted to environmentally friendly and biodegradable composite material from natural fiber. This composite material made from natural fiber is of low density, renewable, biodegradable, and low cost due to these properties area of research on natural fiber-based composite material is increasing day by day instead of synthetic fiber composite

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material [3]. However, many problems with synthetic fiber like not being environmentally friendly and fossil fuel depletion made researchers to search environmentally friendly fibers. Therefore, natural fibers gaining attention because of their properties like they are low in density, biodegradable, good in mechanical properties, sustainable, good thermal, and sound properties, and are cheap easy to process and present in huge quantities [4,5]. Mechanical properties of natural fiber composite material depending on the fiber type and extraction technique, and method of processing, as well as the processing of composite material [6].

Despite the advantages, composite materials made from natural fibers, natural fiber is reinforcement in composite materials comes with a number of serious disadvantages. These fibers are hydrophilic in nature which makes weak matrix/fiber matrix adherence properties. By altering the surface of fiber and removing impurities like hemicellulose, wax, oil wax, and lignin, the moisture resistance and fiber/matrix adhesion characteristics may be improved [7]. Many physical and chemical methods are used to treat the surface of natural fiber for surface modification. Physical methods include calendaring, stretching, treatment by cold plasma electric discharge method of natural fiber, *etc.* [8]. While chemical method includes alkalization, acetylation, benzolylation, bleaching with baking soda, and saline treatments Commonly used [9]. This study focuses mainly on Alkalization also known as the mercerization process. Natural fibers are immersed in a concentrated sodium hydroxide (NaOH) solution during the alkalization process, commonly referred to as the mercerization process. To remove hemicellulose, cellulose, and lignin from the surface of natural fiber alkalization process is used in which reaction of sodium hydroxide (NaOH) takes place with hydroxide-OH of natural fiber, along with that it also increases surface roughness, aspect ratio and reducing fiber diameter as a results in alkalization (mercerized) cellulose structure which interfacial bonding among hydrophobic polymer matrix and hydrophilic natural fiber [10]. Impurities like Lignin, hemicellulose, wax and oils are surround natural fiber [4], natural fiber treated with alkali were becomes clean and rough because of the partial elimination of contaminations like lignin, cellulose and hemicellulose [11]. The cellulose content in the natural fiber increases when treated with alkali [12-14], while impurities like wax and oils along with hemicellulose and lignin are removed [15]. By transforming cellulose, I into cellulose II, alkali treatment has an impact on the crystalline characteristics as well [16]. Studied properties of treated natural fiber with 2% sodium hydroxide (NaOH) and untreated natural fiber noted flexural properties of biodegradable polyester composite material made from treated and untreated natural fiber that flexural properties of treated fiber increased in flexural properties with alkali treatment [17]. Benyahia *et al.* Found that using various concentrations of alkali treatment (1, 3, 5, and 7% for 24 h) Improved Alfa-based polyester composite material tensile and flexural strengths [18], Arrakhiz *et al*., Studied that when Alfa fiber treated with 1.6 M NaOH for 48 hours than the mechanical properties like (tensile strength and Young's modulus) resulted better than untreated natural fiber [19]. Acharya et al. treated bagasse fibers for 2, 4, and 6 h with 5% NaOH. The usage of 5 percent NaOH-treated fibers with a 4-hour immersion time resulted in the bagasse/epoxy composite's best flexural characteristics [28]. According to Vilay *et al*., Higher tensile properties and flexural properties than composite with the untreated natural fibers were obtained from the unsaturated polyester composites after alkali treatment and increased fiber loading of the bagasse fiber [20]. KJ Wong *et al.