One-Step Alkaline-Hydrogen Peroxide Degumming of Hemp Fiber: Optimization for Enhanced Processability and Eco-Efficiency

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Hemp fiber, as a renewable bio-based material, holds significant potential in the textile and paper industries. To unlock this potential, a critical step involves purification of the fibers. However, conventional degumming methods suffer from high energy consumption, severe fiber damage, and environmental pollution. This study evaluated a proposed one-step alkaline-hydrogen peroxide degumming process under mild conditions to achieve high-value utilization of hemp crops. Orthogonal experiments were conducted to optimize reaction conditions, including temperature, time, and liquid-to-solid ratio. Results showed that under optimal conditions (80°C, 4 h, 10:1 liquid-to-solid ratio), the residual gum content was 7.68% and fiber crystallinity increased by 8.33%. Additionally, the proportion of short fibers increased while coarse fibers decreased, yielding paper with a tensile index of 190 N/m and a whiteness of 70.3%. This low-temperature degumming process effectively removed gums while minimizing fiber damage, offering an eco-friendly and industrially viable solution for hemp fiber applications.

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INTRODUCTION

Hemp (*Cannabis sativa L.* subsp. *sativa*), as a natural fiber crop with a long cultivation history, has attracted considerable attention due to its excellent fiber properties and environmental friendliness (Liu *et al*. 2018). China, being the world's largest producer of hemp, holds significant advantages in hemp cultivation and processing. Hemp fibers possess unique structural characteristics: single fiber fineness of approximately 15 to 30 μm, an irregular polygonal cross-section, and a longitudinally distributed porous structure, which endow them with outstanding moisture absorption and perspiration properties (Rani *et al*. 2020). Hemp fibers also exhibit remarkable heat resistance (withstanding temperatures up to 370 °C) and antistatic properties (30 to 90% better than cotton), making them highly valuable functional textile materials (Barberà *et al*. 2011; Jiang *et al*. 2017; Bakshi *et al*. 2024). Moreover, the chemical composition of hemp is dominated by cellulose, with a content ranging from 60% to 86.7%. However, the directional

optimization of components such as hemicellulose and lignin can be achieved through variety breeding or agronomic regulation, thereby adapting to the customized needs of fiber raw materials in different industrial scenarios such as textiles and building materials (Qin *et al*. 2024). Critically, this crop achieves harvest maturity within 3 to 4 months after sowing due to rapid biomass accumulation, supporting multiple annual production cycles in suitable agroclimatic zones. Such accelerated growth kinetics secures sustainable, high-yield raw material provision for industrial supply chains (Crini *et al.* 2020; Zhao *et al.* 2021).

Potential applications of hemp fibers in the paper industry have gradually emerged (Axelrod *et al*. 2023). Lyu *et al*. (2022) demonstrated that hemp pulp can reduce chemical usage by 30% and COD emissions by over 40%. Hemp has a short growth cycle of only 90 to 120 days, with fiber yield per unit area being 2 to 3 times that of fast-growing forests (Huang *et al*. 2024). These advantages position hemp fibers as a potential alternative to address resource scarcity and environmental pollution in the paper industry (Small *et al*. 2003). However, developing efficient and low-consumption degumming processes remains a key challenge for realizing the high-value utilization of hemp fibers (Lyu *et al*. 2021).

Hemp fibers consist mainly of cellulose, accompanied by non-cellulose symbiotic substances such as hemicellulose, pectin, lignin, and the like (Lin *et al*. 2023). These non-cellulose symbiotic substances can impede the high-value utilization of hemp fibers. To purify the fibers and optimize spinnability, components such as hemicellulose and pectin need to be removed *via* a degumming process. Such processes, depending on the conditions employed, may also remove substantial amounts of lignin. During degumming, these dissolved or decomposed non-cellulose symbiotic substances are collectively termed "gum" (Cheng *et al.* 2020). In the papermaking field it can be advantageous to employ a selective process in which the hemicellulose is retained, and the term "degumming" generally is not used. In the course of delignification and bleaching steps used in processing papermaking fibers, only a portion of the hemicellulose becomes solubilized. The hydroxyl groups and non-crystalline nature of hemicellulose can enhance the bonding force between fibers through hydrogen bonds, thereby achieving precise regulation of paper strength (Wang *et al.* 2023). Unprocessed hemp fibers, which are bound by gums into coarse rigid bundles, are unfavorable for subsequent processing, and residual non-cellulosic components directly reduce key performance indicators of the finished products such as mechanical properties and softness. Therefore, a degumming process is a crucial step in hemp fiber processing, directly affecting the spinnability and final product quality (Liu *et al*. 2017). Current degumming methods include enzymatic, chemical, and physical approaches. Enzymatic degumming employs pectinase and xylanase to break down gums. Zhou *et al*. (2017) improved pectate lyase, reducing its optimal temperature from 67.5 to 60 °C, significantly enhancing degumming efficiency. Although enzymatic methods are mild and can reduce chemical usage by 60% (Zhang *et al*. 2021), they require long processing times and have low efficiency (Angelini *et al*. 2015), making them unsuitable for industrial-scale production. Traditional chemical degumming is conducted at temperatures above 90 °C, with energy consumption accounting for 30 to 40% of production costs (Yu *et al*. 2023). While this method achieves low residual gum content (<5%), it is energy-intensive and highly polluting, requiring 1.2 to 1.5 tons of caustic soda and generating 15 to 20 tons of high-concentration wastewater per ton of fiber (Yang *et al*. 2022). Jahan *et al*. found that energy consumption decreases by 25 to 30% for every 10 °C reduction in temperature, and low-temperature treatment effectively removes impurities from lignocellulosic materials (Jahan *et al*. 2016). Physical degumming methods, although using minimal chemicals,

demand specialized equipment and struggle to achieve fiber quality consistency (Ma *et al*. 2020; Nie *et al*. 2020).

