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2,885,308

SPINNABLE TEXTILE FIBERS TREATED WITH COLLOIDAL SILICA

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6 Claims. (Cl. 117—139.5)

The present invention relates to the treatment or processing of spinnable textile fibers, particularly for the purpose of altering their manipulative properties, and more particularly for the purpose of reducing slippage and increasing the tensile strength of yarns formed from such fibers.

It has been proposed in U.S. Patent No. 2,443,512 to Donald H. Powers and William J. Harrison, issued June 15, 1948, to treat textile fibers with certain sols or colloidal solutions of silica thus making it possible to reduce the slippage normally present in the fibers, and as a result to speed up the rate of spinning and increase the tensile strength of the resulting yarn and fabric prepared therefrom. The sols or colloidal solutions employed and described in the above patent contain average particles measuring between about 40 and 180 millimicrons as determined by Hauser's method wherein a supercentrifuging technique is employed, although these average particles apparently comprise agglomerates of small particles having an ultimate size of about 10–20 millimicrons as determined from measurement of the particles in electron microscope photographs. Although the invention described in the above patent has been and is now being practiced on a commercial scale, the process of the present invention gives considerable improvement over the process of the above patent in reducing the slippage of the fibers and in increasing the tensile strength of yarns formed from the fibers.

It has also been proposed in U.S. Patent No. 1,629,241 to Ubbelohde, issued May 27, 1927, to treat textile fibers with silica gels to roughen the surfaces of the fibers. The silica gel particles are relatively large in size being considerably larger than the largest colloidal particles. The latter are usually considered to be colloidal only if their largest dimension is below 1 micron. The use of these large silica gel particles has never been successful in that they give only a slight reduction in slippage of the fibers or only a slight increase in tensile strength of the yarn formed from such fibers by spinning. In this respect, the silica gel particles differ materially from the sols or colloidal solutions of silica disclosed in U.S. Patent No. 2,443,512. Moreover, such silica gel particles cannot be applied uniformly to the fibers because of the difficulty of preparing a uniform suspension of such particles. The process of the present invention, on the other hand, gives reduction in slippage and yarn tensile strength increases which also differ materially from those obtained by the practice of the Ubbelohde patent or the Powers and Harrison patent.

It is one object of this invention to provide an improved process for treating and spinning textile fibers.

It is a further object of this invention to provide an improved process of reducing the slippage of textile fibers and increasing the tensile strength of yarns formed from such fibers by spinning.

Still further objects and advantages of this invention

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will appear from the following description and appended claims.

This invention is based on the discovery that by treating spinnable textile fibers such as spinnable cotton or wool fibers with a colloidal solution or dispersion of silica containing average particles measuring between 250 and 800 millimicrons an unexpected marked decrease in slippage of the fibers is obtained, and when such fibers are subjected to spinning to form a textile yarn such yarn has an unexpectedly high tensile strength, as compared to fibers treated with the silica sols containing average particles of 40 to 160 millimicrons as employed in the aforementioned Powers and Harrison patent. These results are unexpected in view of the prior art since the larger than colloidal size silica gel particles of Ubbelohde do not give any significant improvement, in reduction of fiber slippage or increase in tensile strength of yarns formed from such fibers by spinning. Hence, it could not be predicted that an increase in average particle size of silica above the average particle of the silica sols disclosed by Powers and approaching but not reaching the non-colloidal particles of Ubbelohde would give the results of this invention.

The processes of this invention are applicable to the treatment of all spinnable textile fibers, that is, textile fibers which can be spun to form yarns using the cotton, woolen, or worsted spinning systems. Thus, this invention is applicable in the treatment of spinnable cotton and wool fibers, spinnable synthetic staple fibers of regenerated cellulose, cellulose acetate, nylon, polyacrylonitrile, polyvinylidene chloride, copolymers containing acrylonitrile and the like, and various blends of such fibers, particularly blends of such synthetic staple fibers with wool fibers or cotton fibers or both. Most of the synthetic fibers are quite slippery and the slippage of such fibers is markedly reduced by the processes of this invention, but care must be taken to apply the silica particles on the fiber evenly and uniformly in order to obtain the most uniform and consistent results. On the other hand, the uniform application of the silica particles to spinnable wool fibers is not as essential since the effect on slippage reduction is not as pronounced as, and not equivalent to, the effect produced on shorter staple fibers. Moreover, wool fibers are usually treated with an aqueous emulsion of oil, and amounts of water ranging from 10 to 40% by weight, based on the dry weight of the fibers, are often applied, whereas in the case of spinnable natural vegetable fibers such as cotton fibers or synthetic staple fibers relatively small amounts of water on the order of below 10% by weight, based on the dry weight of the fibers, are permissible during processing. The various spinnable textile fibers are not equivalent in their characteristics, they must be treated in a different although somewhat analogous manner and with varying degrees of care or uniformity. For these and other reasons it is first preferred to treat spinnable wool fibers or blends thereof with other spinnable textile fibers. Spinnable textile fibers of second choice are spinnable cotton fibers or useful blends thereof with other spinnable textile fibers.

