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Preliminary Studies on Conversion of Sugarcane Bagasse into Sustainable Fibers for Apparel Textiles

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Abstract: Owing to increased environmental awareness and the implementation of stringent governmental regulations, the demand for the valorization of natural fibers has increased in recent years. Sugarcane bagasse after juice extraction could be a potential source of natural fibers to be used in textile applications. In this paper, sugarcane bagasse is converted to textile fibers. Sugarcane fibers are extracted through alkali and H_2O_2 treatment with varying concentrations (6, 10, 14) g/L and (8, 12, 16) g/L, respectively. To soften the fibers for textile use, extracted fibers were post-treated with a constant ratio of silicone softener (50 g/L). Treatment of sugarcane fibers with varying concentrations of alkali–H_2O_2 significantly influenced the fiber surface morphology. Furthermore, an increase in the crystallinity of extracted fibers was observed, whereas a reduction in fiber linear density from 54.82 tex to 45.13 tex as well as moisture regain (6.1% to 5.1%) was observed as the ratio of alkali–H_2O_2 treatment was increased. A notable improvement in overall mechanical strength was achieved upon alkali–H_2O_2 treatment, but at a higher concentration (conc.) there was a loss of mechanical strength, and the torsional and flexural rigidity also increased significantly. Based on the results, sugarcane fibers treated with 10 g/L NaOH, 12 g/L H_2O_2 and 50 g/L silicone softener showed the most optimum results. These sustainable fibers have the potential to be used in textile applications due to their enhanced softness, optimum moisture regain, and better mechanical properties.

Keywords: sustainable fibers; alkali treatment; hydrogen peroxide; sugarcane bagasse; textiles

Citation: Jalalah, M.; Khaliq, Z.; Ali, Z.; Ahmad, A.; Qadir, M.B.; Afzal, A.; Ashraf, U.; Faisal, M.; Alsaiari, M.; Irfan, M.; et al. Preliminary Studies on Conversion of Sugarcane Bagasse into Sustainable Fibers for Apparel Textiles. *Sustainability* **2022**, *14*, 16450. https://doi.org/10.3390/su142416450

Academic Editors: Farid Menaa, Roberta Palmieri, Riccardo Gasbarrone and Ludovica Fiore

Received: 28 September 2022
Accepted: 2 December 2022
Published: 8 December 2022

1. Introduction

The higher attention gained by natural plant-based fibers is based on their easy availability, higher specific strength, stiffness, and biocompatibility. Sugarcane (Saccharum officinarum) is a tropical plant and is cultivated all over the world. Most of the sugarcane goes into sugar and alcohol mills. The crushing produces a residue of over 30%, called bagasse [1]. A large quantity of this sugarcane bagasse is utilized as a fuel in industries and as animal feed. In contrast, very little importance is given to the value optimization of these by-products. The conversion of natural plant-based resources to geotextile and biocomposites is more sustainable and environmentally friendly [2–5]. Thus, fiber extraction and its treatment are critical in achieving viable fiber used in fabric. For that reason, alkali treatment is the most-used technique to improve the intrinsic properties of natural fibers. One such commercial process, called "Mercerization", uses sodium hydroxide solution

(20–27 wt. %) under load for the treatment of fibers [6]. The treatment exhibits excellent properties such as improved strength, better sheen, and dyeing [7].

Alkali treatment of natural fibers has also been studied extensively, reporting increased stiffness and strength [8,9]. Alkali treatment also involves hydrogen peroxide (H_2O_2), which helps remove hemicellulose and lignin from the fiber, resulting in the expansion of the surface area, which facilitates the exposure of reactive hydroxyl groups on the fiber surface [10] which can also facilitate the adhesion between fiber and matrix, thus increasing the wear resistance [11]. The change in properties depends on the conc. of sodium hydroxide used as well as the duration of treatment, which may have a wide range of variation. The general concept based on the change in properties is associated with the amorphous parts being dissolved, resulting in a decrease in lignin, hemicellulose, and wax content of fiber. On the other hand, a few researchers disagree with this concept and state that the lignin content remains the same [12].

Sinha et al. [13] carried out the alkali treatment of jute fibers with 5% NaOH solution at room temperature for 2 h, 4 h, and 8 h. The treated fibers showed improved flexural and bending strength, with increased crystallinity over time. The improvement in the mechanical strength and crystallinity was attributed to the removal of hemicellulose and lignin, which made room for the close packing of cellulosic chains. Nevertheless, where the alkali treatment provides strength to fibers, it also increases stiffness. To counter this, using a softener becomes essential to optimize the fineness of the extracted fibers. Therefore, using different softeners becomes necessary to enhance the fineness of fibers. Various softeners are most commonly used to improve the fineness and feel of the fabrics [14,15]. Silicone softeners are used to improve the temperature stability and durability of the treated textiles, which results in value add-on of products [16,17].

All the previous studies on sugarcane fiber extraction are dependent on alkali treatment only, but this work is focused on the treatment of fibers with alkali and hydrogen peroxide and then softening of fibers with softener. Thus, soft and flexible fibers could be prepared for textile applications. The work was done to convert sugarcane bagasse into sustainable textile fibers. Fibers were extracted from sugarcane bagasse through alkali–H_2O_2 pre-treatment and were post-treated with silicone softener. The prepared samples were subjected to evaluate tensile strength, fiber fineness, and moisture regain analytically, and morphological properties were observed on SEM. Subsequently, the fiber sample's crystallinity trend was obtained from XRD testing.