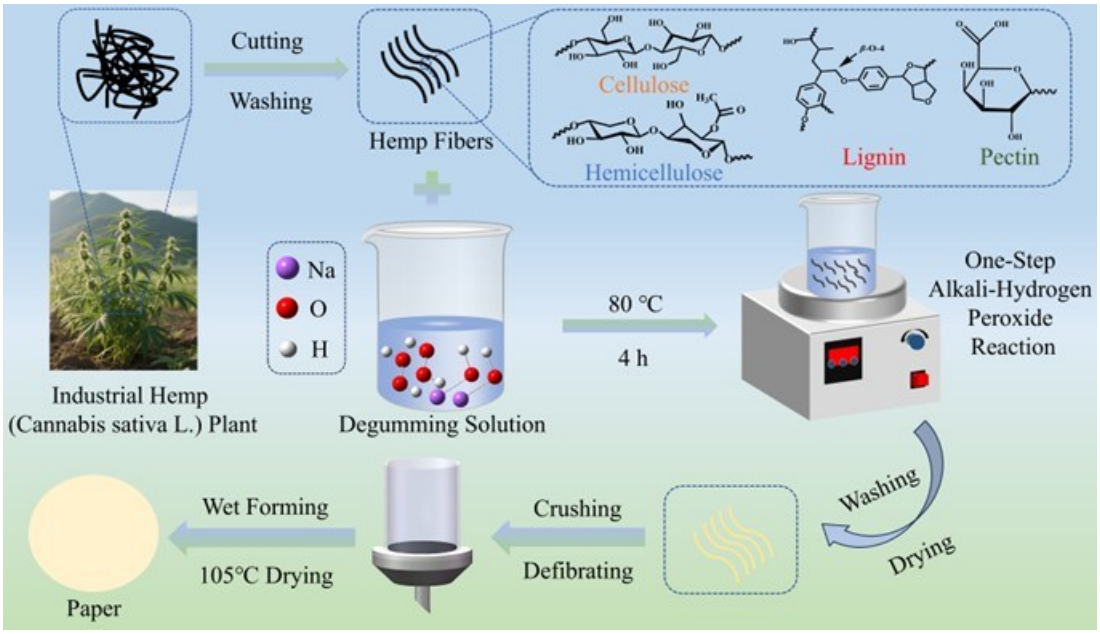

Fig. 1. Process flow diagram of hemp fiber degumming

To address these challenges, this study proposes a novel low-temperature one-step alkaline-hydrogen peroxide degumming process. Based on the differential solubility of cellulose and gum components in alkaline solutions, this method integrates sodium hydroxide and hydrogen peroxide in a single treatment bath, simplifying the process flow. Through systematic orthogonal experiments, this study investigated the effects of key parameters such as alkali and hydrogen peroxide concentrations, treatment temperature, and duration on degumming efficiency, while evaluating the physicochemical properties and papermaking suitability of the resulting fibers. The experimental workflow is schematically shown in Fig. 1. This research provides new insights and methodologies for developing efficient, energy-saving, and environmentally friendly hemp fiber processing technologies.

EXPERIMENTAL

Materials

The hemp fibers were obtained from Heilongjiang Province, China, with their tetrahydrocannabinol (THC) content strictly controlled below 0.3%. All analytical-grade reagents for degumming solution preparation were acquired from Shanghai Macklin Biochemical Technology Co., Ltd. (China), including NaOH, H_2O_2, and other chemicals listed in the Methods section.

Preparation of the Alkali-Hydrogen Peroxide Degumming Solution

This study employed a one-step alkaline-hydrogen peroxide degumming process. The degumming solution (3500 mL total volume) was prepared from 200 g of dry raw hemp, with the formulation detailed in Table 1. The prepared solution was obtained by

dissolving all components in the prescribed ratios at ambient temperature (25 ± 2 °C) with continuous stirring, followed by storage in dark, airtight containers at 4 °C until use. As listed in Table 1, the solution consisted of alkaline agent (NaOH), oxidant (H_2O_2), organic solvents, and auxiliary additives. Specifically, NaOH provided an alkaline environment (final concentration: 22.86 g/L), while H_2O_2 acted as an oxidant for synergistic degumming. Ethanol (C_2H_6O) and isopropanol (C_3H_8O) were added as organic solvents to enhance gum dissolution. Sodium silicate (Na_2SiO_3) served as a pH buffer, magnesium sulfate ($MgSO_4$) inhibited excessive H_2O_2 decomposition, and sodium dodecyl-benzenesulfonate ($C_{18}H_{29}NaO_3S$) improved fiber wettability as a surfactant.

Table 1. Formulation of Alkaline-Hydrogen Peroxide Degumming Solution

Component	Dosage	Final concentration	Function
Hydrogen peroxide (30% H_2O_2)	330 mL	3%	Oxidant
Ethanol (C_2H_6O)	100 mL	-	Organic solvent
Isopropanol (C_3H_8O)	100 mL	-	Organic solvent
Deionized water (H_2O)	2970 mL	-	Solvent
Sodium hydroxide (NaOH)	80 g	22.86 g/L	Alkaline agent
Sodium silicate (Na_2SiO_3)	20 g	5.17 g/L	pH buffer
Magnesium sulfate ($MgSO_4$)	2 g	0.57 g/L	H_2O_2 stabilizer
Sodium dodecylbenzene-sulfonate ($C_{18}H_{29}NaO_3S$)	2 g	0.57 g/L	Surfactant

One-step Alkali-Hydrogen Peroxide Degumming Process for Hemp Fibers

First, 5.0 g of cleaned and dried hemp fibers were placed in a 250 mL conical flask. Different degumming solutions were prepared at a constant bath ratio 1:20 as follows: Group 1 (solid-liquid ratio 1:20) was added with 100 mL of degumming solution; Group 2 (solid-liquid ratio 1:15) was added with a mixture of 75 mL degumming solution and 25 mL deionized water; Group 3 (solid-liquid ratio 1:10) was added with a mixture of 50 mL degumming solution and 50 mL deionized water. The prepared degumming solutions were added to the corresponding conical flasks. After ensuring that the fibers were completely immersed, the mixtures were thoroughly stirred with a glass rod to achieve uniform distribution. The conical flasks were then placed in a water bath preheated to the set temperatures (40, 60, or 80 °C) and sealed. Timing was started after the temperature stabilized. During the reaction, the conical flasks were taken out every 15 min, stirred with a glass rod for 30 s to ensure uniform reaction, and immediately returned to the water bath. After the reaction ended, the conical flasks were quickly taken out, and the fibers were transferred to a clean beaker with forceps. The fibers were repeatedly rinsed with a large amount of deionized water under stirring with a glass rod, after each rinsing cycle, the washing solution and fibers were separated using a 0.125 mm sieve, and the pH value of the washing solution was monitored until it stabilized at 7.0±0.2 to ensure complete removal of the degumming agent. The washed fibers were spread on filter paper to absorb moisture, then transferred to a 60 °C constant-temperature drying oven and dried to constant weight. Finally, the degummed fibers were sealed and stored for subsequent experiments.

