

## Novel Protein Fibers from Wheat Gluten

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Protein fibers with mechanical properties similar to those of wool and better than those of soyprotein and zein fibers have been produced from 100% wheat gluten. Wheat gluten is a low cost, abundantly available, and renewable resource suitable for fiber production. A simple production method has been developed to obtain high-quality wheat gluten fibers, and the structure and properties of the fibers have been studied. Wheat gluten fibers have breaking tenacity of about 115 MPa, breaking elongation of 23%, and a Young's modulus of 5 GPa, similar to those of wool. Wheat gluten fibers have better tensile properties than soyprotein- and casein-based biomaterials. In addition, the wheat gluten fibers have resistance similar to that of PLA fibers to water in weak alkaline and slightly lower resistance in weak acidic conditions at high temperatures.

### Introduction

This paper reports the first successful use of wheat gluten to produce fibers with excellent mechanical properties and with good stability to weak acids and weak alkalis at high temperatures. With a selling price of less than \$0.50 per pound, and a worldwide availability of 500 000 tons every year, wheat gluten is a cheap, abundant, and renewable source for producing protein fibers.<sup>1–3</sup> In addition, wheat gluten has good stability to water and heat, excellent elasticity, and easy degradability, properties that are desirable for fibers.<sup>3–5</sup> The annual world fiber market is about 67 million tons including about 2.3 million tons of the two natural protein fibers, wool and silk.<sup>6</sup> Wool and silk have selling prices of about \$5–8 and \$10–14 per pound, respectively.<sup>6</sup> Therefore, fibrous applications provide an opportunity for high value addition and also offer a large market for consumption of wheat gluten.

Several reports are available on the production of natural cellulose fibers from lignocellulosic agricultural byproducts in an effort to add value to agricultural crops and to make the fiber industry more sustainable in the long run.<sup>7–11</sup> Similarly, attempts have also been made to use plant proteins such as soybeans, corn, and peanut and also milk proteins (casein) for fiber production. Reports are available on the commercial scale production of fibers from these sources during the 1930s and 1940s.<sup>12–14</sup> The higher cost, use of relatively environmentally unfriendly production processes, and inferior properties of the regenerated protein fibers as compared to those of the regenerated cellulose and synthetic fibers led to the abandoning of artificial protein fiber production. However, the increasing use of cereal grains for biofuels and other industrial applications has led to the abundant availability of zein, soyprotein, and also wheat gluten as byproducts at low prices. Therefore, researchers have more recently attempted to produce fibers from zein, casein, and soyprotein and in addition from the blends of these proteins.<sup>15–19</sup> Unfortunately, none of these attempts have been commercially successful to produce 100% protein fibers mainly due to the high cost and poor quality of the fibers.

In addition to fibers, plant proteins including soyproteins and zein and casein have been used for biomedical applications such as temporary replacement implants, tissue engineering scaffolding, and drug delivery carriers.<sup>20</sup> It is said that proteins are ideal templates for biomaterials due to their ease of processability, adhesion to various substrates, and surface active properties.<sup>20</sup> However, soyprotein- and casein-based biomaterials are reported to have poor mechanical properties and hydrolytic stability.<sup>20,21</sup> Similarly, polylactic acid (PLA)-based fibers used for biomaterials have also been reported to have poor stability in water and bulk erosion, resulting in reduced pH that could affect the proteins in the body.<sup>21</sup>

Although wheat gluten has previously not been used for fiber production, it has been used to produce bioplastics especially as films for food packing, as a binder for textile printing pastes, and as nanofibers via the electro-spinning process.<sup>3,5,22–25</sup> The major limitations of the wheat gluten films are their relatively poor mechanical properties and higher cost as compared to those of the synthetic polymer-based films.<sup>4</sup> Using wheat gluten as a binder is relatively expensive and also has limited market potential.

