The Effect of Activated Water Degumming Technique on Alkali-pretreated Banana Fiber

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There is a need for sustainable ways of processing plant fibers. Most chemical degumming methods pose a threat to the eco-system because of the presence of toxins during disposal. This study aimed to find sustainable ways of processing banana fiber with less harm to the environment. Sodium hydroxide (NaOH) and activated water were utilized for degumming and softening of the banana fiber. Activated water is an aqueous solution of electrolyte with strongly oxidizing active substances prepared by an electrochemical method. The alkali-pretreated banana fiber were immersed in activated water to refine banana fiber. The results from the study show that activated water further refined the fibers down to 39.7 µm diameter after NaOH pretreatment and also achieved better whiteness at 74.7%. The electrochemically activated water solutions displayed a promising effect in removing lignin, hemicellulose, and other impurities. The fibers of this quality could be used in the spinning of varn and for other textile applications. Various analytical techniques, including digital microscope, scanning electron microscope, Fourier transform Infrared spectroscopy, X-ray diffractometry, tensile test, and whiteness test were utilized. These tests confirmed that the activated water used on banana fiber enhanced physical, chemical, and morphological properties.

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Keywords: Banana fiber degumming; Alkali pretreatment; Activated water; Fiber diameter; Fiber whiteness

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INTRODUCTION

With the rising demand for biodegradable, eco-friendly fiber materials as alternatives to synthetic fiber for various applications, there is also a growing demand to explore potential sources of natural fiber, such as fibers from banana plants (Balda *et al.* 2021). In many areas, banana pseudo-stems are wasted after harvesting fruits and are left to decompose in gardens, emitting a substantial amount of air pollutants with a bad odor (Subramanya *et al.* 2017; Mostafa 2021). Therefore, banana pseudo-stems must be developed to extract the high-quality fiber to serve a purpose in textiles and other applications.

Previously, some studies explored the traditional degumming technology that improved the mechanical and morphological properties of the various natural fibers. For instance, micro-organisms and enzymes have been widely used in ramie degumming to improve the fiber properties. Microbial action on fiber was found to be safe and environmentally friendly, with minor damage to the fiber (Dijkstra 2010; Xu et al. 2015; Singh et al. 2020). However, due to the lengthy degumming time and expensive equipment required, the use of enzymes has been limited (Hassan and Saifullah 2019; Bruhlmann et al. 2000). Optimization of process parameters by alkali degumming processes in hemp fiber realized the minimum strength loss, effectively reduced lignin content and other impurities in the fiber and shortened the process duration (Ahirwar et al. 2021). Nonetheless, there is a limitation due to environmental hazards associated with the chemical treatments (Mamun et al. 2021). Degumming of fibers is mainly completed using chemicals, including alkalis, bleaching agents, surfactants, acids, and oil-water emulsion (Arifuzzaman Khan et al. 2013). Most by-products of chemical degumming could end up in the water, soil, or general ecosystem, and cause pollution. Some efforts have been made towards innovation for harmless and eco-friendly technology (Xu et al. 2015; Mao et al. 2019).

Some researchers believe that activated water treatment of fiber is a new ecofriendly degumming method. Activated water is prepared by electrochemical catalytic oxidation. It can produce small molecular substances with high activity and strong oxidizing property, which react with lignin, pectin, hemicellulose, and other substances. In the existing textile research, activated water was used to treat hemp fiber, which only took 30 min without boiling; the pretreatment process was short, effective, environmentally friendly, and energy saving (Zhu et al. 2021). The use of electrochemical delignification processing of pulp fiber could achieve good results in terms of delignification rate, bleaching performance, and pulp viscosity (Zhong et al. 2013). It was also reported that the whiteness and dye uptake of the electro-chemically prepared activated water in cotton fiber bleaching only took 10 to 20 min (Das et al. 2016). In addition, it has been extensively used in denim washing, textile dyeing, reduction of indigo and vat dyes, and color removal of textile wastewater (Fukatsu and Kokot 1997, 2000; Bechtold et al. 2006). Thus, the activated water prepared by electrochemical method has the advantages of saving time, high efficiency, low energy consumption, and no environmental hazards (Cao et al. 2018). The use of activated water treatment has a broad application prospect in the textile industry.

