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Structure and properties of high quality natural cellulose fibers from cornstalks

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Abstract

We have developed a fiber extraction method that produces fibers from cornstalks with mechanical properties similar to that of the common textile fibers. The fiber extraction method developed results in partial delignification and produces fibers from cornstalks that are suitable for textile and other industrial applications. The structure of the fibers obtained was investigated using X-ray diffraction and scanning electron microscope. The structure and composition of the natural cellulose fibers obtained from cornstalks are different than that of the common bast fibers such as flax and kenaf. Tensile properties of the fibers were studied using an Instron tensile tester. This study found that cornstalk fibers have relatively lower percent crystallinity but similar microfibrillar angle as that of the common bast fibers. The structure and properties of cornstalk fibers indicate that the fibers are suitable for producing various textile products.

Keywords: Cornstalk; Cellulose fiber; Bast fiber

1. Introduction

Natural cellulose fibers with mechanical properties similar to that of the common textile fibers have been obtained from cornstalks. However, lignocellulosic materials such as cornstalks have structure and composition different from that of the common bast fibers. Lignocellulosic sources such as cornstalks, cornhusks, rice, and wheat straw are composed of single cells of cellulose that are only about 0.5–3.0 mm in length whereas bast fibers such as flax can have single cells as long as 77 mm [1,2]. The short single cells make it difficult to obtain long and fine fibers. In addition, lignocellulosic materials contain up to 20% lignin as in wheat straw compared to 2–3% in flax [1]. The presence of high amounts of lignin affects the structure and properties of the fibers. Fibers with high amounts of lignin are coarse, stiff and have a brownish color that cannot be removed using normal oxidizing bleaching agents [3,4]. However, multicellular fibers with short single cells need lignin to hold the single cells together in the form of a bundle to be useful for textile and other applications. Complete removal of lignin will result in single cells that are too small to be used as textile fibers. Therefore, it is challenging to obtain fibers from lignocellulosic sources such as cornstalks with properties similar to those of the common bast fibers such as flax and kenaf.

Although cornstalks are the largest source of lignocellulosic biomass in the world, there has been limited use of cornstalks for fibrous applications. Traditionally, cornstalks have been used as a source of fibers for manufacturing pulp for paper. Recently, fibers obtained from cornstalks were tried as reinforcements for starch foams for packing materials and also for composites used in the automotive and construction industries [5–7]. Cornstalk fibers used as reinforcement improved the tensile properties of the starch acetate foams in the laboratory [8]. Cornstalks have also been studied to obtain regenerated cellulose [9].

Both chemical and mechanical methods are used to obtain fibers from cornstalks. Chemical methods including alkaline fiber extraction are used to produce pulp from cornstalks for the paper industry [7]. Steam, carbon dioxide

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and ammonia explosion are some of the mechanical methods used to separate fibers from cornstalks [5,6]. However, fibers produced using these methods do not have the properties required for textile and other high value fibrous applications. In addition, there is no literature available describing the structure and properties of the fibers obtained from cornstalks.

As the largest source of lignocellulosic biomass, cornstalks are a cheap and annually renewable resource suitable for producing natural cellulose fibers. Producing textile quality fibers from cornstalks will have several advantages. Using cornstalk fibers for high value applications such as textiles will add value to the corn crops and provide an inexpensive natural cellulose fiber to the fiber industry. Utilizing the byproduct of a major food crop for fibrous applications will save the natural resources required to produce the natural and synthetic fibers and will also benefit the environment. In this paper, we report the process used to produce high quality fibers and the structure and properties of the fibers produced from cornstalks in comparison to three common textile fibers, cotton, flax and kenaf.

2. Experimental

In this paper, a fiber is defined as a bundle of single cells when referring to cornstalk, flax, and kenaf. A single cell is defined as the smallest morphological unit of cellulose in the fiber and a unit cell refers to the smallest size of cellulose crystals in the fiber.

2.1. Materials

Cornstalks were collected from ready-to-harvest corn fields in NE, USA. These stalks were manually cleaned to separate the fibrous tissue from the pith tissue. The pith tissue cannot be used to obtain fibers and was, therefore, mechanically separated from the fibrous parts. Sodium hydroxide, glacial acetic acid, nitric acid, chromic oxide and sulfuric acid used in this study were obtained from VWR international, Bristol, CT.

