

## Preparation and Characterization of Long Natural Cellulose Fibers from Wheat Straw

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Long natural cellulose fibers with properties suitable for textile and composite applications have been obtained from wheat straw. This study aims to understand the potential of using wheat straw as a source for long natural cellulose fibers for textile, composite and other fibrous applications. The presence of wax on the outer layer of the straw and a unique zip-like structure that locks individual fibers makes it difficult to obtain fibers from wheat straw using the common methods of fiber extraction. A novel pretreatment with detergent and mechanical force followed by an alkaline treatment was used to obtain high quality fiber bundles. The structure and properties of the fibers are reported in comparison to common cellulose fibers, cotton, linen, and kenaf. Wheat straw fibers have coarser (wider width) single cells and lower crystallinity than cotton, linen, and kenaf. The breaking tenacity (force at break) of wheat straw fibers is similar to kenaf but lower than that of cotton and linen, % breaking elongation is similar to linen and kenaf but lower than cotton, and Young's modulus of the fibers is similar to cotton but lower than that of linen and kenaf.

**KEYWORDS:** Cellulose; biofibers; wheat straw; fiber extraction

### INTRODUCTION

This paper reports the extraction and characterization of long natural cellulose fibers from wheat straw with properties suitable for composite and textile applications. Wheat straw is available in large quantities (about 540 million tons worldwide) at low cost (\$40 per ton) and is one of the most common lignocellulosic agricultural byproducts investigated as a potential source for pulp, paper, composite, and other industrial applications (1–5). Wheat straw could be a low cost, annually renewable, and environmentally friendly source for natural cellulose fibers that could replace at least a part of the traditional fibers in current use. Such efforts to develop alternative fiber sources could be crucial to the cost, availability, and sustainability of the fiber industry in the near future. This is because of the increasing consumption but decreasing cultivation of natural fibers and dependence on the nonrenewable petroleum resources for synthetic fibers. For example, cotton cultivation in the U.S. has decreased to about 13 million acres in 2005 from about 16 million acres in 1995, whereas cotton consumption worldwide increased by more than 1.7 million tons in 2005 (6). In addition, the increasing use of corn and soybeans for biofuels has led to higher income from these crops and decline in cotton cultivation (7). Cotton cultivation is reported to have an income of \$126 per acre including subsidies, whereas corn

cultivation currently has income of about \$403 per acre (7). These factors could lead to even further decline in cotton production and it will be inevitable to rely on alternative sources for fibers.

Several alternative fiber sources, especially agricultural byproducts, have been used to produce cellulose, protein, and synthetic fibers. Some of the examples include cornhusks, cornstalks, ricestraw, switchgrass, sorghum, pineapple leaves and sugar cane rind for cellulose fibers, wheat gluten, soyprotein and zein for protein fibers, and corn kernels for polylactic acid (PLA) fibers (8–17). However, the fibers obtained from several of these agricultural byproducts have relatively poor properties and efforts are being made to understand and improve the properties of these fibers through various means (15, 18, 19).

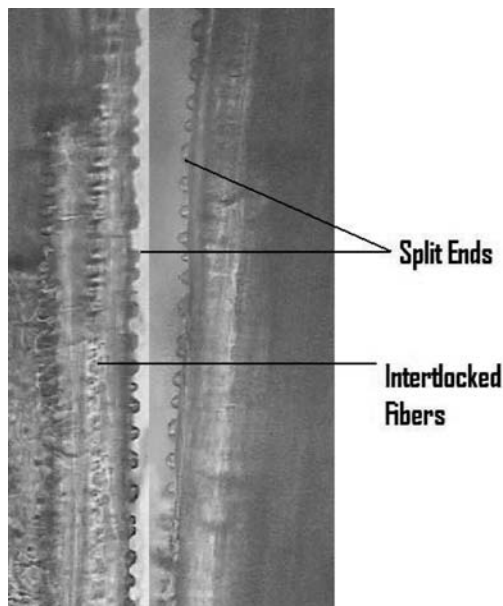
Although long natural cellulose fibers with the length, fineness, strength, and other properties similar to the natural cellulose fibers such as cotton and kenaf and to the fibers obtained from other lignocellulosic agricultural byproducts have not been produced from wheat straw, the structure, morphology, and properties of wheat straw and the single cells or ultimates obtained from wheat straw have been studied (20–23). Wheat straw has also been used to produce regenerated cellulose fibers, and the structure and properties of the fibers has been determined (24).