* According to researches on the effect of alkali treatment (1, 3, and 5 percent) on density and tensile properties of the bamboo fiber-based polyester composite, the density of the composites decreased with the increase in NaOH concentration, leading to higher porosity as a result of the removal of cementing composites. Interfacial shear strength (IFSS) increases, however, the percentage improvement declines as alkali concentration increases, according to their findings [21]. According to Lu *et al*., Tensile and elongation at break properties of Bamboo fiber/epoxy composite were improved by 71% and 53% respectively, when Bamboo fiber is treated with 2% NaOH [22]. Examined treated fiber and untreated fiber of banana, treated with 0.5-20 percent of sodium hydroxide for the time of 30 min when reinforced with epoxy to form composite material. Noticed that the 1 percent treatment of NaOH shows the best mechanical properties additionally, it was also observed that high NaOH concentrations cause the mechanical characteristics to degrade because of the fiber degradation brought on by the alkali treatment [23]. Merlin *et al.* Short fiber/poly-lactic acid composites were exposed to greater concentrations of NaOH (10, 20, and 30 percent), and it was discovered that slowly decrease in tensile modulus with increasing concentration of NaOH. The significant fiber damage brought on by the increasing alkali content was blamed for the drop in tensile modulus [17]. The founded fiber of flex with PLA/PC composite represented great modulus and high tensile strength when treated with 2 Percent sodium hydroxide NaOH compared to 5 and 10 percent sodium hydroxide NaOH concentration [30]. Conducted various treatments of NaOH on flex fiber and noted that greater than 23 percent, both longitude and crosswise tensile strength, decreases of flex fiber epoxy composite tensile strength of the treated fiber/epoxy composite declined notably. At 4% NaOH treatment for 45 s was enough to increase by 30% in these properties. Also noted that beyond 10% causes deterioration in longitude and transverse tensile strength [24]. Studied mechanical properties of Jute fiber decrease with increase in alkali treatment and immersion time when reinforced with vinyl ester composite [25]. When alkali treated Jute fiber reinforced with epoxy composite material had better mechanical properties (tensile, flexural, and impact) than composite made from untreated natural fibers reinforced epoxy composite material. This was the case when the jute fabric was treated with 20 percent NaOH for 25 min at room temperature [26]. Leela *et al.* reported properties of jute fiber when reinforced with polypropylene composite material for 20% loading of fiber along with 10% NaOH, increase in tensile properties of jury fiber showed. Also reported that tensile modulus increases in alkali treatment as well as fiber loading, whereas elongation at break for composite decline for both Properties of fiber loading as well alkali treatment of fiber [27]. With regards to this study natural fabrics of Cordia dichotoma it is present in small size and spreading crown with greenish-brown wrinkled in longitudinal bark [28]. One of these inexpensive and widely accessible fibers is Cordia dichotoma. Using the water-retting procedure, Cordia dichotoma's fibers were removed from its bark. To improve bonding with the matrix, the fiber Cordia dichotoma was alkali treated using a 5 percent solution of sodium hydroxide NaOH. The impact of this treatment on the composite's characteristics was investigated. It was observed that the mechanical properties of the composite improved by treating fiber with 5% NaOH alkali [29].