2.2. Fiber production

The fiber extraction conditions mentioned below are the optimized conditions. Many trials of fiber extraction were conducted by varying the chemical concentration, time and temperature of treatment and the liquor-to-stalk ratio. The optimized conditions were decided based on the quality and yield of the fibers produced. In the optimized condition, the pithed cornstalks were treated with 2% (w/v) sodium hydroxide solution for 45 min at a temperature of 95 °C using a liquor-to-stalk weight ratio of 20:1. The stalks and alkali solution were heated in a closed container on a hot plate with temperature control. After the treatment, the slurry was washed in warm water to remove the dissolved substances and the fibers collected were neutralized using a solution of 10% (v/v) acetic acid. The neutralized fibers were dried under ambient conditions.

The fibers obtained were macerated to obtain single cells using a 1:1 mixture of 10% (w/w) nitric acid and 10% (w/w) chromic acid [10]. The solution was warmed at 60 °C for 5 min to initiate the reaction and then allowed to stay overnight at room temperature. The treated fibers were washed and later centrifuged in water until the pH of the water was 7.0. The centrifuged fibers were dried in ethanol for observing under the scanning electron microscope (SEM).

2.3. Fiber composition

The amount of cellulose in the fiber was determined using the Norman and Jenkins method [11]. The amount of Klason lignin in the fibers was determined by treating 1 g of fiber in 50 ml of 72% (w/w) sulfuric acid for 24 h. Single cell dimensions were measured from SEM pictures. Ten cells were measured for their length and width. The average, percent coefficient of variation (% CV) and the minimum and maximum values are reported. The % CV was calculated using the following formula.

$$\%\ \mathrm{CV} = \frac{\text{Standard deviation}}{\text{Mean}} \times 100 \tag{1}$$

2.4. Crystal structure

Fibers were mounted on a sample holder to obtain the X-ray diffraction data. A Rigaku D-Max/B $\Theta/2\Theta$ X-ray diffractometer with Bragg–Brentano parafocusing geometry, a diffracted beam monochromator, and a copper target X-ray tube set to 40 kV and 30 mA was used to obtain the % crystallinity, crystallinity index (CI) and unit cell dimensions. Diffraction intensities were recorded with 2θ ranging from 5 to 40° with a sample to detector distance of 185 mm.

To determine the % crystallinity, the total diffracted area and the area under the crystalline peaks was determined by integration after correcting the data for absorption, Lorentz-polarization effects, incoherent scatter, and air scatter. The ratio of the crystalline area to that of the total diffracted area is taken as the % crystallinity [12,13].

CI measures the orientation of the cellulose crystals in a fiber to the fiber axis. The CI was determined by using the wide angle X-ray diffraction (WAXD) counts at 2θ angle close to 22 and 18°. The counter reading at peak intensity of 22° is said to represent the crystalline material and the peak intensity at 18° corresponds to the amorphous material in cellulose materials [14,15]. From these readings, the crystallinity index is calculated using Eq. (2).

$$\mathrm{CI} = \frac{I_{22} - I_{18}}{I_{22}} \tag{2}$$

where I_{22} and I_{18} represent the counter readings at 2θ close to 22 and 18°, respectively.

Dimensions of the unit cell of cellulose crystal in the fiber and the lattice distances were determined assuming the monoclinic crystal system and using the equations reported by Hindeleh and Johnson [12,13]. Crystallite size was calculated using the Scherrer equation [13].

$$L = \frac{K\lambda}{P\cos\theta} \tag{3}$$

where λ is the wavelength of the radiation used, θ is the Bragg angle of the diffraction peak, P is the half width of the 002 peak in radians and K is a constant usually considered as 0.89.

A Bruker D8 Discover model diffractometer equipped with an area detector and GADDS software was used to obtain 2-dimensional transmission diffraction patterns of the fibers and to calculate the orientation of the microfibrils to the fiber axis in terms of the microfibrillar angle (MFA). The diffraction patterns were analyzed using the GADDS software to obtain information about the preferred orientation of the microfibrils. The area detector used in this study to obtain the diffraction patterns of the cellulose crystals in the fibers has the advantage that it provides the diffraction patterns of the fibers in both the equatorial and meridional directions. Orientation of the cellulose microfibrils in the fibers was characterized by fitting the intensity distribution around the 002 peak from the area detector frame to two Gaussian curves with a non-linear least squared algorithm using the computer software Microcal ORIGIN. Details of the methods of calculating the MFA are available in literature [16–18].

