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Cotton stalk bark fibers (CSBF) were extracted by steam flash explosion, completed within 0.09 s, and the extracted fibers were compared with those obtained by conventional alkaline treatment. Results indicate that the optimum steam pressure was 2.5 MPa when steaming time was set to 2 min for extracting CSBF. Under the optimized conditions, the obtained CSBF had a cellulose content of 72%, length of 48 mm, fineness of 45 dtex, crystallinity index of 68, moisture regain of 8%, water retention of 98%, and tensile strength of 2.4 cN/dtex, which were similar to results obtained by conventional alkaline treatment. Compared with bark of cotton stalks, CSBF had lower moisture regain and water retention, and higher onset decomposition temperature. The results show that moderate steam flash explosion is a chemical-free, quick, and effective method for exploring the industrial applications of bark of cotton stalks as natural cellulose fibers.

Keywords: Steam flash-explosion; Steam pressure; Bark of cotton stalks; Lignocellulose; Agricultural byproducts; Cotton stalk bark fibers

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INTRODUCTION

Rising environmental concerns and depletion of petrochemical resources has resulted in an increased interest in natural cellulose fibers from lignocellulosic agricultural byproducts (Pan *et al.* 2012, 2013; Smole *et al.* 2013; Thakur *et al.* 2014). Bark of cotton stalks is an abundant lignocellulosic byproduct from cotton production (Yan *et al.* 2013) and is composed of 41 wt% cellulose (Zhou *et al.* 2010, 2012). Natural cellulose fibers from bark of cotton stalks, *i.e.*, cotton stalk bark fibers (CSBF), have significantly better mechanical properties than those from other crop byproducts, such as rice straw and wheat straw (Reddy and Yang 2009; Wu *et al.* 2010). CSBF have mechanical properties between those of cotton and linen (Reddy and Yang 2009). It has been confirmed that CSBF can be used to reinforce composites such as polyester (PET) (Hassan and Nada 2003), polypropylene (PP) (Cao *et al.* 2011; Hou *et al.* 2014), polyethylene (Habibi *et al.* 2008; Qi *et al.* 2012), or poly(butylene succinate) (Tan *et al.* 2011; Qu *et al.* 2011). Bark of cotton stalks is a promising and beneficial renewable resource to produce cellulose fibers at a low cost, with desirable properties and biodegradability (Reddy and Yang 2009; Zhou *et al.* 2010). CSBF are considered a potential replacement for some synthetic fibers.

In the lignocellulosic cell wall, the microfibrils consisting of cellulose are primarily glued by lignin and hemicellulose. To extract the cellulose fiber strands from these cell walls, the lignin and hemicellulose binding them must be partly removed by retting. Several conventional techniques are used for extraction of conventional bast fibers such as flax, ramie, and jute (Smole *et al.* 2013): (1) dew retting by the action of dew, sun, and fungi on the plants spread out on the ground; (2) water retting is conducted in rivers or pools through bacterial action and takes from two to four weeks; (3) chemical retting, which involves solutions of chemicals such as sodium hydroxide, sodium carbonate, soaps, or mineral acids and takes only a few hours; and (4) controlled biological or biochemical retting by the addition of enzymes. Bark of cotton stalks has higher lignin content and lower cellulose content than conventional bast fiber resources (Habibi *et al.* 2008; Reddy and Yang 2009). As one of the conventional chemical retting methods, alkaline treatment with sodium hydroxide concentrations as high as 15 to 100 g/L (Reddy and Yang 2009; Troedec *et al.* 2011; Li *et al.* 2012; Zhou *et al.* 2012; Yan *et al.* 2013) has been used to remove hemicellulose, lignin, and other components from bark of cotton stalks, resulting in fine cellulose fibers. Sodium hydroxide quantities of 0.5 to 2.5 g have been used to treat 1 g of bark of cotton stalks; such treatment results in the generation of large quantities of alkaline waste water that could cause environmental problems. To reduce the negative effects to the environment by alkaline treatment, new efficient methods should be developed to obtain CSBF.

Steam explosion is a novel and green method with a high efficiency to separate biomass, and it can be performed on a large scale (Oliveira *et al.* 2013). It has received substantial attention in pretreatment for both bioethanol and biogas production for more than 10 kinds of lignocellulosic materials (Taherzadeh and Karimi 2008). In early studies, Ruiz *et al.* (2008) concluded that steam explosion pretreatment was an interesting option for the use of sunflower stalks in an ethanol production scheme. To improve the conversion of lignocellulosic material into bioethanol, Wang *et al.* (2009) used a two-step process based on steam explosion pretreatment followed by alkaline ethanol solution post-treatment to fractionate *Lespedeza* stalks. The impact of steam explosion on biogas production from rape straw was studied (Vivekanand *et al.* 2012). Kang *et al.* (2013) confirmed that SO_2-catalyzed steam explosion was an efficient and relatively cost-efficient pretreatment method for the production of bioethanol from softwood. Chang *et al.* (2012) showed that a combination of steam explosion and microbial fermentation increased the nutrient value of corn stover as animal feedstuff, and Pang *et al.* (2013) combined steam explosion and microwave irradiation to pretreat corn stover. Chen *et al.* (2013) investigated a continuous acid-catalyzed steam explosion process to pretreat rice straw on a pilot-scale. Industrial-scale steam explosion was used to pretreat sugarcane straw for enzymatic hydrolysis of cellulose (Oliveira *et al.* 2013). Other investigators (Martín-Davison *et al.* 2014) studied the effects of temperature on steam explosion pretreatment of poplar hybrids with different lignin contents.