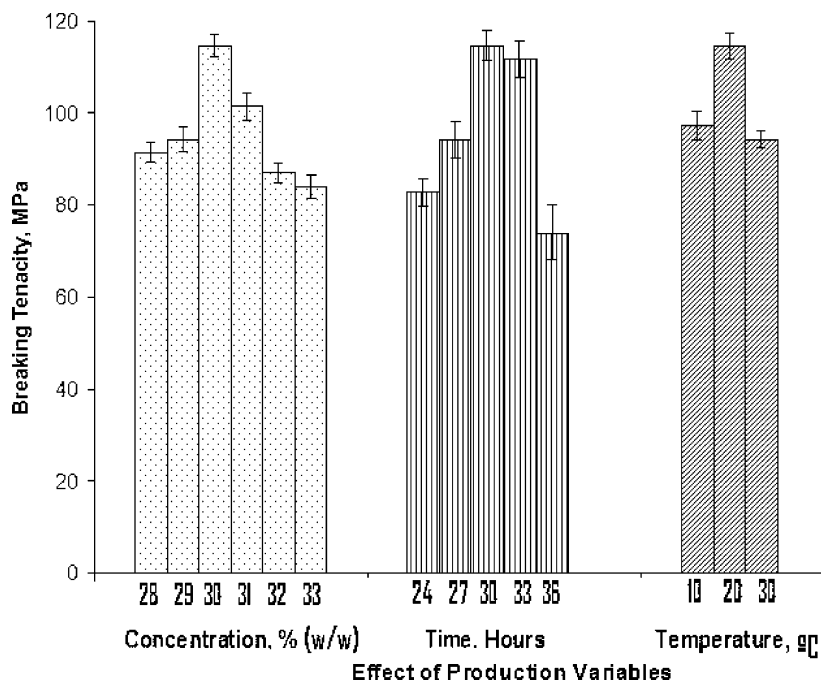
To the best of our knowledge, no reports are available on the use of wheat gluten for biomedical applications similar to the other protein-based materials mentioned above. However, wheat gluten fibers could be an interesting material for biomedical applications such as implants, sutures, scaffolds, and matrices for drug delivery and could overcome the deficiencies of biomaterials produced from zein, soyprotein, casein, and also PLA fibers.<sup>26–29</sup> Also, wheat gluten fibers have resistance similar to that of PLA fibers to water in weak alkaline and slightly lower resistance in weak acidic conditions at high temperatures.

In this paper, we discuss the method of producing 100% wheat gluten fibers, the effect of various production variables on the properties of the fibers, and the structure and properties of the fibers developed. The structure and properties of the fibers have been compared to the most common natural protein fiber, wool, and also to protein fibers produced from 100% zein and soyproteins.

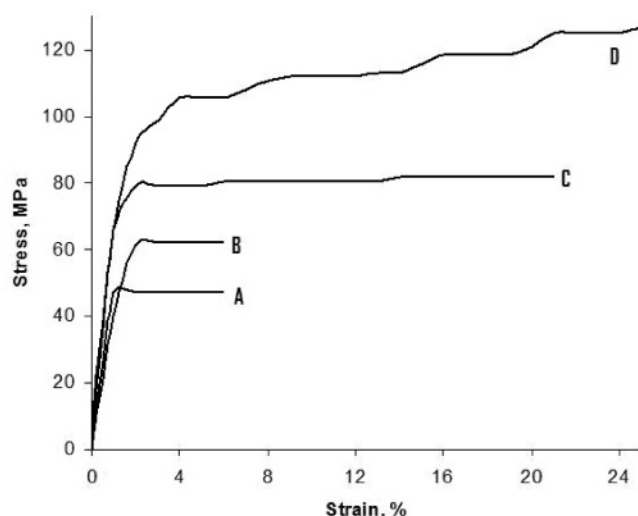
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**Figure 1.** Effect of wheat gluten concentration in, and aging time and temperature of the spinning solution on the breaking tenacity of the spun fibers. Error bars represent standard error of the means.



**Figure 2.** Effect of drawing and dry heating of the solid fibers (annealing) on the stress-strain behavior of wheat gluten fibers: (A) are undrawn and unheated fibers; (B) undrawn heated (125 °C for 90 min) fibers; (C) drawn (about 250% of the initial length) and unheated fibers; and (D) drawn (about 250%) and annealed (125 °C for 90 min) fibers.