2.5. Fiber morphology

A Hitachi model S2000 N scanning electron microscope was used to study the morphology of the untreated cornstalk, alkali extracted fibers and the single cells obtained by maceration. Samples were sputter coated with gold palladium for observing under the SEM. A voltage of 15 kV and a specimen to detector distance of about 10 mm was used for observations.

2.6. Fiber properties

The denier of the fibers was determined by weighing a known length of the fibers. Tensile properties of the fibers were measured using an Instron model 4400 tensile testing machine according to ASTM standards [19]. A gauge length of 25 mm with a crosshead speed of 18 mm/min was used for testing. Five sets of 10 fibers each were tested for strength, breaking elongation and modulus. The mean, percent coefficient of variation (% CV) and minimum and maximum values are reported. The % CV values are representative of the variation in tensile properties of natural fibers and are not the experimental errors. Moisture regain of the fibers was determined according to ASTM standards [19].

3. Results and discussion

3.1. Fiber production

The dimensions of the single cells in cornstalk obtained by maceration are given in Table 1. The lengths of single cells in cornstalks are similar to those in other sources of biomass such as rice and wheat straw [2,22]. The widths of the single cells obtained by us have a larger range of 15–35 μm than the 10–20 μm reported in literature [23]. The wider range of the width of single cells could be because of the different species and maturity of cornstalks used in this study. The single cells in cornstalk are shorter than those in the multicellular vegetable fibers such as flax which has single cells of up to 77 mm in length [1]. Because of the shorter single cells, it is difficult to obtain cornstalk fibers that are long and fine at the same time. To form long fibers, a number of single cells need to be connected together by the binding substances such as lignin. The increase in number of single cells and binding substances increases the width and diameter of the fiber resulting in coarse fibers.

The fibers obtained after the alkaline treatment are composed of single cells of cellulose that are held together in the form of a bundle by binding substances such as lignin and pectin. Lignin forms ester linkages with cellulose and is the major binding material in fibers since most of the pectin will be removed during the alkali treatment [1,20]. Pectin can be removed from the fibers by treating with 0.4% sodium hydroxide at boil [1]. The conditions used in this study to obtain fibers (2% sodium hydroxide, 95 °C for 45 min) are expected to remove most of the pectin. As shown in Table 1, cornstalk fibers contain about 80% cellulose and 8% lignin. The remaining 10–12% will be moisture (8%), minerals and pectin. Therefore, it is reasonable to assume that lignin is the major binding material in cornstalk fibers. In addition, delignification was found to increase the fineness but decreases the strength of the fibers [21]. Complete removal of lignin will result in single cells that are too small to be suitable for high quality fibrous applications.

Although cornstalks contain about 40% cellulose, only about 15–20% by weight of the cornstalks used for extraction is obtained as high quality fibers. The remaining 20–25% of cellulose is in the form of small fibers that are about 1 cm or smaller in length and are, therefore, unsuitable for textile and other high quality fibrous applications. The yield of the fibers obtained depends on the fiber production conditions such as alkali concentration, time, temperature and the stalk-to-liquor ratio used. In addition to the yield of the fibers, the extent of removal of the non-cellulosic substances plays a major role in determining the structure and properties of the fiber. For

Table 1
Single cell dimensions and chemical composition of cornstalk and fibers obtained after chemical treatment

Single cell dimension					% Composition					
	Mean	% CV	Min	Max		Cornstalk [7,20]	Fiber			
							Mean	% CV	Min	Max
Length (mm)	0.8	32	0.5	1.4	Cellulose	38–40	81	4.2	78	84
Width (μm)	27	33	14	35	Hemicellulose	28	–	–	–	–
					Lignin	7–21	8.4	11.9	7.3	9.2

example, higher lignin content means a stiffer and weaker fiber. Cornstalk fibers contain about 8% lignin, lower than that in kenaf (10%) but higher than that in flax (2–3%).