Fibers with lengths of at least 2 cm and with strength greater than 1 g per denier (130 MPa) are required for textile applications. It has not been previously possible to obtain long length fibers from wheat straw. Previous attempts on using wheat straw for paper and composites have either used wheat straw without purification or have hydrolyzed the straw

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**Figure 1.** A digital image of mechanically separated but untreated wheat straw shows the serrated split edges and the zip-like interlocking of the fibers in the straw.

**Table 1.** Composition (% on Dry Weight) of the Wheat, Cotton, Linen, and Kenaf Fibers; Data for Cotton, Linen, and Kenaf are from Refs 27–29

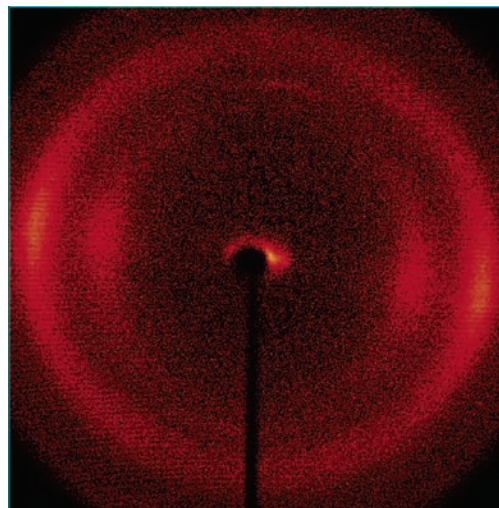
component, %	wheat straw	cotton	linen	kenaf
cellulose	64.8 ± 1.7	85–90	72–82	59–81
lignin	9.0 ± 0.7	0.7–1.6	2–3	15–19
ash	2.9 ± 0.3	0.8–2.0		2–5

**Table 2.** Morphology and Physical Structure of Wheat Straw Fibers Compared with Cotton, Linen, and Kenaf Fibers. Errors are ±1 Standard Deviation; Data for Cotton, Linen, and Kenaf are from Refs 27–29

component	wheat straw	cotton	linen	kenaf
single cell dimensions				
length, mm	0.9 ± 0.3	15–56	4–77	1.5–11
width, μm	12 ± 2.0	12–25	5–76	12–36
physical structure				
crystallinity, %	48	65–70	65–70	61–69
MFA, deg	17	20–30	15	10

into single cells. The single cells or ultimates are only a few millimeters in length and not suitable for high value fibrous applications such as textiles and composites. Longer length fibers provide better reinforcing and therefore higher strength to composites compared to short length fibers. Typically, long length fibers have a number of single cells held together to form a fiber bundle and are obtained from agricultural byproducts and basts or stems of plants. Fiber bundles are obtained through partial removal of the binding materials that hold the single cells together either by natural retting or by using chemical and/or enzymatic treatments. Such long length fibers have previously not been produced from wheat straw.

For the first time, we have developed a method of obtaining long length fiber bundles from wheat straw by removing the surface wax, disrupting the mechanical interlocking, and by partially removing the binding substances that hold the single cells together. A simple pretreatment using detergent and mechanical force followed by alkaline extraction has been used to obtain cellulose rich fiber bundles. The structure and properties of the fibers obtained has been studied in comparison to cotton, linen, and kenaf.



**Figure 2.** Diffraction patterns of wheat straw fibers show long and less intense diffraction arcs indicating poor orientation of cellulose crystals to the fiber axis.