2. Materials and Methods

The local fields provided sugarcanes, and sugarcane bagasse was extracted by crushing the sugarcane on a crusher. Sodium hydroxide (NaOH) "molar mass (40 g/mol)" and Hydrogen peroxide (H_2O_2), 30 wt% solution purchased from Sigma Aldrich were used to separate the lignin and hemicellulose from fibers. Silicone softener was also purchased from Rudolf GmbH (®RUCOFIN SIQ) to improve the softness and fineness of extracted fibers.

2.1. Extraction of Sugarcane Fibers

Fresh sugar cane stalks were washed to remove soil, dirt, and other foreign material and passed through the crusher to obtain bagasse. The soft part of the bagasse was removed, and the outer rind part was further processed and cut 4 cm long and 1–2 mm wide to extract the fibers.

The cut rind samples were subjected to hot water treatment for 60 min to remove coloring matter and sugar traces (sample A). They were then subjected to alkaline treatment for 4 h at 90 °C temperature with continuous stirring with different ratios of NaOH and H_2O_2, as shown in Table 1, to remove the lignin and other binding material from the fibers. Finally, the fibers extracted in this process were washed and sun-dried.

Table 1. Design of experiment for sugarcane fiber softness treatment.

Sample ID	The Concentration of Alkali/H_2O_2/Silicone Softener
A	No treatment (Simply extracted fibers)
B	Silicone softener 50 g/L
C	H_2O_2 12 g/L
D	(6 g/L NaOH, 8 g/L H_2O_2)–(50 g/L silicone softener)
E	(10 g/L NaOH, 12 g/L H_2O_2)–(50 g/L silicone softener)
F	(14 g/L NaOH, 16 g/L H_2O_2)–(50 g/L silicone softener)

2.2. Treatment of Sugarcane Fibers

The alkali–H_2O_2 treated sugarcane fibers were further post-treated with a constant silicone softener (50 g/L) ratio to make the fibers soft and flexible. Untreated raw fibers (sample A) were directly post-treated with 50 g/L of silicone softener with curing and drying (sample B). Untreated raw fibers (sample A) were also directly post-treated with 12 g/L of H_2O_2 curing and drying (sample C). These samples (A, B, C) were prepared for comparison purposes. The alkali–H_2O_2-treated fibers were dipped in a solution containing silicone softener for 5 min with continuous stirring. Afterward, the fibers were dried in an oven at 100 °C to remove moisture and subsequently cured at 150 °C for better penetration and adhesion of silicone softener with the fibers. After the treatment, fibers were washed and thoroughly dried in the oven at 100 °C temperature. The processes were carried out according to the experiment design shown in Table 1.

2.3. Fiber Fineness

Fiber fineness (linear density) was calculated according to ASTM D-1577. Among each type of treated fibers, 5 random fibers were selected for length measurement using a knitmeter, the weight of samples was observed on an electronic balance, and the linear density was calculated accordingly.

2.4. Moisture Regain

Moisture regains of various fiber samples were calculated following the protocol set by ASTM D-2495. First, the samples were oven dried completely and then placed under standard conditions to let the moisture be absorbed. The difference in weight measured on a weight balance before and after absorption gave the value of moisture absorption for different fibers.

2.5. Tensile Properties

The fiber's tensile properties (tenacity and elongation at break) were determined on a Lloyd LRX Tensile Tester according to ASTM D-3822. The gauge length of the sample was kept at least 10 mm so that the fiber to be tested should be long enough to clamp between the testing assemblies.

The torsional rigidity of various fiber samples was measured by calculating the oscillations of the torsion pendulum, which comprised a bar suspended by fiber. The fiber samples were subjected to four torsional oscillations, and the time was observed accordingly, where the damping was negligible. The value of torsional rigidity was obtained from the following formula:

T.R. = $8\pi^3 IL/T^2$, where I represents the moment of inertia ($I = m\,(l^2/12 + r^2/4) = 0.267$ g cm^2) of the rod.

The flexural rigidity was measured according to the pierce method, comprised of the study of loop deformation under applied load. A circular fiber ring loaded with a rider was suspended, and the deformation of the ring as compared to its original circular shape (increased diameter) was measured using the formula given below.

FR = 0.0047 mg (2 π r) 2(cosθ/tanθ); where mg is the weight of the rider and r is the radius of the ring (0.9 cm).

2.6. Characterization of Treated and Untreated Samples

Scanning electron microscopy (SEM) results were produced on Quanta 250—FEG (ASTM E-2809-13) for each fiber sample at a voltage of 5 kV. The fiber samples were sputtered with gold to make them conductive and placed on carbon tape to be fixed on aluminum stubs. The surface morphology was observed at a magnification of 300 µm to 550 µm. In addition, x-ray diffraction was performed (ASTM D-2809) to evaluate the crystalline structure of fiber samples. The traces were recorded on powder samples with Cu radiation at 40 kV and 35 mA anode excitation in the 2θ range between 1 and 70°.

3. Results and Discussions

3.1. Linear Density of Sugarcane Fibers

Figure 1 shows the variation in fiber's linear density for multiple treated and untreated sugarcane fiber samples. Samples A, B, C, D, E, and F have linear densities ranging from 61.23 tex to 45.13 tex.