3.2. Crystal structure

The crystalline parameters for cornstalk fibers are given in Table 2. Cornstalk fibers contain 52% crystalline cellulose, lower than that of flax, cotton, and kenaf which have crystallinities of about 70, 65, and 60%, respectively, [24–27]. The percent crystallinity of the fibers affects the chemical absorptions of a fiber. Lower crystallinity means higher amorphous regions, which are more accessible to chemicals and water. Crystallinity is also related to strength and generally, the higher the crystallinity the higher is the strength of the fibers if the polymer structures are the same. Flax, cotton, and kenaf have strength of about 6, 3.5, and 2.5 g per denier, respectively whereas cornstalk fibers have an average strength of 2.2 g per denier. In addition to the crystallinity, the angle or the orientation of the cellulose microfibrils to the fiber axis (MFA) and the order of the cellulose crystals in the fiber (CI) influence the strength and stiffness of fibers [28–30].

Cornstalk fibers have an MFA of about 11°, lower than that of cotton which has MFA in the range of 20–30° depending on the maturity and species of cotton [28]. Multicellular bast fibers such as flax typically have lower MFA's of about 6–10° [30]. A lower MFA means a stronger but stiffer fiber with lower elongation. On the other hand, a lower CI means poor order of cellulose crystals in the fiber. CI of cornstalk fibers at 74 is higher than that of cotton at 60 but similar to that of flax at 80 [18]. Although MFA and CI give a quantitative measure of the orientation of the cellulose microfibrils and the crystals in fibers, X-ray diffraction patterns are visual indicators of the orientation of the cellulose crystals.

The intensity, size, and shape of the diffracting arcs in a fiber diffraction pattern are determined by the size and orientation of the cellulose crystals in the fibers. Fig. 1(a) and (b) shows the diffraction patterns of cornstalk and cotton fibers, respectively. The diffraction of cornstalk fibers produces narrow and bright patterns that are characteristic of oriented crystals [30,31]. On the other hand, the diffraction pictures of cotton shows long diffracting arcs that end sharply. This is due to misorientation of the cellulose crystals to the fiber axis as indicated by the lower CI in cotton fibers. In Fig. 1(b), the distinct meridional reflections of cotton are seen in addition to the typical equatorial reflections. The diffraction patterns of cornstalk fibers show bright and narrow equatorial reflections indicating that the cellulose crystals are better oriented in cornstalk fibers than in cotton.

The crystal size and dimensions of a unit cell of cellulose in cornstalk are compared with that of the various forms of cellulose in Table 2. The major difference between the four forms of cellulose is the β angle. As seen from the table, cornstalks have unit cells of cellulose with dimensions of the *b*- and *c*-axis and the β angle same as that of cellulose I [32]. The difference in the value of the *a*-axis of cornstalk cellulose and that of cellulose I could be because of the different sources of cellulose being compared. Based on our knowledge, a value of 8.02–8.35 Å have been reported for the *a*-axis in cellulose I [1,32,33]. The value of *a*-axis of cornstalk cellulose (8.54 Å) determined by us is close to the

Table 2
Crystal structure of cellulose in the cornstalk fibers

Crystal structure		Unit cell dimensions				
		Cellulose	*a* (Å)	*b* (Å)	*c* (Å)	β (°)
Crystallinity (%)	52	Cornstalks	8.54 ± 0.044	10.26 ± 0.009	7.93 ± 0.009	84
Crystallinity index	74	Cellulose I	8.35	10.3	7.9	84
Crystal size (nm)	3.8	Cellulose II	8.1	10.3	9.1	62
MFA (°)	10.9	Cellulose III	7.74	10.3	9.9	58
		Cellulose IV	8.11	10.3	7.9	90

Unit cell dimensions for cellulose I, II, III and IV are from Ref. [32].