The experimental design consisted of a three-factor (time, temperature, and solid-to-liquid ratio), three-level orthogonal array, as presented in Table 2.

Table 2. Factors and Levels of the Orthogonal Experiment for One-Step Alkali-Hydrogen Peroxide Degumming of Hemp Fibers

Factors	A: Temperature (°C)	B: Time (h)	C: solid-to-liquid ratio (m:v)
Level 1	40	2	1: 20
Level 2	60	3	1: 15
Level 3	80	4	1: 10

Where "m" was the dry weight of hemp fibers (g); "v" was the volume of the degumming liquid (ml).

Fabrication of Hemp Fiber Sheets

The hemp fibers treated by one-step alkali-hydrogen peroxide degumming were first pulverized for 4 min using a high-speed crusher (Model DFY-500). The crushed fibers were then mixed with water at a fiber-to-water ratio of 1:600 (w/v) to prepare a suspension, which was soaked for 4 h before separation of the fibers for 10 min using a standard fiber disintegrator (Model ZCX-200). Subsequently, handsheets with a basis weight of 100±2 g/m² were prepared following the ISO 5269-2 standard (2004).

Determination of Gum Content in Hemp Fibers

The chemical composition of hemp fibers was analyzed according to GB/T 5889 (1986). The hemp fiber raw materials were prepared by removing impurities, washing, and drying. Then, 5 g of absolutely dry hemp fibers with a length of 2 to 3 cm were placed in a sodium hydroxide solution (20 g/L) and a liquor ratio of 1:30, followed by boiling for 1 h. The solution was replaced with a fresh sodium hydroxide solution of the same liquor ratio and mass concentration, and the boiling process was continued for another 2 h. After the reaction was completed, the fibers were thoroughly washed under running water until neutral and then dried to a constant weight. In the present work, non-fibrous impurities including hemicellulose, pectin, the extractable portion of lignin, water-soluble substances, waxy lipids, and ash were all classified under the category of gums. The gum content of hemp fibers was defined as the ratio of the difference in the absolute dry mass of the sample before and after the gum content test to the absolute dry mass of the sample before the test. The calculation is shown in Eq. 1,

$$W_1 = \frac{G_0 - G_1}{G_0} \times 100\% \quad (1)$$

where W_1 is gum content of the sample (%), G_0 is absolute dry mass of the sample before the gum content test (g), and G_1 is absolute dry mass of the sample after the gum content test (g).

By weighing and calculating the gum content of hemp fibers and the weight loss rate (gum removal rate) of hemp fibers after alkali-hydrogen peroxide degumming, the residual gum content of hemp fibers is obtained as the difference between the gum content before degumming and the gum removal rate, as shown in Eq. 2,

$$W_2 = W_1 - \frac{G_0 - G_2}{G_0} \times 100\% \quad (2)$$

where W_1 is gum content of the sample (%), W_2 is residual gum content of the sample (%), G_0 is dry weight of the sample before gum removal (g), and G_2 is dry weight of the sample after gum removal (g). All experimental data represent the mean values of three independent replicate experiments.

Determination of Chemical Components in Hemp Fibers

In accordance with GB/T 5889 (1986), the contents of cellulose, hemicellulose, pectin, lignin, water-soluble substances, and waxy lipid in the hemp samples were determined. In accordance with the component determination standards, the flowchart of gum removal using the weight loss method is shown in Fig. 2. Each determination was carried out three times, and the average value was taken.

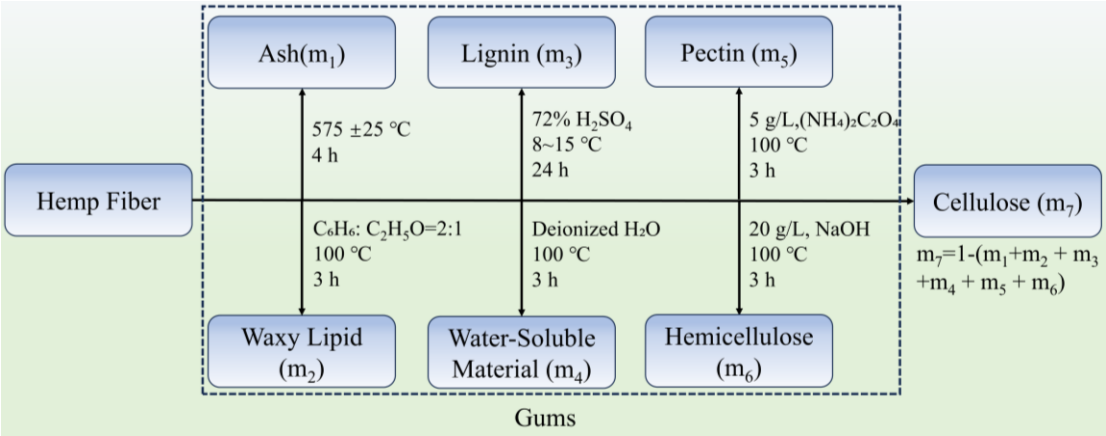

Fig. 2. Flow chart of degumming using the weight loss method in accordance with the component determination standards

Fiber Morphology Analysis

The fiber length, width, coarseness, kink angle, curl index and other indexes were measured by fiber quality analyzer (Techpap, Grenoble, France). These values can be used to identify fiber raw materials, control refining conditions, predict pulp coating, and aid in pulping the process, among other things.

SEM Analysis

A SU1510 desktop scanning electron microscope was employed to observe the surface morphology and microstructure of the samples (Hitachi, Japan). The samples of hemp fibers before and after degumming were dried and then attached to conductive adhesive. Surface gold-spraying treatment was carried out, and then the micro-morphology of the samples was observed and photographed. The accelerating voltage was 15 kV.