The colloidal solutions or dispersions of silica as employed in the process of this invention generally are alkaline in nature, that is, they generally have a pH between about 7.5 and 10.5 and a SiO_2 to M_2O ratio in excess of 10:1, and usually between 40:1 and 150:1, where M is a monovalent cation. However, stable acidic sols or dispersions may also be used, particularly in those instances where the acidity of the sol does not have a detrimental effect on the properties of the textile fibers employed.

The silica sols or colloidal dispersions employed in the process of this invention contain average particles measur-

ing between 250 and 800 millimicrons, and preferably between 300 and 650 millimicrons. The size of the particles is determined according to the general principles proposed by Hauser and Lynn, pages 141 to 149 of their treatise entitled "Experiments in Colloid Chemistry" (1940), published by McGraw-Hill Book Company, Inc., N.Y. The technique employed, the mathematical treatment of the method used and other considerations involved in adaptation of supercentrifuges for the measurement of particle size of colloids are described in greater detail by Hauser et al., J. Phys. Chem. 40, 1169 (1936); Hauser et al., J. Phys. Chem. 44, 584 (1940); Hauser et al., J. Ind. and Eng. Chem. 32, 659 (1940); and Fancher et al., J. Ind. and Eng. Chem. Anal. Ed. 14, 552 (1942). In the literature references cited, Hauser and his co-workers describe the preparation of a particle size distribution curve from the particle size data obtained in accordance with their methods. Since the entire area under the particle size distribution curve represents the total weight of the particles in the colloidal system tested, the area under the curve which is included between any two different particle diameters represents the weight of particles within that particular particle diameter size range. It is possible by using such particle size distribution curve to select a particle diameter at which 50% of the area under the particle size distribution curve, that is, 50% of the total weight of particles, includes particles of a smaller particle diameter and 50% of the area under the curve includes particles of a larger diameter. The particle diameter which meets this condition represents the average particle size of all the particles in the colloidal solution or dispersion. It is in this sense or meaning that the terms "average particle" or "average particle size" are used in the description of this invention.

The silica sols or colloidal dispersions employed herein may be prepared in various ways. One method of preparation comprises dispersing a silica powder having the proper average particle size, as defined herein, in water to form a colloidal dispersion. A colloid mill, or high speed agitator may be used to facilitate proper dispersion of the silica powder. The silica powder may be silica aerogel which has been comminuted to the proper average size or it may be a fume silica prepared by burning a combustible silicon compound such as silicon tetrachloride under conditions suitable for producing the desired average particle size silica. Sols or dispersions prepared from such sources of silica usually are slightly acidic, that is, have a pH between about 2 and 5, but may be rendered alkaline by the addition of dilute solutions of an alkali such as sodium silicate or sodium hydroxide. In such cases the SiO_2 to M_2O ratio is usually greater than 100:1.

Suitable silica sols may also be prepared by heating a stable alkaline silica sol containing from about 0.4 to 1.3% by weight (calculated as sodium ion) of a free basic compound such as sodium hydroxide or sodium silicate, and average particles measuring between about 10 and 180 millimicrons, at a temperature of about 160 to 220° C. until a pasty mass or thixotropic agglomerate of colloidal silica containing average particles of 250 to 800 millimicrons is obtained. The duration of heating is usually from 15 to 240 minutes with the longer heating periods corresponding to the lower temperatures. The pasty mass or thixotropic agglomerate is readily dispersed, with or without added water, by means of intense shear force such as is obtained with a colloid mill, Benet mill, Waring blender or the like, to form a stable alkaline sol having a pH between about 8.0 and 11 and containing average particles of the desired size. Usually the sols contain average particles measuring between 300 and 600 millimicrons. A more detailed description of preparing sols or colloidal dispersions by the preceding process will be found in the copending U.S. application Serial No. 73,215, filed January 27, 1949, which application is assigned to the same assignee as the present application,

and is now U.S. Patent No. 2,741,600 of Lyman S. Allen, issued April 10, 1956.