There have been a few investigations of steam explosion technology for the preparation of cellulose fibers from lignocellulosic byproducts. The influence of the moisture content of cotton stalks before steam-explosion and the duration of steaming treatment on the mechanical prosperities of PP composites were investigated by Cao *et al.* (2011). Hou *et al.* (2014), Dong *et al.* (2014), Tan *et al.* (2011), and Qu *et al.* (2011) pretreated bark of cotton stalks by steam explosion and the obtained CSBF were used as reinforcing fibers of composites or textile fibers. Ibrahim *et al.* (2010) isolated cellulose from different lignocellulosic biomass sources including cotton stalk, corn cob, banana plant, and cotton gin waste using steam explosion technology followed by alkaline peroxide bleaching. A novel method of steam explosion that coupled mechanical carding in order to fractionate cornstalk long fibers for the production of cornstalk dissolving pulp was proposed by Wang and Chen (2013). Cherian *et al.* (2010) employed a steam explosion process to extract cellulose nanofibrils from pineapple leaf fibres for biomedical and biotechnological applications.

Special care should be taken in selecting the severity factor of a steam explosion treatment to avoid excessive degradation of the physical and chemical properties of the cellulose. The severity factor is determined by a correlation between time and temperature of the process. In very harsh conditions, lower enzymatic digestibility of lignocelluloses may be observed after steam explosion. For instance, generation of condensation substances between the polymers in steam explosion of wheat straw may lead to a more recalcitrant residue (Taherzadeh and Karimi 2008). Steam explosion speed also has an important effect on the separation of biomass and energy consumption (Yu *et al.* 2012; Zhao *et al.* 2012). High steam explosion speed can provide enough force to separate the compact structure of biomass and avoid a long period of violent treatment under high temperature or pressure. At present, there are two primary steam explosion modes: the valve blow mode and the catapult mode. The catapult mode can complete the explosion within 0.0875 s, while the valve blow mode needs at least 0.5 s (Yu *et al.* 2012). Catapult-mode steam explosion, called steam flash-explosion, is a sustainable and practical pretreatment for the extraction of feather keratin (Zhao *et al.* 2012).

Based on our best knowledge, there have been no detailed investigations of the effect of steam pressure in flash-explosion treatment on the structures and properties of the obtained CSBF. In this investigation, the steaming time was set to 2 min and the steam pressure was changed from 1.5 to 3.5 MPa. The influence of steam pressure on the composition, crystallinity, morphology, moisture regain, water retention, mechanical properties, and thermal stability of the obtained CSBF was investigated. The exploded CSBF were also compared with those obtained by conventional alkaline treatment.

EXPERIMENTAL

Materials

Cotton (*Gossypium hirsutum*) stalks were obtained from a farm in Yancheng city, Jiangsu Province, China. After the side branches of cotton stalks were removed, the outer bark was stripped manually, air dried, and cut into segments with a length of 10 cm. Sodium hydroxide and hydrochloric acid (37% w/w) of AR grade were purchased from Sinopharm Chemical Reagent Co., Ltd., Shanghai, China.

Steam flash-explosion treatment

Bark of cotton stalks was steam exploded using a QBS-200B test bed from Gentle Science & Technology Co., Ltd., China. The test bed with the catapult mode can complete an explosion within 0.0875 s (Yu *et al.* 2012). The bark of cotton stalks was first immersed in water with a bath ratio of 10:1 at room temperature for 2 h. The wet bark of the cotton stalks was steam flash-exploded under the conditions shown in Table 1. The steam pressures in Table 1 were all the gauge pressures of saturated steam. The corresponding severity factor was calculated according to Eq. 1 (Jacquet *et al.* 2011). All steam flash-exploded fibers were rinsed with tap water at a bath ratio of 20:1 at 80 °C for 1 h.

$$S = Log_{10}\left[t \cdot \exp\left(\frac{T - 100}{14.75}\right)\right] \quad (1)$$

where S is the severity factor, T is steam temperature corresponding to pressure, and t is steaming time.

Table 1. Steaming Conditions of Steam Flash-Explosion Treatment and Corresponding Severity Factors

Steam pressure (MPa)	Time (min)	Temperature (°C)	Severity factor
1.5	2	198	3.3
2.0	2	212	3.6
2.5	2	223	4.0
3.0	2	233	4.3
3.5	2	242	4.5

Alkaline treatment

Bark of cotton stalks was treated at 98 °C for 1 h with a concentration of sodium hydroxide of 80 g/L and a bath ratio of 20:1 (Reddy and Yang 2009). After the alkaline treatment, CSBF were rinsed and neutralized by adding hydrochloric acid (37% w/w). The fibers were rinsed again with tap water at a bath ratio of 20:1 for 5 min and air dried.

Measurement of yield, moisture regain, and water retention of CSBF

The yield of CSBF from bark of cotton stalks was calculated according to Eq. 2,

$$Y\ (\%) = \frac{W_{df}}{W_{db}} \times 100 \quad (2)$$

where Y is the yield of CSBF, W_{db} is the dry weight of the bark of cotton stalks, and W_{df} is the dry weight of CSBF.

Moisture regain of CSBF was determined according to ASTM D2654-89a (1998) and calculated according to Eq. 3,

$$MR\ (\%) = \frac{W_{sf} - W_{df}}{W_{df}} \times 100 \quad (3)$$

where MR is moisture regain and W_{sf} and W_{df} are the standard weight and the dry weight of the obtained CSBF, respectively.

Water retention of CSBF was determined by the method described by Jacquet *et al.* (2012). Approximately 0.5 g of samples was immersed in deionized water for 24 h at ambient temperature. Samples were then placed in a filter centrifugation tube (pore diameter 4.5 to 9 μm) and centrifuged at 4000 xg for 10 min with high-speed Avanti J-E refrigerated centrifuge (Beckman Coulter, USA). Wet samples were then weighed (W_{fw}), dried in an oven at 105 °C for 8 h and cooled in a desiccator. The water retention was then calculated using Eq. 4,

$$WR\ (\%) = \frac{W_{wf} - W_{df}}{W_{df}} \times 100 \qquad (4)$$

where *WR* is water retention and W_{wf} and W_{df} are the wet weight and the dry weight of CSBF, respectively.

For moisture regain and water retention of each kind of CSBF obtained by different steam pressure, five fiber samples were measured.