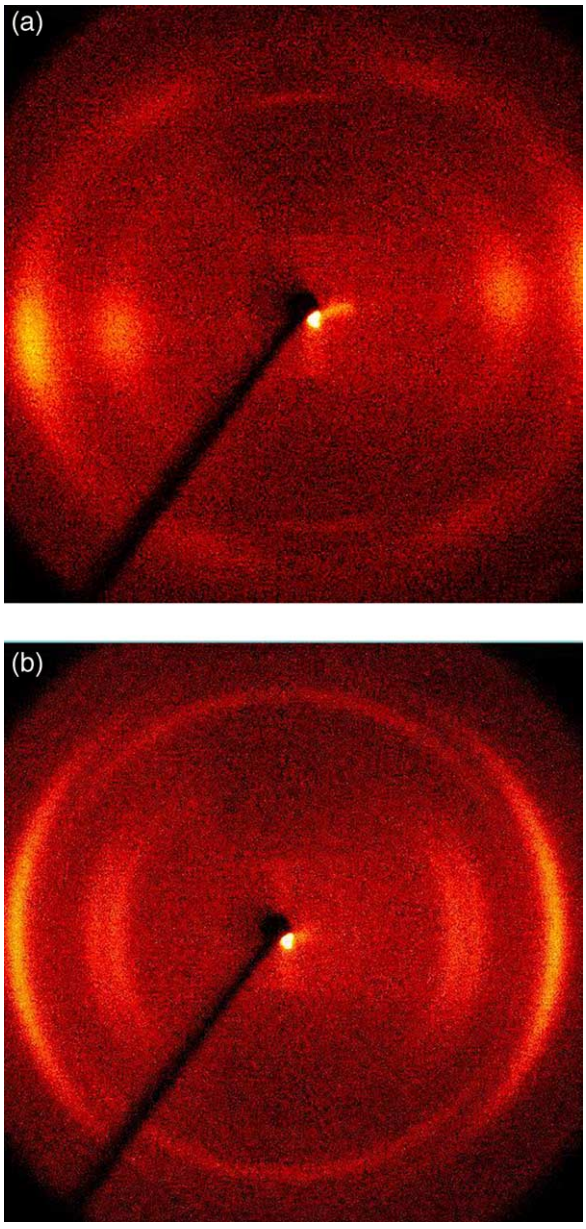

Fig. 1. (a) X-ray diffraction pattern of a cornstalk fiber. The bright diffraction spots in the picture are due to better orientation of cellulose crystals in cornstalk fibers. (b) X-ray diffraction pattern of cotton obtained using an area detector. The diffraction pattern shows both the equatorial and meridional reflections. The long diffracting arcs indicate the higher MFA in cotton.

value recognized for cellulose I (8.35 Å). Based on the dimensions of the *b*- and *c*-axis and the β angle, we believe that the cellulose in cornstalk is more close to cellulose I structure than the other forms.

Apparent crystal size of cellulose in cornstalk fibers is about 3.8 nm, smaller than that of cotton but larger than that in flax. Flax has cellulose crystals that are about 2.8 nm whereas the cellulose crystals in cotton are about 5.5 nm [25]. A large crystal means reduced surface area. Lower surface area decreases the moisture and chemical absorptions of the fibers.

3.3. Morphological structure

Cornstalks have an outer layer consisting of hemicellulose, lignin and other substances as shown in Fig. 2(a). The outer layer protects the cellulose in the stalks from being damaged by the microorganisms in the environment. Most of these surface materials are removed during the alkaline fiber extraction. However, as seen from Fig. 2(b), the fibers obtained from cornstalks are composed of single cells that are held together by binding materials that have not been removed by the alkali during fiber extraction. Stronger treatment conditions remove most of the binding substances resulting in single cells that are too small to be used for high value fibrous applications. The single cells of cornstalk in Fig. 2(c) have lengths of about 0.7–1.5 mm and width of about 20 μm at the widest part. The single cells have convolutions along their length with tapered ends but are broader and ribbon like at the center.

3.4. Fiber properties

The properties of the cornstalk fibers are summarized in Table 3. Fibers obtained from cornstalks have fineness (denier) similar to that of kenaf, which has denier of about 50. Cornstalk fibers have strength ranging from 1.5 to 4.5 g per denier. The fibers in the lower strength range are similar to kenaf (1.5–2.5 g per denier) whereas the higher strength fibers are similar to those of cotton (3.5 g per denier) [1,34]. As discussed earlier, the strength and elongation of multicellular fibers are dependent on the % crystallinity of the fibers, orientation of the microfibrils and cellulose crystals in the fibers. In addition, the number, length and width of single cells and the binding agents affect the strength of a fiber. The single cells in cornstalk fibers are shorter and have smaller widths than the single cells in flax and kenaf. Flax fibers can have single cells that are up to 77 mm in length whereas kenaf has smaller single cells of about 3.3 mm [1]. Therefore, for a given size of the fiber, there will be greater number of single cells in cornstalk fibers than in flax or kenaf. The higher number of single cells means a greater number of binding spots which could have more weak links that break relatively easily during tensile testing resulting in reduced strength.