The specific sols employed in the process of this invention are applied to the spinnable textile fibers at some stage of their processing prior to spinning, preferably prior to drawing operations, and may be applied in a variety of ways. Thus such sols may be sprayed or dripped on the fibers and the fibers may then be allowed to dry or may be dried by heating. The fibers may also be immersed in the sols or colloidal dispersions either in the form of loose fibers or as a lap or in the form of a sliver or roving. Fibers thus treated are dried to the desired moisture content before further processing. The sols may also be applied to the fibers after the fibers have been carded and while they are being formed into a sliver. This may be accomplished by delivering the sol to an applicator device which includes a hollow tube through which the sol is supplied into the center of the sliver as the sliver is formed by condensing the web of carded fibers moving from the card. The sol is thus transferred from the hollow tube of the applicator device to the fibers by the wiping action of the latter as they move past and around the hollow tube of the applicator and are condensed. This method of application is usually restricted to the treatment of textile fibers which are processed and spun according to the cotton system, and is described, together with the means used, in greater detail in U.S. Patent No. 2,115,218 to Hughes L. Siever, granted April 26, 1938. In this method of application the amount of liquid applied to the fibers is not excessive and the fibers are allowed to dry at normal temperatures during the subsequent processing operations, and usually without heating ovens or other equipment.

The particular mode of applying the silica sols used is governed to some extent by the particular textile fibers which are being treated. In the case of cotton fibers or textile fibers which are processed and spun by the cotton system, it is preferred to apply the silica sols to the fibers by spraying the silica sols on the fibers in the form of fine droplets while the fibers are being conveyed in a gas stream, for example, in a trunk line prior to the formation of such fibers into a lap, and particularly during the stage subsequent to the opening and cleaning of the fibers and prior to the formation of a lap of the fibers. However, the silica sol may be applied prior to or subsequent to this stage, but prior to the actual twisting of the fibers during the spinning step. In such instances, however, there is a greater tendency for non-uniform distribution of the silica, the formation of neps and for dusting of the silica from the fibers. The application of the silica sols to the fibers by spraying while the fibers are being conveyed in a gas stream may be carried out using the method or apparatus described in U.S. Patent No. 2,568,499 to Ralph S. Hood, issued September 18, 1951 or, preferably, using the method or apparatus described in copending U.S. application Serial No. 239,054, filed July 28, 1951, which application is assigned to the same assignee as the present application, and is now Patent No. 2,805,640.

In the case of spinnable textile fibers which are processed and spun according to the woolen system, it is preferred to apply the silica sols on the fibers by incorporating them with the oil or oil emulsion which is normally applied to the fibers at the picker. In the case of spinnable textile fibers which are processed and spun according to the worsted system, that is, either the Bradford or French systems, it is preferred to apply the silica sols on the fibers by incorporating the sols in the oils or oil emulsions which are normally applied to the fibers at the gill box.

As is noted above, the silica sols may be combined with emulsifiable or self-emulsifiable oils normally used in the processing of wool fibers by the woolen or worsted systems, and in such cases compositions comprising stable oil emulsions containing silica of average particle size

between 250 and 800 millimicrons are readily obtained. The silica sols may be combined with other additives such as compatible wetting agents, oils and softening agents, for example, those described in U.S. Patent No. 2,443,512 hereinbefore referred to, or the silica sols may be combined with polyhydric compounds such as those described in U.S. Patent No. 2,635,056 to Donald H. Powers, issued April 14, 1953, or the silica sols may be combined with an oil and an organic hygroscopic agent such as those described in U.S. Patent No. 2,590,659 to Basil G. Skalkas, issued March 25, 1952, or the silica sols may be combined with condensation products of an alkylene oxide and an ester of a fatty acid and a polyhydric alcohol such as those described in U.S. Patent No. 2,696,444 to Elmer H. Rossin, issued December 7, 1954. The additives described in the last three mentioned patents are particularly suitable for incorporation in the silica sols employed herein to form compositions which may be applied to textile fibers which are processed and spun according to the cotton system so as to minimize dusting of the silica particles from the fibers.