Analysis of composition of CSBF

Cellulose and lignin contents of CSBF were determined according to the Chinese National Standard GB 5889-86 (1986). For each kind of CSBF obtained by different steam pressure, five fiber samples were measured.

Analysis of crystallinity of CSBF

A D8 Advance X-ray diffractometer (Bruker AXS Co., Germany; wavelength 1.54 Å, Cu Kα radiation) was used to analyze the crystallinity of the CSBF. The intensity and current of the generator were 40 kV and 40 mA, respectively. The powdered fiber samples were scanned from 3 to 60° at a rate of 4°/min and a step size of 0.02°. The crystallinity can be characterized using the crystallinity index (I_c) calculated according to Eq. 5 (Moran *et al.* 2008; French 2014; Dong *et al*. 2014).

$$I_c = \frac{I_{200} - I_{am}}{I_{200}} \times 100 \qquad (5)$$

where I_c is the crystallinity index, I_{200} is the peak intensity at a 2θ angle close to 22.5° representing crystalline cellulose, and I_{am} is the peak intensity at a 2θ angle close to 15.5° representing the amorphous components in CSBF (*i.e.*, amorphous cellulose, hemicellulose, and lignin).

Measurement of length and fineness of CSBF

Length and fineness of CSBF were measured after they were conditioned in a standard atmosphere of 21 °C and 65% relative humidity for at least 24 h. For each kind of cotton stalk fiber obtained by different steam pressure, the lengths of 100 fibers were measured by a stainless steel ruler; then, the total weight of these 100 fibers was measured. Fineness of fibers was characterized in terms of dtex, which is defined as the conditioned weight of the fibers in grams *per* 10,000 m.

Morphological observation of CSBF

A Hitachi SU1510 scanning electron microscope (SEM) (Japan) was used to observe the morphologies of CSBF. The cross-sections of CSBF were prepared by slicing with a Harrington slicer (Y172, Nantong Hongda Experiment Instruments Co., Ltd., China). The fracture surfaces of the CSBF were obtained by immersion in liquid nitrogen and breaking.

All fiber samples were mounted on an aluminum stub with conductive adhesive tape, sputter coated with gold palladium, and observed under an accelerating voltage of 5 kV.

Measurement of tensile properties of CSBF

After being conditioned in a standard atmosphere of 21 °C and 65% relative humidity for at least 24 h, the tensile properties of the CSBF were measured by a tensile testing machine (Model YG004; Changzhou No. 2 Textile Machinery Co., Ltd., China). A gauge length of 10 mm and crosshead speed of 20 mm/min were used. At least 100 fibers were tested for each kind of cotton stalk fiber.

Analysis of thermal properties of CSBF

Thermogravimetric (TG) analysis of the CSBF was performed on a thermogravimetric analyzer (TGA/SDTA 851e; Mettler Toledo; Switzerland) in a nitrogen atmosphere at a flow rate of 10 mL/min. The samples were heated from 30 to 700 °C at a heating rate of 20 °C/min. The masses of the samples ranged from 5 to 10 mg.

Statistical analysis

The data for cellulose content, length, fineness, moisture regain, water retention, tensile strength and lignin content were analyzed using SAS software, version 8.1 (Cary, NC). The confidence interval was set at 95% with $\alpha = 0.05$, and a p value of < 5% was considered to be a statistically significant difference.

RESULTS AND DISCUSSION

Effect of Steam Pressure on the Composition of CSBF

Cellulose content of CSBF first significantly increased with increasing steam pressure up to 2.5 MPa; there was no obvious change from 2.5 to 3.5 MPa, as shown in Fig. 1.

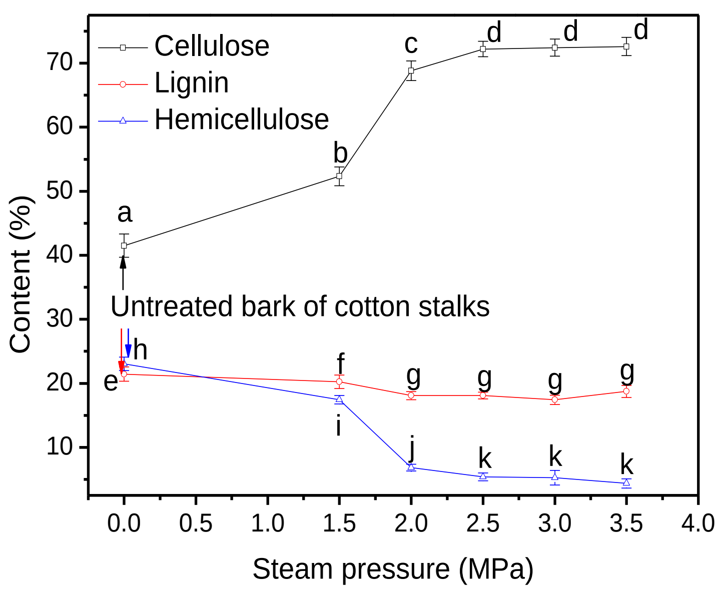

Fig. 1. Effect of steam pressure on constituent of CSBF by steam flash-explosion for 2 min. Note that data points with different letters represent statistically significant differences (p<0.05). Data reported as mean ± standard deviation

When the steam pressure was lower than 2.5 MPa, the removal of hemicellulose, lignin, and other impurities increased with increasing steam pressure; the cellulose content of CSBF significantly increased ($p<0.05$) from 41 to 72%. When the steam pressure was above 2.5 MPa, the cellulose content (about 72%) of CSBF did not change at pressures of 2.5, 3, and 3.5 MPa. On the other hand, the content of lignin and hemicellulose presented an opposite tendency respectively. Specifically, they were decreased with the steam pressure increasing from 1.5 to 2.5 MPa, and leveled off after that. In other words, the cellulose and residual non-cellulosic impurities (*i.e.* lignin and hemicellulose) in CSBF were so closely connected that they could not be adequately separated by increasing steam pressure.