In this research, the influence of alkali (untreated, 5%, 10%, and 15% NaOH) was investigated over morphology, functional groups, and physical and mechanical properties of fiber obtained from Cardia Dichotoma. SEM represents the surface morphology of untreated and alkali-treated natural fiber. The FTIR analyzed the structure & functional group which were present in the fiber. TGA indicated the thermal stability of the treated and untreated fibers. It was inferred that the use of alkali (NaOH) receded the hemicellulose in its raw form. Tensile Force & Elongation at intervals showed that the mechanical properties of the increasing amount of alkali reduced the mechanical properties of the fiber by 5%. The above research shows that treated fiber is more effective in removing the lignin hemicellulose in the comparison of untreated fiber.

MATERIALS AND METHODS

Materials

Sodium hydroxide (NaOH) pellets were purchased from Merck. The fiber was extracted from branches of the Cordia dichotoma tree found locally in Sindh province, Pakistan.

Fiber Extraction and Treatment

The fiber was extracted from the tree branches following the study [30]: Initially, the skins of the branches were peeled off and submerged in water for 15-20 days in a process called as the ratting process. The skin was gently beaten with hammer to extract fiber easily and dirt was removed. The extracted fiber was washed with water and then sun-dried for 24 h followed by oven drying at 105 °C for 24 h. This removed the moisture from the fiber completely. To treat the fiber solution of Sodium hydroxide NaOH was used with different (w/v) ratios for 2 h at room temperature. Then fiber was washed with distilled water to remove excessive NaOH and heated in the oven at 105 °C for 24 h. Figure 1 shows the schematic representation of fiber treatment and characterization.

Characterization of Fiber

Micrographs of untreated fiber and alkali treated with NaOH with 5, 10, and 15 % respectively were obtained using SEM (Jeol Jsm-6490LV). For FTIR analysis, KBr and

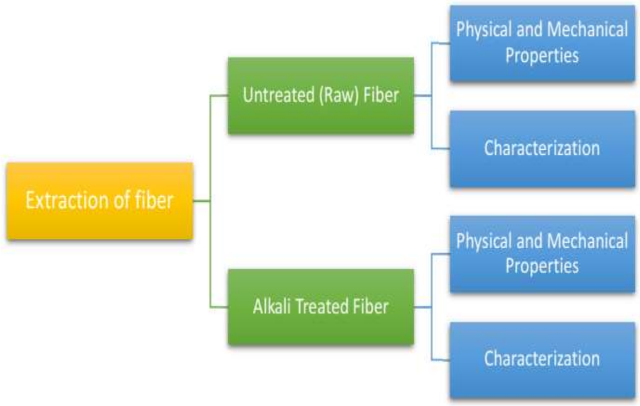

Fig. 1. Schematic Representation of Fiber Treatment and Characterization

powdered samples were mixed and pressed with hydraulic press to form pellets. Mechanical properties such as tensile strength, elongation at break, and Young's modulus of treated and untreated fiber were determined by using Titan universal testing machine at a cross-head speed of 3 mm per min and a gauge length of 75 mm. For characterization and tensile properties, names are given as S1 represents untreated sample, S2 treated sample with5% NaOH, S3 represents treated sample with 10% NaOH and S4 represents treated with 15% NaOH respectively [30]. The linear density of fiber was determined using ASTM D 1577-96 by weighing the length of fiber. Water absorption was found by immersing the known weight of the sample in distilled water and after 5 min, the sample was taken out and weighed again. The percent of water absorption capacity was determined by using the water absorption formula given below:

$$\%\ \text{Water Absorption} = \frac{\text{weight after immersion} - \text{water before immersion}}{\text{weight before immersion}} \times 100 \quad (1)$$

RESULT AND DISCUSSION

Morphology and Structure (SEM)

Figure 2 shows the SEM results of untreated and alkali (NaOH) treated fiber. As can be seen from the figure, the