## EXPERIMENTAL PROCEDURES

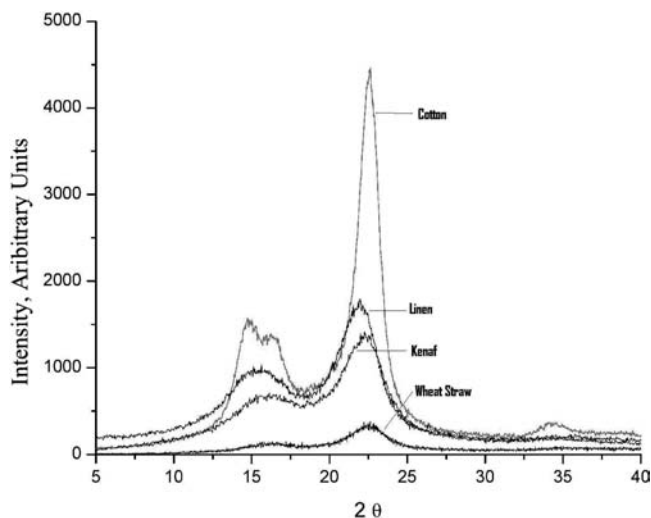
**Materials.** Straw from hard white winter wheat was obtained from a research field in Lincoln, Nebraska. The straw was used as is without any purification or pretreatments. Sodium hydroxide, acetic acid, chromic acid, and nitric acid were reagent grade chemicals purchased from VWR International, Bristol, CT. The data for cotton, linen, and kenaf are from the literature

**Fiber Extraction.** Several experiments were conducted to develop optimum conditions of fiber extraction. The optimization process included varying the pretreatment and extraction conditions such as the concentration of sodium hydroxide, time, and temperature of treatment and the liquor to material ratio used based on our previous experiences in obtaining fibers from various lignocellulosic sources. (8, 11, 12, 14) The optimization was based on the fineness and yield of the fibers obtained. Our aim was to obtain the finest possible fibers with the highest yield and with good tensile properties.

Unlike the lignocellulosic sources such as cornhusks, cornstalks, and rice straw, wheat straw has high resistance to alkali treatment. This resistance was found to be due to the presence of wax on the surface of the straw. Wheat straw was treated in a 0.2% (on weight of the straw) detergent (American Association of Textile Chemists and Colorists (AATCC) standard detergent, without optical brightener) solution in a laundrometer at 70 °C for 30 min with 20 steel balls weighing about 21 g to remove the wax on the surface of the straw. The steel balls were used to provide the mechanical force required to disrupt the zip like interlocking of the individual fibers. The treated straw was then subject to an alkali treatment using 2% sodium hydroxide solution at 95 °C for 45 min with a liquid to material ratio of 15 to 1. The treated straw was thoroughly washed in water to remove the dissolved substances. The fibers collected were neutralized using 3% (w/w) acetic acid solution and later washed and dried under ambient conditions. The air-dried fibers were then heated in an oven at 105 °C for 5 h. The dried fibers were allowed to equilibrate in a conditioning room maintained at standard conditions of 21 °C and 65% relative humidity for 24 h. The weight of the conditioned fibers was determined to know the % yield of the fibers based on the initial weight of the wheat straw used for extraction.

The wheat straw fibers were macerated to obtain the single cells in the fiber. Maceration was done using 10% (w/w) nitric acid and 10% (w/w) chromic acid solution. Fibers were dipped in equal volumes of the two solutions for about 24 h after initiating the reaction by heating the solution at 60 °C for 5 min (25).

**Fiber Composition.** The amount of cellulose in the wheat straw fibers was determined using the Acid Detergent Fiber (ADF) method according to AOAC method 973.18 (26). Lignin in the fibers was determined as Klason lignin according to ASTM standard method



**Figure 3.** Diffractograms of wheat straw fibers have similar peak positions but are less intense than cotton, linen, and kenaf fibers.

D1106–96. Ash in the fibers was determined according to ASTM standard E1755–01. Two replications were done for each compositional analysis and the average and one standard deviations are reported.