To date, there has been no report on the banana fibers that have been degummed with activated water. Therefore, the combination of alkali pretreatment and activated water treatment was used for degumming banana fiber in this study. Alkali pretreatment can remove most of the impurities from the fiber, and the subsequent activated water treatment can further remove the impurities and improve the fiber whiteness remarkably. The combination of the two enhances the quality of the fibers, resulting in fibers that can be used in yarn production and other textile applications by cutting to about 4 cm, and have great industrial application potential. The structure of the fiber was characterized by scanning electron microscopy (SEM), Fourier transform infrared (FTIR) spectroscopy, and X-ray diffraction (XRD). The fiber properties, including diameter, tenacity, chemical composition, and whiteness, were analyzed to determine the optimum conditions for activated water treatment of the fiber.

EXPERIMENTAL

Materials

The banana fiber was extracted from fresh pseudo-stem sheaths by a machine decorticator from Texfad Ltd. in Mukono, Uganda, East Africa. The detergent (Tide powder) used in the experiment was purchased from Hebei Huayatu Co., Ltd., Hebei province (China). Sodium hydroxide (NaOH) was supplied by Tianjin Chengyuan Co., Ltd. (China). Tiangong University in China provided activated water with a pH of 9 and a hydrogen peroxide concentration greater than or equal to 1000 mg/L (Zhu *et al.* 2021).

Experimental Design

Based on the previous research (Twebaze *et al.* 2022), banana fiber alkali pretreatment was conducted with two steps, which were boiling the distilled water and alkali treatment.

Alkali pretreatment

First, the untreated fibers were boiled in water and emulsified using a 1% concentration of Amway home concentrated multipurpose detergent for one hour and then dried at 80 °C for one hour. Next, the banana fibers were boiled at 95 °C for 3 h in distilled water containing 10% NaOH with a liquor ratio of 1:20 (w/v). Then, the alkali in pretreated fiber was neutralized by 1% HCl solution and the fibers were dried in the oven at 80°C for 3h.

Activated water treatment

The active water used for banana fiber refining was prepared using an electrochemical device (Zhu *et al.* 2021). Sodium chloride was selected as the electrolyte, the concentration was 30g/L, graphite was used as the electrode, the current intensity was controlled at 2A, and the voltage range was maintained at about 10V. It was believed that there were some substances with strong oxidation activity, such as OH–, HClO, ClO–, and H₂O₂, in the aqueous solution generated by electrolysis, which could react with lignin, pectin, hemicellulose, and other substances, and play a role in degumming and bleaching of banana fiber to a certain extent.

Experiments A and B involving different bath ratios and treatment times were treated with activated water at 30 °C. In experiment A, to detect the effect of bath ratio on degumming and fiber properties, the pretreated fibers were placed in activated water with various bath ratios (1:60, 1:70, 1:80, 1:90, and 1:100 w/v) continued for 50 min at 30 °C to degum the fibers. The fibers were then rinsed to remove residual chemicals and then dried in an oven for 1.0 h at 80 °C. In experiment B, activated water with a constant bath ratio of 1:80 was added for different times (30, 40, 50, 60, and 70 min) according to a similar treatment procedure. Figure 1 explains the banana treatment, processing steps and their outcomes.

Note: "UT" represents the untreated fiber sample; "AT" represents the alkali pretreated fiber sample; "AWT" represents the activated water treated fiber sample; and "AWT1" represents the fiber sample treated with activated water at a bath ratio of 1:80 and a duration of 50 min.

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Fig. 1. Schematic diagram showing the raw fiber obtained from pseudostems, alkali pretreatment, and activated water treatment processes

Fiber Fineness Test

The LLY-27 analyzer (Laizhou Electronic Instrument Co., Ltd., Shandong Province, China) was used to determine the fiber diameter (fineness) by taking on the single fiber. A total of 80 fibers were tested. The distribution pattern of fiber diameter and number was obtained. The weighted average values of fiber diameter and number were calculated for the concentrated distribution in the diameter range. This fiber average diameter acted as the fiber fineness.

Mechanical Properties Test

Individual fiber mechanical properties were measured using a computer-controlled YM-06A type electronic single fiber strength machine (Shaoxing Yuan Mao Electromechanical Equipment Co., Ltd., Zhejiang Province, China). An approximate 5 cm fiber length, 90 cN pre-tension, 20 mm gauge, and 100 mm/min were used. A total of 30 fibers were tested. The tested samples were from those that had been tested to obtain the fiber fineness.