FTIR Analysis

An FTIR 8400S Fourier transform infrared spectrometer was used to characterize and analyze the changes in functional groups of hemp fibers before and after degumming (Shimadzu Corporation, Japan). The vacuum-dried sample powder was mixed with potassium bromide powder and fully ground. After tableting, detection was carried out. The detection wavelength range was 500 to 4000 cm^{-1}, and the cumulative scanning was 32 times.

XRD Analysis

An Ultima IV X-ray diffractometer was used to characterize the crystal phase structure of hemp fibers before and after degumming (Nippon Kuraray Co. Japan). Test conditions: A Cu target and Kα rays were used. X-ray diffraction analysis of the samples was performed under the conditions of a tube voltage of 40 kV and a current of 20 mA.

The incident wavelength of the X-rays was 0.154 nm, and scanning was carried out between 5° and 55° at a scanning speed of 4°/min. The crystallinity index (I_{cr}) of the hemp fibers was calculated according to Eq. 3 (Zhu *et al*. 2022),

$$I_{cr} = \frac{I_{002} - I_{am}}{I_{002}} \times 100\% \quad (3)$$

where I_{002} represented the diffraction intensity of the crystalline region at 2θ=22.6°, and I_{am} was the diffraction intensity of the amorphous region at 2θ=19.1°.

TG Analysis

The thermal stability of hemp fibers was analyzed using a TA Discovery SDT 650 synchronous thermogravimetric (TG) analyzer (TA Instruments, USA). 8 mg of the sample was weighed and heated to 550 °C at a heating rate of 10 °C/min under a nitrogen atmosphere.

Tensile Strength Measurement

The tensile strength of the paper was measured with reference to the national standard GB/T 12941 (2018), using a WZL-B horizontal computerized tensile tester (Hangzhou Qingtong Bok Automation Technology Co., Ltd. China). The test sample was a slender strip with a width of 15 mm and a length of more than 100 mm. The calculation formula for tensile strength (*TS*) was shown in Eq. 4.

$$TS = \frac{\bar{F}}{b} \quad (4)$$

where $\bar{F}$ represented the average value of the maximum tensile force (N), and b was the width of the sample (mm). All experimental data represent the mean values of three independent replicate experiments

Whiteness Measurement

The whiteness of the paper was measured with reference to GB/T 22879 (2008), using a YQ-Z-48B whiteness tester (Hangzhou Qingtong Bok Automation Technology Co., Ltd., China). The test sample was a whole piece of paper. All experimental data represent the mean values of three independent replicate experiments.

RESULTS AND DISCUSSION

Optimization of Process Conditions for Alkaline-Hydrogen Peroxide One-step Degumming

The parallel experiments conducted on raw hemp fibers yielded gum contents of 25.73%, 25.37%, and 25.36%, with a calculated average of 25.5%. To investigate the effects of temperature (A), time (B), and degumming solution concentration (C) on the alkali-hydrogen peroxide degumming efficiency of hemp fibers, an orthogonal experimental design was employed, with residual gum content as the evaluation metric (Table 3). Generally, a lower residual gum content indicates more effective gum removal and better degumming performance (Fan *et al*. 2021).

Table 3. Orthogonal Experimental Design and Results of the Alkali-Hydrogen Peroxide Degumming of Hemp Fibers

Number	A Temperature (°C)	B Time (h)	C m:v	#RGC (%)	#TS (N/m)	#W (%)
1	40	2	1: 20	13.62±0.36	170±3.50	68.6±0.10
2	40	3	1: 15	12.43±0.19	190±1.37	66.6±0.21
3	40	4	1: 10	12.20±0.21	130±3.40	65.6±0.27
4	60	2	1: 15	10.21±0.20	120±1.50	64.6±0.31
5	60	3	1: 10	9.82±0.24	190±2.00	59.1±0.27
6	60	4	1: 20	8.79±0.32	180±1.75	70.0±0.32
7	80	2	1: 10	8.17±0.34	230±1.22	66.3±0.23
8	80	3	1: 20	7.63±0.17	310±3.30	73.2±0.15
9	80	4	1: 15	7.36±0.25	180±3.90	71.4±0.20

#RGC: Residual Gum Content; #TS: Tensile Strength; #W: Whiteness

Range analysis revealed that the influence of the three factors on residual gum content (Table 4) followed the order: A (temperature) > B (time) > C (concentration). Temperature exerted the most significant effect on alkali-hydrogen peroxide degumming efficiency, followed by time, while concentration had the least impact. Based on the orthogonal test results of hemp fiber alkaline-hydrogen peroxide degumming, raising the temperature promoted the swelling and dissolution of pectin and hemicellulose, while accelerating the oxidation rate of lignin. The concentrations of sodium hydroxide and hydrogen peroxide reflected the supply of effective chemical reaction reagents in the alkaline-hydrogen peroxide system, and the length of reaction time was a key factor determining the degree of degumming (Bhattacharya and Shah 2007). Using the residual gum rate as an evaluation indicator, the optimal solution for the orthogonal test was judged to be A3B3C2.

Table 4. Range Analysis Table of the Orthogonal Test Data for the Alkali-Hydrogen Peroxide Degumming of Hemp Fibers (Residual Gum Content)

Mark	Level Value	A	B	C
Residual Gum Content	K_1	38.250	32.001	30.039
	K_2	29.121	29.880	30.000
	K_3	23.160	28.350	30.189
	R	5.030	1.217	0.470

Note: K1, K2 and K3 denoted the cumulative sums of the residual gum content for the corresponding factors (A, B, and C) across different levels, respectively; R was defined as the difference between the maximum and minimum of the K values at different levels of each factor.

The results of the ANOVA were calculated and analyzed using IBM SPSS Statistics 27.0 software. As could be seen from the analysis of Table 5, the effects of three factors, namely temperature, time, and *m*:*v* ratio, on the residual glue rate all reached a significant level ($\alpha = 0.05$). Among them, temperature had the most significant impact ($P = 0.001$), with the sum of squares between groups being 38.37, much larger than the sum of squares within groups of 4.19. This indicated that temperature change was the main factor leading to differences in the residual gum content, which might be related to the fact that increased temperature accelerated the reactions of alkali and hydrogen peroxide, thereby promoting the removal of gums. Time also had a relatively significant impact on the residual gum content ($P = 0.013$), reflecting that the length of degumming time directly affected the

sufficiency of gum removal. Additionally, the *m*:*v* ratio had a significant impact on the residual gum content (P = 0.023), suggesting that the ratio of reagents to fibers in the reaction system affected the efficiency of gum dissolution and detachment.