### Experimental Section

**Materials.** Commercially available wheat gluten, Whetpro 80 with about 80% protein content, was a gift from Archer Daniels Midland Co., Decatur, IL. Urea, sodium sulfite, and sodium sulfate were reagent grade chemicals obtained from VWR International, Bristol, CT. Polylactic acid (PLA) fibers used for the water stability study were obtained from Fiber Innovation Technology Inc., Johnson City, TN. The PLA fibers were of 7 denier with an average breaking tenacity of 2.7 grams per denier.

**Fiber Extrusion.** Protein solutions with wheat gluten concentrations of 28–32% (w/w) were prepared by dissolving wheat gluten using 8 M urea solution as a swelling agent and 1% (w/w) sodium sulfite on total weight of the bath as the reducing agent. The wheat gluten solutions were aged for various times (12–36 h) at three different temperatures (10, 20, and 30 °C) to study the effect of protein

concentration, time, and temperature of aging on fiber properties. Fibers were extruded from the aged solution into a coagulation bath consisting of 10% (w/w) sodium sulfate and 10% (w/w) sulfuric acid using a normal syringe and needle. The fibers formed were allowed to stay in the coagulation bath for about 15 min and later rinsed in warm water and air-dried.

**Drawing.** The fibers formed were dipped in water and drawn by hand to about 200–300% of their original length.

**Annealing.** The dry fibers obtained before and after drawing were dried in an oven at 85 °C for 1 h and later annealed at 125 °C for about 1 h. Annealing was done to improve the mechanical properties of the fibers.

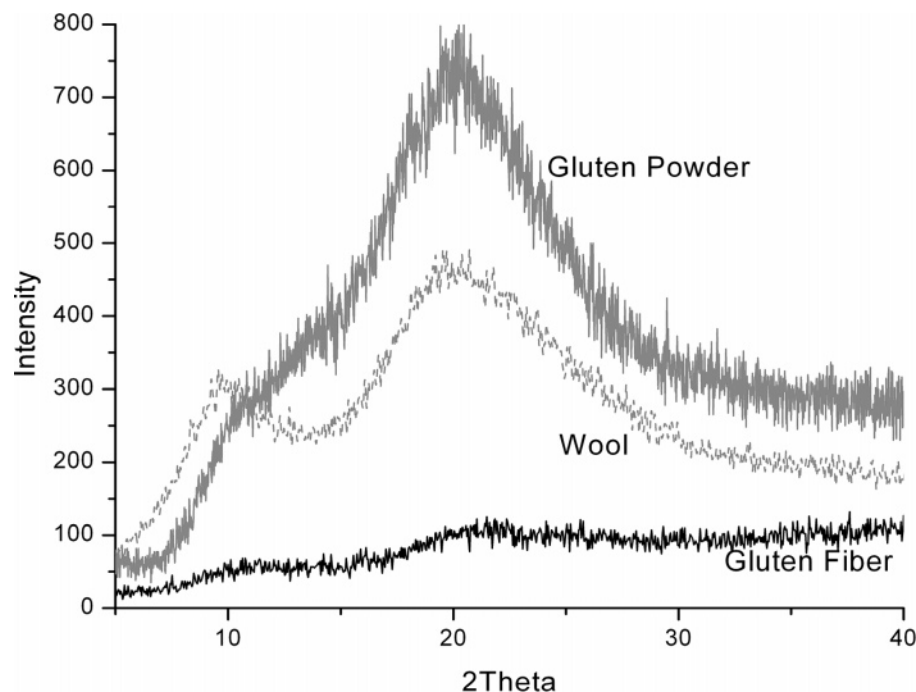
**Hot Water Stability.** Wheat gluten and PLA fibers were heated in water at 90 °C for 60 min in various pH (3, 5, 7, 9, and 11) conditions to test their stability. The hot water treated fibers were tested and compared to the untreated controls for the changes in tensile properties. After the fibers were heated in water, the fibers were conditioned for 24 h at standard testing conditions of 21 °C and 65% relative humidity. About 50 fibers were tested for each condition, and the average and standard error of the means are reported.

