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(54) **PROCESS FOR THE PRODUCTION OF  
HIGH QUALITY FIBERS FROM WHEAT  
PROTEINS AND PRODUCTS MADE FROM  
WHEAT PROTEIN FIBERS**

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(57) **ABSTRACT**

A series of new manufactured protein fibers from wheat proteins, a method and kit for production of these wheat protein fibers is provided. The method may include dissolving wheat proteins by use of at least one of an alkali, acid, alcohol, reducing agent, amino acid, chemical additive, or salt in weight ratios ranging between approximately 0.5 percent and 50 percent by weight of a single wheat protein or a combination of one or more wheat proteins. Further, the method may involve aging the dissolved wheat proteins at temperatures from approximately minus thirty degrees Celsius ( $-30^{\circ}\text{C.}$ ) to one hundred and fifty degrees Celsius ( $150^{\circ}\text{C.}$ ) for approximately one to eighty hours to obtain a spinnable solution. In addition, the method entails extruding the dissolved wheat proteins into a coagulation bath to precipitate the wheat protein fibers in which the coagulation bath includes at least one of an alkali, acid or salt.

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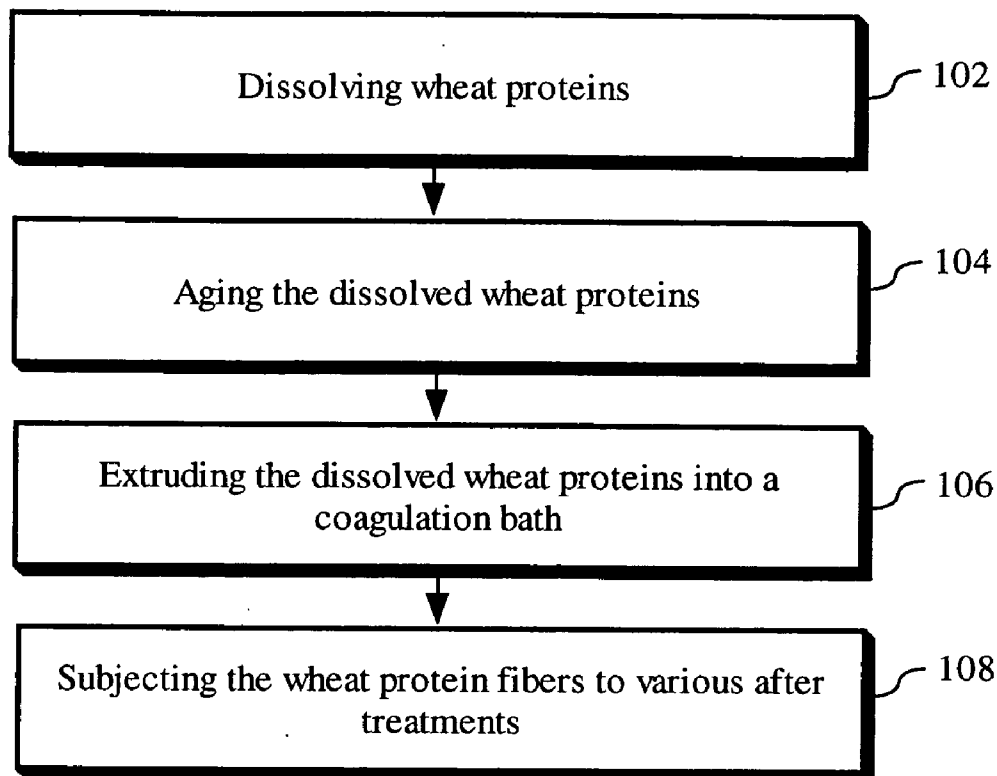
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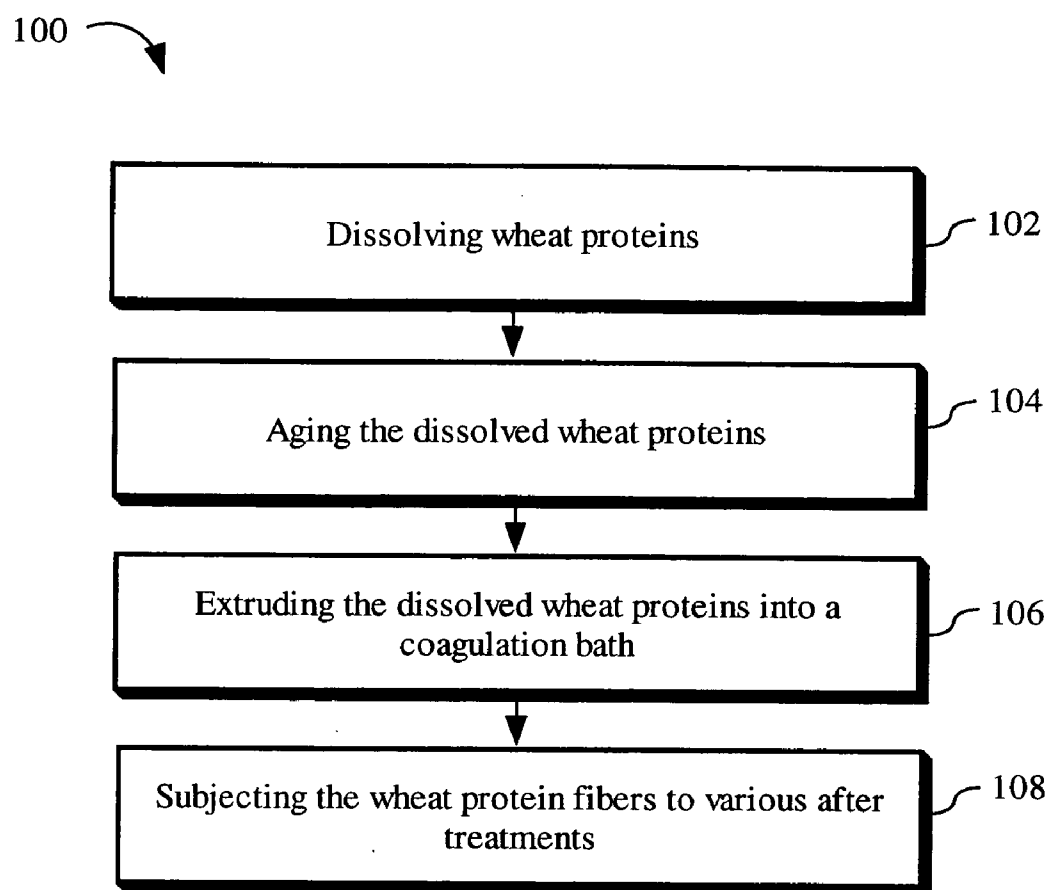
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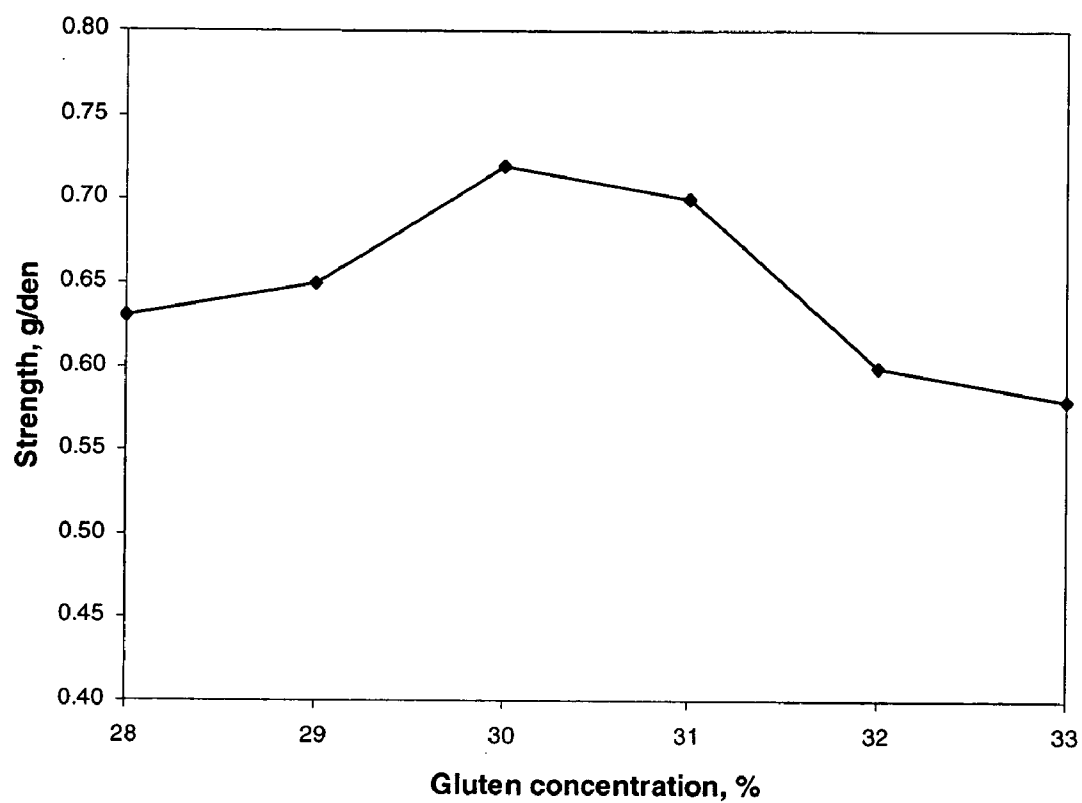
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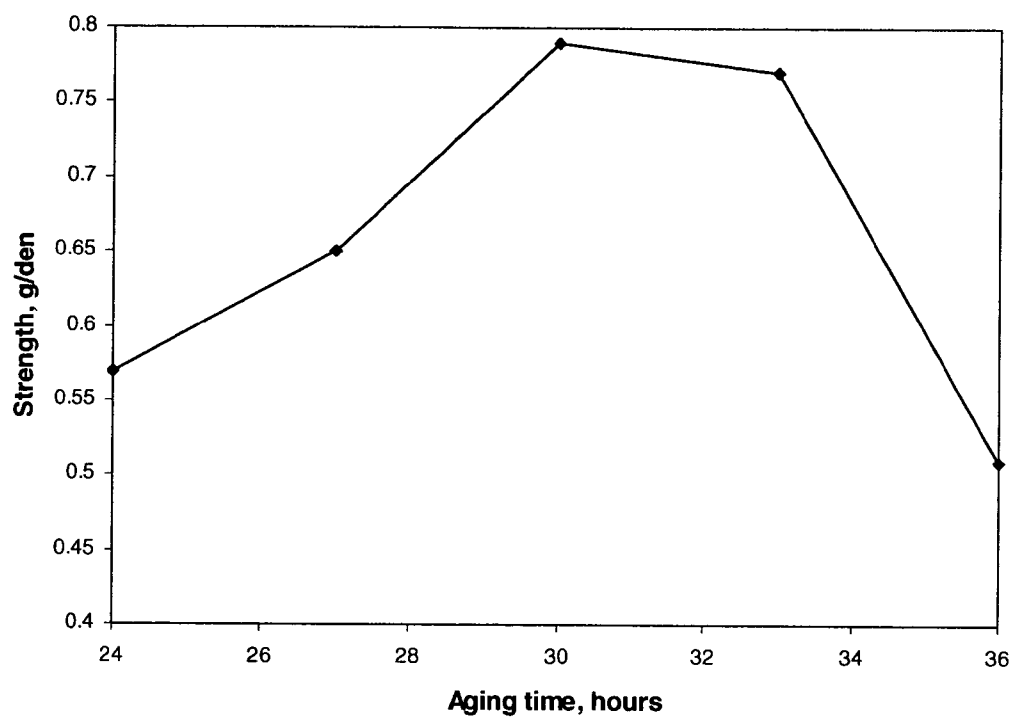