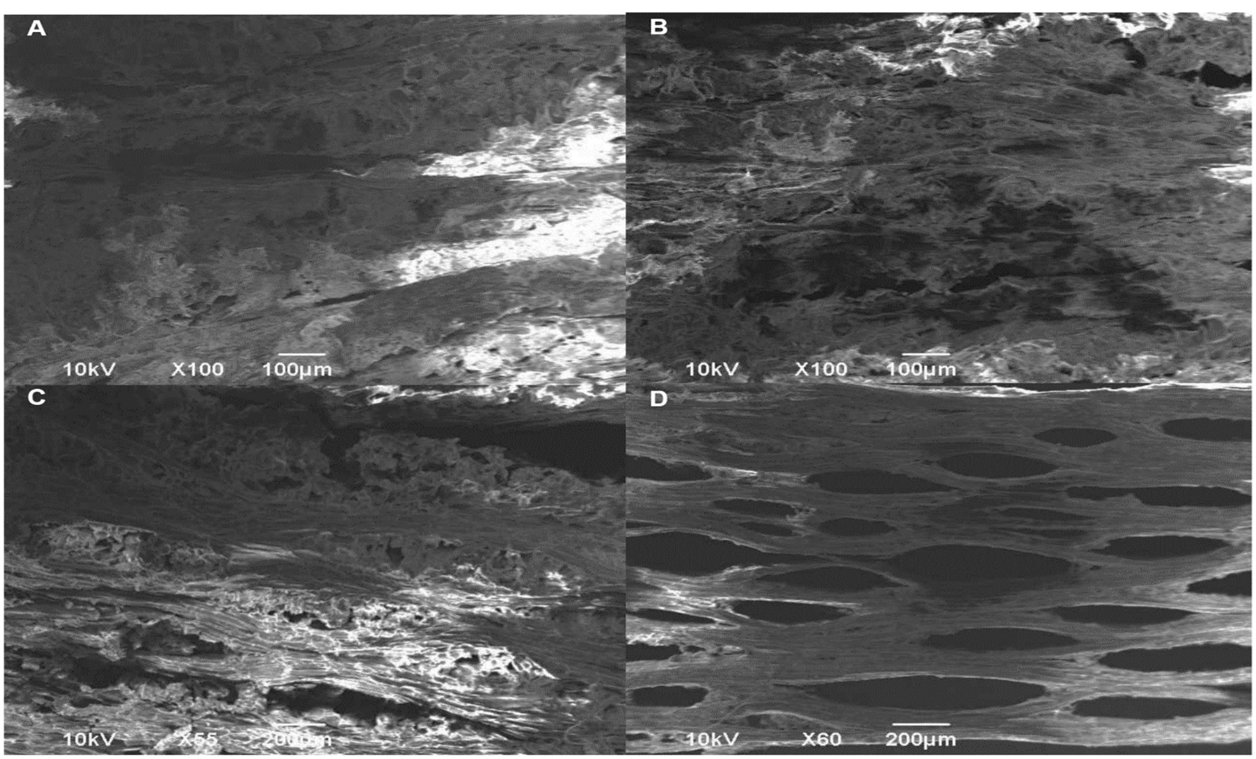

Fig. 2. SEM images (A) untreated fiber (B) 5% NaOH treated fiber (C) 10% NaOH treated fiber (D) 15% NaOH treated fiber.

untreated sample is the least porous in structure with smaller pore size. As the concentration of the NaOH is increased, surface area, porosity, and pore size increase. This is validated by the vivid pores shown in the sample treated with 15% NaOH. Moreover, it can be seen that gummy polysaccharides of lignin, pectin, and hemicellulose were present on the surface of untreated natural fiber which are reduced with alkali (NaOH) treatment. Similarly, the alkali (NaOH) treatment is shown to reduce the thickness of the fiber but increase the surface area and roughness with etched and striation. This is in agreement with the previous studies [32,33]. The rough and clean surface of the fiber can be a potential candidate for synthesizing green composites [30].

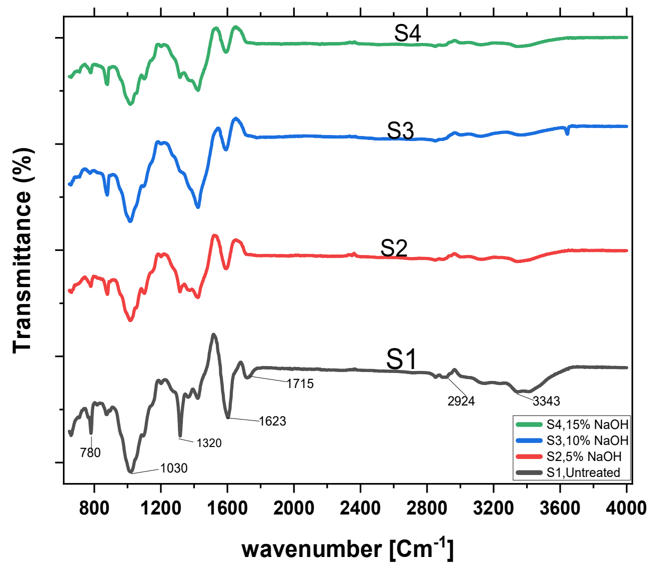

Fig. 3. FTIR of untreated and alkali (NaOH) treated fiber samples of fiber.

