Effect of a promising CSESE pretreatment on the morphological structure and properties of jute fibers

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Abstract. Plant fiber has a complex aggregated structure, high crystallinity and a large number of hydrogen bonds, and has low chemical reaction activity. Therefore, in order to improve the reactivity, the plant fiber needs to be pretreated before chemical modification. Therefore, the pretreatment method has an important influence on the chemical modification of plant fibers. In this paper, Jute fibers were successively treated by a particular continuous screw-extrusion steam explosion (CSESE). The effects on the morphological structure, composition, thermal and crystalline properties of jute fibers before and after treated were investigated. Scanning electron microscopy (SEM) showed that the morphological structure changed significantly after CSESE pretreatment, the diameter was smaller, the surface cracks and the specific surface area increased. There was no new functional groups appeared in fourier transform infrared spectroscopy (FTIR). Composition analysis demonstrated that the content of hemicellulose and extractive decreased, but cellulose and lignin increased. The crystallinity of JSE increased from 66.31 % to 70.19 %. X-ray photoelectron spectroscopy (XPS) suggested that the cellulose and lignin content increased. Thermogravimetric analysis (TGA) demonstrated that the initial decomposition temperature of JSE increased from 334.4 to 355.2 °C. DMA indicated that the Tg of JSE reduced from 92.6 to 90.0 °C. Therefore, for jute fiber, the CSESE pretreatment is an effective and promising pretreatment method for further chemical modification, biofuels and chemical products.

1 Introduction

The growing demand for energy and decreasing petroleum-based transportation fuels have stimulated tremendous interest in finding alternative renewable energy sources [1]. Recently, the utilization of plant fibers is increasing due to its natural, renewable, low cost, environmentally friendly [2]. For example, plant fibers can be used to prepare biogas, biofuels and other chemicals through fermentation, chemical modification and other methods. Nevertheless, the main components of plant fiber include cellulose, hemicellulose and lignin [3], because three major components of the fibers structure was denser and complicated [4-6], it was

difficult to carry out fermentation [7], dissolution and processing [8], especially chemical modification [9,10]. Appropriate pretreatment of plant fibers can remarkably improve utilization efficiency of plant fibers for the production of chemicals, biofuels and degradable biomaterials. The main pretreatment procedure include biological methods; physical methods such as milling and chemical methods such as acid pretreatment, alkaline pretreatment. Among these, steam explosion (SE) pretreatment is a sustainable method with advantages of cost-effective, energy-efficient and environmentally friendly [11,12]. The SE is a kind of physical and chemical method, rapid development and low cost plant fibers separation technology in recent years, which can effectively realize the separation of

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chemical composition of plant fibers, and no chemicals, no environmental pollution, low energy consumption, low cost [13-15]. In the process of SE, high pressure steam penetrated into the fibers, and rapidly released it from the closed pore in the way of air flow, which caused the fibers to be broken. At the same time, when the pressure was released, the explosion effect was produced, the fibers structure became loose, and cellulose was partial stripped from lignin and hemicellulose. In addition, high temperature and pressure aggravated the destruction of hydrogen bonds, freed the new hydroxyl groups, so that the ordered structure of cellulose was changed [16,17]. After steam explosion, partly hemicellulose was hydrolyzed to low molecular substances, some lignin was dissolved, lignin and cellulose were separated, the fibers was loose and porous, and the accessibility can be increased [15]. Therefore, SE is an effective pretreatment for disrupting the structure of biomass materials and increasing their chemical reactivity for further modification and conversion.

Continuous screw-extrusion steam explosion (CSESE) pretreatment have been developed for the continuous pretreatment of various biomass materials [18]. CSESE is high-throughput pretreatment methods, suitable for large scale processing and desirable for the industrial production of value-added materials from lignocellulosic biomass. Moreover, in our group, the eucalyptus fibers [18] and eulaliopsis binata [19] were treated with CSESE, and the effect of CSESE pretreatment on the morphological structure and properties were studied. It was found that CSESE pretreatment is a continuous high-efficiency pretreatment that dramatically decreases the fiber size and increases specific surface area. It could therefore be used to improve accessibility for further chemical modification and chemical products.

In this work, in order to discuss effect of CSESE pretreatment on the morphological structure and properties of jute fibers, the laboratory-designed CSESE device was used to treat jute fibers. Then, the jute fibers before and after treated (JSE) were characterized by SEM, FTIR, component analysis, XPS, XRD, TGA and DMA. These results were used to evaluate the effectiveness of CSESE pretreatment for further chemical modification, biofuels and chemical products.