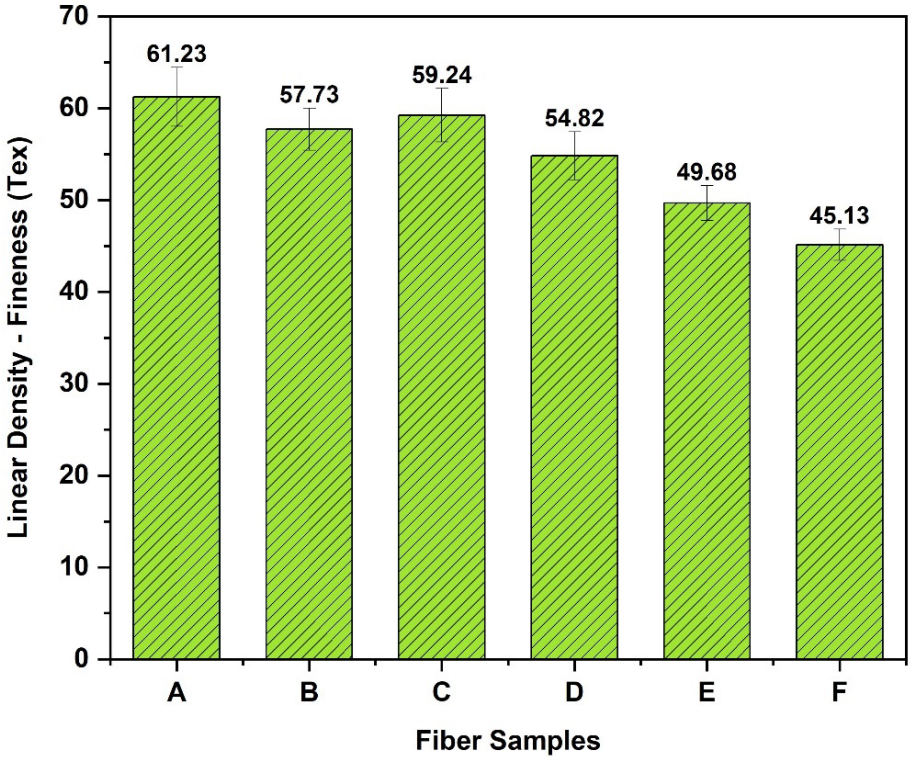

Figure 1. Comparison of fiber linear density of developed samples.

A decreasing trend can be observed (54.82 tex to 45.13 tex) as the fibers are treated with an increasing conc. of alkali–H_2O_2, as shown in Figure 1 (Sample D, E, and F). In the case of raw fibers treated with 50 g/L silicone softener (Sample B) and 14 g/L H_2O_2 (Sample C) separately, the fiber linear density shows a minor decline compared to untreated fibers (Sample A). Sample A (untreated fibers) offers the highest linear density (61.23 tex), as the fibers are coarser than other samples because they contain a high amount of lignin and hemicellulose [18]. Alkali–H_2O_2 treatment with continuous stirring packs the cellulosic chain closely by removing lignin and hemicellulose. It ultimately results in a close packing of chains and improved fiber fineness [19]. Post-treatment with a constant amount (50 g/gL) of silicone softener also helps decrease the value of alkali–H_2O_2-treated fiber's linear density and increase the fiber fineness, as shown in Figure 1 (D, E, and F). The treatment of 50 g/L silicone on raw fibers (Sample B) also demonstrates a minor fineness improvement over untreated Sample A. Similarly, H_2O_2-treated raw fibers (Sample C) also demonstrated a minor reduction in fiber's linear density as compared to untreated sample

(A) due to a minor amount removal of lignin and hemicellulose, and these treatments did not cause any significant damage to raw fibers [20].

3.2. Moisture Regain of Sugarcane Fibers

Moisture management in fibers plays a vital role in enhancing the comfort properties of the fibers and their subsequent yarns and fabrics [21,22]. Natural fibers usually have a round shape and plenty of void spaces between the packed fiber bundles, which act as pockets for moisture retention, resulting in higher moisture content [23]. Sugarcane fibers are also moisture-absorbing due to hemicellulose, which inherently possesses a hydrophilic nature [24]. Figure 2 compares moisture regain (%) of raw and other treated sugarcane fibers. It has been found that the raw sugarcane fibers (sample A) show the highest value of moisture regain (7.1%) as compared to all other samples because they contain a high amount of hemicellulose in their structure [24]. The presence of hemicellulose in raw sugarcane fibers readily absorbs moisture (as described above), resulting in high moisture regain% values [25]. After alkali–H_2O_2 treatment, the lignin/hemicellulose linkage is destroyed, resulting in a loss of inherent hydrophilicity of fibers as exhibited in Figure 2 (C, D, E and F) [10,24]. Furthermore, the declining trend can be observed in moisture regain values of samples D, E, and F of 6.1% to 5.1%, respectively, which can be linked to increasing conc. of alkali–H_2O_2 used in the treatment of fibers. The application of silicone softener also plays a vital role in increasing the hydrophobicity of the fibers. Silicone softener acts as a coat on the fiber surface, which also aids the restriction of moisture absorption in samples B, C, and D [26]. However, raw fibers treated with 50 g/L silicone softener alone (Sample B) show a moderately low value of moisture regain (6.5%) when compared to Sample A, but still higher than Sample D, E, and F due to the presence of intact hydrophilic hemicellulose structure. Moreover, H_2O_2 treatment of raw fibers (Sample C) shows an equally low moisture regain value (5.6%) as samples D, E, and F due to the destruction of hemicellulosic structure.