**Physical Structure.** The physical structure of the fibers was studied in terms of the % crystallinity, crystallinity index (CI), multifibrillar angle (MFA), and the orientation of the cellulose crystals by observing the diffraction patterns of the fibers. Two types of X-ray diffraction machines were used to determine the physical structure of the fibers. 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 determine the % crystallinity and CI. These measurements were taken for a  $2\theta$  range from 5 to 45° on fibers that were made into pellets of about 5 mm thick. To make the pellets, fibers were powdered in a Wiley mesh to pass through a 250  $\mu\text{m}$  mesh and the powdered fibers were pressed into a pellet using a hydraulic press operated at about 12000 PSI.

A Bruker D8 Discover model diffractometer equipped with an area detector and GAADS software was used to obtain the diffraction patterns and calculate the MFA. To obtain these measurements, a parallel bundle of fibers was mounted vertically in a specially designed sample holder with the axis of the fiber perpendicular to the X-ray beam. To obtain the MFA, the 002 peak intensities in the diffraction patterns were fit into two Gaussian curves using a nonlinear least square algorithm using the software program Microcal ORIGIN.

**Morphological Structure.** A Hitachi S3000N model variable pressure scanning electron microscope (SEM) was used to observe the morphological features of the untreated wheat straw and the fibers and single cells obtained. The specimens to be observed were mounted on conductive adhesive tape, sputter coated with gold palladium and observed in the SEM using a voltage of 15 kV. The lengths of the single cells were measured using a Motic image plus digital microscope. About 100 single cells were measured for their length and the average and standard deviations are reported. The widths of the single cells were measured from the SEM pictures.

**Tensile Properties.** Length of about 100 fibers was measured using a ruler, and the average and standard deviations were calculated. Denier (weight in grams per 9000 m of the fibers) of the fibers was determined by weighing the known length of fibers. All the tensile tests were performed after conditioning the samples for at least 24 h in a standard conditioning atmosphere of 21 °C and 65% relative humidity. The tensile properties of the fibers in terms of the strength, % elongation and modulus were determined using an Instron (model 4000) tensile testing machine. A gauge length of 25 mm and a crosshead speed of 18 mm/min were used for the testing. About 100 fibers were tested and the average and standard deviations are reported.

**Moisture Regain.** The moisture regain of the wheat straw fibers was determined according to ASTM method 2654.

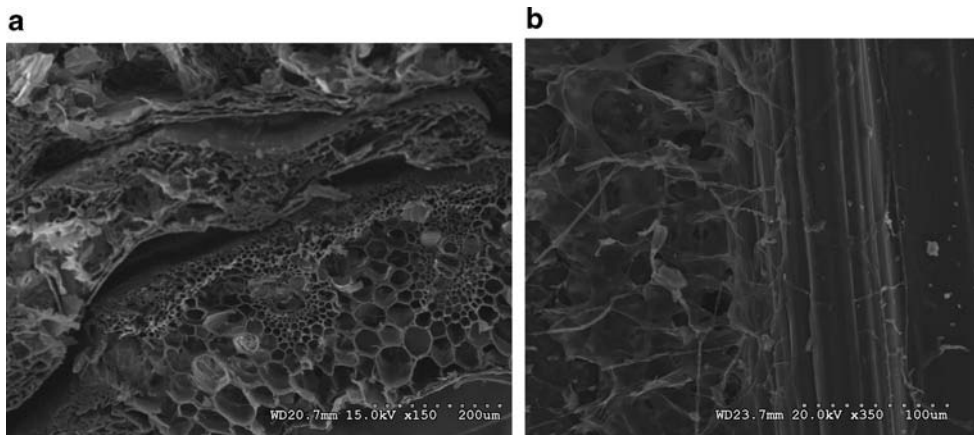
## RESULTS AND DISCUSSION

**Fiber Extraction.** Figure 1 depicts the unique interlock structure in wheat straw that is not seen in other agricultural byproducts such as cornstalks, rice straw, and sugar cane. (8, 11–14) As seen from the figure, the serrations at the separated edges lock into each other and form a zip-like interlock structure that is difficult to be separated using normal alkaline and/or enzyme treatments. The interlock structure is said to provide better longitudinal and lateral strength to the fibers (23, 27, 28). It is necessary to disrupt this interlock structure and remove most of the wax on the surface of the wheat straw to allow the alkali to penetrate the straw, remove the noncellulosic substances and produce high quality fiber bundles that have lengths greater than 2 cm and strength greater than 1 g per denier. The detergent and the mechanical treatment are able to remove the wax and disrupt the interlock structure without damaging the long fibers in the straw. Although high concentrations of alkali alone can saponify the wax and remove the noncellulosic substances, strong alkali will damage the cellulose fibers.