Table 5. Results of One-Way ANOVA for Residual Gum Content

Factors	Category	Sum of squared	Degree of freedom	Mean square	F value	Significance (P)
Temperature	Between groups	38.37	2	19.18	27.4	0.001
	Within groups	4.19	6	0.698	—	—
	Total	42.56	8	—	—	—
Time	Between groups	12.38	2	6.19	8.84	0.013
	Within groups	4.2	6	0.7	—	—
	Total	16.58	8	—	—	—
m:v	Between groups	10.54	2	5.27	6.98	0.023
	Within groups	4.52	6	0.75	—	—
	Total	15.06	8	—	—	—

As shown in Table 6, range analysis revealed that the influence of the three factors on tensile strength was in the following order: A (temperature) > B (time) > C (concentration). Temperature exerted the most significant effect on tensile strength, followed by time, while concentration had the least impact. Effective removal of gums facilitated the formation of more hydrogen bonds and physical entanglements between fibers. When fibers remained undamaged, more intact original fibers of high tenacity are preserved, which was the potential reason for the paper to exhibit relatively high tensile strength. While increasing temperature accelerated the degumming rate, prolonged exposure to high temperatures tended to induce excessive degumming. This reduced the number of hydrogen bonding sites on the fiber surface, ultimately leading to a decrease in the tensile strength of the paper. Using the tensile strength as an evaluation indicator, the optimal solution for the orthogonal test was A3B2C1.

Table 6. Range Analysis Table of the Orthogonal Test Data for the Alkali-Hydrogen Peroxide Degumming of Hemp Fibers (Tensile Strength)

Mark	Level Value	A	B	C
Tensile strength	K_1	0.489	0.519	0.660
	K_2	0.489	0.690	0.489
	K_3	0.720	0.489	0.549
	R	0.077	0.067	0.057

The results of the ANOVA were calculated and analyzed using IBM SPSS Statistics 27.0 software. As revealed by the analysis of Table 7, with regard to tensile strength, the effects of temperature and time were significant, whereas the influence of the m:v was not significant ($\alpha = 0.05$). Temperature exerted an extremely significant effect on tensile strength (P = 0.003), with the sum of squares between groups being 19266.67, much larger than the sum of squares within groups of 3333.33, indicating that temperature changes

significantly affected the mechanical structure of the fibers. Time also had a significant impact on tensile strength (P = 0.018), which suggested that either excessively long or short degumming time might affect the integrity of the fibers. In contrast, the effect of the *m*:*v* ratio on tensile strength was not significant (P = 0.068), indicating that within the range of liquid-to-material ratios employed in this experiment, its influence on the tensile properties of the fibers was limited.

Table 7. Results of One-Way ANOVA for Tensile Strength

Factors	Category	Sum of squared	Degree of freedom	Mean square	F value	Significance (P)
Temperature	Between groups	19266.67	2	9633.33	17.43	0.003
	Within groups	3333.33	6	555.56	—	—
	Total	22600	8	—	—	—
Time	Between groups	13866.67	2	6933.33	7.67	0.018
	Within groups	5400	6	900	—	—
	Total	19266.67	8	—	—	—
m:v	Between groups	7466.67	2	3733.33	4.13	0.068
	Within groups	5400	6	900	—	—
	Total	12866.67	8	—	—	—

As shown in Table 8, range analysis revealed that the influence of the three factors on whiteness followed the order C (concentration) > A (temperature) > B (time). Concentration exerted the most significant effect on whiteness, followed by temperature, while time had the least impact. In terms of whiteness index, compared with the untreated hemp fibers, the alkali-hydrogen peroxide-treated fibers showed higher whiteness, indicating that the combined bleaching of sodium hydroxide and hydrogen peroxide endowed the hemp fibers with lightness. Lignin was related to the dark color of hemp fibers, and its removal improved the whiteness, brightness, and gloss of the fibers (Pandey *et al*. 2019; Cao *et al*. 2024). Moreover, this process did not generate toxic substances (Treimanis 2009). Using the whiteness as an evaluation indicator, the optimal solution for the orthogonal test was found to be A3B3C1.

Table 8. Range Analysis Table of the Orthogonal Test Data for the Alkali-Hydrogen Peroxide Degumming of Hemp Fibers (Whiteness)

Mark	Level Value	A	B	C
Whiteness	K_1	200.799	199.500	211.800
	K_2	193.701	198.900	202.599
	K_3	210.900	207.000	191.001
	R	5.733	2.7	6.933

The results of the ANOVA were calculated and analyzed using IBM SPSS Statistics 27.0 software. As indicated by the analysis of Table 9, the effects of temperature, time, and m:v on whiteness were all non-significant ($\alpha = 0.05$), with their respective p-values (0.135, 0.347, and 0.295) all exceeding 0.05. Furthermore, the sum of squares between groups for each of the three factors was either smaller than or close to the sum of squares within groups. This suggested that under the experimental conditions employed, whiteness was less affected by fluctuations in these three factors.

Table 9. Results of One-Way ANOVA for Whiteness

Factors	Category	Sum of squared	Degree of freedom	Mean square	F value	Significance (P)
Temperature	Between groups	107.15	2	53.58	2.64	0.135
	Within groups	121.77	6	20.29	—	—
	Total	228.92	8	—	—	—
Time	Between groups	61.77	2	30.88	1.23	0.347
	Within groups	150.33	6	25.05	—	—
	Total	212.1	8	—	—	—
m:v	Between groups	58.17	2	29.08	1.45	0.295
	Within groups	120.33	6	20.05	—	—
	Total	178.5	8	—	—	—

The optimal process parameters were determined by comprehensively evaluating three different indicators, yielding the configuration of A3B3C1: temperature of 80 °C, time of 4 h, and solid-to-liquid ratio of 1:10. Experiments on the optimal process showed that the residual gum content was 7.68%, the tensile strength was 190 N/m, and the whiteness was 70.3%. Based on the above analysis, the A3B3C1 experimental group was identified as the optimal condition. Consequently, all subsequent characterizations of alkali-hydrogen peroxide degumming were performed using samples from A3B3C1 group.