FTIR Spectroscopy

Fourier-transform infrared spectroscopy was performed to analyze the change of chemical functional groups, which was employed to determine the disappearance of certain gums and chemical form of new substances. The analysis was obtained on Nicolet iS50 Spectrometer (Thermo Fisher Technologies, Boston, MA; USA). The spectra were read in the absorption mode in the range of 4000 to 400 cm⁻¹ with a resolution of 4 cm⁻¹.

X-ray Diffractometry

An X-ray diffractometer (D8 Discover, Bruker, Germany) aided the samples' crystallinity analysis. The diffraction intensities ranged between 10° and 43° (2θ angle range). The crystallinity index (CrI) of the material was calculated using Eq. 1,

$$CrI = (I_{002} - I_{am})/I_{002} \times 100\%$$
⁽¹⁾

where *CrI* represents the relative degree of crystallinity (%), I_{002} is the maximum intensity of the (002) crystal lattice diffraction at $2\theta = 22.6^{\circ}$, and I_{am} is the intensity of diffraction at around $2\theta = 15.3^{\circ}$.

Scanning Electronic Microscopy

Scanning electron microscopy (Hitachi Model S-4800 Tokyo, Japan) revealed the surface morphology of samples. The fiber was coated with gold before testing (SBC-12, Shanghai Minyi Electronics Co., Ltd., Shanghai, China). All micrographs were taken under the operating voltage of 15 kV and magnification of 1000 times.

Fiber Chemical Composition Test

The chemical composition of the fiber sample was analyzed using the wet traditional standard method GB/T 5889 (1986). Every constituent was dried at 105 $^{\circ}$ C before the weight was measured. The weight of each component was determined, and the percentage was recorded.

Fiber Whiteness Index Test

The whiteness index of all tested samples was determined using a whiteness meter (WSD -3U, Beijing Kangguang Instrument Co., Ltd., Beijing, China). The fiber samples were wound and aligned parallel in a paper card carefully so that an opaque sheet of fiber was prepared for instrumental assessment. Each group of samples was measured five times, and the average was calculated as the final result.

RESULTS AND DISCUSSION

Banana Fiber Mechanical Properties and Diameter Analysis

The mechanical properties and diameter were analyzed to determine the effects of bath ratios, treatment time, and degumming technique on the fiber.





The effects of bath ratio on mechanical properties and diameter are shown in Figs. 2a and 2b. It can be seen that the mechanical properties of banana fiber progressively

decreased with the increasing bath ratio. The diameter decreased with the increase in bath ratio and fluctuated slightly after 1:80. The effect of time on diameter and mechanical properties is shown in Figs. 2c and 2d. The mechanical properties and diameter of banana fiber decreased with increasing treatment time, and the fiber diameter tended to be flat after 50 min.

The lignin within the banana fiber was the filler bonding material linking the cellulose microfibril structural units to each other and the single cells, and was an important factor in maintaining the fiber mechanics. As the gum was removed, the lignin content decreased and the corresponding breaking tenacity also decreased. Under the continuous action of activated water, the effective active substances in the activated water could fully penetrate the banana fiber, causing further breakdown of the gums and thus effectively decreasing the diameter of the fibers. However, when the bath ratio was higher than 1:80 and the time exceeded 50 min, the residual gum in the fibers tended to stabilize, as a result, the fiber diameter also tended to level off. To meet the needs of downstream processing, such as yarn spinning, certain tenacity and breaking elongation values of banana fibers are desired after the degumming treatment. The greater tenacity the degummed fibers have, the better quality of banana yarn can be obtained. Therefore, the most favorable treatment for banana fibers was when the activated water bath ratio was 1:80 and the treatment time was 50 min.

As shown in Figs. 2e and 2f, as the degumming process advanced step by step, the diameter and breaking tenacity of the banana fiber gradually decreased. The reason was that under the action of alkali and activated water, a large amount of gum was removed from the banana fiber, which promoted the radial stripping of single fiber along with the fiber bundle, making the fiber diameter and tenacity decrease. According to the Chinese national standard GB/T 20793 (2015), the single fiber strength of ramie for spinning should not be less than 3.50 cN/dtex. However, after 50 min of treatment with an activated water bath ratio of 1:80, the fiber diameter was reduced to 39.7 μ m and the breaking tenacity was 4.82 cN/dtex. Therefore, the banana fibers processed using alkali pretreatment and activated water treatment in this study can offer better qualities for textile applications.