In general, the silica sols or compositions containing colloidal silica having the average particle size described and claimed herein are applied to the fibers in amounts sufficient to provide from about 0.05 to 3%, and preferably from 0.1 to 1.8%, by weight of colloidal silica based on the dry fibers.

A further understanding of the processes and products of this invention will be obtained from the following specific examples which are intended to illustrate the invention but not to restrict the scope thereof, parts and percentages being by weight unless otherwise specified.

EXAMPLE 1

A stable, aqueous colloidal dispersion of silica containing 10% of SiO_2 and having a pH of about 10.6, an SiO_2 to Na_2O ratio in excess of 25:1 and containing average particles measuring about 350 millimicrons was applied to $1\frac{1}{16}$ inch Strict-Middling cotton fibers, as the web was being condensed into a sliver, by means of a hollow tube which was inserted in the central portion of the sliver as it was formed. The dispersion was deposited on the fibers by a wiping action as the sliver passed by the tube. The type of apparatus and the method of application employed are described in detail in U.S. Patent No. 2,115,218, hereinbefore referred to. The colloidal dispersion was supplied to the tube at a rate sufficient to provide 0.5% silica on the weight of dry fibers. The resulting sliver was drawn twice and then made into 4 hank roving in the normal manner. This roving is hereinafter referred to as roving A.

A second roving was prepared in the same manner as described in the preceding paragraph with the exception that 0.25% of silica was applied on the weight of the dry fibers. This roving is hereinafter referred to as roving B.

A third roving was prepared in the same manner as described in the first paragraph of this example with the exception that the silica dispersion used was a commercial silica sol containing 10% of SiO_2 of an average particle size of 160 millimicrons and having a pH of about 9.5 and an SiO_2 to Na_2O ratio in excess of 50:1. The amount of silica applied was 0.5% as in the first paragraph of this example. This roving is hereinafter referred to as roving C.

A fourth roving was prepared in the same manner as described in the preceding paragraph with the exception that 0.25% of silica was applied on the weight of the dry fibers. This roving is hereinafter referred to as roving D.

A fifth roving was prepared in the same manner as described in the first paragraph of this example with the exception that the fibers were not treated with a silica dispersion or with any other liquid. This roving is hereinafter referred to as the control.

The break factor of each roving was determined and the results are given in Table I below:

Table I

Roving Tested	Break	Percent Increase in Break Factor ¹
Control.....	19.2	-----
Roving A.....	137.0	613
Roving B.....	58.5	205
Roving C.....	88.4	360
Roving D.....	41.6	117

¹ The percent increase in break factor was determined by the following equation:

$$\text{Percent Increase in Break Factor} = \frac{\text{Break Factor of Treated Roving} - \text{Break Factor of Control}}{\text{Break Factor of Control}} \times 100$$

From the above table, it is apparent that the rovings obtained from fibers treated with the sol containing average particles measuring 350 millimicrons (rovings A and B) have a materially higher break factor and thus a materially higher breaking or tensile strength than rovings obtained from fibers treated with the sol containing average particles measuring 160 millimicrons (rovings C and D), at equivalent silica concentrations.

EXAMPLE 2

A colloidal dispersion of silica was prepared by dispersing 10 parts of colloidal silica particles having an average particle size of 600 millimicrons in 90 parts of water with a Waring Blendor (a high speed stirrer) until the particles remained colloidal dispersed for at least one day. The resulting colloidal dispersion contained silica particles having an average particle size of 600 millimicrons. This colloidal dispersion was applied to $1\frac{1}{16}$ inch Strict-Middling cotton fibers, as the web was being condensed into a sliver, by means of a hollow tube which was inserted in the central portion of the sliver as it was formed. The dispersion was deposited on the fibers by a wiping action as the sliver passed by the tube. The type of apparatus and the method of application employed are described in detail in U.S. Patent No. 2,115,218, hereinbefore referred to. The colloidal dispersion was supplied to the tube at a rate sufficient to provide 0.5% silica on the weight of dry fibers. The resulting sliver was drawn twice and then made into a 4 hank roving. This roving is referred to hereinafter as roving E.