Effect of Alkali-Hydrogen Peroxide One-Step Degumming Process on the Compositional Characteristics of Hemp Fibers

Compositional analysis results (Fig. 3) revealed that the untreated hemp fibers contained 64.2% cellulose, which was notably lower than the cellulose content of cotton fibers (92 to 95%).

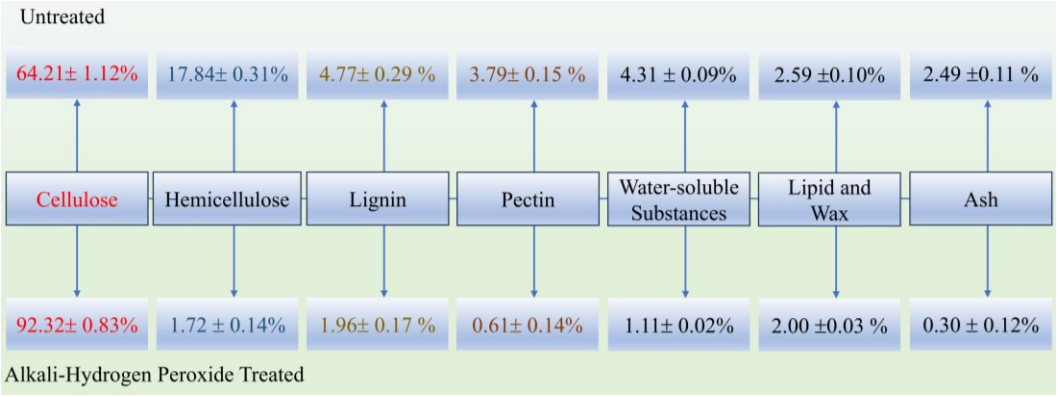

Fig. 3. Content of components in hemp fibers before and after treatment

While the gum components initially provided structural rigidity to the fibers, they substantially compromised the flexibility and processability of the material. Remarkable changes were observed after the one-step alkali-hydrogen peroxide treatment. The gum components, including hemicellulose, pectin, and lignin, were significantly reduced to 1.72%, 0.61%, and 1.96%, respectively, corresponding to removal efficiencies of 90.4%, 83.9%, and 58.9%.

Concurrently, the cellulose content increased to 92.3%, representing a 28.1% enhancement compared to untreated fibers. The effective removal of these gum substances substantially improved the processability of hemp fibers, thereby enhancing their potential for industrial applications (Jahan *et al*. 2016; Ma *et al*. 2023)

Impact of Alkali-Hydrogen Peroxide One-Step Degumming on the Chemical Structure and Crystalline Morphology of Hemp Fibers

The X-ray diffraction (XRD) patterns of hemp fibers before and after degumming are shown in Fig. 4(a), exhibiting distinct diffraction peaks within the 2θ range of 10° to 50°. All samples displayed a dominant peak at 22.6°, corresponding to the (200) crystallographic plane (I_{200}) of cellulose Iβ, along with a characteristic peak at 15.3° attributed to the (101) plane (I_{101}) of cellulose I, confirming the typical cellulose I crystalline structure (Vilaro *et al*. 2024). These results indicate that the alkali-hydrogen peroxide one-step treatment did not alter the crystalline form of cellulose, suggesting its structural stability under mild processing conditions.

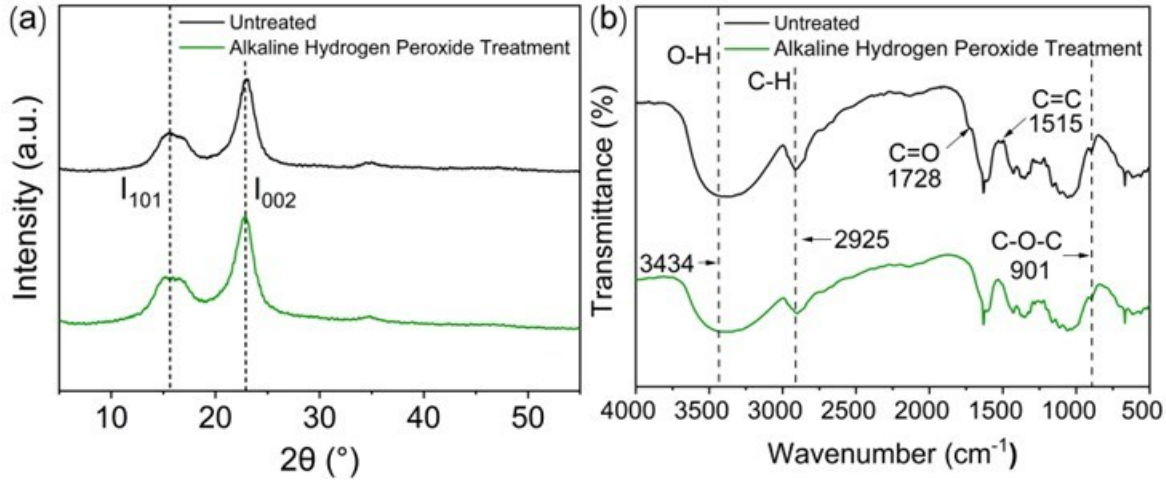

Fig. 4. (a) XRD and (b) FTIR of untreated hemp fibers and alkali-hydrogen peroxide treated hemp fibers

Quantitative analysis of XRD data revealed that the crystallinity index of hemp fibers increased from 74.6% to 82.9% after the alkali-hydrogen peroxide treatment (Pratiwi *et al*. 2024). This enhancement can be attributed to the effective removal of gummy components (*e.g.*, pectins and hemicellulose), which promotes the separation of fiber bundles into individual fibers. Concurrently, the relative cellulose content increased, while the amorphous components (*e.g.*, hemicellulose and lignin) decreased, leading to the observed rise in crystallinity (Fan *et al*. 2021). This trend aligns well with the compositional analysis, further supporting the dual role of alkali-hydrogen peroxide treatment in achieving efficient degumming and chlorine-free bleaching.