Elongation of cornstalk fibers (1.1–3.5%) is similar to that of flax (2–3%) but lower than that of kenaf (3.5–5.5%)

Table 3
Cornstalk fiber properties

Fiber property	Mean	% CV	Min	Max
Length (cm)	3.0	55	1.5	8.5
Fineness (denier)	70	50	35	120
Strength (g/denier)	2.2	45	1.5	4.5
Elongation (%)	2.2	34	1.1	3.5
Modulus (g/denier)	127	44	65	264
Work of rupture (g/denier)	0.04	100	0.01	0.08
Moisture regain (%)	7.9	5.4	7.5	8.4

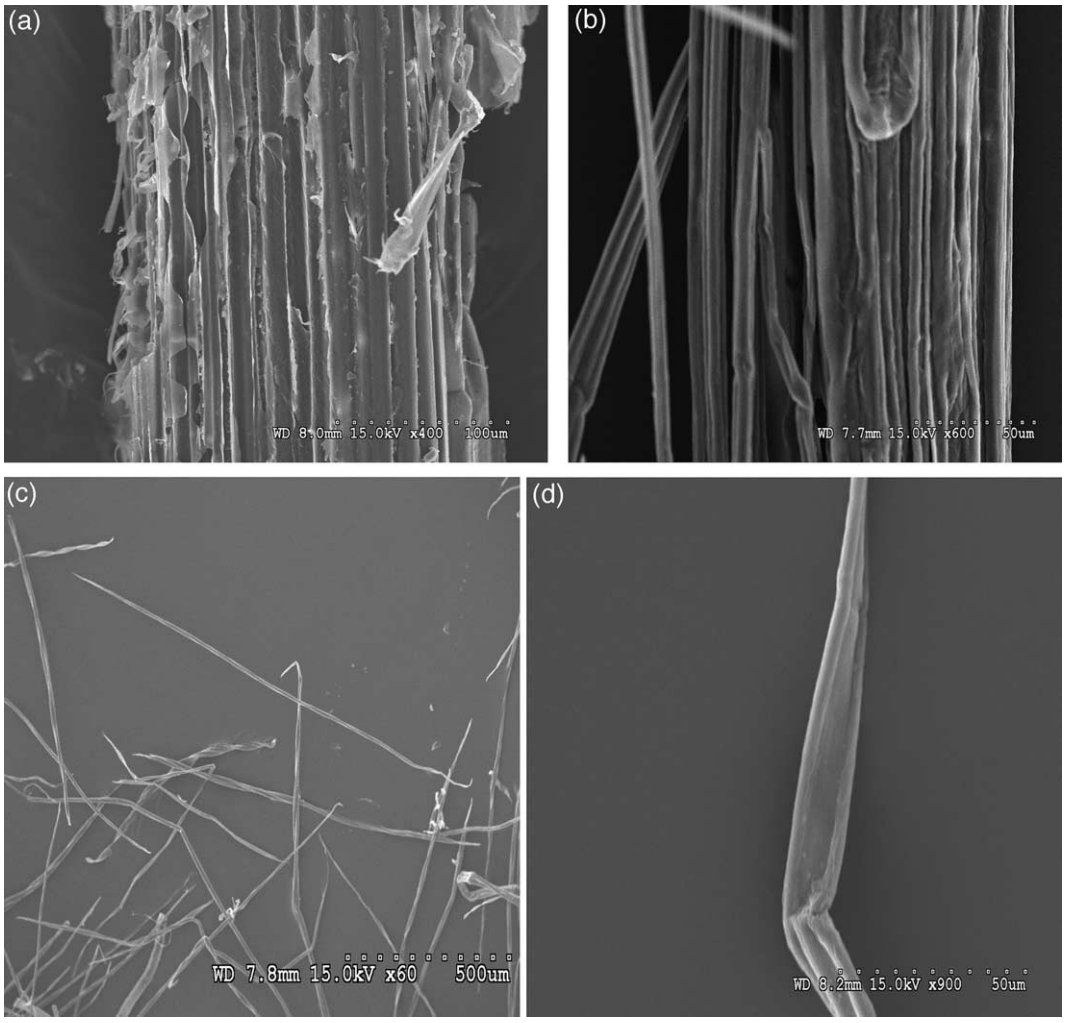

Fig. 2. (a) SEM picture of an untreated outside layer of cornstalk. (b) SEM picture of a cornstalk fiber obtained after alkali treatment. (c) SEM images of a unit cell in cornstalk. The higher magnification picture at the right shows the clean surface of the unit cell without any surface deposits.

and cotton (6–10%) [1,34]. Elongation of the fibers is mainly dependent on the MFA of the cellulose microfibrils in the fibers [29]. Cornstalk and flax fibers both have similar MFA of about 10° and, therefore, have similar elongation. Cotton has an MFA of about 30° and, therefore, higher elongation. Fig. 3 depicts the stress–strain curves for cornstalk, flax, kenaf and cotton from our laboratory. As shown in Fig. 3, cornstalk fibers have lower strength than flax and lower elongation than cotton and kenaf. However, the modulus of cornstalk fibers at about 127 g per denier is between that of flax (200) and cotton (50).

The lower modulus of cornstalk fibers than flax indicates that products made from cornstalk fibers will be more flexible and soft to hand than products from flax. In addition to the better flexibility and soft hand, cornstalk fibers also have good durability, measured in terms of the work of rupture. A higher work of rupture means a more durable fiber. Cornstalk fibers have work of rupture similar to kenaf (0.03 g per denier) but lower than that of cotton (0.13 g per denier) and flax (0.09 g per denier) [1]. Overall, the