Effect of Steam Pressure on Crystallinity of CSBF

The X-ray diffraction curves depicted in Fig. 2 show that untreated bark of cotton stalks and all CSBF presented the two major cellulose I peaks at 2θ angles of approximately 22° and 35°, which is corresponding to the (200) and (004) lattice planes, respectively. The crystallinity index was increased from 58.1 for untreated bark of cotton stalks to 72.9 for CSBF obtained by a steam pressure of 3.0 Mpa. This is due to the removal of the amorphous components and the recrystallization process of the amorphous parts in cellulose by heating (Yano *et al.* 1976). An increase in the crystallinity index of microcrystalline cellulose from corn stover after moderate steam explosion treatment (severity factor below 5.2) (Jacquet *et al.* 2012; Pang *et al.* 2013) has also been reported. However, the crystallinity index decreased when increasing the steam pressure from 3.0 to 3.5 MPa, which could be a combined effect of the decrease in amorphous components and disruption of the crystal structure in cellulose.

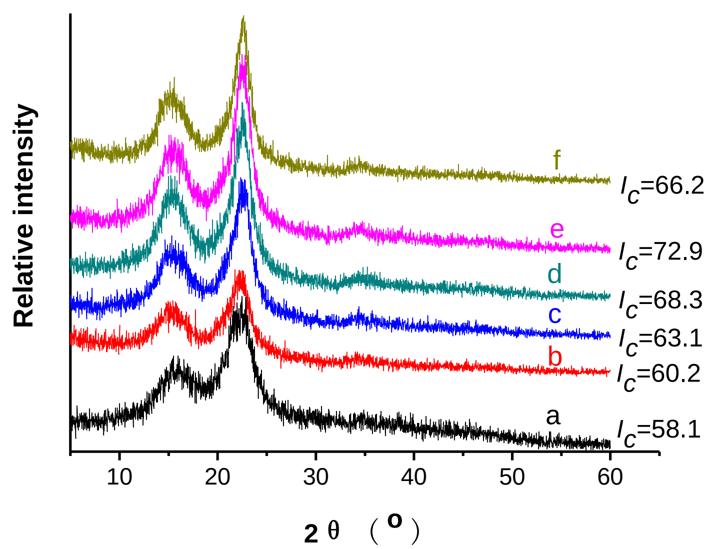

Fig. 2. X-ray diffraction curves and crystallinity index (I_c) for (a) untreated bark of cotton stalks and CSBF by steam flash-explosion under the steam pressures of (b) 1.5 MPa, (c) 2.0 MPa, (d) 2.5 MPa, (e) 3.0 MPa, and (f) 3.5 MPa for 2 min

Effect of Steam Pressure on Morphology and Yield of CSBF

The length, fineness, and yield of CSBF decreased, but the surface cleanness and color depth of CSBF increased, with increasing steam pressures, as shown in Table 2 and Figs. 3 and 4. The digital and SEM micrographs for untreated bark of cotton stalks and CSBF by steam flash explosion under different steam pressures (Figs. 3 and 4) indicate that higher steam pressures can separate bark of cotton stalks into finer fibers, whose surfaces were cleaner and had more longitudinal grooves.

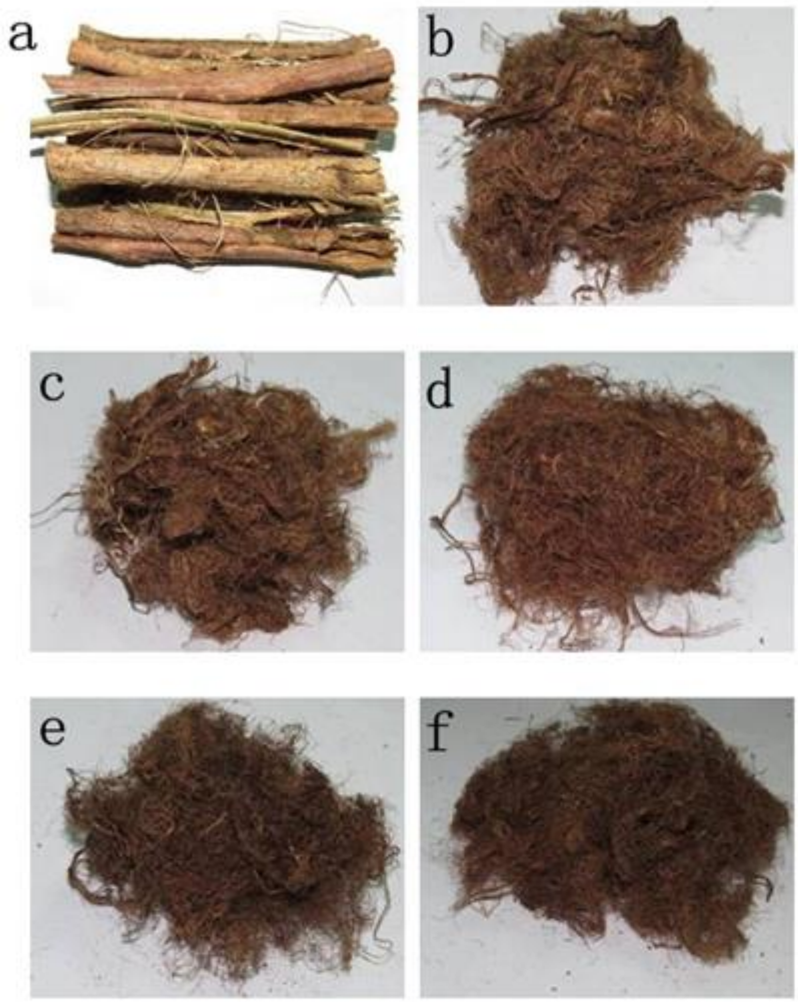

Fig. 3. Digital images for (a) untreated bark of cotton stalks and CSBF by steam flash-explosion under the steam pressure of (b) 1.5 MPa, (c) 2.0 MPa, (d) 2.5 MPa, (e) 3.0 MPa, and (f) 3.5 MPa for 2 min

From Table 2, the fineness value of CSBF decreased from 55 ± 7 dtex at 1.5 MPa to 42 ± 4 dtex at 3.5 MPa, which resulted from the effective removal of the non-cellulosic components bonding cellulose microfibrils together. The fineness of fibers is one of the most important parameters in determining the application value of the fibers. Finer CSBF are softer and can be applied to the spinning industry.