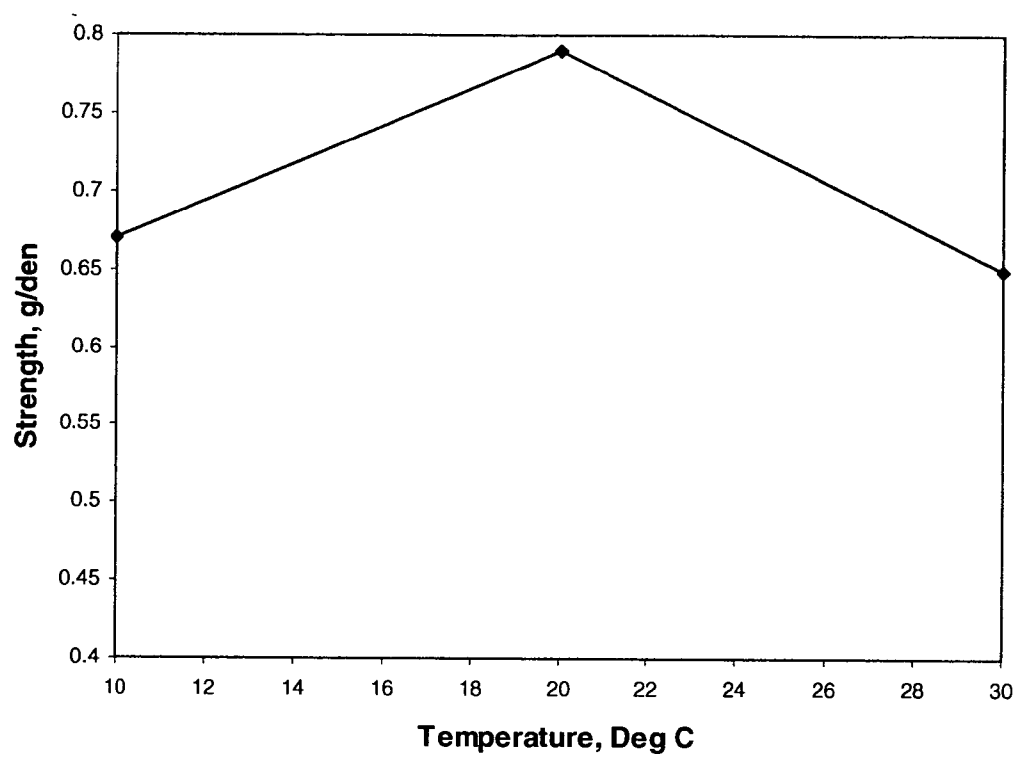
**FIG. 1**



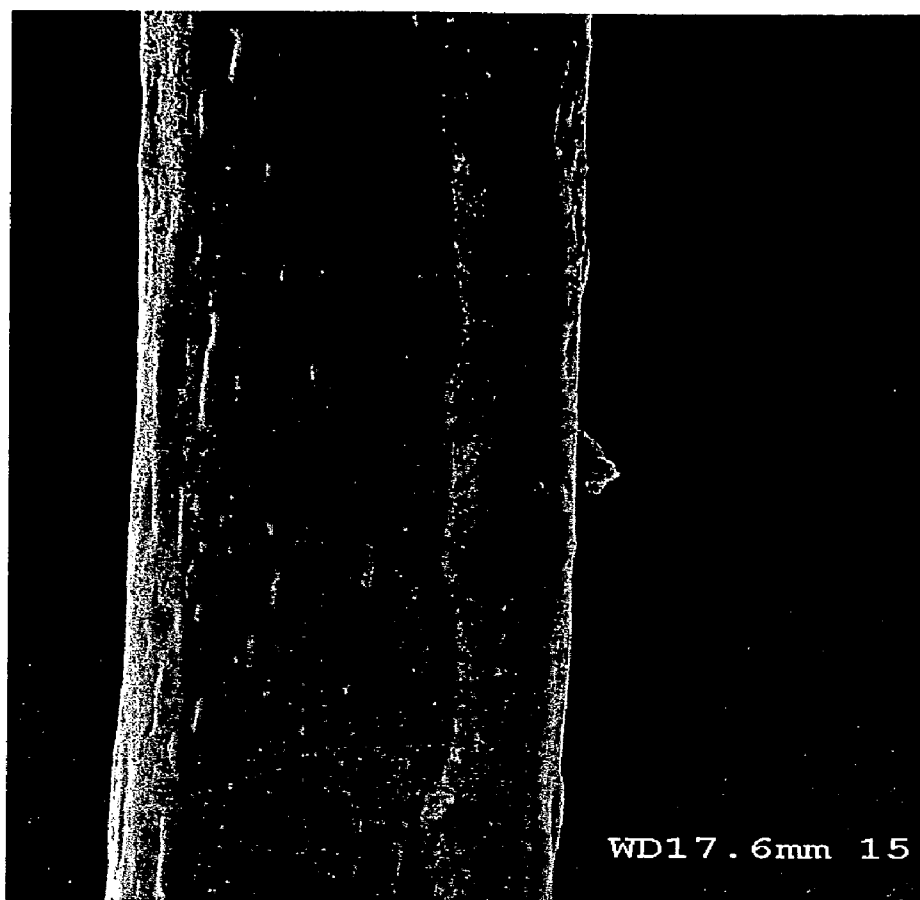
**FIG. 2**



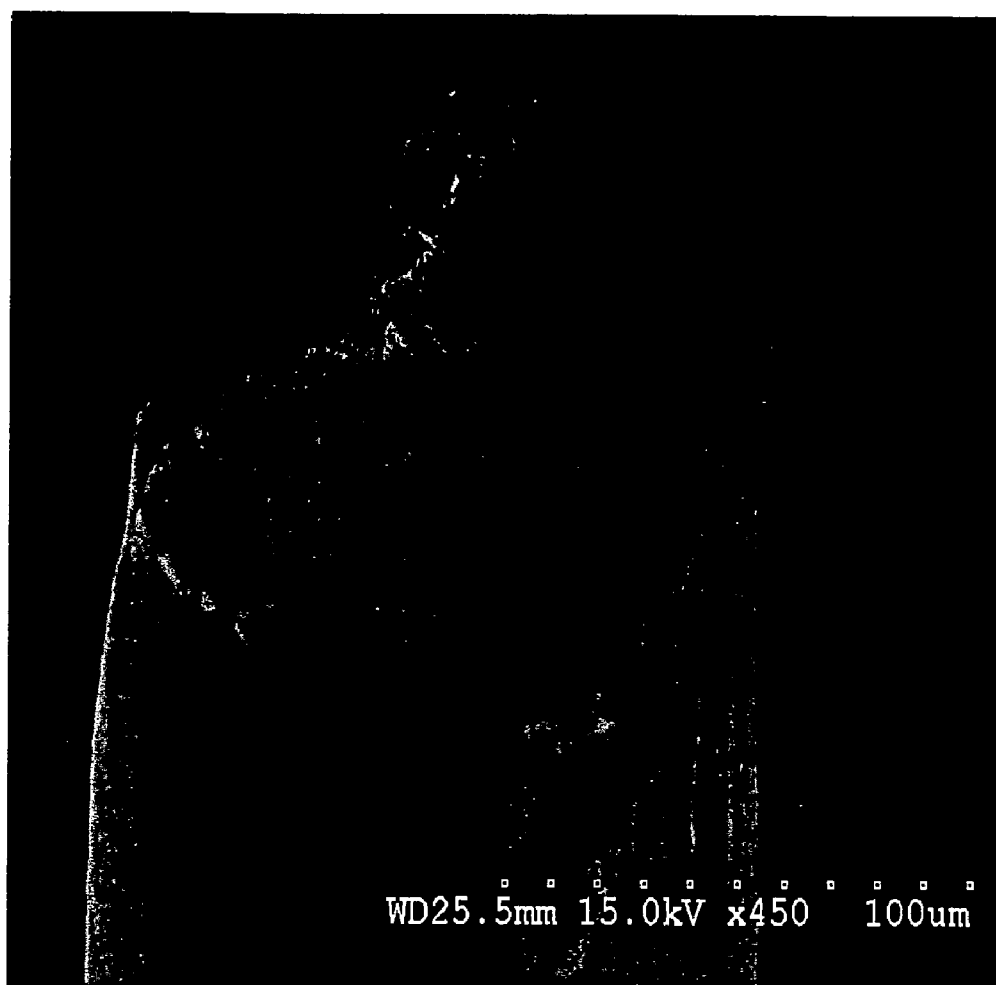
***FIG. 3***



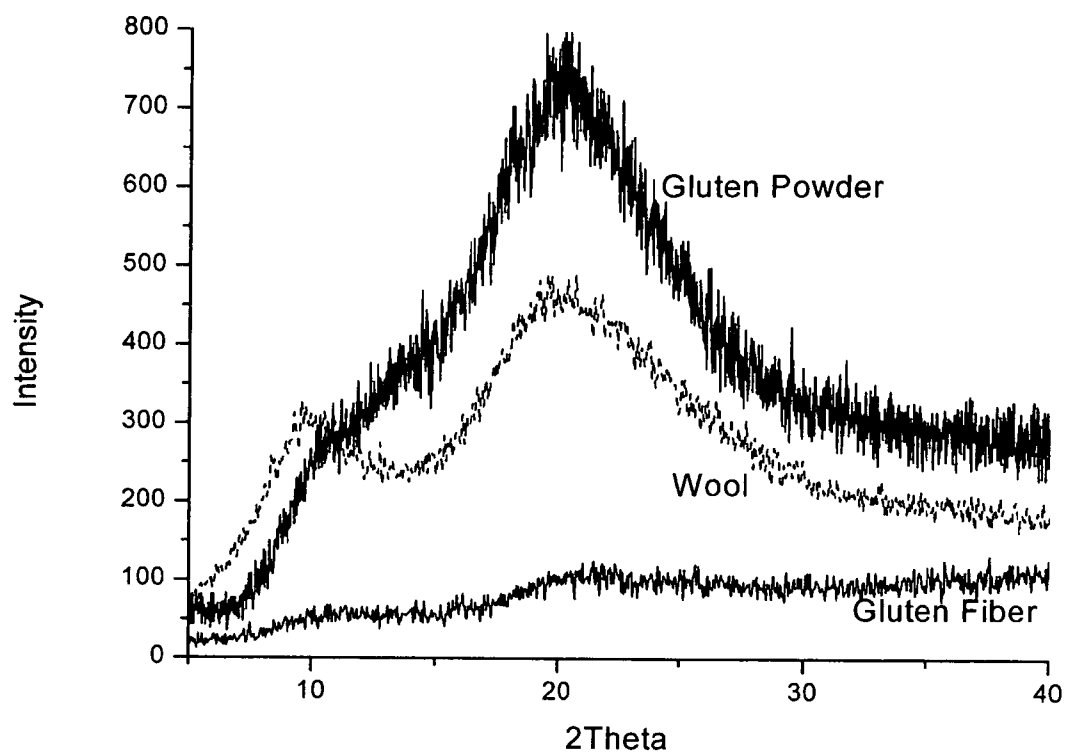
***FIG. 4***



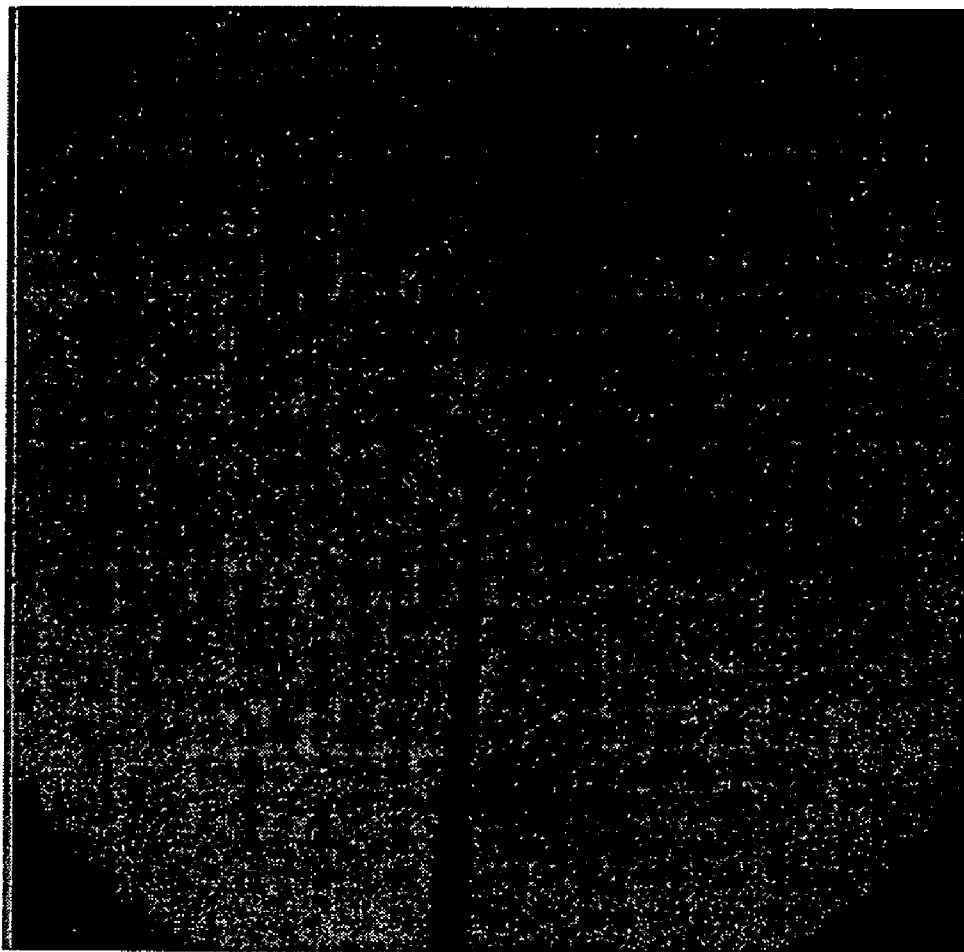
***FIG. 5***



***FIG. 6***



**FIG. 7**



***FIG. 8***

**PROCESS FOR THE PRODUCTION OF HIGH  
QUALITY FIBERS FROM WHEAT PROTEINS AND  
PRODUCTS MADE FROM WHEAT PROTEIN  
FIBERS**

**CROSS REFERENCE TO RELATED  
APPLICATIONS**

[0001] The present application claims the benefit under 35 U.S.C. § 119(e) of U.S. Provisional Patent Application Ser. No. 60/691,816, entitled "100% Regenerated Protein Fibers and Products Made From Wheat Gluten," filed Jun. 17, 2005 which is herein incorporated by reference in its entirety.

**FIELD OF THE INVENTION**

[0002] The present invention relates generally to protein fiber production, and particularly to a method of producing protein fibers from proteins in wheat grains to be used in various products including textiles, films, composites, and the like.

**BACKGROUND OF THE INVENTION**

[0003] Protein fibers are commonly preferred over synthetics and natural cellulose fibers for several applications. The unique properties of protein fibers such as their high extensibility, moisture absorption, better warmth retention, soft hand, luxurious appearance and durability make them preferred fibers for several applications. However, wool and silk are the only two natural protein fibers available on the market. The total world production of protein fibers is about 2.5 million tons, only about 4% of the total world fiber production. The limited availability of wool and silk makes them to be relatively expensive fibers. For instance, medium quality wool costs about \$5 per pound and silk costs about \$12 per pound competing against cotton selling at \$0.60 per pound and most synthetic fibers selling at less than \$1 per pound. This makes wool and silk to be premium fibers used for high value applications.

[0004] Over the past several years, numerous attempts have been made to produce fibers with properties similar to that of the protein fibers, but by using a more cost effective material. First, the regenerated cellulose fiber rayon often referred to as "artificial silk" was employed to produce products with similar qualities as those with protein fibers from silk and wool. However, limitations in the quality of the fibers, introduction of cheap synthetic fibers and environmental concerns on the production of rayon led to a gradual decline in rayon production and use. In addition, attempts have been made to use proteins in the agricultural products and byproducts such as soybeans, corn, milk and peanuts as a source for protein fibers. For instance, regenerated protein fibers generally called "Azlons" were commercially produced from proteins in corn, soybean, peanuts and milk. The poor quality of such protein fibers coupled with the use of toxic chemicals (e.g., formaldehyde) during fiber production and the introduction of cheap regenerated cellulose and synthetic fibers caused such fibers to fall out of favor.

[0005] Thus, despite the several attempts to produce protein fibers with properties similar to those of protein fibers found in silk and wool from more cost effective agricultural products and byproducts, the need remains. Therefore, it would be desirable to provide a method of production of

protein fibers which produces fibers that have similar properties to that of wool or silk and is cost effective.