**Fiber Fineness.** The fineness of the fibers was measured in terms of denier, which is defined as the weight in grams of 9000 m of the fibers. The weight of a known length of about 50 wheat gluten fibers was measured to calculate the fiber fineness.

**Tensile Testing.** All of the fiber samples were conditioned before testing in a standard testing atmosphere of 21 °C and 65% relative humidity. Fibers were tested for their tensile properties using an Instron tensile tester. A gauge length of 1 in. and a crosshead speed of 18 mm/min were used for tensile testing. About 50 fibers were tested for each condition, and the average and standard error for the means are reported.

**Morphological Structure.** The morphological features in terms of the longitudinal and cross-sectional appearance of the wheat gluten fibers were observed using a Hitachi model S3000 N scanning electron microscope. A coarse fiber of about 100 denier was intentionally selected to observe the morphological features. Samples were sputter coated with gold palladium, mounted on conductive adhesive tapes, and observed under the microscope at a voltage of 15 kV.

**Physical Structure.** The physical structure of wheat gluten and wheat gluten fibers in terms of % crystallinity was measured using X-ray diffraction. A Rigaku D-Max/B Θ/2Θ X-ray diffractometer with



**Figure 3.** Diffractograms of wheat gluten powder and wheat gluten fibers as compared to wool.

Bragg–Brentano parafocusing geometry, a diffracted beam monochromator, and a copper target X-ray tube set to 40 kV and 30 mA was used for the X-ray measurements. The measurements were taken on powdered pellets made by grinding the fibers to pass through a 250  $\mu\text{m}$  mesh and pressing the powder into a pellet using a hydraulic press operated at about 20 000 psi. The % crystallinity of the samples was calculated as the ratio of the integral area under the crystalline peaks to the total area after subtracting the background and air scatter using the software Microcal ORIGIN.<sup>30</sup> A Bruker D8 Discover model diffractometer equipped with an area detector and GADDS software was used to obtain two-dimensional transmission diffraction patterns of the fibers. A bundle of fibers were mounted on a specially designed sample holder perpendicular to the X-ray beam to obtain the diffraction patterns.

**Moisture Regain.** The moisture regain of the fibers was studied according to ASTM method 2654 under standard atmospheric conditions of 65% relative humidity and 21 °C.

## Results and Discussion

**Fiber Production.** The effect of wheat gluten concentration in the solution, time of aging, and temperature of aging on the breaking tenacity of the wheat gluten fibers is shown in Figure 1. As seen from Figure 1, the properties of the fibers are influenced by the concentration of proteins in the solution and the time and temperature of aging the solution. Fibers produced from solutions with wheat gluten concentrations of less than 25% and/or aging time of less than 24 h have poor drawability. Solutions with wheat gluten concentrations between 25% and 27% had relatively good drawability, but the fibers had poor mechanical properties. Increasing the concentration of the wheat gluten in the solution above 27% to 33% gave the solutions excellent drawability, and the fibers obtained had good properties. As seen from Figure 1, fibers with the highest breaking tenacity were obtained from a 30% wheat gluten solution. Temperature of aging influenced the aging time, spinnability of the protein solution, and the breaking tenacity of the fibers. Generally, higher temperatures decreased the aging time and also gave solutions better spinnability. Of the three temperatures studied, the solutions aged at 20 °C produced fibers that had

about 15% higher breaking tenacity than the fibers aged at 10 and 30 °C with a wheat gluten concentration of 30% and aging time of 32 h as shown in Figure 1.