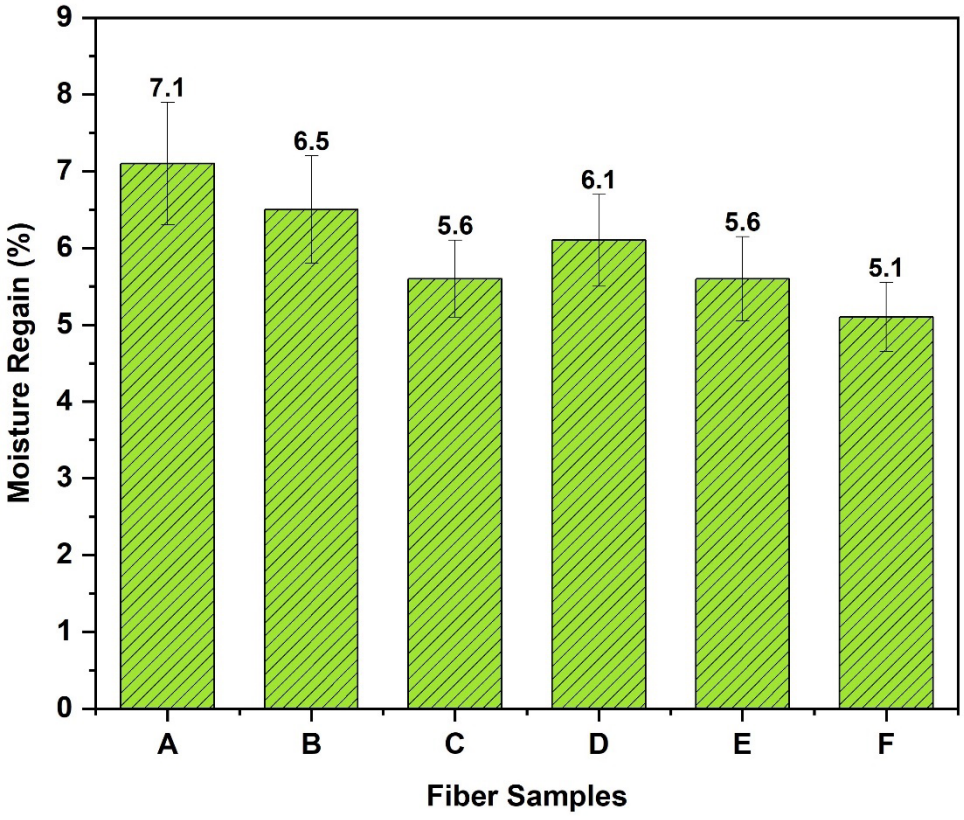

Figure 2. Comparison of moisture regain of developed fiber samples.

3.3. Tensile Properties of Sugarcane Fibers

Fiber strength plays an important role in the production of yarns with properties that are required to transform them into fabric [27–29]. The tensile properties of various treated and untreated sugarcane fibers were evaluated, and a comparison of variation in mechanical properties was plotted, as shown in Figure 3. Sample A shows the mechanical behavior of untreated raw sugarcane fibers. The silicone treatment on the raw fibers (Sample B) improves the tenacity, and a major improvement in the elongation at break can also be observed. The improved elongation of treated fibers (Sample B) and tenacity is due to the ductility that silicone softener imparts to the fibers by filling up the void spaces within fiber bundles [30]. Comparatively, a minor decline (as compared to Sample A and B) is evident in the tenacity of H_2O_2-treated sugarcane fibers (Sample C), which could be attributed to the damage caused to fiber structure [31]. In contrast, the alkali–H_2O_2-treated fiber samples (B, C, D) with 50 g/L silicone softener conc. show an increase in the tenacity and force at break (cN) values. In untreated raw fibers (Sample A), the dispersion of lignin and hemicellulose in the interfibrillar structure causes a constraint to cellulosic chains, which is the reason for lower tenacity. The alkali–H_2O_2 treatment removes the lignin and hemicellulose content, and the cellulosic chains become more compact, resulting in improved overall fiber strength and mechanical properties. Alkali–H_2O_2 increases the strength (Sample B, D and E) but also results in rigid and brittle fibers, which causes a significant reduction in elongation at break of fibers, as observed in Figure 3 (Sample C, D, E, and F) [31]. Increasing the alkali–H_2O_2 treatment conc. does improve the tenacity (Sample D and E), but at higher concentrations of alkali–H_2O_2 treatment (14–16) g/L, the tenacity reduces significantly [32]. The reduction in mechanical strength at higher conc. of alkali–H_2O_2 treatment is also supported by SEM results.