FTIR Analysis

The FTIR spectra of fiber before and after degumming using alkali and activated water are shown in Fig. 3. The spectra revealed absorbance bands corresponding to cellulose and non-cellulosic components in natural fibers (Fan *et al.* 2012). The activated water-treated fiber spectra showed bands at 2907 and 3336 cm⁻¹, which signified strong absorptions for many hydroxyls (O–H) and C–H stretching vibrations belonging to the –CH group of cellulose and hemicellulose components. The intense and broad absorption appearance indicated many hydroxyl groups or the fiber's hydrophilic behavior (Parre *et al.* 2020). The very weak bands found around 1648 cm⁻¹ meant –C=O stretching, which confirmed the presence of hemicellulose and lignin in UT that decreased after treatment (Haque *et al.* 2021). The peak at 1737 cm⁻¹ also shows the C=O stretching vibration found in hemicellulose or carboxylic acid of lignin (Dalmis *et al.* 2020). The peak at 1322 cm⁻¹ indicated O–H in-plane bending. O–H stretching of the hydrogen bond network, which became less intense, after alkali treatment. This decrease was due to breaking of hydrogen bond between O–H groups of cellulose and hemicellulose molecules.

The peaks at 1022 to 1158 cm⁻¹ showed C–O–C asymmetrical stretching and C–O/C–C stretching vibrations of cellulose and hemicellulose. The peak at 1022 and 1158 cm⁻¹ also belong to β -glycosidic linkages in cellulosic fibers. The peak at 1104 cm⁻¹

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showed ring stretching vibrations belonging to cellulose. The band located at around 662 cm^{-1} corresponds to C–OH out of plane deformation in cellulose. The bands at 553 cm^{-1} showed S-S stretching of sulfides originating from thio-substituted compounds.

The FTIR analysis of treated fibers showed that the band at 1600 to 1700 cm⁻¹ had varnished, indicating the removal of lignin from the fibers as observed in Fig. 3. In addition, the α -cellulose content in the fiber and the formation of inter- and intra-molecular hydrogen bonds in the cellulose strongly influenced fiber crystallinity, which affected the mechanical properties.



Fig. 3. FTIR spectra of (a) UT, AT, and 50 min' AWT at various bath ratios; (b) UT, AT, and AWT at 1:80 bath ratio at various times

XRD Analysis

This analysis determined the crystallinity and diffraction intensities (Subramanya *et al.* 2017). The lignin part is amorphous, and the cellulosic part is crystalline in nature. The XRD spectra revealed that removal of non-cellulosic contents made the fiber more crystalline (Aseer *et al.* 2013).



Fig. 4. X-ray diffractometric spectra of (a) UT, AT, and 50 min AWT at various bath ratios; (b) UT, AT and AWT at 1:80 bath ratio at various times

The values of 2θ angle ranged between 10° and 43°. All samples, including UT, AT, and AWT, showed typical cellulose fiber spectral peaks at 15.3° at crystal plane I_{01} and 22.6° at I₀₂, confirming the presence of primary cellulose I (Dominic et al. 2020). Thus, the spectra indicated that all the lignocellulosic fibers exhibited similar crystallinity structures. The crystallinity of AWT was as high as 62%, corresponding to the first peak $I_{01} = 5.47$ and the second peak $I_{02} = 16.11$ in Fig. 4a. Figure 4b displays the crystallinity of the fiber at 53%, corresponding to the first peak $I_{01} = 6.08$ and the second peak $I_{02} = 11.58$. The unusual peak at 2θ angle 28.9° could be due to precipitation of some Na⁺ cations with other compounds (Nergis et al. 2020). During the 1:80 bath ratio and 50 min treatment duration, the crystallinity index of the fiber was remarkably improved from 53% to 62%, indicating that the activated water treatment greatly enhanced the cellulosic content of banana fiber. This is because lignin, hemicellulose, and other extractives were removed, the macromolecular chains of fibers were re-arranged, and the molecules were more orderly arranged. Therefore, it is confirmed that better crystallinity signified improved quality of fibers after activated-water treatment. The crystallinity improved as the fibers experienced continuous degumming by alkali and activated water treatment.