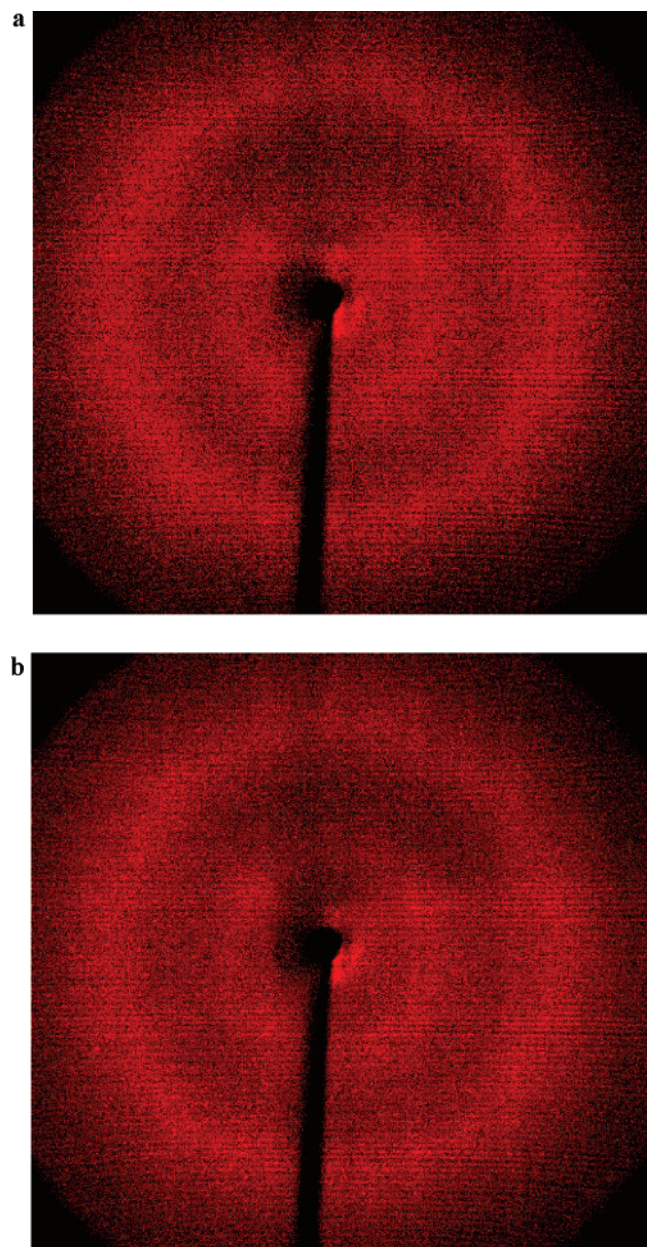
Solutions with wheat gluten concentrations studied here needed at least 24 h of aging to form a viscous and spinnable solution. Increasing the solutions aging time above 24 h and to 30 h increased the breaking tenacity of the fibers, and above 33 h of aging the breaking tenacity of the fibers decreases gradually. A solution aging time between 30 and 33 h was found to produce fibers with relatively high breaking tenacity as seen from Figure 1. Based on the concentration of wheat gluten, time, and temperature of aging studied in this research, a wheat gluten concentration of 30% and an aging time between 30 and 33 h at 20 °C was found to be the most optimum condition to obtain fibers with relatively high breaking tenacity without affecting other properties of the fibers.

As observed in this study, a narrow range of concentration of the protein solution, aging time, and temperature is necessary to obtain a viscoelastic and spinnable solution that can produce good quality fibers. This is because too low of an aging time and/or temperature does not break the natural cross-links in the proteins to the required extent and therefore results in a solution with poor drawability. On the other hand, too high of a time and/or temperature will result in excessive breaking of the polymers also resulting in poor drawability.

**Effect of Drawing and Annealing.** The effect of drawing and annealing (dry heating of the solid fiber) on the stress–strain properties of the fibers is shown in Figure 2. Drawing increased the breaking tenacity of the fibers by about 72% and the breaking elongation of the fibers by about 250% (curves a and c in Figure 2). Drawing increases the length, decreases the diameter, and also aligns the polymers in the fibers toward the axis of the fiber. The better alignment of the polymers in the fibers is expected to improve the breaking tenacity and breaking elongation of the fibers.