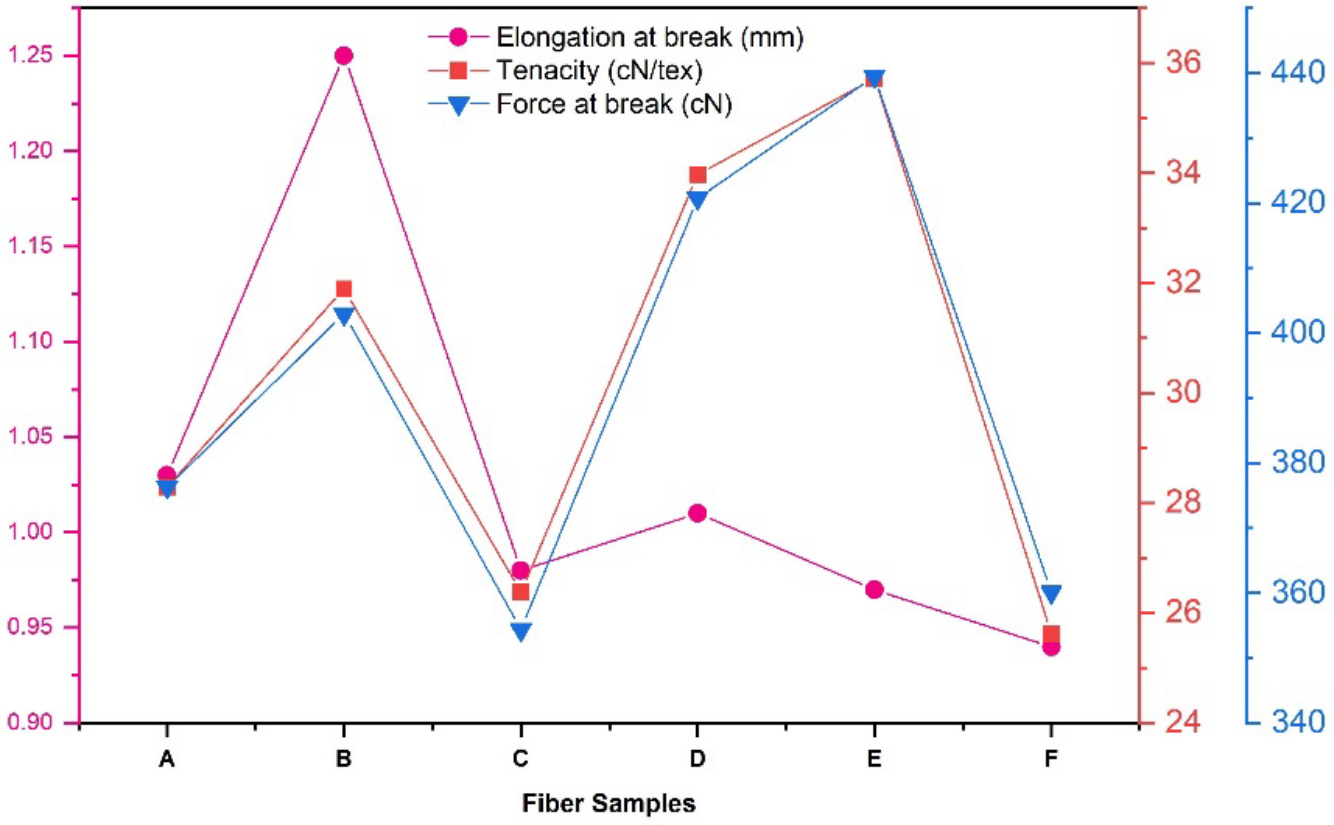

Figure 3. The variation of elongation at break, tenacity, and force at break of various fiber samples.

The torsional rigidity and flexural rigidity of various sugarcane fibers can be observed in Figure 4. The alkali conc. directly influences the rigidness of extracted fibers, whereas

the purpose of silicone softener is to eliminate that rigidness, as described above. Herein, the high torsional and flexural rigidness of alkali–H_2O_2 treated sugarcane fibers and their subsequent increase in rigidness as the ratio of aklai-H_2O_2 treatment increases, which is well in accordance with the claims made earlier. In contrast, the fibers treated with a constant ratio of silicone softener help decrease sugarcane fibers' rigidness [30]. The raw sugarcane fiber treated with only silicone softener treatment reduces the fiber rigidity (Sample B) as compared to untreated raw fibers (Sample A). In contrast, the treatment of raw fibers with H_2O_2 treatment only (Sample C) does increase the fibers' torsional and flexural rigidity significantly. The increasing conc. of alkali–H_2O_2 and the constant ratio of silicone softener (Sample D, E and F) shows an increasing trend of fiber's flexural and torsional rigidity because of the removal of hemicellulose and lignin, which causes the fiber molecules to pack closely, thus increasing the fiber's rigidity, whereas silicone softener does fill in the void spaces and helps in reducing the torsional and flexural rigidity [13] but not to a great extent because of the low conc. of silicone softener used. The increase in the amount of alkali–H_2O_2 treatment subsequently increases the rigidity of fibers, thus hampering the use of fibers in woven textiles but still allowing them to be used in non-woven textiles [33].