The aspect ratio is another important parameter for textile fibers. However, the higher steam pressures could result in negative effects such as shorter length and lower yield for CSBF. The length of CSBF decreased from 51.3 ± 2.5 mm at 1.5 MPa to 39.8 ± 0.8 mm at 3.5 MPa; the fiber yield decreased from 51.1% at 1.5 MPa to 27.6% at 3.5 MPa. The fiber length and yield presented their highest decrease, and the color of the fibers obviously changed to dark brown with increasing steam pressure from 2.5 to 3 MPa, as shown in Table 2 and Fig. 4, respectively. These negative effects could have occurred because some of the cellulose was damaged during steam flash explosion under steam pressures above 2.5 MPa.

Table 2. Length, Fineness, and Yield of CSBF by Steam Flash Explosion at Various Steam Pressures for 2 min

Steam pressure (MPa)	1.5	2.0	2.5	3.0	3.5
Length (mm)	51.3 ± 2.5[a]	49.1 ± 2.1[a]	47.5 ± 1.6[b]	42.7 ± 0.6[c]	39.8 ± 0.8[d]
Fineness (dtex)	55 ± 7[a]	49 ± 6[b]	45 ± 6[c]	43 ± 5[d]	42 ± 4[d]
Yield (%)	51.1	48.2	41.3	31.6	27.6
Note: In each row, data with different letters indicate statistically significant differences (P<0.05)					

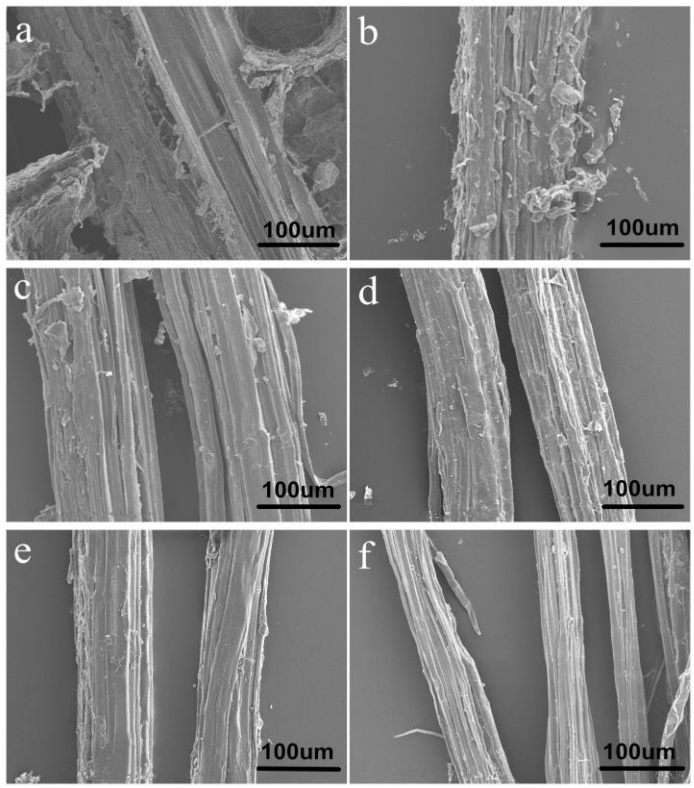

Fig. 4. SEM micrographs for (a) untreated bark of cotton stalks and CSBF by steam flash-explosion at the steam pressure of (b) 1.5 MPa, (c) 2.0 MPa, (d) 2.5 MPa, (e) 3.0 MPa, and (f) 3.5 MPa for 2 min

Effect of Steam Pressure on Moisture Regain and Water Retention of CSBF

The moisture regain and water retention of CSBF decreased with increasing steam pressure, as shown in Fig. 5. Compared with untreated bark of cotton stalks, CSBF had lower moisture regain and water retention. Because lignin is composed of aliphatic and aromatic hydrocarbons and is hydrophobic in nature (Thakur *et al.* 2014), the decrease in hydrophilic properties resulted from the removal of the hydrophilic components such as pectin and hemicellulose, indicating that CSBF contained primarily cellulose and lignin, while the majority of hemicelluloses and pectin were degraded and/or solubilized. The decrease in hydrophilic properties also resulted from the decrease in the amorphous regions that water molecules could enter. With increasing steam pressure, the crystallinity index of CSBF increased and water molecules could not enter the crystalline regions, leading to lower adsorption and retention of water. The decrease in hydrophilic properties can be helpful when CSBF are used as reinforcing fibers in hydrophobic thermoplastic matrices such as PP, PET, and PLA (Troedec *et al.* 2011).

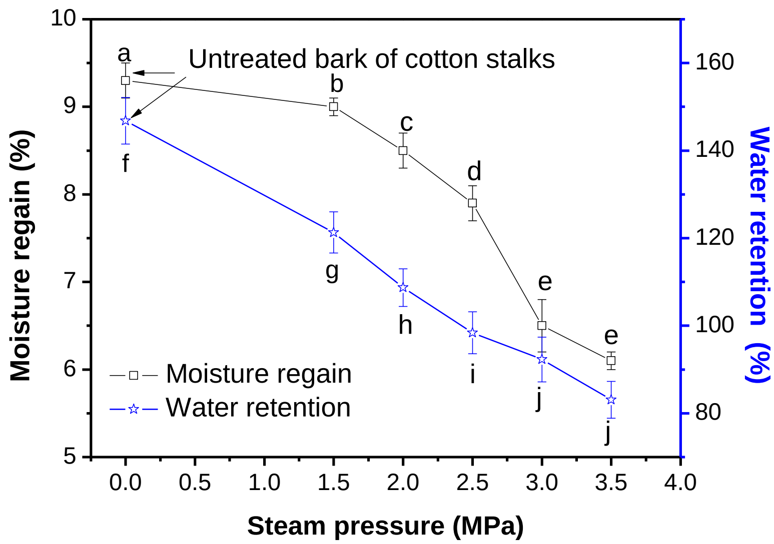

Fig. 5. Effect of steam pressure on moisture regain and water retention of CSBF by steam flash explosion for 2 min. Note that data points with different letters represent statistically significant differences ($p<0.05$). Data reported as mean ± standard deviation