2 EXPERIMENTAL

2.1 Materials and preparation of JSE

Jute fibers were used in this paper was obtained from Nanjing Xinhe Textile Co., Ltd. A moisture analyzer (MB25, Ohaus Instruments Co., Ltd., Shanghai, China) was used to measure the moisture content of materials. The moisture of jute fibers was about 12 wt.%. Jute fibers were cutted into the length of approximately 10 mm. Then, the moisture of jute fibers is adjusted to about 50 wt.% by addition of tap water, and the wet jute fibers was placed in a sealed plastic bag for 24h at room temperature and follow treated by continuous screw-

extrusion steam explosion (CSESE) [3, 18] with our laboratory-designed steam explosion equipment. Briefly, the screw continuously conveyed the jute fiber chips with about 50% moisture content forward, where they were squashed and compacted by the screw, and gradually heated due to friction between the chips, screw, and barrel. The pressure and temperature of the compacted jute fiber reached approximately 1–1.5 MPa and 120–150°C, respectively, the screw speed was about 300 rpm, when they conveyed to the die. The compacted jute fiber was continuously discharged from the die, with a slit width of 1 mm. Pressurized water in the fiber bundles was instantaneously vaporized, which resulted in destruction of the tissue structure of the fiber bundles. This pretreatment process was repeated three times to obtain pretreated jute fiber, and the output reached 100–150 kg/h. Then the pretreated jute fiber was washed with tap water and dry naturally, CSESE pretreated jute fiber is hereafter referred to as JSE.

2.2 Methods

2.2.1 Chemical composition analysis

The chemical composition of jute fibers and JSE were analyzed using standard National Renewable Energy Laboratory method [20,21]. Samples were dried at 105 °C, weighed to obtain dried weight, and then extracted with benzene and ethanol (2:1) prior to acid hydrolysis. The standard method uses a two-step acid hydrolysis. Then, high performance liquid chromatography (Prominence LC-20 A, Shimadzu, Japan) was used to determine the cellulose and hemicellulose contents in the hydrolysis liquid. Next, the lignin was fractionated into acid-insoluble and acid-soluble materials. The results were calculated on an dry weight basis.

2.2.2 Fourier transform infrared spectrometer (FTIR)

Jute fibers and JSE were dried in electro thermal oven at 60 °C. Subsequently, jute fibers and JSE were cut into smashed particles, mixed with dried potassium bromide solid in agate mortar and were ground into powder, respectively. The powder was pressed into slices then dried in infrared box. Finally, the dry slices of jute fibers and JSE were used to test the infrared spectrum (Nicolet-Nexus 670 infrared spectrophotometer).

2.2.3 Scanning electron microscopy (SEM)

Jute fibers and JSE were dried in electro thermal oven at 60 °C. Then, samples were coated with a thin layer of gold using a vacuum coater before analysis. Images of the surface morphology of jute fibers and JSE were obtained using a Quanta FEG 250 field emission scanning electron microscope (FEI, Hillsboro, Oregon, USA) at room temperature operated in secondary electron mode with a beam current of 100 mA and accelerating voltage of 5 KV.

2.2.4 X-ray diffraction (XRD)

X-ray diffractograms of jute fibers and JSE were obtained with a Bruker D8 ADVANCE (Bruker, Germany) diffractometer (40 kV, 40 mA) by the refraction method using nickel-filtered Cu K α radiation ($\lambda = 1.54 \text{ \AA}$). Scans were performed from 4 to 60° with increments of 0.04° and 0.2 s per step. Prior to testing, samples were ground to powder in agate mortar. Then, the powder samples were dried and pressed into a sheet with a diameter of approximately 10 mm and a thickness of approximately 2 mm. The collected data were analyzed using MDI Jade (Materials Data Incorporated, US). The crystallization peaks and amorphous peaks were obtained by means of XRD-peak-different-imitating analysis with parameters for all samples, and the fitness error was about 3%.

2.2.5 Dynamic thermomechanical analysis (DMA)

The samples were molded into a disk having a diameter of approximately 15 mm and a height of 2 - 3 mm in a self-made mould. Dynamic thermomechanical analysis were carried out on a Dynamic thermomechanical analyzer (Germany, NETZSCH, DMA242C). Test Conditions: Compression mode; -80 - 180 °C; 3 °C/ min; 10 Hz; amplitude: 10 μm .