SEM Analysis

Figure 5 shows the micrographs obtained from SEM analysis. The morphological features of the fibers were observed to determine the extent of the degumming effect on the fiber physical surfaces (Senthilkumar *et al.* 2018; Subagyo and Chafidz 2018).



Fig. 5. SEM micrographs of (a) UT, (b) AT, AWT with bath ratio (c) 1:60, (d) 1:70, (e) 1:80, (f) 1:90, (g) 1:100, and AWT at various times (h) 30 min, (i) 40 min, (j) 50 min, (k) 60 min, (l) 70 min

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The UT in Fig. 5a contains wax, pectin, and lignin, and its surface is rough and has a poor hand feel compared to cotton fibers due to the presence of gum. After NaOH treatment, some gum was removed from the surface, as shown in Fig. 5b. In the next images Fig. 5c to Fig. 5l, a combination of sodium hydroxide and activated water treatments revealed better results by changing the activated water bath ratios and treatment durations. The increase in bath ratios and treatment time revealed reduced diameter and more degummed surfaces. The surfaces of fibers treated with the combination of two methods yielded the best results at a bath ratio of 1:80 and 50 min duration. The surface became smoother and cleaner, indicating that alkali and activated water combined treatments effectively removed lignin, hemicellulose, pectin, and other surface impurities.

Chemical Composition Analysis

The chemical composition of AWT was measured through the compositional analysis as shown in Tables 1 and 2. The results of UT and AT were compared with those of the AWT in Tables 1 and 2.

Table 1. Fiber Chemical Composition of UT, AT, and 50 min' AWT at Various
Bath Ratios

Method	Bath Ratio	Wax (%)	Water Extractives (%)	Pectin (%)	Hemicel- lulose (%)	Lignin (%)	Cellulose (%)
UT	_	1.900 ± 0.036	6.500 ± 0.608	2.780 ± 0.044	22.220 ± 0.751	21.300 ± 0.500	45.300 ± 0.626
AT		1.800 ± 0.021	0.980 ± 0.046	0.980 ± 0.017	3.920 ± 0.400	19.610 ± 0.300	72.710 ± 1.200
AWT	1:60	1.740 ± 0.036	0.835 ± 0.012	0.970 ± 0.020	3.610 ± 0.108	17.050 ± 0.436	75.795 ± 0.901
	1:70	1.630 ± 0.026	0.830 ± 0.009	0.900 ± 0.060	3.470 ± 0.105	16.700 ± 0.721	76.470 ± 0.990
	1:80	1.350 ± 0.020	0.759 ± 0.026	0.794 ± 0.004	3.070 ± 0.058	15.840 ± 0.458	78.187 ± 0.656
	1:90	1.410 ± 0.053	0.790 ± 0.026	0.755 ± 0.010	3.123 ± 0.061	15.610 ± 0.529	78.312 ± 0.700
	1:100	1.390 ± 0.010	0.810 ± 0.035	0.730 ± 0.009	3.220 ± 0.100	15.420 ± 0.346	78.430 ± 1.249

Before activated water degumming, the cellulose content in banana fiber accounted for only 45.3%. After alkali pretreatment, the pectin content in the fiber decreased from 2.78% to 0.98%, the hemicellulose content decreased from 22.2% to 3.9%, and the cellulose content in banana fiber increased to 72.7%. This increase of cellulosic content signified that the alkali pretreatment process could remove impurities from the banana fiber.