Effect of Steam Pressure on Mechanical Properties of CSBF

The tensile strength of the CSBF slightly increased as the pressure increased from 1.5 to 2.0 MPa, and then significantly decreased as the pressure increased from 2.5 to 3.0 MPa, as shown in Fig. 6. The slight increase resulted from the higher cellulose content and crystallinity index of cellulose because the tensile strength of the lignocellulosic fibers is primarily due to the cellulose component (Smole *et al.* 2013). When the steam pressure was above 2.5 MPa, the tensile strength significantly decreased, which was explained by the fact that the cellulose components in CSBF were partly destroyed by higher temperatures. Wang *et al.* (2009) demonstrated that steam explosion under steam pressures higher than 20 kg/cm^2 for 4 min (severity factor > 3.95) could induce cellulose degradation to a certain degree for Lespedeza stalks. Jacquet *et al.* (2011) also indicated that thermal degradation of cellulose fibers was considerable when the severity factor of steam explosion was above 4.0. Compared with the published paper (Yzombard *et al.* 2014), the tensile strength of the untreated CSBF in this paper was lower, which was mainly due to the different growing environment of cotton stalk.

Tensile stress-strain curves of CSBF, as shown in Fig. 7, indicate that the breaking elongations and initial modulus of the CSBF decreased with increasing steam pressure. The lower initial modulus implied that the fibers were more easily deformed by tensile force. The SEM images of the fracture surfaces of untreated bark of cotton stalks and CSBF obtained by liquid freezing and breaking (Fig. 8a1, Fig. 8b1, and Fig. 8c1) all present lumens in cells. However, the lumens for untreated bark of cotton stalks were smaller and the lumens for the CSBF all disappeared when the cross-sections were obtained by slicing with a Harrington slicer, as shown as Fig. 8a2, Fig. 8b2, and Fig. 8c2. These differences between the SEM images indicated that the lumens were destroyed by the Harrington slicer. The cell walls for CSBF were softer and were more easily deformed by compression and shear stress by the Harrington slicer than those of bark of cotton stalks due to the lower lignin content and looser structures.

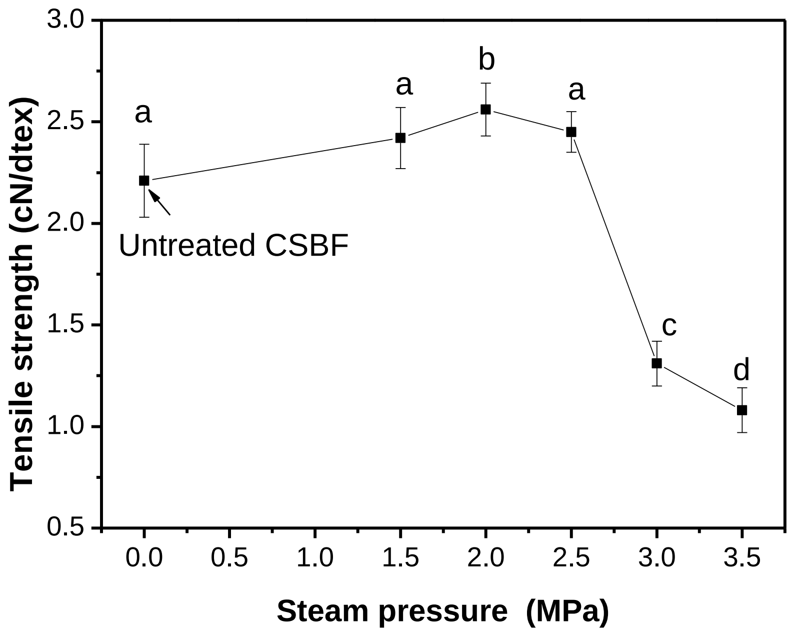

Fig. 6. Effect of steam pressure on tensile strength of CSBF by steam flash-explosion for 2 min. Note that the data points with different letters represent statistically significant differences ($p<0.05$). Data reported as mean ± standard deviation

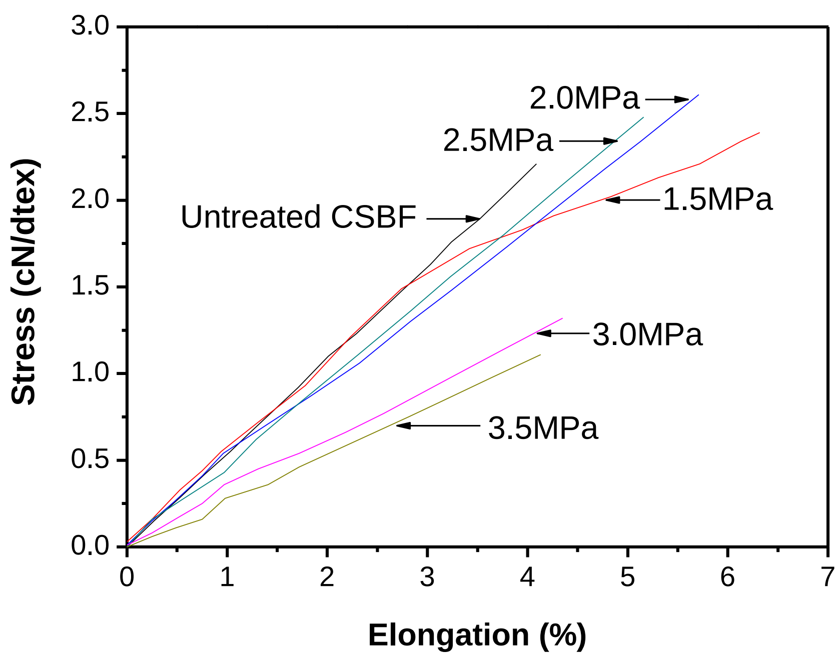

Fig. 7. Tensile stress-strain curves of untreated CSBF and CSBF by steam flash-explosion under different steam pressures from 1.5-3.5 MPa for 2 min