2.2.6 Thermogravimetric analysis (TGA)

The thermal stability of jute fibers and JSE were investigated using a thermogravimetric analyzer (TG209 F3, Netzsch, Germany). Approximately 5 mg of sample was heated from room temperature to 700 °C at a rate of 10 °C/ min. Nitrogen gas at a flow rate of 50 ml/ min was used to protect samples from oxidation. Prior to thermal analysis, the samples were dried at 60 °C.

2.2.7 X-ray photoelectron spectroscopy (XPS)

Surface composition analysis of dried jute fibers and JSE was performed with an XPS instrument (AXIS Ultra DLD, Shimadzu Corporation, Japan) equipped with a monochromatic Al K α X-ray source (15 kV, 5 mA) operated at 150 W and with an electron flood gun for charge compensation. Because the objective was to study the influence of JSE on the composition of fibers, samples were not extracted before analysis. Measurement were taken at two different locations on the surface of each samples, and the analyzed area of each location was 300 $\mu\text{m} \times 700 \mu\text{m}$. Low-resolution spectra were taken with a 1-eV step and 160-eV pass energy, while high-resolution spectra were taken with a 0.1-eV step and 40-eV pass energy. The oxygen to carbon atomic ratio (O/C) was determined from low-resolution spectra, and the relative amounts of differently bound carbons were determined from high-resolution C1s spectra. The relative sensitivity factors of carbon and oxygen are 0.278 and 0.780 respectively. The collected data were analyzed using CASA XPS version 2.3 (Casa Software Ltd., UK).

3 RESULTS AND DISCUSSION

3.1 The morphological structure of jute fibers and JSE

The surface morphology of jute fibers and JSE are showed in Fig.1. The morphological structures of Fig.1a. and Fig.1b. are significantly different. As observed in Fig.1, the untreated jute fibers take on fibrous structure and JSE show a rough, loose, and broken surface structure. This difference may be due to CSESE pretreatment. This is because that in CSESE process, the wet jute fibers in high temperature and pressure, steam penetrated into the fibers material, fibers pore and cell

wall, formed hydrogen bonds with some hydroxyl groups of fibers [22]. Under the combined action of water vapor and heat, the fibers began to soften and degrade, low molecular weight substance began to dissolve, and the connection between fibers began to weaken. Hereafter, high pressure steam rapidly released from the fibers gap, softened fibers to produce mechanical separation, faulting, breaking down cellulose intramolecular hydrogen bonds, greatly reduce the connection in the fibers and the fibers are separated from each other [23]. Principle of pretreatment of plant fibers by SE was formed with high temperature and pressure water vapor environment of the blasting chamber, softened plant fibers, and through a sudden pressure relief, resulted in "blasting", so that fibers component separated and structural dissociated. It was generally believed that the process of SE has the following aspects [24]: acidic hydrolysis and thermal degradation, hydrogen bonds destruction, structural rearrangement and mechanical fracture.

Fig.1 .The photos of jute fibers (a) and JSE (b).

SEM was used to investigate the samples to determine the effect of CSESE on the microstructure of materials. SEM images of jute fibers and JSE are shown in Fig.2. The results of SEM indicated that the diameter of untreated jute fibers (Fig.2 a, b, c) (70-100 μm) were larger than JSE (20 -30 μm) (Fig. 2 d, e, f), the surface of JSE was rough, and had impurity particles. Moreover, there were some protruding structures which may be produced by the cell cavity in Fig.2c. After CSESE treatment (2 d, e, f), the surface impurities had been removed, the fibers morphology changed obviously, the diameter were smaller, cracks increased, and what's more, some plant fibers were destroyed and produced some debris, so the specific surface area increased. This indicated that CSESE pretreatment can not only removes impurities on fibers surface, but also makes fibers bundle

binding force weakened, cellulose, hemicellulose and lignin are separated, fibers structure obvious changed [14]. So it can greatly improve the accessibility of the chemical reagents, and it is beneficial to further chemical modification and chemicals.

Fig. 2 SEM images of jute fibers (a, b, c) and JSE(d, e, f).