Generally, drawing increases strength by improving the alignment and interaction of the polymers inside the fiber, and the better alignment decreases the breaking elongation of fibers. However, we see an increase in both the tenacity and the

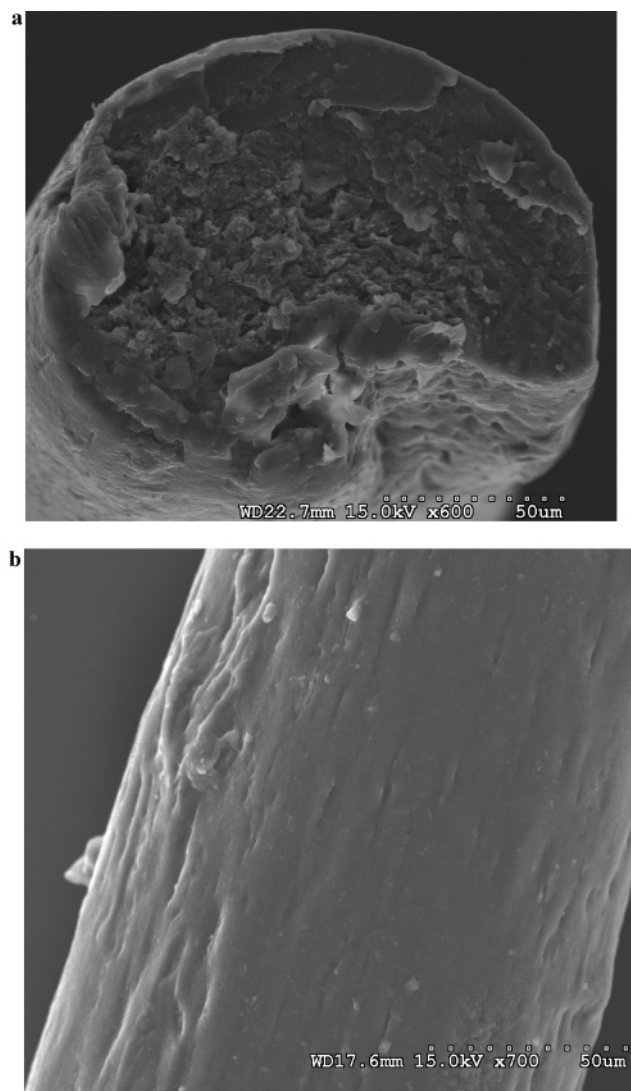




**Figure 4.** (a) Diffraction pattern of undrawn and unheated wheat gluten fiber shows the absence of strong diffracting arcs indicating poor orientation of proteins crystals in the fibers; (b) diffraction pattern of drawn and annealed wheat gluten fiber also shows no increase in the orientation of the protein crystals as compared to (a).

breaking elongation of wheat gluten fibers after drawing. This is due to the fact that drawing of wheat gluten fibers is done in water. In the swollen state, polymers in the wheat gluten fibers have a better chance of getting aligned along the fiber axis as compared to fibers drawn in a dry state. The better aligned polymers can stretch more during tensile testing, and therefore the breaking elongation of the fibers drawn in a wet state increases after drawing.

Annealing (dry heating) increased the breaking tenacity of the undrawn and drawn fibers by about 32% and 54%, respectively. Although annealing did not improve the breaking elongation of the undrawn fibers, an increase in breaking elongation of about 19% after annealing was observed for the drawn fibers. Annealing of wheat gluten has been reported to enhance the S–S interactions in wheat gluten and therefore improves the breaking tenacity of the fibers.<sup>32</sup> It is also reported that annealing forms new disulfide bonds and cross-links the



**Figure 5.** (a) SEM image of the cross-section of a wheat gluten fiber shows a solid structure; (b) SEM image of the surface of the wheat gluten fiber shows it is smooth.