Functional Group Analysis (FTIR)

Figure 3 shows the FTIR spectra of untreated and treated fiber samples with 5%, 19%, and 15 % of NaOH solutions. It may be seen from the figure that the untreated and treated fiber contains lignin, hemicellulose, and α- cellulose due to their well-defined peaks in the graph at 3343 cm^{-1} for O-H stretching, 2924 cm^{-1} for alkyl C-H stretching for α- cellulose, 1715 cm^{-1} stretching for C=O of hemi cellulose, 1623 cm^{-1} for C=O stretching of lignin, 1320 cm^{-1} for stretching of C-O, 1030 cm^{-1} stretching for S=F and 780 cm^{-1} stretching of lignin for aromatic C-H. it is interesting to see that the peaks at 3343 cm^{-1}, 2924 cm^{-1}, 1715 cm^{-1}, 1623 cm^{-1}, 1320 cm^{-1}, 1030 cm^{-1} and 780 cm^{-1} decreased when compared with untreated fabrics [25]. These peaks indicate the reduction of hemicellulose and lignin with alkali (NaOH) treatment.

XRD

Figure 4 shows the XRD of the fiber treated with 5% NaOH. One broad peak and one sharp peak can be observed at 15.69° and 22.29°, indicating the crystallographic planes. The broad peak indicates the presence of non-cellulosic materials while the sharp peak shows the content of α-cellulose respectively. The crystallinity indices are a function of surface treatment through alkali (NaOH).

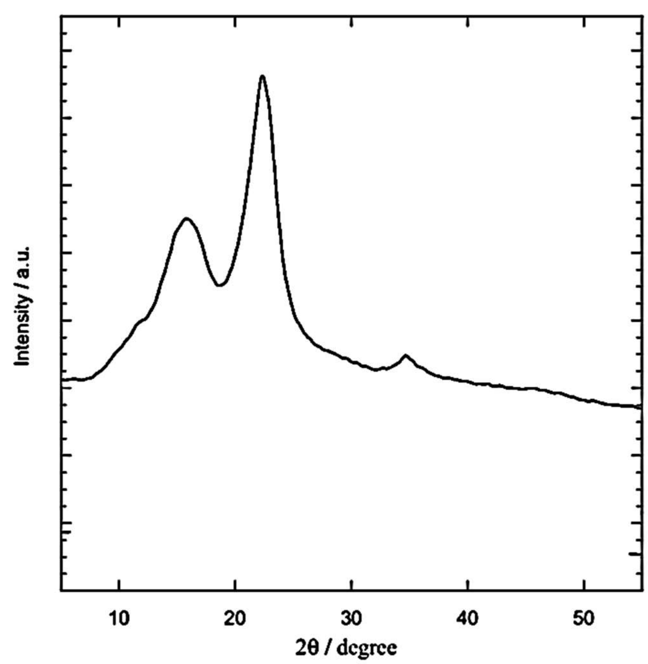

Fig. 4. XRD of 5% NaOH treated fiber.

TGA

Figure 5 shows the thermogravimetric analysis of untreated and 5% NaOH-treated fiber samples. As can be seen, alkali (NaOH) treatment has resulted in enhancing the thermal stability of the respective fiber. This can be attributed to the removal of hemicellulose and lignin from the sample during the treatment, which resulted in alkali (NaOH) treated fiber losing weight at a lower temperature than the untreated sample. Finally, both the fiber samples are found to be thermally stable up to 400 °C.