A second roving was prepared in the same manner as described in the preceding paragraph with the exception that the silica dispersion employed was a commercial silica sol having a pH of about 9.5 and a SiO_2 : Na_2O ratio in excess of 50:1 and containing 10% of colloidal silica of an average particle size of 180 millimicrons. The amount of silica applied was 0.5% as in the preceding paragraph. This roving is hereinafter referred to as roving F.

A third roving was prepared using the general procedure described in the first paragraph of this example with the exception that no silica sol or liquid was applied to the fibers. This roving is hereinafter referred to as the control.

The break factor of each roving was determined and the results are given in Table II below:

Table II

Roving Tested	Hank ¹	Break Factor ²	Percent Increase in Breaking Strength
Control.....	3.71	17.1	-----
Roving E.....	3.48	170.8	899
Roving F.....	3.56	73.6	331

¹ Hank = 500 divided by weight in grains of 60 yard skein.

² Break Factor = Hank multiplied by breaking strength.

From the above table it is apparent that the roving

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prepared from fibers treated with the colloidal dispersion having average particles measuring 600 millimicrons (roving E) exhibits materially greater breaking or tensile strength than a roving prepared from fibers treated with the colloidal dispersion having average particles measuring 180 millimicrons (roving F). Thus, this indicates materially less fiber slippage in the case of roving E.

Rovings identical with those described above were spun by the cotton system to form single yarns of 30 count with a twist of 25 turns per inch, the yarns being hereinafter referred to as the control, yarn E and yarn F which correspond respectively, to the control roving, roving E and roving F.

The breaking strength of each yarn was determined and the results are given in Table III below:

Table III

Yarn Tested	Count ¹	Breaking Strength	Break Factor ²	Percent Increase in Break Factor
Control.....	29.8	61.1	1,820	-----
Yarn E.....	26.8	77.5	2,075	14
Yarn F.....	28.6	70.6	2,020	11

¹ Count=1,000 divided by weight in grains of 120 yard skein.

² Break Factor=Count multiplied by breaking strength.

It is obvious from the above table that the yarn prepared from fibers treated with a colloidal dispersion containing average particles measuring 600 millimicrons (yarn E) had a percentage increase in break factor which is 27% greater than the percentage increase in break factor of the yarn prepared from fibers treated with a colloidal dispersion containing average particles measuring 180 millimicrons over the control.

It is possible according to the process of this invention, as is shown in the examples, to produce stronger yarns of the same construction as those prepared by the use of the silica sols or colloidal dispersions employed

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in the prior art at equivalent silica concentration. It is also possible in accordance with the process of this invention to produce a yarn having a tensile strength which is equal to that prepared from sols containing average particles measuring 40 to 180 millimicrons, by using a lower concentration of silica on the fibers, usually one-quarter or one-half that of the prior art processes, or by using a lower twist multiple which results in increased production and loftier yarns.

What is claimed is:

1. Spinnable textile fibers comprising spinnable textile fibers containing from about 0.05 to 3% by weight, based on the weight of the dry fibers, of colloidal silica particles having an average particle size between 250 and 800 millimicrons.

2. Spinnable textile fibers as in claim 1, but further characterized in that said fibers comprise wool fibers.

3. Spinnable textile fibers as in claim 1, but further characterized in that said fibers comprise cotton fibers.

4. Spinnable textile fibers comprising spinnable textile fibers containing from about 0.1 to 1.8% by weight, based on the weight of the dry fibers, of colloidal silica particles having an average particle size between 300 and 650 millimicrons.

5. Spinnable textile fibers as in claim 4, but further characterized in that the fibers comprise wool fibers.

6. Spinnable textile fibers as in claim 4, but further characterized in that the fibers comprise cotton fibers and also contain a hygroscopic agent.

References Cited in the file of this patent

UNITED STATES PATENTS

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2,443,512	Powers et al.	June 15, 1948
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2,590,659	Skalkeas	Mar. 25, 1952
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2,734,835	Florio et al.	Feb. 14, 1956
2,741,600	Allen	Apr. 10, 1956

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 2,885,308

May 5, 1959

John J. Healy, Jr.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 3, line 43, for "average size" read -- average particle size --;
column 6, Table I, heading to second column thereof, for "Break" read
-- Break Factor --.

Signed and sealed this 22nd day of September 1959.

(SEAL)

Attest:

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