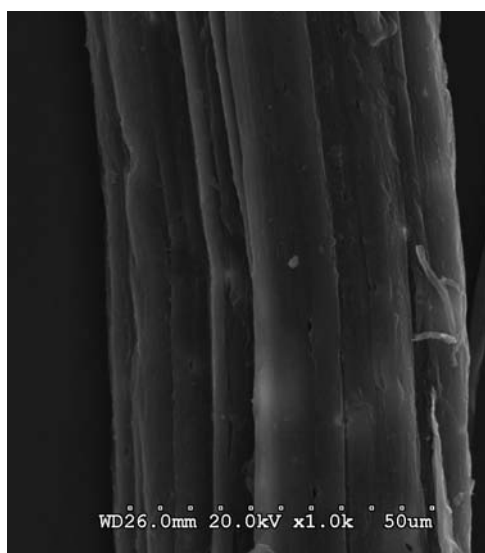
At the optimized condition of fiber extraction, about 20% of the wheat straw used can be obtained as long fibers with the properties described in this research. Although it is possible to obtain higher yields from the straw, the fibers from higher yields will be coarser than obtained at the optimized condition. The fiber extraction conditions not only affect the yield and quality but determine the composition of the fibers as well. Stronger extraction conditions are expected to remove more of the hemicellulose and lignin and provide fibers with higher cellulose contents. However, excess removal of noncellulosic substances is also not desirable since the noncellulosics are required to bind the single cells together and form a fiber bundle.

**Fiber Composition.** The compositions of the fibers obtained from wheat straw are compared to the composition of cotton, linen, and kenaf fibers in Table 1. Wheat straw fibers have lower cellulose content than cotton and linen but in the range of cellulose content reported for kenaf (29–31). As mentioned earlier, the inherent composition of the straw and extraction conditions employed are the major factors contributing to the cellulose content of the fibers. However, cotton is a single cell fiber whereas wheat, linen and kenaf are multicellular fibers. The multicellular fibers need noncellulosic substances such as lignin to hold the single cells together and form a fiber bundle. The lignin content in wheat straw fibers is lower than the lignin content of kenaf but higher than that in linen. Generally, bast fibers such as kenaf are lignified to a higher extent than lignocelluloses from agricultural byproducts such as wheat straw, rice straw and cornhusks, and therefore, the fibers from the byproducts have lower lignin contents than some of the common bast fibers (8, 11, 12, 14). The ash content in wheat fibers is similar to cotton but lower than that of kenaf fibers.

**Physical Structure.** The physical structure of wheat straw fibers in terms of % crystallinity and MFA are given in Table 2 in comparison to cotton, linen, and kenaf. As seen from the table, the wheat straw fibers have lower % crystallinity than the other three fibers. Previous studies on the structure of cellulose in wheat straw have reported % crystallinity in the range of 43–47% for cellulose obtained from various parts of wheat straw (23). Since wheat straw fibers have lower cellulose content and crystallinity, wheat straw fibers may have lower strength but better moisture and chemical absorptions compared to cotton, linen, and kenaf. The cellulose crystals in wheat straw fibers are poorly oriented along the fiber axis compared to cotton and linen as indicated by the long and less intense diffraction



**Figure 4.** SEM picture of the cross-section (a) shows cellular cross-section and longitudinal surface (b) shows the rough outer surface.