3.2 Chemical composition of jute fibers and JSE

The main components of jute fibers and JSE are shown in Table1. The content of lignin are the sum of acid-insoluble and acid soluble lignin. After CSESE pretreatment, lignin and cellulose content of JSE

Table.1 The main chemical composition of jute fibers and JSE.

sampl es	Cellulose %	Hemicellulos e%	Lignin %	extracti ve%
Jute fibers	52.35	13.20	11.45	2.20
JSE	53.72	11.35	13.15	1.58

increased from 11.45 to 13.15 % and from 52.35 to 53.72%, respectively, and hemicellulose content decreased from 13.20 to 11.35 %. Compared to jute fibers, cellulose and lignin content of JSE increased about 3% while the hemicellulose content decreased approximately 2%. A series of hydro-thermal reactions can be triggered in high-temperature and acidic environment where the hemicellulose can be hydrolyzed into monomeric and oligomeric sugars while partial soluble sugars will subsequently be degraded into small molecular products by the catalysis of acidic water, acetic and other acids derived from acetyl groups [7,24]. Therefore, the loss of hemicellulose was attribute to the degraded hemicellulose to small molecule in CSESE process, subsequent, small molecule were partly removed by washing with tap water, so hemicellulose content of JSE decreased. SE was usually carried out at relatively high temperature (140-220 °C). The elevated temperature decreased the water pK_w and the releases of organic acids from biomass components created a mild

acidic condition. Although lignin degraded into lignin fragments during CSESE process, repolymerization also occurred simultaneously [25, 26] and produced new products. In addition, extractive content decreased, so relative content of lignin increased. Meanwhile, cellulose content of JSE was increased. This is because, in CSESE process, almost no degradation of cellulose, so cellulose content did not change, but hemicellulose and extractive content decreased, therefore, the relative content of cellulose was increased.

3.3 Fourier transform infrared spectroscopy (FTIR) of jute fibers and JSE

FTIR spectra of plant fibers was very complex and generally used for simple qualitative evaluation. According to some literatures [27-30], the broad peak at approximately 3400 and 2920 cm⁻¹ were attributed to hydroxyl stretching vibration and C-H stretching of methyl and methylene groups, respectively. The peak at 1736 cm⁻¹ was the characteristic peak of hemicellulose, the characteristic peak of lignin was the absorption of aromatic ring at approximately 1510 and 1600 cm⁻¹, the peak at 1426 cm⁻¹ was originated from the C-H stretching of cellulose and lignin, the peak at 1375 cm⁻¹ was originated from the C-H bending vibration of cellulose and hemicellulose, the peak at 1160 and 1060 cm⁻¹ was the C-H stretching vibration of cellulose and hemicellulose, the characteristic peak of β-D-glucoside was 897 cm⁻¹. FTIR spectra of jute fibers and JSE were shown in Fig.3. As can be seen from Fig.3, jute fibers and JSE had also many peaks of 3425, 2924, 1739, 1600, 1505, 1378, 1060 and 897 cm⁻¹. This is similar to the above-mentioned literatures. Compared with jute fibers, there was no new functional groups of JSE appeared, so the chemical structure of the samples was not significantly changed. Then, the peak area at 1739, 1505 and 897 cm⁻¹ values were normalized by the peak area of the C-O-C ether stretching of the anhydroglucose unit at 1060 cm⁻¹ [31]. However, compared to jute fibers, the absorption peak at 1739 cm⁻¹ decreased, and the peaks of 897 and 1505 cm⁻¹ increased. And the peaks of 897, 1505 and 1739 cm⁻¹ were represent cellulose, lignin and hemicellulose [28], respectively. So, the results showed that the content of hemicellulose decreased, while the content of cellulose and lignin increased after CSESE pretreatment, which corresponded to the data on compositional analysis determination presented in Table1. The repolymerization of sugar degradation products and polymerization with lignin to form a lignin-like material termed pseudolignin or condensed lignin fragments might be the reasons for apparent increase in lignin content [32,33]. In addition, the results were consistent with the results of component analysis and SEM of jute fibers and JSE.

Fig.3 FTIR spectrum of jute fibers and JSE.

Fig.4 XPS spectra of jute fibers and JSE.

Table.2 XPS analysis of jute fibers and JSE.