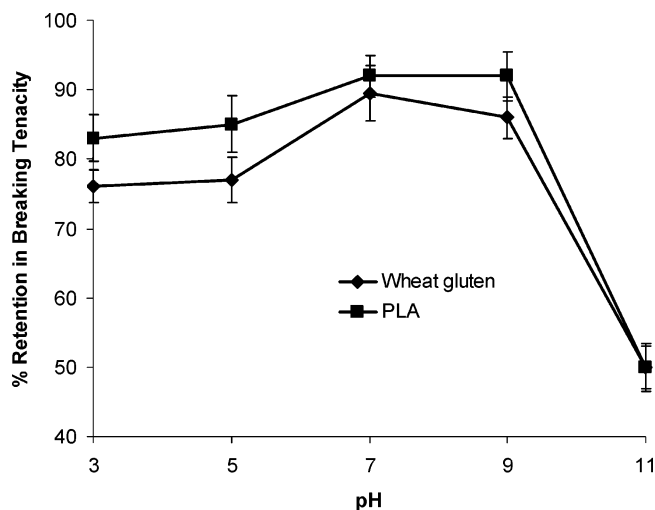
**Table 1.** Tensile Properties of Wheat Gluten Fibers As Compared to Soyprotein, Zein, and Wool<sup>a</sup>

fiber	denier	breaking tenacity, MPa	breaking elongation, %	modulus, GPa	moisture regain, %
wheat gluten	34 ± 4	115 ± 7	23 ± 2.7	5 ± 0.2	18
soyprotein		37–104	0.4–5.9		
zein		36–60	1.8–5.0		
wool	8–15	174–260	30–40	4.3–6.5	16

<sup>a</sup> Data for soyprotein, zein, and wool are from refs 12, 15, and 16. The data are for the fibers without chemical cross-linking treatments, and errors represent standard error of the means.

protein molecules and also increases the hydrophobic interaction by unfolding the protein molecules leading to higher breaking tenacity.<sup>2</sup>

**Physical Structure.** The wheat gluten fibers obtained in this research have a percent crystallinity of about 20%, lower than the crystallinity of wool fibers (26%), as seen from Figure 3. However, the wheat gluten powder has about 35% crystallinity. The lower crystallinity of wheat gluten fibers as compared to the gluten powder is probably due to the denaturing of the protein polymers during dissolution. The possible hydrolysis of protein molecules during fiber production and the presence of impurities and different proteins in wheat gluten result in a



**Figure 6.** % retention in dry strength of drawn and annealed wheat gluten and PLA fibers after heating at various pH conditions in water at 90 °C for 1 h. The tensile tests of the water treated fibers were performed after conditioning the fibers at 21 °C and 65% relative humidity for 24 h. Error bars are standard error of the means.

broad and diffuse diffracting pattern for the wheat gluten fibers. It is difficult and also inaccurate to index the various peak positions and obtain the crystal structure of the proteins in the fibers from such diffracting patterns. We therefore have not attempted to elucidate the crystal structure of the proteins in wheat gluten or the fibers.

In addition to the percent crystallinity, the orientation of the protein polymers along the axis of the fiber contributes to the properties of the fibers. Fibers with oriented polymers are expected to show sharp, bright, and short diffracting arcs and provide better tensile strength. As seen from Figure 4a and b, the undrawn and the drawn wheat gluten fibers do not show any diffracting arcs, indicating poor orientation of the polymers in the fibers. Similarly, regenerated protein fibers produced from soybean and zein do not show any preferred orientation either.<sup>15,18</sup> The low % crystallinity and the poor orientation of the protein polymers with respect to the fiber axis should be the main reasons for the relatively low breaking tenacity of the regenerated protein fibers as compared to the natural protein fibers wool and silk.

**Morphological Structure.** The morphological features of wheat gluten fibers are shown in Figure 5a and b. Wheat gluten fibers obtained in this research have a solid and smooth surface with a circular cross-section. The fineness and length of wheat gluten fibers are compared to those of soyprotein, zein, and wool in Table 1. Wheat gluten fibers have fineness similar to that of wool, but the wheat gluten fibers reported here are produced using a syringe and needle. Wheat gluten fibers with deniers similar or even finer than that of wool should be producible using a fiber extrusion system with fine spinnerets. The length of wheat gluten fibers can be controlled to the required extent depending on the end use application and processing method chosen to develop products from wheat gluten fibers.