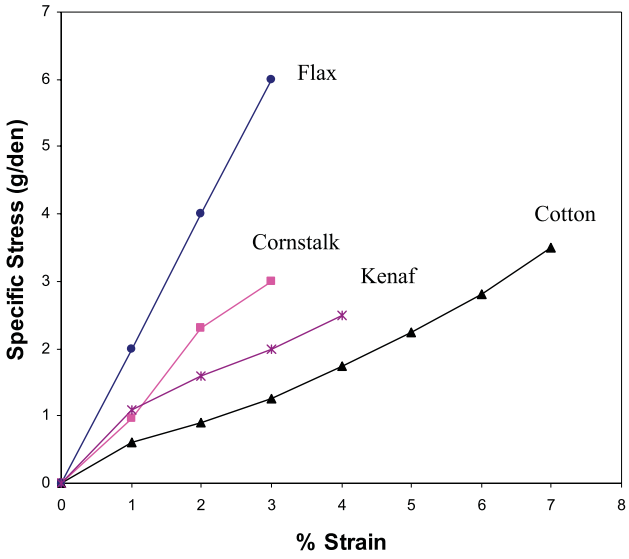

Fig. 3. Stress–strain curve for cornstalk fibers compared with cotton, flax, and kenaf.

mechanical properties of cornstalk fibers in terms of the denier, strength, elongation, and modulus are similar to that of kenaf. Kenaf fibers have been blended with cotton and processed on the conventional textile machinery to produce various textile products [34]. Therefore, cornstalk fibers may also be suitable for processing on the conventional textile machinery and also for blending with the other common fibers.

Moisture regain of cornstalk fibers at about 7.9% is similar to that of cotton but lower than flax (12%) and kenaf (17%), respectively, [1,34]. Having moisture absorption similar to that of cotton means products made from cornstalk fibers will be comfortable to wear.

4. Conclusions

Natural cellulose fibers obtained from cornstalks have the structure and properties required for textile and other industrial applications. The single cells of cellulose in cornstalks have dimensions smaller than those in flax and kenaf. Cellulose crystals in cornstalks have the typical cellulose I structure but the size of the crystals is smaller than that in cotton and larger than that in flax. Although cornstalk fibers have low crystallinity, the relatively high orientation of the microfibrils and crystals provides the fibers high strength but low elongation. The properties of the cornstalk fibers are similar to that of kenaf and, therefore, it is expected that cornstalk fibers would be suitable for blending and processing with other common textile fibers to produce various products.

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