Samples	C1s=100%				O/C
	C1(%)	C2(%)	C3(%)	C4(%)	
Jute	56.74	18.91	14.10	10.25	0.33
JSE	57.93	26.63	9.91	5.53	0.34

3.4 Surface composition of jute fibers and JSE

X-ray photoelectron spectroscopy (XPS) is a useful tool for analyzing chemical composition of fibre surface. The core of XPS method for fibre surface research lies in oxygen and carbon analyses. Typical XPS spectra of jute fibers and JSE are shown in Fig.4. Jute fibers and JSE had the similar curve shape and peak position. As expected, oxygen and carbon can be clearly identified and peak position at about O1s:532 eV, C1s:284.8 eV, respectively. The carbon of plant fibers including lignin, cellulose, hemicellulose and extractives, had four combinations for C1, C2, C3 and C4. The C1 (C-C, C-H), binding energy at 285 eV, derived from lignin and extractives on the surface of plant fibers. The C2 (-C-O), binding energy at 286.5 eV, existed in hydroxyl of cellulose and hemicellulose. The C3 (C=O/O-C-O), binding energy at 288.3 eV, was due to a product of the oxidation products of cellulose molecules or carbonyl, aldehyde in lignin molecules. The C4 (O-C=O), binding Energy at 289.5 eV, arised from carboxylic acids [28, 34, 35]. The C1 content in extractives is very high. The relative content of C1, C2 and C3 in lignin are 49 %, 49 % and 2%, respectively. The relative amount of C2 and C3 in cellulose are 83 % and 17 %, respectively [36]. The XPS high resolution C1s spectra of jute fibers and JSE are shown in Fig.5. The C1, C2, C3, C4 content and O/C atomic ratios were presented in Table 2. The C1 and C2 content of JSE were higher than that of jute fibers, which may be attributed to the high concentration of lignin and cellulose on the surface of sample. In addition, the C2 content of JSE was significantly higher than that of jute fibers. This may be due to that after CSESE pretreatment, more cellulose of JSE was exposed. The higher the ratio of O/C, the higher the cellulose content [37]. So, the content of lignin and cellulose can be indirectly reflected by the ratio of O/C. The O/C atomic ratio for JSE is 0.34, while jute fibers is 0.33, suggested that the JSE surface had more cellulose content than jute fibers. This change is also confirmed by the increasing in the amount of cellulose from 52.35 % to 53.72 % for JSE, as shown Table.1. Therefore, it can be speculated that after CSESE treatment, the structure of jute fibers was disrupted, cellulose exposed, the relative content of cellulose increased. This is consistent with the results of SEM, chemical composition analysis and FTIR.

Fig.5 The XPS spectra of C1s for the jute fibers (a) and JSE (b).

3.5 The crystallinity of jute fibers and JSE

The crystallinity of polymers is commonly known to significantly affect their mechanical properties and X-ray diffraction provides significant structural information for polymers and composites [38]. Therefore, it is important that the crystallinity of jute fibers and JSE be precisely determined. The crystal structure of natural cellulose is

mainly cellulose I, and its peak position is about at $2\theta = 16.6, 22.6$ and 34.6° [39]. The XRD curves of jute fibers and JSE are shown in Fig.6. It is evident from chart that there are three diffraction peaks. Jute fibers showed peaks at $16.1, 22.5$ and 34.8° , respectively. Similarly JSE showed peaks at $16.0, 22.5$ and 34.6° , respectively. So these peaks indicated that the specimens contained cellulose I. In addition, it can be seen from the figure that there was no new peaks appeared and old peaks disappeared of JSE, but the peak area and intensity changed slightly, the type of crystallization did not change. The crystallinity was calculated according to formula 1 [38-41]. Where I_{200} and I_{am} were the intensity of the peak at 2θ about 22.5 and 18.0° , respectively.

Fig.6. XRD curves of the jute fibers and JSE.

The percentage crystallinity of jute fibers and JSE comes out to be 66.31 % and 70.19 %.

$$CI = (I_{200} - I_{am})/I_{200} \times 100\% \quad (1)$$

It has been observed that there is a remarkable increasing in crystallinity of JSE. Crystallinity increased may be caused by two reasons, on the one hand, part of crystalline and amorphous cellulose molecular rearrangement in the chain, forming a new crystalline structure, so the crystallinity increased. on the other hand, the crystalline structure of cellulose was destroyed by acidic substance produced from the degradation of lignin, hemicellulose and mechanical destruction, therefore the crystallinity decreased [39]. While the former plays an important role, therefore, the overall result was crystallinity increased. Moreover, the result was consistent with the results of SEM, chemical composition analysis, FTIR and XPS.