Method	Time (min)	Wax (%)	Water Extrac- tives (%)	Pectin (%)	Hemicell- ulose (%)	Lignin (%)	Cellulose (%)
UT	_	1.900 ± 0.036	6.500 ± 0.608	2.780 ± 0.044	22.220 ± 0.751	21.300 ± 0.500	45.300 ± 0.626
AT		1.800 ± 0.021	0.980 ± 0.046	0.980 ± 0.017	3.920 ± 0.400	19.610 ± 0.300	72.710 ± 1.200
AWT	30	1.780 ± 0.017	0.960 ± 0.026	0.924 ± 0.000	3.526 ± 0.076	17.515 ± 0.625	75.295 ± 0.703
	40	1.482 ± 0.010	0.808 ± 0.003	0.876 ± 0.006	3.233 ± 0.069	16.499 ± 0.400	77.102 ± 1.054
	50	1.350 ± 0.011	0.759 ± 0.005	0.794 ± 0.012	3.070 ± 0.082	15.840 ± 0.483	78.187 ± 0.557
	60	1.370 ± 0.030	0.735 ± 0.013	0.802 ± 0.009	3.107 ± 0.107	15.779 ± 0.529	78.207 ± 0.580
	70	1.352 ± 0.029	0.768 ± 0.024	0.841 ± 0.020	3.033 ± 0.118	15.921 ± 0.608	78.085 ± 0.800

Table 2. Fiber Composition of UT, AT, and 1:80 Bath Ratio's AWT at Various

 Times

The contents, such as lignin, hemicellulose, pectin, and other extracts decreased gradually as activated water bath ratios and time increased, as reflected in Tables 1 and 2. The higher the lignin content, the fiber became less flexible to handle and had higher diameter. After activated water treatment, the contents of wax, water extractives, pectin, hemicellulose, and lignin were decreased. For instance, a bath ratio of 1:80 and 50 min treatment revealed lower levels of impurities. These impurities included wax at 1.35%, water extractives at 0.76%, pectin at 0.79%, hemicellulose at 3.07%, and lignin at 15.8% as a result of activated water treatment. The impurities were lower in AWT than that in AT and UT (Tables 1 and 2).

At the treatment time of 50 min and the bath ratio of 1:80, the cellulose composition was optimized. The yield percentage of cellulose was 78.2%, which remarkably improved the quality of banana fiber. The improved fiber fineness or smaller diameter was caused by reduced impurities from the fiber.

Whiteness Analysis

Figure 6 explains activated water action on banana fibers. As shown in Fig. 6a, when the activated water bath ratio and treatment time were increased, the whiteness of the fiber improved. When the bath ratio exceeded 1:80 and the treatment time surpassed 50 min, the whiteness tended to be stable. At lower bath ratios and durations, activated water interaction with fiber was insufficient, and the whiteness value was low. When the bath ratio and time increased, the fiber was thoroughly immersed in the solution. The activated water contains more effective active substances, making the pigment and lignin easier to be removed, so the fiber whiteness is enhanced. This is attributed to the fact that there were strong oxidizing substances such as OH^- and H_2O_2 present in the activated water. The bleaching principle is that the benzene ring and side-chain in the lignin structural unit were broken by HOO⁻ produced by H_2O_2 dissociation. The conjugated double bonds of some chromophore groups, such as *p*-quinone and *o*-quinone side chains, were oxidized and broken into colorless groups to increase the whiteness of the fiber (Gierer 1990; Behrooz *et al.* 2012). The chromogenic group in fiber reacts with H_2O_2 , as shown in Fig. 6b.



Fig. 6. (a) The influence of both various bath ratios at 50 min, and various times with 1:80 bath ratio on fiber whiteness, (b) reaction of chromogenic groups in fibers with H_2O_2 , and (c) the whiteness of AT and AWT1

Figure 6c shows the AT and AWT1. The whiteness of AT increased from 32.4% to 74.7% when treated by the activated water bath ratio of 1:80 at 50 min. The results revealed that activated water had a tremendous bleaching capacity and degumming effect that improved the whiteness of the banana fibers.

CONCLUSIONS

- 1. A method of combined degumming of banana fibers by alkali pretreatment supplemented with activated water was developed. The best overall result in preparing the fibers for textile applications was a fiber diameter of $39.7 \,\mu\text{m}$ and breaking tenacity of $4.82 \,\text{cN/dtex}$ using a 10% NaOH solution and then an activated water bath ratio of 1:80 for 50 min.
- 2. Due to the high oxidation capacity of activated water, the whiteness of the fibers increased from 32.4% to 74.7%. A smooth fiber surface and good separation between fibers were observed by SEM, and the FTIR spectrum indicated the removal of lignin and hemicellulose components.
- 3. Activated water treatment improved the performance of alkali pretreated fibers and provided an alternative for processing high quality banana fibers. This processed fiber can be utilized for yarn production and other textile applications.

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