**SUMMARY OF THE INVENTION**

[0006] Accordingly, the present invention provides a process of fiber production and modification that produces fibers from wheat proteins that have mechanical properties similar to that of wool, and appearance and handle similar to that of silk. The unique properties and low cost advantage of wheat gluten and other wheat proteins have been utilized to produce high quality 100% protein fibers. There are several advantages of using wheat proteins when compared to the other common protein sources such as zein and soy proteins. Wheat proteins, particularly wheat gluten, are relatively low cost in comparison to soy proteins and zein. Purified zein is reported to cost \$8 to \$12 per pound and soy protein costs about \$1.20 per pound. Wheat gluten costs about \$0.50 per pound. Other advantages of wheat proteins include excellent water and thermal stability and oxygen barrier properties. In addition, wheat proteins have excellent spinnability and can be used to form fine fibers.

[0007] In a first aspect, a method for production of wheat protein fibers is provided. In such aspect, the method may include dissolving wheat proteins by use of at least one of an alkali, acid, alcohol, reducing agent, chemical additive, or salt in weight ratios ranging between approximately 0.5 percent and 50 percent by weight of a single wheat protein or a combination of one or more wheat proteins. Further, the method may include aging the dissolved wheat proteins at temperatures from approximately minus thirty degrees Celsius ( $-30^{\circ}\text{C.}$ ) to one hundred and fifty degrees Celsius ( $150^{\circ}\text{C.}$ ) for approximately one to eighty hours to obtain a spinnable solution. In addition, the method entails extruding the dissolved wheat proteins into a coagulation bath to precipitate the wheat protein fibers in which the coagulation bath includes at least one of an alkali, acid or salt.

[0008] In further aspects, a textile product or a composite product is provided. The textile or composite product may include extracted wheat protein fibers, wherein the extracted wheat protein fibers include at least one of a fineness ranging from approximately eight deniers to one hundred and fifty deniers, a strength ranging from approximately 0.1 grams to five grams per denier or elongation from two percent to fifty percent. The textile product may be yarn, woven material, non-woven material, apparel, carpet, automotive fabric, or a medical textile.

[0009] In an additional aspect, a protein fiber production kit is provided. In such aspect, the kit may include a dissolving solution. The dissolving solution includes at least one of an alkali, acid, alcohol, reducing agent, chemical additive, or salt in weight ratios ranging between approximately 0.5 percent and fifty percent by weight of a single protein source or a combination of one or more protein sources. The protein source may vary including a wheat protein, a soy protein, a peanut protein, zein, or a chicken feather protein. The kit may also include a coagulation bath to precipitate the fibers. For instance, the coagulation bath may include at least one of an alkali, acid or salt to allow the fiber proteins to be precipitated. Treatment of the protein source with the dissolving solution followed by a protein aging time period and treatment with the extruding solution allows protein fibers to be precipitated from the protein

source. The yielded protein fibers are suitable for use in a variety of products including, but not limited to, woven fabrics, non-woven materials, composites, powders and films. Further, the products may include varying amounts of wheat protein fibers ranging from one hundred percent form or as blends with other fibers or polymers in ratios ranging from approximately five percent to ninety five percent.

[0010] It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory only and are not restrictive of the invention as claimed. The accompanying drawings, which are incorporated in and constitute a part of the specification, illustrate an embodiment of the invention and together with the general description, serve to explain the principles of the invention.

#### BRIEF DESCRIPTION OF THE DRAWINGS

[0011] The numerous advantages of the present invention may be better understood by those skilled in the art by reference to the accompanying figures in which:

[0012] **FIG. 1** is a flow chart of a method of production of wheat protein fibers in accordance with an exemplary embodiment of the present invention;

[0013] **FIG. 2** is a graphical depiction of the effect of wheat gluten concentration on the strength of the fibers in accordance with an exemplary embodiment of the present invention;

[0014] **FIG. 3** is a graphical depiction of the effect of aging time on the strength of the fibers in accordance with an exemplary embodiment of the present invention;

[0015] **FIG. 4** is a graphical depiction of the effect of aging temperature on the strength of wheat fibers in accordance with an exemplary embodiment of the present invention;

[0016] **FIG. 5** is a scanning electron microscope image of a wheat protein fiber in accordance with an exemplary embodiment of the present invention, wherein the wheat protein fiber includes a smooth outer surface;

[0017] **FIG. 6** is a scanning electron microscope image of a wheat protein fiber in accordance with an exemplary embodiment of the present invention, wherein the wheat protein fiber includes a solid cross-section;

[0018] **FIG. 7** is a graphical depiction of the X-ray diffraction pattern of wheat gluten, wheat gluten fibers in comparison to wool in accordance with an exemplary embodiment of the present invention; and

[0019] **FIG. 8** is an X-ray diffraction image of a wheat gluten fiber in accordance with an exemplary embodiment of the present invention.

#### DETAILED DESCRIPTION OF THE INVENTION

[0020] Reference will now be made in detail to the presently preferred embodiments of the invention, examples of which are illustrated in the accompanying drawings.

[0021] Referring to **FIG. 1**, a method **100** for production of wheat protein fibers from wheat proteins including gluten, glutenin, gliadin or other like wheat proteins is provided.

Commercially available wheat gluten in "as is" form is preferred for producing fibers to avoid additional costs in purifying the wheat proteins. However, the wheat gluten may be purified to separate the two major components glutenin and gliadin and these components may be used separately or in any combination to produce the protein fibers.

[0022] In an exemplary embodiment, the method **100** may include dissolving wheat proteins **102** by use of at least one of an alkali, acid, alcohol, reducing agent, chemical additive, or salt in weight ratios ranging between approximately 0.5 percent and fifty percent by weight of a single wheat protein or a combination of one or more wheat proteins. The at least one alkali, acid, alcohol, reducing agent, chemical additive, or salt dissolves the proteins by breaking the disulfide linkages and hydrogen bonds in the wheat proteins. Thus, any solvent that dissolves proteins including alkalis, alcohols and acids can be used for dissolving the wheat proteins. Examples of wheat gluten solvents include, but are not limited to ethanol, isopropyl alcohol, urea, mercaptoethanol, sodium hydroxide, urea, formaldehyde, and like reagents. These solvents can be used alone or as a combination of one or more solvents to achieve complete dissolution of the proteins and form a highly viscous and spinnable solution. Reducing agents such as sodium sulfite, sodium bisulfite, mercaptoethanol, dithiothreitol and cysteine may also be used in weight ratios of 0.05 to twenty percent on weight of the proteins used. In addition to the solvents and reducing agents, other chemicals such as plasticizers may also be added to improve the spinnability and properties of the fibers. Examples of plasticizers include but not limited glycerol and ethanol in ratios of 0.1 to fifty percent, preferably between approximately one and twenty percent and more preferably between approximately one and ten percent of the weight of proteins used. The protein dissolution can be carried out at temperatures ranging from approximately minus thirty degrees Celsius and one hundred degrees Celsius, preferably at room temperature and more preferably between approximately fifty and ninety degrees Celsius.