3.6 The thermal stability of jute fibers and JSE

The TG and DTG analysis of jute fibers and JSE were obtained in order to evaluate their thermal stability and the results are shown in Fig.7. There was no significant difference between the TG and DTG curves of jute fibers and JSE, because the main components of jute fibers did not change before and after CSESE treatment. As can be seen from Fig.7, the thermal degradation of jute fibers and JSE can be divided into three parts: an evaporation stage, a thermal pyrolysis stage, and a thermal polycondensation stage [42]. An evaporation stage was from 30 to 130 $^\circ\text{C}$, there was little mass loss, which was caused by the evaporation of water. A thermal pyrolysis stage from 200 to 500 $^\circ\text{C}$, and was the main stage of

thermal degradation of jute fibers and JSE. The pyrolysis of hemicellulose, lignin and partial cellulose mainly occurred from 200 to 330 $^\circ\text{C}$ [43]. And the weight loss from 330 to 500 $^\circ\text{C}$ was due to bulk pyrolysis of cellulose and lignin. A thermal polycondensation

Fig.7. TG (a) and DTG (b) curves of jute fibers and JSE stage was from 500 to 700 $^\circ\text{C}$, which was caused by lignin containing aromatic ring structure, when the temperature rises to a certain extent, the aromatic ring structure will be the condensation reaction, and the weight loss rate is very small [3].

Initial decomposition temperature of jute fibers and JSE were 334.4 and 355.2 $^\circ\text{C}$, respectively. The results indicated that the thermal stability of JSE was higher than jute fibers. There may be two reasons: on the one hand, after CSESE pretreatment, the content of hemicellulose reduced, so the thermal stability of JSE increased. On the other hand, JSE had a higher crystallinity after CSESE pretreatment, which improved the thermal stability of JSE. In addition, we can also draw that the temperature of jute fibers and JSE at the maximum decomposition rate were 362.6 and 370.1 $^\circ\text{C}$, respectively. Compared to jute fibers, JSE was higher. It may be attributed to two aspects. First, the cellulose content of JSE was higher, and cellulose has higher pyrolysis temperature, so the temperature of the maximum decomposition rate of JSE was higher. Second, the crystallinity of JSE was higher, so the temperature of the maximum decomposition rate of JSE was higher. And, the result was consistent with the results of SEM, chemical composition analysis, FTIR, XPS and XRD.

3.7 DMA of jute fibers and JSE

The glass transition temperature (T_g) is one of the characteristic temperature of polymers. It is very important for process and application of materials. Therefore, it is generally known that the thermal transition behavior of the amorphous components in plant fibers is beneficial to the design and performance improvement of the materials [3]. It is usually considered that the glass transition behavior of plant fibers is caused by lignin. The DMA curves of jute fibers and JSE are shown in Fig.8. The T_g of jute fibers (92.6 °C) is higher than JSE (90.0 °C). Compared to jute fibers, the lower T_g of JSE may be due to three factors: 1) CSESE pretreatment disrupted the compacted structure of the fibers cells, and the resulting degradation of

Fig.8 DMA curves of the jute fibers and JSE.

and lignin reduced the number of chemical bonds between these components, so steric hindrance reduced. This increases the mobility of the molecular chain of lignin, thus the T_g appears at lower temperature [44]. 2) The T_g and molecular weight are closely related. Degraded lignin has a lower molecular weight and thus a lower T_g [3]. 3) Studies have shown that lignin degradation and repolymerization occur simultaneously during CSESE process [25]. Repolymerization of lignin fragments produces new low-molecular weight products with lower T_g , so JSE has lower T_g .

4 Conclusion

In the paper, jute fibers were successively pretreated by CSESE method. The jute fiber before and after pretreatment was characterized by chemical composition analysis, SEM, FTIR, XPS, XRD, TGA and DMA. SEM showed that the morphological structure of jute fiber has changed significantly; FTIR, XPS and chemical composition analysis showed that the content of cellulose and lignin increased, and the hemicellulose decreased; XRD showed that the crystallinity was significantly increased from 66.31% to 70.19%; TG showed that the thermal stability increased significantly from 334.3°C to 355.2°C; DMA indicated that the T_g decreased from 92.6 to 90.0°C. In summary, the pretreatment destroyed the structure of the jute fiber, increased the specific surface area, exposed the cellulose, and increased the chemical reaction activity. Therefore, the CSESE pretreatment method is feasible for further

chemical modification of jute fiber. In addition, the CSESE pretreatment will be widely used in the pretreatment of various biomass materials, which is conducive to the further chemical modification of biological materials, and the preparation of biofuels and chemical products.

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