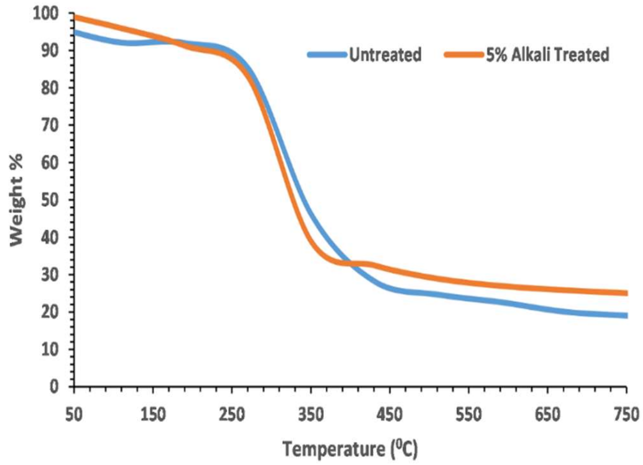

Fig. 5. TGA analysis of untreated and 5% NaOH treated fiber.

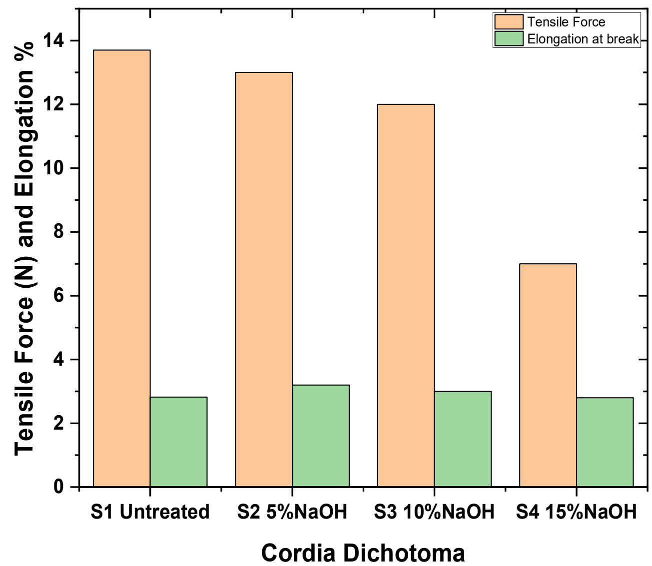

Fig. 6. Tensile Force and Elongation at Break of Untreated and Treated with NaOH samples of fiber.

Tensile Strength

Natural fibers have high tensile strength but have lower elongations at break. The tensile force and elongation at break of natural fiber as given in Table 1 and their values are plotted in Fig. 4. While the actual space occupied by the fibers in the textiles may be much less, the area occupied by the empty areas was also taken into consideration, so the actual tensile properties may be higher than the table. It is evident from Table 1 and Fig. 6 that the tensile properties of fiber increase if we treat with alkali (NaOH). It is also noted that tensile properties are highest at 5% NaOH treatment as the percent of NaOH increases tensile properties decrease [11]. The tensile force (N) of the respective fiber samples is shown to reduce from 13.7 N to 7 N as the alkali (NaOH) concentration is increased from 0-15% respectively.

Furthermore, it is also noted that the percent elongation increases as the treatment % of NaOH increases from 2.82% to 3.2% for 5% NaOH treatment then decreases to 3% for 10% NaOH and 2.9% for 15% NaOH. In conclusion, 5% NaOH gives the best properties.

Linear Density

As shown in Table 2 and Fig. 7, linear density of fiber increased slightly as the percent of alkali treatment increases at 5% of NaOH with the value of linear density as 3.08. When the alkali percentage was increased to 10% and 15%, the value of linear density slightly increased from 3.08 to 4.16 and 4.07 in mg mm^{-1} respectively. Hence it can be stated that higher alkali concentration increases the linear density of the fiber, which is consistent with the previous study [25].