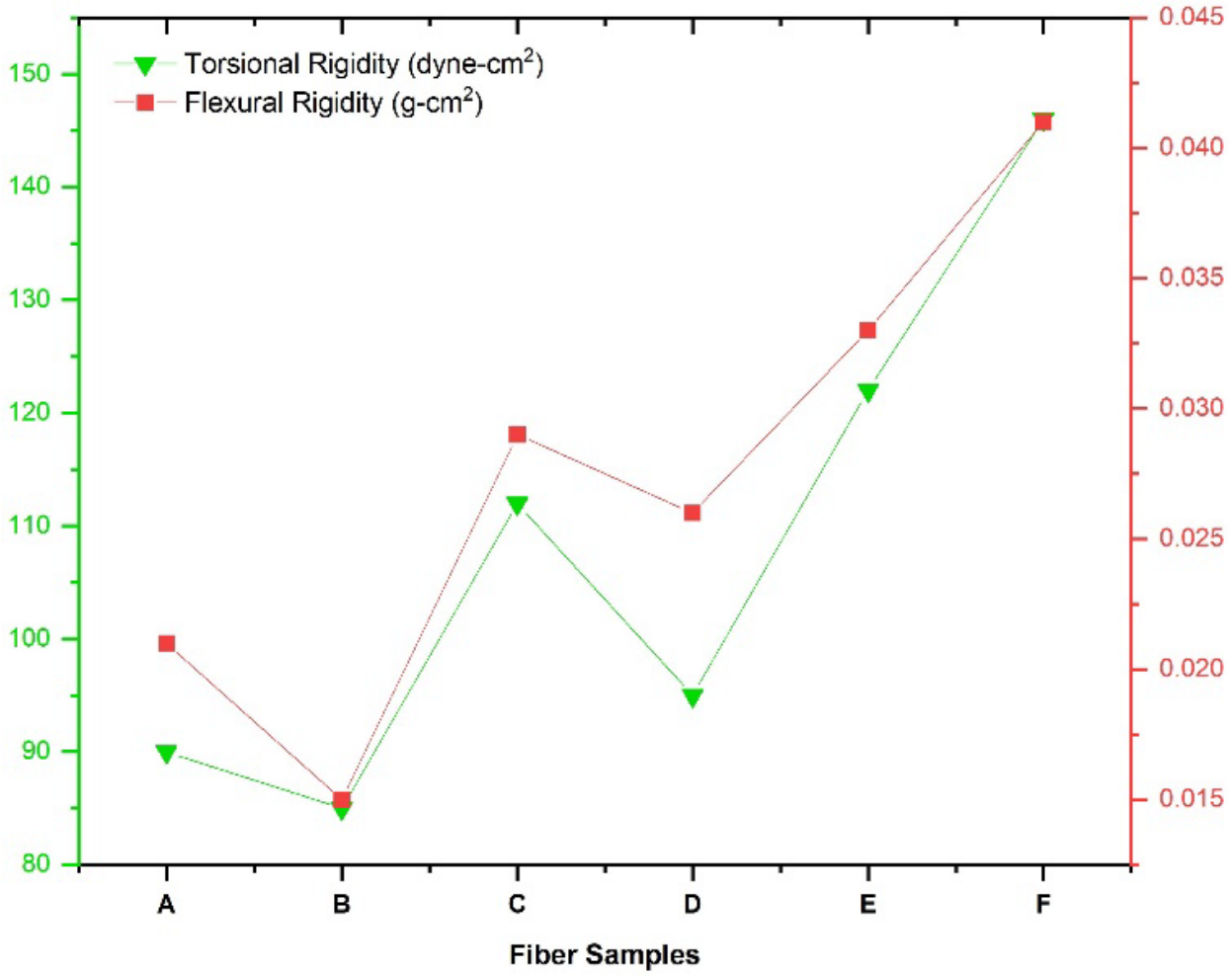

Figure 4. The variation of torsional and flexural rigidity of various fiber samples.

3.4. Morphology and Crystallinity of Sugarcane Fibers

The morphological properties of different sugarcane fibers were evaluated through Scanning Electron Microscopy (SEM), as shown in Figure 5. The untreated fiber Sample (A) SEM image exhibited a smooth, intact surface with a well-ordered fiber structure. This is because of the large amount of lignin and hemicellulose, which makes the structure

coarse. In contrast, alkali–H_2O_2-pretreated fiber samples with varying concentrations from (6–14) g/gL to (8–16) g/gL and const. silicone softener conc. (50 g/gL) as shown in Figure 4, Sample (C, D, E), which manifests a rather rough fiber surface with a significant presence of cracks and debris with a loose and rough surface [34]. These changes could be ascribed to the increasing conc. of alkali–H_2O_2 treatment, which destroys the hemicellulose and lignin linkages and causes the swelling of fibers [35]. The increase in conc. of alkali–H_2O_2 treatment affects the fiber surface, producing cracks, and any further increment in conc. destroys the fiber structure and causes the structure to open, which can be observed in Sample F. However, the application of 50 g/L silicone softener treatment on raw and alkali–H_2O_2-treated fibers coats the fibers' surface and produces a layer of grease on the edges of fibers [36], which can be more prominently observed in raw fibers coated with 50 g/L silicone softener (Sample B).