[0023] As illustrated in **FIG. 1**, the method **100** may include aging the dissolved wheat proteins **104** at temperatures from approximately minus thirty degrees Celsius ( $-30^{\circ}\text{C.}$ ) to one hundred and fifty degrees Celsius ( $150^{\circ}\text{C.}$ ) for approximately one to eighty hours, more preferably between approximately twenty and forty hours, and more preferably between approximately twenty four and thirty eight hours, in order to form a viscous, spinnable solution.

[0024] In addition, as demonstrated in **FIG. 1**, the method **100** may entail extruding the dissolved wheat proteins into a coagulation bath **106** to precipitate the wheat protein fibers in which the coagulation bath includes at least one of an alkali, acid or salt. The aged solution will be used to produce fibers using the dry or wet spinning system depending on the protein solvent used. For wet spinning, a suitable coagulation bath will be formulated to precipitate the solvents and obtain the fibers. Spinning of the fibers can be carried out at atmospheric or elevated temperatures. Most suitable temperatures are about twenty five to eighty five degrees Celsius and more preferably between approximately sixty and one hundred degrees Celsius. Moreover, the fibers from the spinning system can be air dried in the case of dry spinning or treated in a coagulation bath for the fibers to precipitate if wet spinning is used. The coagulation bath for wet

spinning is composed of acids, alkalis, salts and other chemicals depending on the solvent system used. Examples of acids used include, but are not limited to sulfuric acid, hydrochloric acid and formic acid. Examples of salts used include sodium chloride, sodium sulfate, ammonium sulfate, and aluminum sulfate. The temperature of the coagulation bath could be between approximately zero and one hundred degrees Celsius, preferably between approximately twenty and fifty degrees Celsius and more preferably between approximately forty and eighty degrees Celsius. In an additional embodiment, wheat proteins may be used as a blend to produce bicomponent or multicomponent fibers using the wet or dry spinning system. Fibers in various configurations such as sheath core, island-in-sea or other configurations can be produced.

[0025] In further embodiments, the method 100 may include subjecting the wheat protein fibers to various after treatments 108 to improve the properties of the fibers. Several after treatments may be used to improve the properties of the wheat protein fibers. One or more of these processes may be necessary to obtain fibers suitable for a particular end use. For example, wheat protein fibers may be crosslinked either during or after fiber production. Crosslinking agents can be dissolved during protein dissolution or in the coagulation bath as a one-step process or the fibers may be crosslinked after fiber formation as a two-step process. Examples of crosslinking agents that may be used for crosslinking wheat protein fibers include but not limited to Poly(carboxylic acids) containing more than two carboxylic groups such as Butanetetracarboxylic acid (BTCA) and citric acid, carbodiimides, aldehydes such as formaldehyde and glutaraldehyde, cysteine and enzymes such as peroxidase, glucose oxidase and transglutaminase (Tgase). The concentration of the crosslinking agents used can be between approximately 0.001 to thirty percent based on the weight of the fibers, preferably between approximately 0.01 to twenty percent and more preferably between approximately three and ten percent. Crosslinking can be performed between zero to one hundred degrees Celsius, preferably between twenty to seventy degrees Celsius. The time of crosslinking is usually between approximately one and three hundred and sixty minutes, preferably between ten and two hundred minutes. The crosslinked fibers may be dried and cured. Some of the crosslinking chemicals need to be cured for the crosslinking reaction to occur. Curing temperatures may be from one hundred to three hundred degrees Celsius depending on the crosslinking chemicals used. The time of curing is usually between approximately one and one hundred and twenty minutes, preferably between approximately one and thirty minutes and more preferably between approximately one and ten minutes depending on the type of chemicals and the concentration of chemicals used for crosslinking. The crosslinking chemicals and enzymes listed here may be used in combination with the others. Any suitable catalyst may also be used to accelerate the crosslinking reaction.

[0026] In an alternative embodiment, the wheat protein fibers may be subjected to heat treatment to improve the strength of the wheat protein fibers. For instance, wheat protein fibers are heated in dry air or in water at temperatures between ambient to approximately two-hundred degrees Celsius for approximately two to six-hundred minutes and preferably between eighty five to one hundred and forty degrees Celsius for sixty to one hundred and twenty minutes

and more preferably between one hundred and one hundred and twenty degrees Celsius for about sixty to eighty minutes. In a further embodiment, drawing may be utilized to increase the alignment of the polymers along the fiber axis thereby leading to increased fineness, strength and elongation. For example, the wheat protein fibers formed may be drawn by hand or any suitable means to lengths from approximately two to five hundred percent of their original length after extrusion. The drawing may be carried out during extrusion or coagulation as a one step process or after fiber formation as a two step process. Moreover, drawing may be carried in the dry or wet state. The wet state could be achieved using water or any other solution that does not dissolve wheat proteins.

[0027] In another embodiment, wheat protein fibers may be subject to physical treatments such as ultra-violet (UV) light treatment and  $\gamma$ -irradiation to improve the fiber properties. UV and  $\gamma$ -irradiation can be done using standard equipment for any lengths of time as required to improve the properties of fibers.

[0028] In accordance with an additional embodiment of the present invention, a protein fiber production kit is provided. In such aspect, the kit may include a dissolving solution. The dissolving solution includes at least one of an alkali, acid, alcohol, reducing agent, chemical additive, or salt in weight ratios ranging between approximately 0.5 percent and fifty percent by weight of a single protein source or a combination of one or more protein sources. The protein source may vary including a wheat protein, a soy protein, a peanut protein, zein, or a chicken feather protein. The kit may also include an extruding solution for dissolving the proteins source into a coagulation bath. For instance, the coagulation bath may include at least one of an alkali, acid or salt to allow the fiber proteins to be precipitated. Treatment of the protein source with the dissolving solution followed by a protein aging time period and treatment with the extruding solution allows protein fibers to be precipitated from the protein source. The yielded protein fibers are suitable for use in a variety of products including, but not limited to, woven fabrics, non-woven materials, composites, powders and films. Further, the products may include varying amounts of wheat protein fibers ranging from one hundred percent form or as blends with other fibers or polymers in ratios ranging from approximately five percent to ninety five percent.

[0029] Thus, the present method and kit yield high quality one hundred percent protein fibers and products which may be utilized to produce numerous products including, but not limited to, woven fabrics, non-woven materials, composites, powders and films, the products including wheat protein fibers in one hundred percent form or as blends with other fibers or polymers in ratios ranging from approximately five percent to ninety five percent. Wheat protein fibers obtained according to this invention have the fineness, length, strength and elongation required for textile (e.g., yarn, woven material, non-woven material, apparel, carpet, automotive fabric, or a medical textile) and other fibrous applications. For instance, wheat protein fibers with a fineness ranging from approximately eight deniers to one hundred and fifty deniers, a strength ranging from approximately 0.1 grams to five grams per denier or elongation from two percent to fifty percent may be produced by the presently disclosed method.

[0030] Some of the specific examples in accordance with exemplary embodiments of the present invention are provided below.