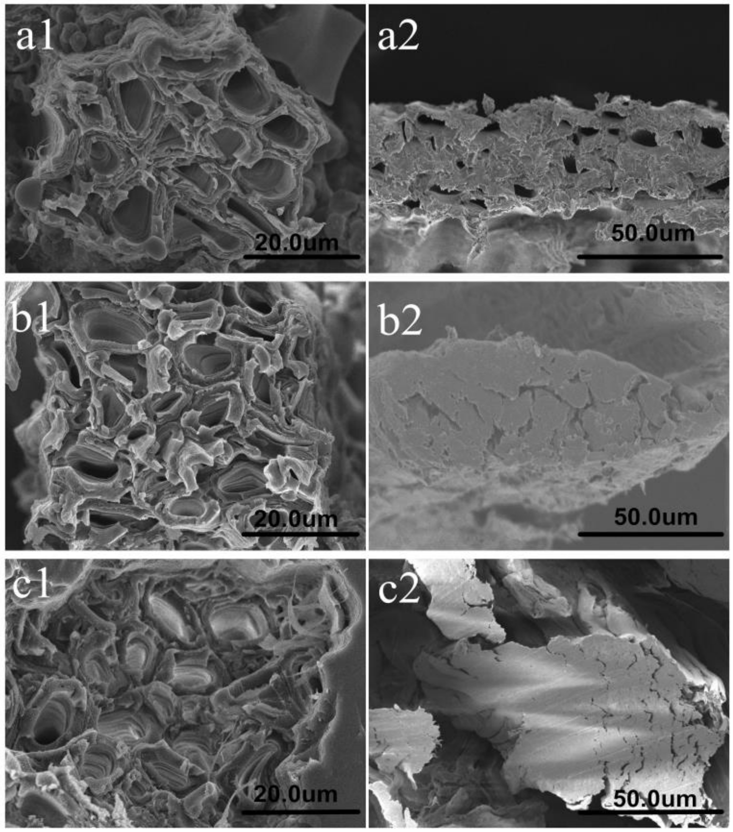

Fig. 8. SEM micrographs of (1) the fracture surfaces and (2) the cross-sections of (a) untreated bark of cotton stalks and CSBF by steam flash-explosion at steam pressures of (b) 2.5 MPa and (c) 3.5 MPa for 2 min

Effect of Steam Pressure on Thermal Stability of CSBF

Figure 9 shows the TG curves and the differential thermogravimetric (DTG) curves for untreated bark of cotton stalks and CSBF. Table 3 shows their TG and DTG data for the leading decomposition steps. The CSBF presented higher temperatures for onset decomposition and maximum decomposition compared to untreated bark of cotton stalks. Specifically, for untreated bark of cotton stalks, the onset decomposition temperature was 225 °C and the decomposition peaks occurred at 348 °C. For the CSBF, the onset decomposition temperatures were 235 to 237 °C and the decomposition peaks occurred at 376 to 390 °C, respectively. Decomposition of hemicelluloses in hemp and jute occurs at approximately 220 to 320 °C (Das *et al.* 2000; Ouajai and Shanks 2005; Yang *et al.* 2007; Moran *et al.* 2008). The maximum thermal decomposition occurs at 268 °C for xylan, which was a representative component of hemicellulose in pyrolysis processes (Yang *et al.* 2007), at 355 °C for commercial cellulose from Sigma-Aldrich Chemie GmbH (Yang *et al.* 2007), and at 370 °C for standard microcrystalline cellulose (Jacquet *et al.* 2011). The decomposition temperature of commercial cellulose occurs at 330 to 400 °C, and cellulose is entirely decomposed at 400 °C (Yang *et al.* 2007). The weight loss after 400 °C can be attributed to the decomposition of lignin, which occurs in a wide temperature range from 160 to 900 °C (Yang *et al.* 2007). The differences in the inherent structures and chemical natures of lignin possibly account for the different behaviors of untreated bark of cotton stalks and CSBF obtained at different steam pressures. The higher temperatures of onset decomposition and maximum decomposition for CSBF indicated that the unstable hemicellulose component had been effectively removed from CSBF and the CSBF had higher thermal stability.