Table 1. Tensile Force and Elongation at Break of Untreated and Treated with NaOH Samples of Fiber

Cordia dichotoma fiber	Tensile force (N)	Elongation at break (%)
S1 (Untreated fiber 0% NaOH)	13.7	2.82
S2 (Treated with 5% NaOH)	13.0	3.2
S3 (Treated with 10% NaOH)	12.0	3.0
S4 (Treated with 15% NaOH)	7.0	2.8

Table 2. Linear Density of Untreated and Alkali (NaOH) Treated Fiber Samples of Fiber

Sample	Linear density approximately (mg mm^{-1})
S1 (Untreated fiber 0% NaOH)	3.07
S2 (Treated with 5% NaOH)	3.08
S3 (Treated with 10% NaOH)	4.16
S4 (Treated with 15% NaOH)	4.07

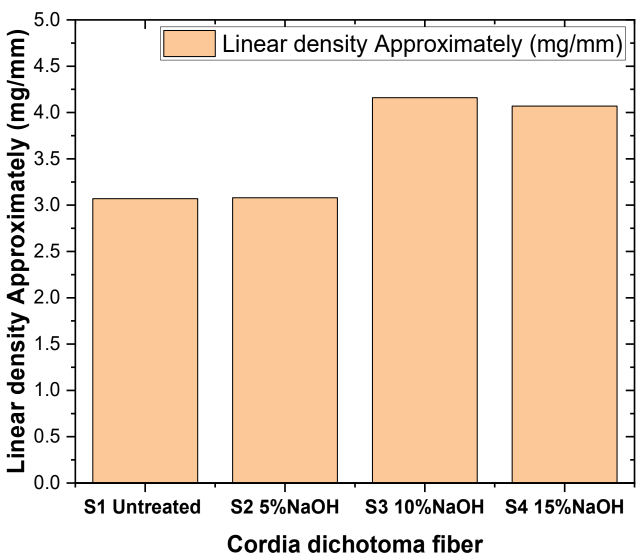

Fig. 7. Linear density of untreated and alkali treated with NaOH samples of fiber.

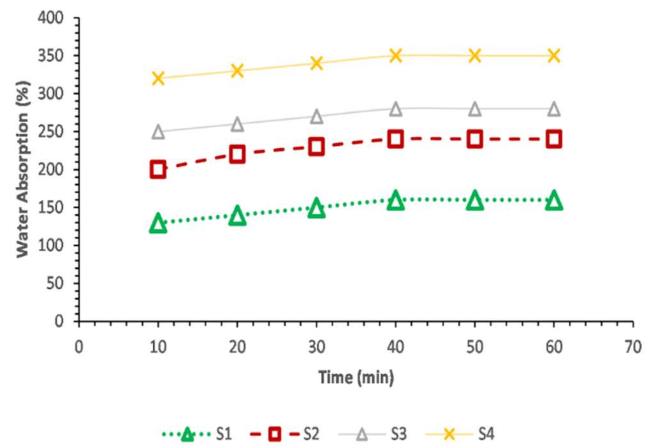

Fig. 8. Water absorption (%) of untreated and alkali treated fiber samples.

Water Absorption

Figure 8 shows the water absorption capacity of the untreated and alkali-treated fiber samples. As can be seen, alkali treatment enhances the water absorption capacity of the fiber. The lowest water absorption of 160% is observed in the untreated sample (S1). When this sample is treated with 5% alkali, its water absorption capacity increases to 240. Similarly, when the alkali concentrations are increased to 10% and 15%, the water absorption capacities of the respective fiber samples increase to 280% (S3) and 350% (S4) respectively. This can be attributed to a number of reasons. As also confirmed by SEM, alkaline treatment increases the surface area of the fiber, hence the water absorption is increased. NaOH treatment also removes the impurities such as cellulose and hemicellulose, thereby making the fiber more hydrophilic. Alkaline treatment exposes more hydroxyl groups in the cellulose molecules of the fibers. These hydroxyl groups have a strong affinity for water, leading to enhanced water absorption. The treatment can improve the crystallinity of the fibers, making them more capable of holding water molecules within their structures [34,35].