**Tensile Properties.** The tensile properties of wheat gluten fibers are compared to those of soyprotein, zein, and wool in Table 1. The breaking tenacity of wheat gluten fibers is about 75% of that of wool but about 10% and 50% higher than the tenacity of 100% soyprotein and zein fibers reported in literature.<sup>15,16</sup> The breaking elongation of wheat gluten fibers is similar to that of wool but much higher than that of the soyprotein and zein fibers as given in Table 1. Although soyprotein- and zein-based fibers with strength and breaking

elongations similar to those of wool have been reported during the 1940s, neither the soyprotein nor the zein fibers with properties reported during the 1940s have been produced lately.<sup>12,15–19,33</sup> The Young's modulus and moisture regain of wheat gluten fibers are similar to those of wool, and, therefore, products made from wheat gluten fibers should be soft and flexible as wool if similar denier fibers are used. No reports are available on the modulus of 100% soyprotein and zein fibers.

Wheat gluten fibers also have better tensile properties than casein- and soyprotein-based biomaterials intended for biomedical applications reported earlier. Casein and soyprotein made into tensile bars of 2 × 4 mm<sup>2</sup> cross section had strength, elongation, and modulus of 49 MPa, 2.2%, and 2.5 GPa for the casein bars, respectively, and the corresponding values for the soyprotein bars were 33 MPa, 16%, and 1.3 GPa.<sup>20</sup> The tensile properties of wheat gluten fibers reported in Table 1 are higher than both the casein- and the soyprotein-based biomaterials.

Based on the properties of the fibers, it can be inferred that wheat gluten offers better promise to produce high-quality fibers and biomaterials than do zein and soyproteins. Furthermore, using purer wheat gluten for fiber production, gluten from other varieties of wheat, and cross-linking are some of the approaches that could improve mechanical properties and the stability of the fibers to water and make them suitable for most fibrous applications.

**Stability to Acidic and Basic Conditions.** Figure 6 depicts the changes in the breaking tenacity of the wheat gluten and PLA fibers after heating in water at various pH conditions. The wheat gluten and PLA were conditioned under standard testing atmosphere before testing the fibers in the dry state. The wheat gluten fibers have the least breaking tenacity loss of about 10% at pH 7, and the breaking tenacity loss increases when the pH is increased or decreased. As can be predicted, the fibers are more stable in acidic conditions and lose only about 25% of their breaking tenacity at pH 3 as compared to 50% strength loss at pH 11. Similarly, the PLA fibers have the least loss in breaking tenacity of about 8% at pH 7 and 9, but the tenacity loss increases sharply to 50% at pH 11. Previous reports also indicate that PLA loses substantial strength (60%) when heated in pH 8 solution at 110 °C for 90 min.<sup>34</sup> Although the low resistance of PLA fibers to high pH has limited the use of PLA fibers for high value fibrous applications, several approaches are being considered to improve the resistance to hydrolysis of PLA.<sup>35,36</sup> Similarly, cross-linking the wheat gluten fibers using environmentally friendly chemicals is expected to provide better water resistance to the fibers.

## Conclusions

Protein fibers with mechanical properties similar to those of wool and better than those of 100% soyprotein and zein fibers have been produced from wheat gluten. It has been found that only a narrow range of concentration of wheat gluten, time, and temperature of aging is required to produce good quality fibers, and the properties of the fibers are improved by drawing and annealing. Although the fibers have low % crystallinity and poor orientation as compared to wool, they have good stability to weak acidic and weak alkaline conditions at high temperatures. The increasing availability of wheat gluten at low prices will provide an opportunity to develop cheap and environmentally friendly protein-based bioproducts. Wheat gluten fibers are suitable for biomedical applications because they have better properties than those of soyprotein-, zein-, and casein-based materials.



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