#### EXAMPLE 1

##### Dissolving Wheat Proteins

[0031] The wheat proteins specifically, wheat gluten, glutenin and gliadin were dissolved using various solvents and reducing agents. Wheat gluten and glutenin were dissolved using a urea solution with three percent sodium sulfite to form a twenty percent solution. The solution was aged for twenty-four hours and fibers extruded using a syringe and needle. Fibers were extruded into a coagulation bath including ten percent sodium sulfate and ten percent sulfuric acid in equal proportions. The fibers were allowed to stay in the coagulation bath for fifteen minutes and were later dried under ambient conditions. Fibers obtained under these conditions had deniers of about two hundred and seventy, strength of 0.14 to 0.23 grams per denier and an elongation of 1.5 to three percent. Gliadin purified from wheat gluten was dissolved using aqueous alcohol. A twenty five percent gliadin solution was prepared using a seventy percent alcohol solution. The gliadin solution was aged for about 15 hours and later extruded in air. Gliadin fibers obtained had strength of about 0.75 grams per denier and an elongation of eight percent.

#### EXAMPLE 2

##### Adding Plasticizers

[0032] Plasticizers such as glycerol were added into the wheat gluten solution and fibers were extruded. Adding the plasticizers increased the viscosity of the solution and fine fibers were produced. Fibers of seventy deniers with strength of 0.3 grams per denier and elongation of 1.5 to three percent were obtained.

#### EXAMPLE 3

##### Effect of Drawing

[0033] Fibers obtained from Examples 1 and 2 were dipped in water and then stretched by hand to about two hundred to three hundred percent of their original length. The fibers were dried in tension to retain the extended length. The drawn fibers had increased fineness by about two hundred percent, strength of about one hundred percent and elongation by about three hundred to five hundred percent compared to the corresponding undrawn fibers.

#### EXAMPLE 4

##### Studying the Effect of Concentration of Wheat Gluten

[0034] Various concentrations of wheat gluten were prepared to study the effect of wheat gluten concentration on fiber properties. The solutions were aged for thirty-two hours at a temperature of twenty degrees Celsius. FIG. 2 shows the effect of increasing wheat gluten concentration on the strength of the fibers. As seen from the figure, increasing the concentration of wheat gluten increases the strength of the fibers up to thirty percent and the strength decreases

upon further increase in concentration. A concentration of thirty percent was found to be optimum for obtaining fibers with high strength.

#### EXAMPLE 5

##### Studying the Effect of Aging Time

[0035] The aging time of the wheat gluten solution was varied from twenty-four to thirty-six hours to obtain a spinnable solution that could provide fibers with the highest possible strength. The effect of aging time on the strength of the fibers is shown in FIG. 3. As illustrated in FIG. 3, an aging time of thirty hours provides fibers with the highest strength. However, there is relatively less change in strength from thirty to thirty-three hours of aging and this interval of time is preferred in terms of obtaining fibers with the desired strength.

#### EXAMPLE 6

##### Studying the Effect of Aging Temperature

[0036] Three temperatures, ten, twenty, and thirty degrees Celsius were used to study the effect of temperature on the spinnability of the wheat gluten solutions and the strength of wheat gluten fibers. The results of the temperature study are shown in FIG. 4. As illustrated in FIG. 4, a temperature of twenty degrees Celsius provides fibers with the best strength.

#### EXAMPLE 7

##### Producing Fibers from Glutenin and Gliadin

[0037] The two major components of wheat gluten, glutenin and gliadin were separated and fibers produced from each of the individual components. Gliadin fibers were produced by dissolving gliadin in alcohol and glutenin fibers were produced using urea as a solvent. Gliadin fibers produced had strength of 0.75 grams per denier and an elongation of 8.6 percent. Similarly, fibers produced from glutenin had strength of 0.6 grams per denier and an elongation of eighteen percent.

#### EXAMPLE 8

##### Crosslinking Using Poly(Carboxylic Acids)

[0038] Wheat gluten fibers were crosslinked using butanetetracarboxylic acid (BTCA). A three percent BTCA solution with sodium hypophosphite as a catalyst was used for crosslinking. Fibers were allowed to stay in the crosslinking solution for thirty minutes and were later dried and cured. Fibers were cured at a temperature of one hundred and seventy degrees Celsius for three minutes. The BTCA crosslinked fibers were of forty-two denier with strength of 0.60 grams per denier and an elongation of thirty-two percent.

#### EXAMPLE 9

##### Crosslinking with Gluteraldehyde

[0039] Wheat gluten fibers were crosslinked using gluteraldehyde at varying temperatures and pH's. In a typical experiment, about 0.02 grams of wheat gluten fibers were dipped in 6.5 ml of twenty-five percent of gluteraldehyde

solution. The pH of the solution was adjusted to 5.1 using a buffer. The fibers in the solution were allowed to react at fifty degrees Celsius for forty-five minutes in an oven. The crosslinked fibers were removed from the solution and redipped in a solution containing 3 ml of twenty-five percent glutaraldehyde and allowed to stay in this solution for about thirty minutes at ambient temperature. The crosslinked fibers were then drawn by hand to about 100 to 200% of their original length. The drawn fibers were dried in an oven at eighty-five degrees Celsius for one hundred and twenty minutes and were later conditioned at twenty-one degrees Celsius and sixty five percent relative humidity (RH) for twenty-four hours before testing for the fiber properties. A specific example of crosslinking wheat gluten with glutaraldehyde and the changes in the properties of the fibers are given in Table 1.

TABLE 1

Properties of glutaraldehyde crosslinked wheat gluten fibers				
Fiber	Denier	Strength (g/den)	Elongation (%)	Modulus (g/den)
Control	54	0.74	24.6	36.3
Glutaraldehyde	61	1.13	18.3	46.0

## EXAMPLE 10

## Crosslinking Using a Single Enzyme

[0040] Three types of enzymes, peroxidase, glucose oxidase and Tgase were used for crosslinking wheat gluten fibers. Crosslinking conditions such as the concentration of enzymes used, time and temperature of treatment were varied to obtain fibers with the highest possible strength and elongation without affecting other fiber properties. Either single enzymes or a combination of two or more enzymes was used to crosslink the fibers. In a typical example of crosslinking using a single enzyme, about 0.3 grams of anhydrous glucose and 0.08 grams of glucose oxidase or Tgase was dissolved in 20 ml of water and the pH of the solution was adjusted to 5.1. About 0.02 grams of wheat gluten fibers were added into the solution and the fibers were allowed to stay in the solution for 2.5 hours at a temperature of 25° C. The fibers were then drawn by hand to about 200% of their initial length, dried at 85° C. for 2 hours. The changes in the properties of the fibers after this treatment for each enzyme are given in Table 2.

TABLE 2

Properties of wheat gluten fibers crosslinked using single enzymes.				
	Denier	Strength (g/den)	Elongation (%)	Modulus (g/den)
Control	62	0.61	24.4	26.6
Tgase	59	0.77	33.8	32.2
Glucose oxidase	52	0.72	23.6	31.2

## EXAMPLE 11

## Crosslinking Using Multiple Enzymes

[0041] Single enzymes i.e. glucose oxidase and Tgase used to crosslink wheat gluten did not improve the proper-

ties of the wheat gluten fibers to the required extent. It was desired to have wheat gluten fibers with strengths higher than 1 gram per denier. To achieve higher strength, a combination of enzymes was used. After the initial treatment with a single enzyme as described in example 10, the fibers were retreated with 0.5 g of Tgase in 15 mL of water for 2 hours at 45° C. The fibers were later dried at 85° C. for 2 hours. The changes in the properties of the fibers when a combination of enzymes was used are given in Table 3 below.