DISCUSSION

Natural fibers have gained significant attention in various industries due to their eco-friendly nature, low cost, and potential for a wide range of applications. Cordia dichotoma, commonly known as Clammy Cherry, is a tree that grows abundantly in tropical and subtropical regions. The fiber obtained from Cordia dichotoma has promising properties that make it suitable for use in textile, composite, and paper industries. This discussion focuses on the influence of NaOH treatment on the fiber obtained from Cordia dichotoma, highlighting its effects on the fiber's characteristics and potential applications. NaOH treatment, also known as alkaline treatment, is a common method used to modify natural fibers. It involves the immersion of fibers in a sodium hydroxide (NaOH) solution, which results in the removal of impurities and the modification of fiber surface properties. The treatment alters the chemical composition of the fiber, leading to changes in its physical, mechanical, and chemical properties. NaOH treatment causes structural changes in the fiber obtained from Cordia dichotoma. It leads to the removal of lignin, hemicellulose, and other impurities, resulting in increased fiber purity. The treatment also induces the swelling of cellulose, exposing more hydroxyl groups and increasing the fiber's surface area. These changes enhance the fiber's hydrophilicity, making it more receptive to chemical modifications and enhancing its dyeability. One of the significant benefits of NaOH treatment is its positive influence on the mechanical properties of Cordia dichotoma fiber. The treatment improves the fiber's tensile strength, modulus, and elongation at break. The removal of impurities and the enhanced interfacial bonding between cellulose chains contribute to the increased strength and stiffness of the treated fibers. These improved mechanical properties make the fiber suitable for applications that require high strength and durability, such as reinforcement in composites. NaOH treatment also influences the thermal stability of Cordia dichotoma fiber. The treatment reduces the thermal degradation of the fiber and increases its resistance to high temperatures. The removal of impurities and the alteration of the fiber's structure contribute to improved thermal stability. This property is advantageous for applications that involve exposure to heat, such as in flame-retardant textiles and insulation materials. Scanning electron microscopy (SEM) analysis of NaOH-treated Cordia dichotoma fibers reveals changes in surface morphology. The treatment results in a smoother and cleaner fiber surface, with reduced surface irregularities and protrusions. These morphological changes increase the fiber's surface area, allowing for better interaction with other materials in composite systems or improved adhesion in papermaking. The influence of NaOH treatment on Cordia dichotoma fiber opens up new possibilities for its application in various industries. The enhanced fiber properties make it suitable for textile applications, including clothing, home textiles, and technical textiles. The improved mechanical strength enables its use as a reinforcing material in composites for the automotive, construction, and aerospace industries. Furthermore, the modified fiber can be utilized in the production of specialty papers, biodegradable packaging materials, and bio-based additives. NaOH treatment plays a crucial role in modifying the fiber obtained from Cordia dichotoma. The treatment enhances the fiber's properties, including improved mechanical strength, enhanced thermal stability, and increased surface area. These modifications expand the potential applications of Cordia dichotoma fiber in various industries. Further research and development are necessary to unlock its full potential and optimize its properties for specific applications. The influence of NaOH treatment on Cordia dichotoma fiber opens doors for sustainable, eco-friendly alternatives in textile, composite, and paper industries.

CONCLUSION

In this work, the influence of alkali (NaOH) treatment over chemical, physical and mechanical properties of fiber was investigated. The fiber was obtained and extracted from the Cordia dichotoma tree which is locally abundant in Pakistan. SEM images showed that by increasing the amount of alkali the fiber creates a number of etched, rough, and possess wide regions. Moreover, alkali treatment receded lignin, hemicellulose, and cellulose, thereby imprinting roughness onto the surface. FTIR results confirmed the removal of lignin and hemicellulose. In terms of mechanical properties, it was shown that 5% alkali treatment is the optimum value that enhances the overall properties. In terms of water absorption, the higher concentration of alkali treatment results in fiber being more hydrophobic and absorbing lesser water. Conclusively, the above research shows that treated fiber is more effective in removing the lignin hemicellulose in the comparison of untreated fiber. However, further research is needed to explore the long-term effects of NaOH treatment on fiber durability and the optimization of treatment parameters to achieve desired properties. Additionally, the investigation of chemical modifications and the development of hybrid fibers by blending Cordia dichotoma fiber with other natural or synthetic fibers can lead to advanced applications and performance improvements.

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