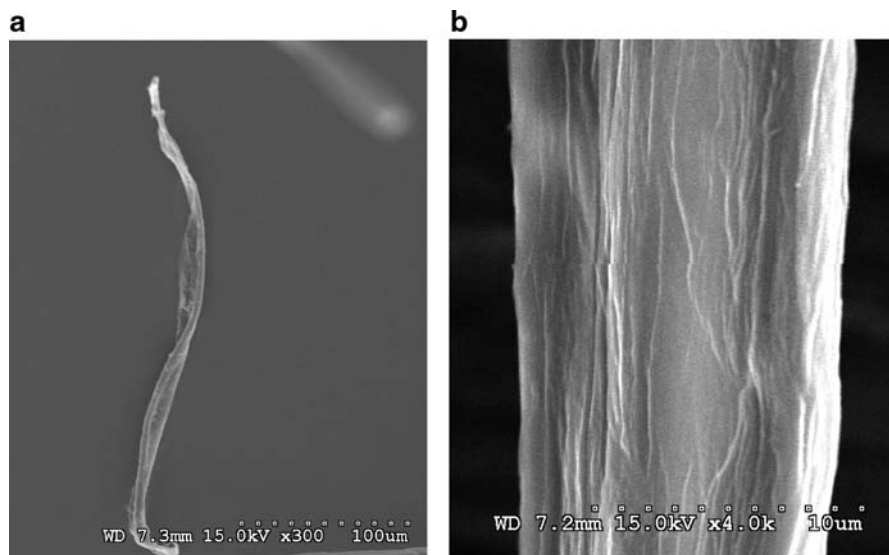
**Figure 5.** SEM picture of the cross-section (a) shows cellular cross-section and longitudinal surface (b) shows the rough outer surface.

patterns shown in **Figure 2**. However, the cellulose crystals in wheat straw fibers have better orientation that could provide relatively higher strength to wheat straw fibers compared to cornhusk fibers (8, 11).

**Figure 3** shows the diffractogram of wheat straw fibers compared to cotton, linen, and kenaf fibers. As seen from the figure, the wheat straw fibers have very similar diffraction pattern to that of linen and kenaf, but the intensity of the peaks produced by wheat straw fibers is lower than that of cotton, linen, and kenaf. All the fibers have the prominent cellulose peak at a  $2\theta$  angle of  $22^\circ$  representing the 002 plane. However, the characteristic 101 and  $10\bar{1}$  peaks found in cotton ( $2\theta$  between 15 and 17 degrees) are not distinct in wheat, linen, or kenaf fibers. Instead, the two peaks have combined into one broad peak as seen from **Figure 3**. This is said to be due to the presence of noncellulosic substances in the fibers (30).

**Morphological Structure.** Single cells in wheat straw fibers are shorter than in cotton, linen, and kenaf fibers as given in **Table 2**. However, the dimensions of the single cells will be dependent on the particular variety of straw used. Wheat straw has been reported to have single cell lengths and widths in the range of 0.4–3.2 mm and 8–34  $\mu\text{m}$ , respectively. (5) Recently, an average single cell length of 1.9 mm and diameter of 94  $\mu\text{m}$  has been reported for mechanically processed wheat straw fibers and a single cell length and diameter of 1.5 mm and 84  $\mu\text{m}$  for microbial retted fibers (21).

**Figures 4, 5, and 6** show the morphological features of wheat straw, the fibers, and single cells obtained from wheat straw. Like all the other lignocellulosic sources, wheat straw also has

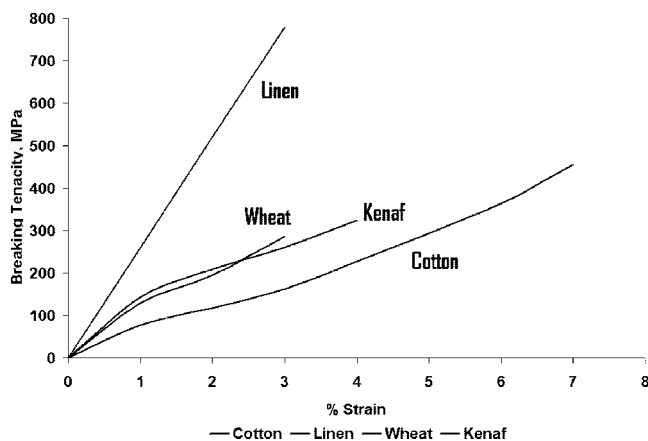


**Figure 6.** Single cells in wheat straw fibers (a) show convolutions and a higher magnification image of the single cells (b) shows a smooth and clean surface.