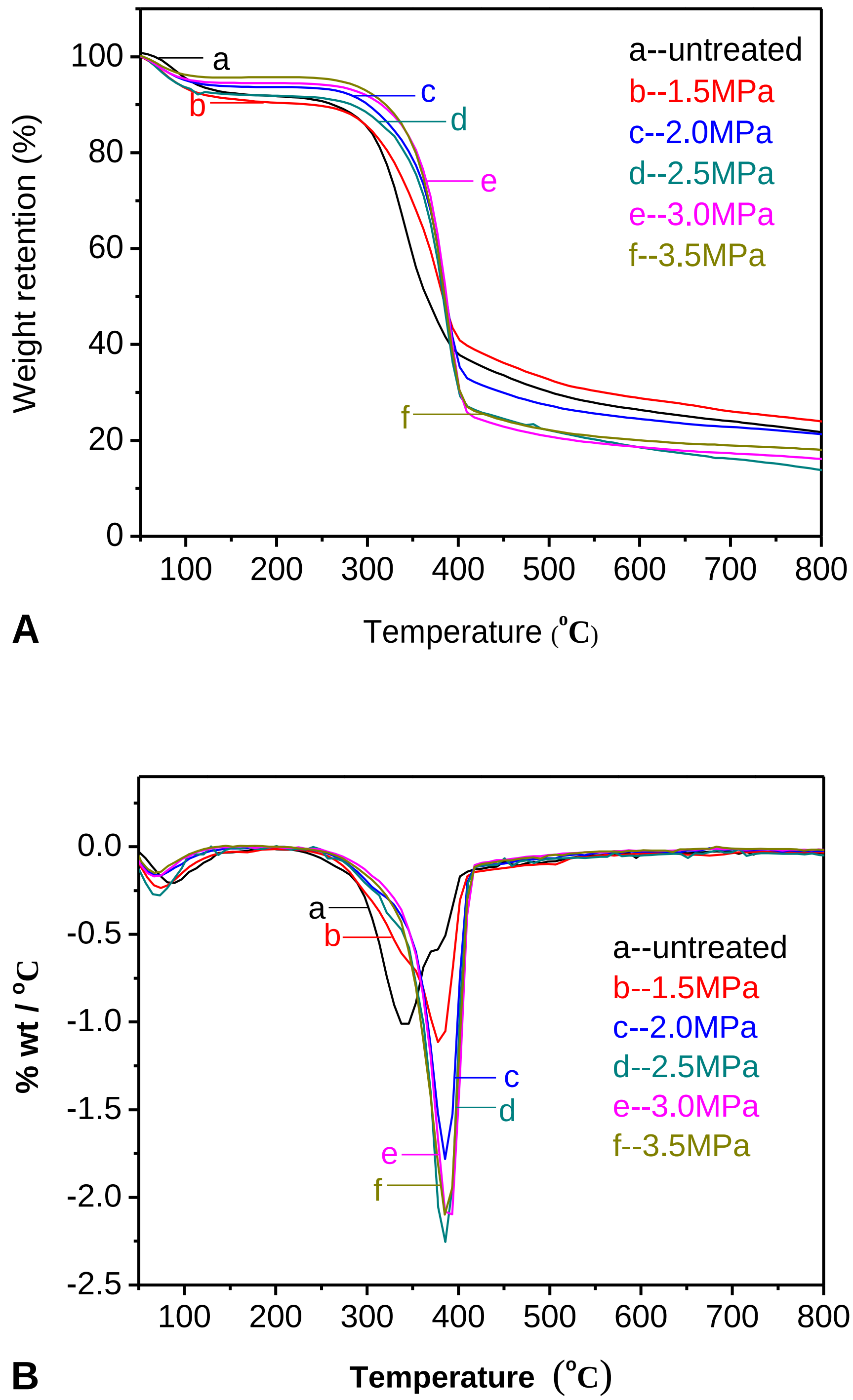

Fig. 9. (A) TG and (B) DTG curves for (a) untreated bark of cotton stalks and CSBF by steam flash-explosion at steam pressures of (b) 1.5 MPa, (c) 2.0 MPa, (d) 2.5 MPa, (e) 3.0 MPa, and (f) 3.5 MPa for 2 min

Table 3. TG and DTG Data for Untreated Bark of Cotton Stalks and CSBF by Steam Flash Explosion at Steam Pressures of 1.5 to 3.5 MPa for 2 min

Materials	Steam pressure /MPa	Onset decomposition temperature /°C	Temperature of decomposition peak /°C
Untreated bark of cotton stalks	Without steam explosion	225	348
CSBF	1.5	235	376
	2.0	237	387
	2.5	237	387
	3.0	237	390
	3.5	237	387

Comparison of Extraction of CSBF by Steam Flash Explosion and Alkaline Treatment

Table 4 shows that the CSBF obtained by steam flash explosion had higher lignin content and yield, but similar crystallinity index, length, fineness, moisture regain, water retention, and tensile strength compared with those obtained by conventional alkaline treatment. Steam flash explosion is chemical-free and uses less energy and water compared to conventional alkaline treatment, as shown in Table 5, which results in a lower production cost.

Table 4. Structures and Properties of CSBF Obtained by Steam Flash Explosion Compared with Those Obtained by Alkaline Treatment

Method	Steam flash explosion	Alkaline treatment
Cellulose content (%)	72.2 ± 0.9	74.0 ± 0.7
Lignin content (%)	18.1 ± 0.5[a]	16.5 ± 0.6[b]
Crystallinity index	68.3	67.4
Length (mm)	47.5 ± 1.6	50.1 ± 2.1
Fineness (dtex)	45 ± 6	43 ± 4
Moisture regain (%)	7.9 ± 0.2	7.7 ± 0.2
Water retention (%)	98.4 ± 4.8	96.0 ± 0.4
Tensile strength (cN/dtex)	2.45 ± 0.10	2.49 ± 0.16
Onset decomposition temperature (°C)	237	240
Yield (%)	41.3	30.7
Note: In each row, data with different letters indicate statistically significant differences (p<0.05)		

Table 5. Comparison of Estimated Consumption for Extracting 1 kg of Bark of Cotton Stalks by Steam Flash Explosion and Alkaline Treatment

Method	Steam flash explosion	Alkaline treatment
Heat energy (kJ)	5170	6130
Water (L)	40	60
NaOH (kg)	No	1.6
HCl (37% w/w) (L)	No	0.6

CONCLUSIONS

1. Steam flash explosion can open the tight lignocellulosic structures and remove the amorphous and non-cellulosic components from bark of cotton stalks. However,

steam pressures that are too high can destroy the crystal structures of cellulose and even decompose the cellulose. When the steam pressure was below 2.5 MPa, CSBF had a higher content of cellulose, cleaner and smoother surfaces, lower fineness values, and higher crystallinity index with increasing steam pressure. When the steam pressure was above 3.0 MPa, the CSBF showed lower tensile strength, lower yield, darker color, and lower crystallinity index.

2. Under the optimized steam pressure of 2.5 MPa for 2 min, the obtained CSBF had a cellulose content of 72%, length of 48 mm, fineness of 45 dtex, crystallinity index of 68, moisture regain of 8%, water retention of 98%, tensile strength of 2.4 cN/dtex, and yield of 41%, similar to those of CSBF obtained by conventional alkaline treatment.
3. Moderate steam flash explosion is a chemical-free, quick, effective, and feasible treatment for extracting CSBF with desirable properties.

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