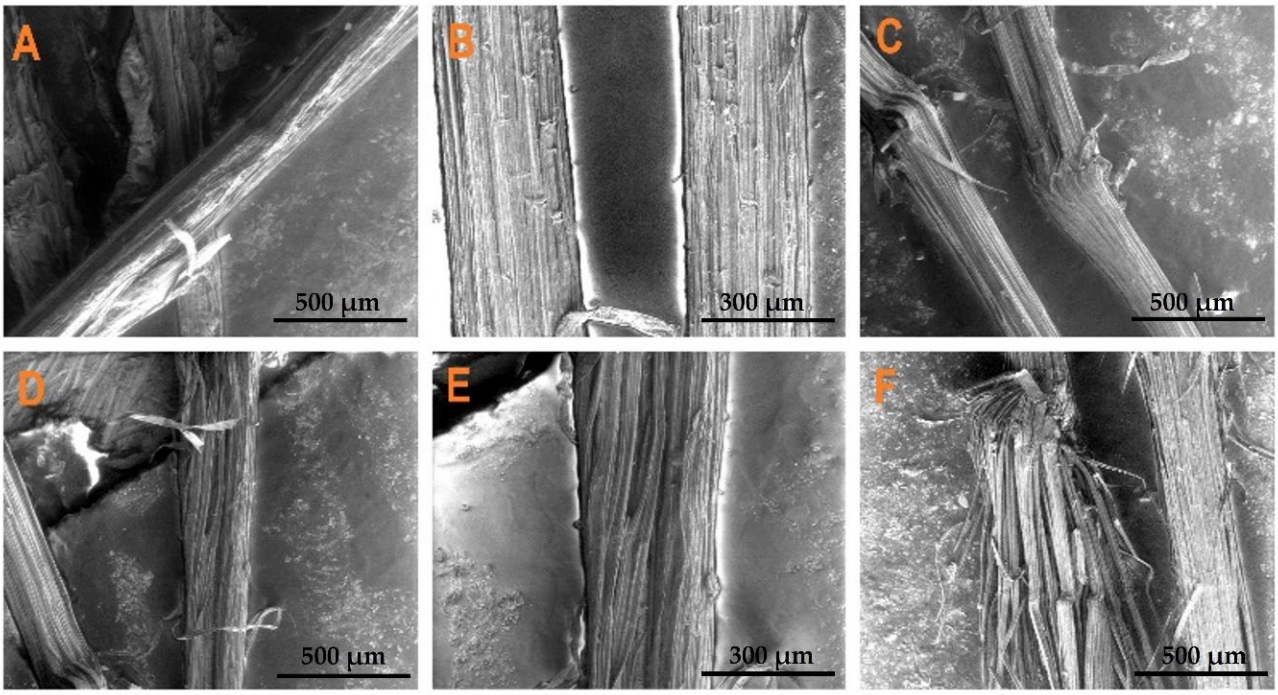

Figure 5. Scanning electron microscopy (SEM) results for different sugarcane fibers.

Figure 6 shows the X-ray diffraction pattern of various treated and untreated sugarcane fiber samples at room temperature. XRD results show some very prominent sharp peaks around the 30° position, attributed to the high crystalline region in alkali–H_2O_2-treated fiber samples (C, D, E, and F), as shown in Figure 6. In the case of untreated sugarcane fiber (Sample A), the crystalline region is not as sharp as in alkali–H_2O_2-treated fibers. This is due to the dispersion of lignin and hemicellulose in the interfibrillar fiber structure, which restricts Sample A's compact cellulosic chain arrangement. After alkali–H_2O_2 treatment, the fibrils become more capable of rearranging themselves in a compact packing due to the removal of lignin and hemicellulose [13]. An increase in the conc. Of alkali–H_2O_2 treatment also increases the crystallinity, as can be observed for fiber Samples D, E, and F, whereas silicone softener treatment neutralizes the stiffness of the alkali–treated fibers [37]. The loss of crystalline peak as observed in silicone-softener-treated raw fiber (Sample B) at 29.37 ° is related to the ductility and softness it imparts to the fiber, increasing the amorphosity of fibers [38]. The H_2O_2 treatment of raw fibers increases the crystallinity of the fibers because of the degumming process. The degumming of fibers makes room for the rearrangement of molecules and helps achieve high crystallinity [39], as shown in Sample F (Figure 6).