**Table 3.** Tensile Properties and Moisture Regain of Wheat Straw Fibers Compared Cotton, Linen and Kenaf Fibers<sup>a</sup>

fiber properties	wheat straw	cotton	linen	kenaf
denier	35–100	3–8	1.7–17.8	50
length, cm	4–8	1.5–5.6	20–140	150–180
strength, MPa	273 ± 26	351–455	598–793	130–299
elongation, %	2.7 ± 0.1	6.0–9.0	1.6–3.3	1.3–5.5
modulus, GPa	13 ± 1.5	7.1–11.7	26	12–30
moisture regain, %	9.5	7.5	12.0	9.5–10.5

<sup>a</sup> Errors are ± one standard deviations. Data for cotton, linen and kenaf are from references 27–29.

**Figure 7.** Stress–strain curves of wheat straw fibers compared to cotton, linen, and kenaf.

a thick layer of surface deposits on the surface mostly composed of lignin and hemicellulose that protect the cellulose fibers inside (Sun, 2004). However unlike other straws, wheat straw has a layer of hydrophobic wax on the outside surface that protects the straw from degradation to moisture (27). The alkaline treatment removes most of the surface material resulting in fibers that have a relatively clean surface as seen from **Figure 5**. The fiber has a few holes on its surface. These holes assist in the ventilation and metabolism for the wheat straw plants (23, 29, 30). The single cells in the wheat straw fibers have natural convolutions and tapered ends as seen from **Figure 6a** and a smooth surface as seen from the higher magnification image in **Figure 6b**.

**Fiber Properties.** **Table 3** gives the mechanical properties and **Figure 7** shows the stress–strain properties of wheat straw fibers in comparison to cotton, linen, and kenaf. Wheat straw fibers have similar lengths to cotton but are much coarser than cotton and linen mainly due to the relatively shorter and wider width single cells in wheat straw fibers. The wheat straw fibers have lower breaking tenacity than cotton and linen but similar to kenaf fibers. The % breaking elongation of the wheat straw fibers is similar to both linen and kenaf but all the three fibers have lower % elongation than cotton. Cotton has higher MFA than wheat, linen, and kenaf fibers and therefore has higher % breaking elongation. The wheat straw fibers have low modulus similar to cotton but lower than linen and kenaf fibers. The lower modulus of wheat straw fibers compared to linen and kenaf fibers indicates that the wheat straw fibers will be more soft and flexible than linen and kenaf fibers. Although the wheat straw fibers have lower % crystallinity, the moisture regain of the fibers is similar to that of linen and kenaf and higher than cotton.

**Potential Applications.** The length of the wheat straw fibers indicates that the fibers are suitable for processing on the short staple spinning machines to develop textile products. However,

the fibers are coarser than cotton and linen and may not be suitable for producing fine yarns for high value applications such as apparels. Wheat straw fibers have the minimum strength (1 g per denier, 130 MPa) and % breaking elongation (1.5–2%) of commercial fibers currently in use. The Young's modulus and moisture regain are similar to common natural cellulose fibers. Overall, wheat straw fibers have the properties required for textile and composite applications.

Wheat straw shows potential to be a cheap, abundant, and annually renewable source for long natural cellulose fibers for textile and composite applications. The wax layer on the surface of wheat straw needs to be removed and the interlock structure that locks single cells together needs to be disrupted to enable the alkali to partially remove the noncellulosic substances and produce cellulose rich fiber bundles. The fibers obtained from wheat straw have similar cellulose and lignin contents but lower ash content than kenaf fibers. Wheat straw fibers have low modulus and good % elongation that could provide flexibility to products. Utilizing wheat straw as a source for natural cellulose fibers will add value to wheat crops, provide a sustainable base for fibers, and benefit the environment.

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