The FTIR spectra of hemp fibers in the wavenumber range of 500 to 4000 cm^{-1} are shown in Fig. 4(b). The untreated sample exhibited characteristic absorption peaks at 3434 cm^{-1} (O-H stretching vibration), 2925 cm^{-1} (C-H stretching vibration), 1728 cm^{-1} (C=O stretching vibration), 1515 cm^{-1} (aromatic C=C vibration), and 901 cm^{-1} (glycosidic bond C-O-C vibration). Significant spectral changes were observed after the alkali-hydrogen peroxide one-step treatment (Zhao *et al*. 2024) : (1) The decreased intensity of the hydroxyl peak at 3434 cm^{-1} indicated the reduction of surface free hydroxyl groups; (2) The complete

disappearance of the carbonyl peak at 1728 cm^{-1} confirmed effective removal of hemicellulose and pectin components (Zhao *et al*. 2024); (3) The remarkable attenuation of the lignin characteristic peak at 1515 cm^{-1} demonstrated the breakdown of aromatic structures (Nie *et al*. 2020). Notably, the cellulose-related peaks at 901 and 3434 cm^{-1} remained stable (Zhao *et al*. 2023), confirming the preservation of cellulose's fundamental chemical structure (Huang *et al*. 2024). These spectroscopic changes were well correlated with chemical composition analysis, demonstrating that the alkali-hydrogen peroxide process could selectively remove non-cellulosic components (hemicellulose, lignin, pectin, *etc*.) while maintaining cellulose integrity.

Effect of One-Step Alkali-Hydrogen Peroxide Degumming on Hemp Fiber Morphology

Fiber dimension analysis of hemp fibers before and after alkali-hydrogen peroxide one-step treatment was characterized using a fiber analyzer, with experimental results shown in Fig. 5.

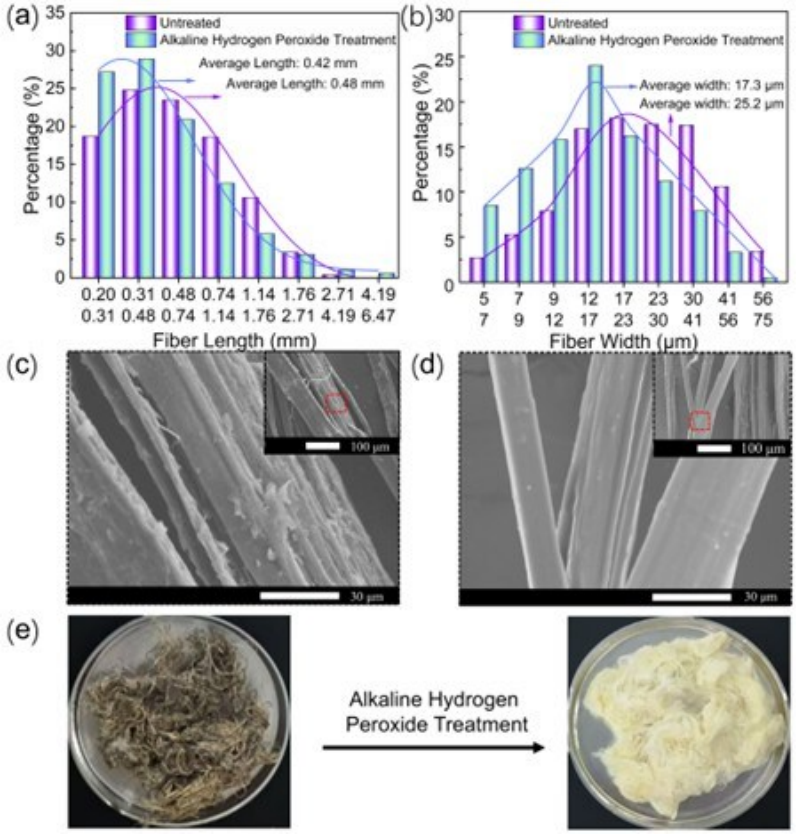

Fig. 5. a) Length of untreated and alkaline-hydrogen peroxide treated hemp fibers; b) width of untreated and alkaline-hydrogen peroxide treated hemp fibers; c) SEM Images of untreated hemp fibers; d) SEM Images of alkali-hydrogen peroxide hemp fibers; e) photographs of untreated and alkaline-hydrogen peroxide treated hemp fibers

Regarding length distribution (Fig. 5a), the treated fibers exhibited a pronounced shortening trend: the proportion of fibers in the 0.20 to 0.74 mm range increased by 9.5 to 12.1%, while the fraction of longer fibers (1.14 to 6.47 mm) decreased by 14.3 to 22.8% compared to the raw material. This bimodal shift can be attributed to two mechanisms: (1)

Selective erosion of fiber ends by the degumming solution, leading to fiber shortening (Zhu *et al*. 2013); and (2) Disintegration of the fiber-bonding network due to gum dissolution, promoting the separation of fiber bundles into individual fibers (Chen *et al*. 2022). Notably, this altered length distribution directly impacted paper properties: while increased short fibers enhance formation uniformity, they may compromise mechanical strength. In terms of diameter distribution (Fig. 5b), the treated fibers showed a significant refinement: the proportion of fibers in the 5 to 17 μm range surged from 32.9% to 60.9%, while those in the 17 to 75 μm range decreased correspondingly. This phenomenon primarily results from: (1) liberation of individual fibers from gum-bonded bundles upon gum removal (Beltran *et al*. 2002); and (2) surface etching during alkali-hydrogen peroxide treatment, further weakening interfiber bonding (Huang *et al*. 2022). Such diameter refinement substantially increases the specific surface area of fibers, facilitating fiber entanglement and hydrogen bond formation in subsequent papermaking processes.

To investigate the effect of one-step alkali-hydrogen peroxide treatment on the surface morphology of hemp fibers, scanning electron microscopy (SEM) was employed to characterize the fibers before and after degumming (Fig. 5c-d). Untreated hemp fibers (Fig. 5c) exhibited a rough and hardened surface due to the deposition of gummy substances such as hemicellulose, pectin, lignin, and wax, which caused severe fiber aggregation and made individual fiber separation impossible (Ahirwar *et al*. 2019). Although these interconnected gums provided protective benefits against environmental damage, enhanced inter-fiber bonding, and improved compressive strength, their excessive presence negatively impacted thermal stability, air permeability, and subsequent dyeing/processing performance. After alkali-hydrogen peroxide treatment (Fig. 5d), the fiber bundles were thoroughly separated with minimal residual gums on the surface, exposing more cellulose structures. Compared to raw fibers, the treated fibers displayed brighter coloration, softer texture, and distinct inter-fiber separation (Fig. 5e). These findings confirm that degumming effectively removed most non-cellulosic gums, thereby optimizing the mechanical properties of hemp fibers for pulping processes and significantly improving their spinnability for textile applications.