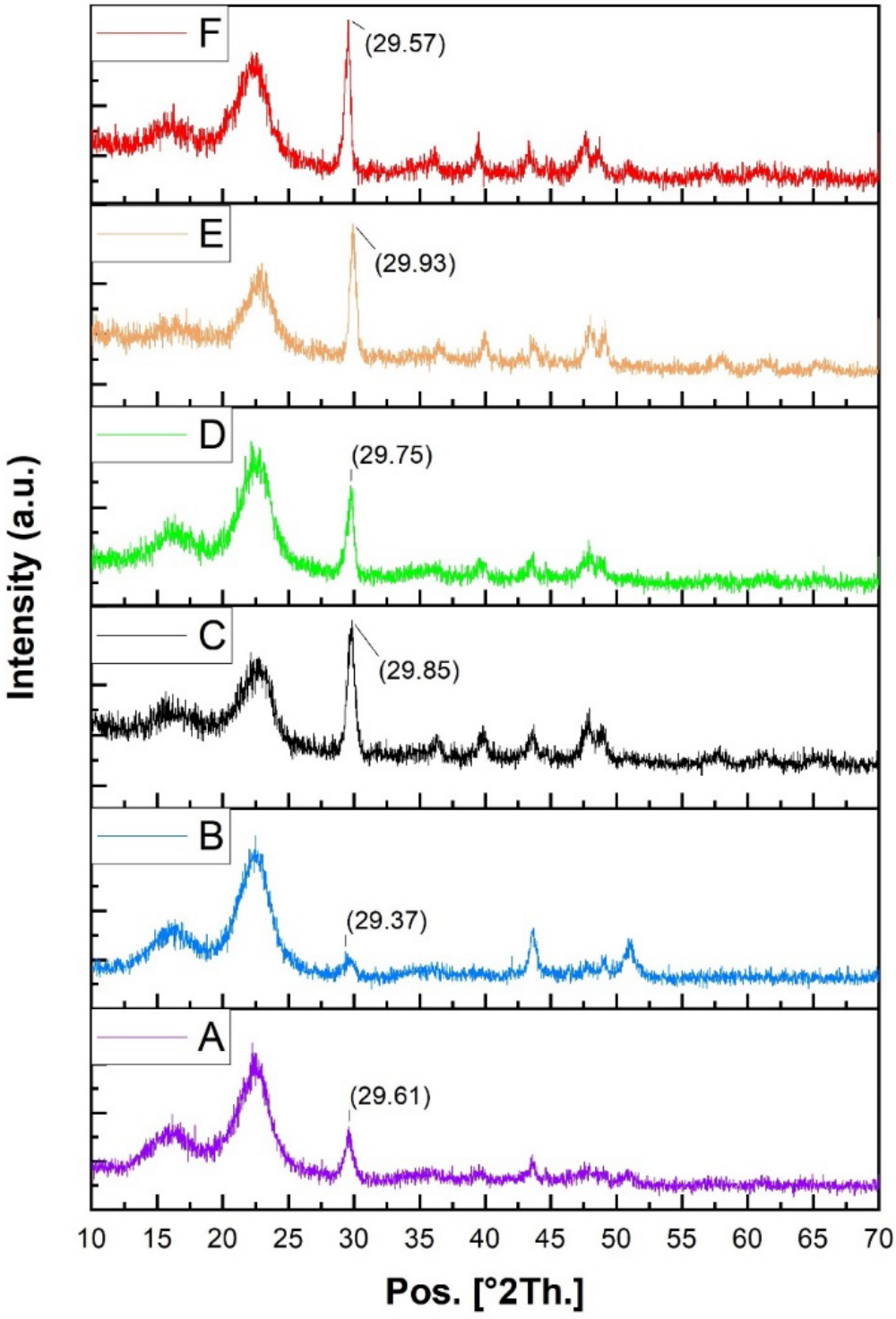

Figure 6. X-ray diffraction results of fiber samples.

4. Conclusions

The sugarcane fibers were extracted through alkali–H_2O_2 pre-treatment with varying concentrations, and post-treated with a constant ratio of silicone softener. Raw fibers were also treated solely with H_2O_2 and silicone softener to modify and evaluate the properties and draw a comparison. Treated and untreated fibers were tested mechanically and analytically. The fibers were also characterized to find the fiber morphology and change in crystallinity over pre- and post-treatment of fibers. A reduction in the fiber's linear density from 54.82 tex to 45.13 tex, as well as moisture regain (6.1% to 5.1%), was observed as the

ratio of alkali–H_2O_2 treatment was increased. The alkali–H_2O_2 pre-treatment also affected the fiber morphology and mechanically strengthened the fibers, but further increase in alkali and H_2O_2 conc. Resulted in the reduction of mechanical strength; overall torsional and flexural rigidity was also increased as the conc. Of alkali–H_2O_2 treatment was increased. Post-treatment with silicone softener further strengthened the fibers and improved their overall fineness by reducing the fiber's linear density and rigidity. A minor decrease in performance, especially in fiber's mechanical properties, was observed upon treatment with H_2O_2. Upon increasing the conc. Of alkali–H_2O_2, the fiber samples showed a substantial decrease in fiber moisture regain values. An increase in fiber crystallinity was observed as the conc. Of alkali–H_2O_2 increased. Overall, an improvement in fiber fineness and excellent mechanical properties make the fibers a viable choice for textile applications, which will be a stepping stone towards cheaper, sustainable, and environmentally friendly products.

Author Contributions: Methodology and funding acquisition, M.J.; supervision and validation, Z.K.; project administration and writing—original draft, M.B.Q.; data curation and writing—review & editing, Z.A.; conceptualization, A.A. (Ali Afzal); investigation, review, and editing, U.A.; software, A.A. (Adnan Ahmad); visualization, M.F.; resources, M.A.; data Curation, M.I.; resources and investigation, S.A.A.; formal analysis, F.A.H. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the Deanship of Scientific Research at Najran University grant code (NU/RG/SERC/11/6).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data will be available upon request.

Acknowledgments: The authors are thankful to the Deanship of Scientific Research at Najran University for funding this work under the Research Groups Funding Program: grant code (NU/RG/SERC/11/6).

Conflicts of Interest: All authors certify that they have no affiliations with or involvement in any organization or entity with any financial interest (such as honoraria; educational grants; participation in speakers' bureaus; membership, employment, consultancies, stock ownership, or other equity interest; and expert testimony or patent-licensing arrangements), or non-financial interest (such as personal or professional relationships, affiliations, knowledge or beliefs) in the subject matter or materials discussed in this manuscript.

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