TABLE 3

Properties of wheat gluten fibers crosslinked with single and multiple enzymes				
	Denier	Strength (g/den)	Elongation (%)	Modulus (g/den)
Control	59	0.70	28.8	30.6
Glucose oxidase	51	0.81	25.2	38.7
Glucose oxidase and TG	57	0.90	27.7	40.8

[0042] Referring to FIGS. 5 and 8, examples of the morphological and physical structure of the fibers produced by the aforementioned methods are provided. First, the longitudinal and cross-sectional features of wheat gluten fibers were observed using a scanning electron microscope. FIGS. 5 and 6 show the longitudinal and cross-sectional view of a wheat gluten fiber, respectively obtained using scanning electron microscopes. Wheat gluten fiber has a smooth surface and a solid cross-section as seen from FIGS. 5 and 6, respectively. The physical structure of the wheat gluten fibers was studied using X-ray diffraction in terms of the percent crystallinity and orientation of the protein crystals in the fibers. FIG. 7 shows a picture of the diffraction pattern of wheat gluten, wheat gluten fibers in comparison to wool. As seen from the picture, wheat gluten fibers have the lowest percent crystallinity compared to gluten and wool. The fiber forming process may have hydrolyzed the wheat proteins leading to fibers with lower percent crystallinity. The wheat gluten fibers also have poor orientation as demonstrated by the weak and broad diffracting arcs in FIG. 8. Based on the diffraction presented in FIG. 7, wheat gluten fibers have a crystallinity of about twenty percent compared to thirty-five percent for the gluten powder and about twenty-five percent for wool.

[0043] It is believed that the present invention and many of its attendant advantages will be understood by the foregoing description. It is also believed that it will be apparent that various changes may be made in the form, construction and arrangement of the components thereof without departing from the scope and spirit of the invention or without sacrificing all of its material advantages. The form herein before described being merely an explanatory embodiment thereof. Further, it is to be understood that the claims included below are merely exemplary of the present invention and are not intended to limit the scope of coverage which has been enabled by the written description.

What is claimed is:

1. A method for production of protein fibers, comprising: dissolving wheat proteins by use of at least one of an alkali, acid, alcohol, reducing agent, chemical additive,

or salt in weight ratios ranging between approximately 0.5 percent and fifty (50) percent by weight of a single wheat protein or a combination of one or more wheat proteins;

aging the dissolved wheat proteins at temperatures from approximately minus thirty degrees Celsius ( $-30^{\circ}\text{C.}$ ) to one hundred and fifty degrees Celsius ( $150^{\circ}\text{C.}$ ) for approximately one to eighty hours to obtain a spinnable solution; and

extruding the dissolved wheat proteins into a coagulation bath to precipitate the wheat protein fibers, the coagulation bath including at least one of an alkali, acid or salt.

2. The method as claimed in claim 1, further comprising treating the precipitated wheat protein fibers with at least one of ultraviolet light or thermal treatment to enhance such protein fiber properties.

3. The method as claimed in claim 1, further comprising crosslinking the precipitated wheat protein fibers to enhance such protein fiber properties.

4. The method as claimed in claim 1, further comprising bleaching the precipitated wheat protein fibers to enhance such protein fiber properties.

5. The method as claimed in claim 1, further comprising dyeing the precipitated wheat protein fibers to enhance such protein fiber properties.

6. The method as claimed in claim 1, wherein the at least one of alkali, acid, alcohol, reducing agent or salt include ethanol, isopropyl alcohol, urea, mercaptoethanol, sodium hydroxide, urea, formic acid or formaldehyde solutions, each solution being one hundred percent pure or a blend with other solvents.

7. The method as claimed in claim 1, wherein the chemical additive includes at least one of a plasticizer or a stabilizer in a weight ratio of approximately 0.05 percent to fifty percent to weight of wheat protein used.

8. The method as claimed in claim 1, wherein the method is implemented to produce wheat protein fibers for products selected from the group consisting of woven fabrics, non-woven materials, composites, powders and films, the products including wheat protein fibers in one hundred percent form or as blends with other fibers or polymers in ratios ranging from approximately five percent to ninety five percent.

9. The method as claimed in claim 1, wherein the method is implemented to produce wheat protein fibers at least one of a fineness ranging from approximately eight deniers to one hundred and fifty deniers, a strength ranging from approximately 0.1 grams to five grams per denier or elongation from two percent to fifty percent.

10. A textile product, comprising:

extracted wheat protein fibers, wherein the extracted wheat protein fibers include at least one of a fineness ranging from approximately eight deniers to one hundred and fifty deniers, a strength ranging from approximately 0.1 grams to five grams per denier or elongation from two percent to fifty percent.

11. The textile product as claimed in claim 10, wherein the textile product is at least one of yarn, woven material, non-woven material, apparel, carpet, automotive fabric, or a medical textile.

12. A composite product, comprising:

extracted wheat protein fibers, wherein the extracted wheat protein fibers include at least one of a fineness ranging from approximately eight deniers to one hundred and fifty deniers, a strength ranging from approximately 0.1 grams to five grams per denier or elongation from two percent to fifty percent.

13. An extracted wheat protein fiber with a fineness ranging from approximately eight deniers to one hundred and fifty deniers, a strength ranging from approximately 0.1 grams to five grams per denier and an elongation from two percent to fifty percent.

14. A protein fiber production kit, comprising:

a dissolving solution, the dissolving solution including at least one of an alkali, acid, alcohol, reducing agent, chemical additive, or salt in weight ratios ranging between approximately 0.5 percent and 50 percent by weight of a single protein source or a combination of one or more protein sources, the protein source being at least one of a wheat protein, a soy protein, a peanut protein, zein, or a chicken feather protein; and

an extruding solution for dissolving the proteins source into a coagulation bath, the coagulation bath including at least one of an alkali, acid or salt,

wherein treatment of the protein source with the dissolving solution followed by a protein aging time period and treatment with the extruding solution allows protein fibers to be precipitated from the protein source, the protein fibers being suitable for use in products selected from the group consisting of woven fabrics, non-woven materials, composites, powders and films, the products including wheat protein fibers in one hundred percent form or as blends with other fibers or polymers in ratios ranging from approximately five percent to ninety five percent.

15. The kit as claimed in claim 14, wherein the kit is implemented to produce wheat protein fibers at least one of a fineness ranging from approximately eight deniers to one hundred and fifty deniers, a strength ranging from approximately 0.1 grams to five grams per denier or elongation from two percent to fifty percent.

16. The kit as claimed in claim 14, wherein the chemical additive includes at least one of a plasticizer or a stabilizer in a weight ratio of approximately 0.05 percent to fifty percent to weight of wheat protein used.

17. The kit as claimed in claim 14, wherein the at least one of alkali, acid, alcohol, reducing agent or salt include ethanol, isopropyl alcohol, urea, mercaptoethanol, sodium hydroxide, urea, formic acid or formaldehyde solutions, each solution being one hundred percent pure or a blend with other solvents.

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