Thermal Stability Analysis of Hemp Fibers

A comparative analysis of the TG-DTG curves (Fig. 6) between raw hemp fibers and those treated by the one-step alkali-hydrogen peroxide method reveals distinct thermal degradation behaviors. In the initial stage (50 to 150 °C), both samples exhibited a minor mass loss (~5%), which can be attributed to the evaporation of adsorbed water, low-molecular-weight volatiles, and bound water. As the temperature increased to 250 to 350 °C, a pronounced mass loss occurred, corresponding to the thermal decomposition of amorphous components (*e.g.*, pectin and hemicellulose) within the fibers. A secondary degradation peak emerged in the range of 350 to 400 °C, reflecting the breakdown of cellulose backbones. Notably, the untreated sample displayed an additional mass loss near 360 °C, resulting from the progressive pyrolysis of lignin's complex aromatic structures. The DTG curves (Fig. 6b) provided further insights into the thermal stability of cellulose. Both untreated and degummed fibers exhibited a sharp DTG peak within 300 to 400 °C, associated with the rapid thermal degradation of cellulose (Ahmed *et al*. 2022). Importantly, the alkali-hydrogen peroxide treated sample showed a slight shift (~5 to 10°C) of the cellulose decomposition peak toward higher temperatures, indicating enhanced thermal stability. This phenomenon can be ascribed to the selective removal of amorphous regions (*e.g.*, pectin, hemicellulose, and partial lignin) during alkali-hydrogen peroxide

treatment, which increased the crystallinity of cellulose (Liu *et al*. 2018). The higher crystallinity promotes tighter molecular packing and a stronger hydrogen-bond network, thereby delaying the thermal degradation process.

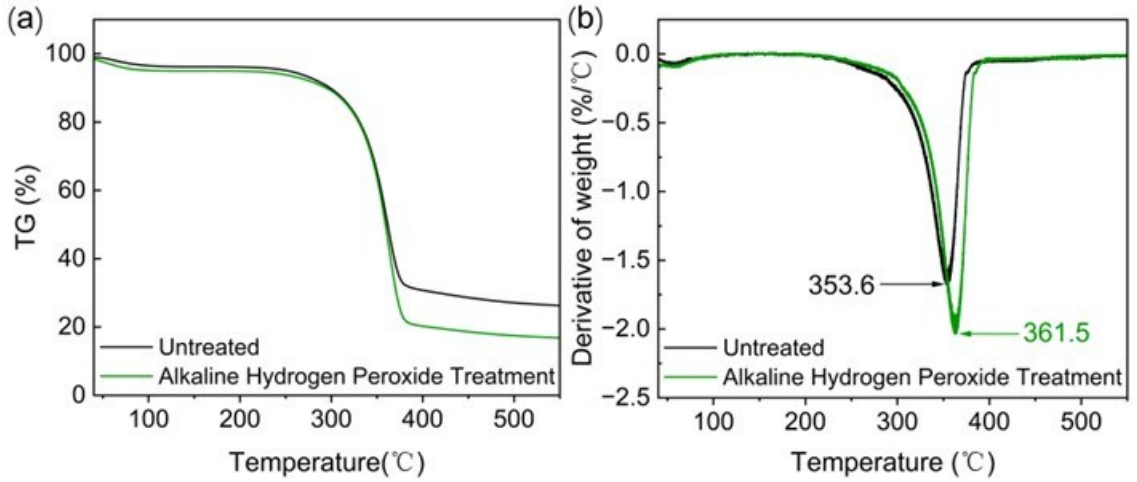

Fig. 6. Thermogravimetric analysis of hemp fiber before and after treatment. (a) TG and (b) DTG

Although this study effectively demonstrated the improvement of fiber properties by degumming treatment through papermaking technology, such as paper strength and whiteness under different treatment conditions, it must be recognized that there are substantial differences between papermaking technology and textile technology in many key aspects. In the papermaking process, the residual gum components would affect the performance of paper. For example, papers with high residual lignin not only exhibit low initial whiteness due to the aromatic structure inherent in lignin itself, but also the aldehydes, carboxylic acids, and other products generated during its oxidation process can accelerate fiber aging through mechanisms such as damaging the cellulose molecular structure and weakening interfiber bonding (Małachowska *et al*. 2021). In the textile field, the structural stability of textiles depend on the twisting and binding of yarns and the design of weaving structure, and fibers need to maintain the integrity of the aggregate under axial stress. Since yarns have to withstand large tension and friction, in order to maintain the strength, wear resistance and other properties of fibers, it is necessary to determine the optimal degumming threshold during hemp fiber degumming to avoid excessive degumming leading to a decrease in fiber cohesion and an increase in spinning breakage rate. Insufficient degumming would cause an increase in yarn hairiness due to residual gum. Therefore, indicators such as fiber gum content, surface friction characteristics and elongation at break were particularly important.

CONCLUSIONS

1. The one-step alkali-hydrogen peroxide degumming process at relatively low temperature, achieved through the combined action of sodium hydroxide and hydrogen peroxide, was found to effectively remove non-cellulose components from hemp fibers. The treated fibers exhibited favorable characteristics, including a higher proportion of fine fiber widths and shorter fiber lengths. This not only optimizes the fiber structure but also contributes to better processing performance in subsequent applications. Additionally, the crystallinity increased from 74.6% to 82.9% after the alkali-hydrogen peroxide treatment and the enhanced thermal stability indicates the improvement in fiber quality, which is crucial for its use in various industries.
2. Chemical and morphological analyses confirmed that the degummed hemp fibers had a clean and impurity-free surface, with lignin, hemicellulose, and other non-cellulose components effectively removed. This impurity removal not only improved the appearance of the fibers but also had a positive impact on their physical and chemical properties. The degummed fibers displayed a residual gum content of 7.68%, and the paper produced therefrom exhibited a whiteness of 70%. The paper further exhibited a tensile index of 190 N/m. Such properties endow degummed hemp with high potential in the papermaking applications.

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