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TREATMENT OF TEXTILE FIBERS

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This invention relates to the treatment of textile fibers for the purpose of improving their properties, and particularly relates to the treatment of fibers for the purpose of reducing slippage, improving their manipulative characteristics and increasing the tensile strength of the material resulting from the processing of the fiber.

In the past numerous attempts have been made to accomplish the above results by treatment of the fibers or raw stock with various substances such as organic resins, gelatin, casein and other albuminous materials. However, such treatment has been unsatisfactory and in many instances has entirely prevented proper operation of the processing machinery. Thus, resins frequently become sticky or rigid, depending on the type of resin employed, and tend to coat the card teeth and drafting rolls and otherwise interfere with the drafting and spinning operations.

It is accordingly a primary object of the present invention to provide a method of treating textile fibers which avoids the difficulties encountered in prior methods and at the same time produces fibers of improved properties which can be readily spun into yarn of exceptionally high tensile strength.

Still further objects and advantages of the invention will appear from the following description and appended claims. Before explaining in detail the present invention, however, it is to be understood that the invention is not limited in its application to the details described herein, since the invention is capable of other embodiments and of being practiced or carried out in various ways.

The present invention is based on the discovery that by treating textile fibers with certain sols or colloidal solutions of silica, it is possible to reduce markedly the slippage normally present in fibers, and as a result to speed up the rate of spinning and greatly increase the tensile strength of the resulting yarn and the fabric prepared therefrom. In general, the invention is carried out by applying the silica sol to the fibers in any desired manner. Preferably, the sol is employed in a relatively dilute condition, but it may be used in widely different concentrations, depending upon the type of apparatus employed to apply it to the fibers. After application to the fiber, the sol is dried, or allowed to dry. This may be accomplished automatically for example by evaporation, during manipulation of the fibers prior to spinning, but in some instances a special drying step may be employed.

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Prior to application to the fibers, it is usually preferable to add a small amount of a suitable wetting agent to the colloidal solution or sol, as this tends to increase the penetration of the solution and enhance its slip-proofing action and other properties. However, good results can frequently be obtained in the absence of such an agent. Moreover, whether or not a wetting agent is employed, it is sometimes desirable to add a softening agent to the solution.

The colloidal solutions used in accordance with the invention may be made in a variety of ways, but are preferably made by reacting an acid, such as a mineral acid or any other acid capable of forming salts by reaction with silicates, with a water-soluble silicate in the manner customarily employed to form silica gel, washing the resulting gel with water to remove the electrolytes formed during the reaction, covering the gel with a weak aqueous solution of a substance capable of forming hydroxyl ions and, after removing the gel from the solution, heating the gel, while avoiding evaporation of water, until substantially all of the gel is converted to a sol. A more complete description of the manufacture of the above type of sol may be obtained in Patent No. 2,375,738, granted to John F. White May 8, 1945.

Solutions prepared in the above manner are preferred, since they are stable for an indefinite period of time. Moreover, the silica contained therein has a larger particle size than the silica contained in other types of sols, and since they are usually prepared in a neutral or slightly alkaline state, they are admirably adapted for the purposes of this invention. It is possible, however, to prepare the colloidal solutions as described above, then to acidify them and use them in a slightly acid condition, when desired. Other types of colloidal solutions or sols of silica may also be used, provided it is not necessary to store the sol for an extended length of time prior to use. For example, it is possible to use a sol prepared by reacting a water-soluble silicate with acid and subjecting the acidified silicate to treatment with alcohol and/or to cooling to remove the electrolyte, as described in the U. S. patent to Morris D. Marshall, No. 2,285,449, and the U. S. patent to John F. White, No. 2,285,477. It is also possible to use sols prepared by treatment of an alkali silicate with ion-exchange material, as described in the U. S. patent to Paul G. Bird, No. 2,244,325, as well as anhydrous organosols. However, the anhydrous sols are not as suitable as those containing a preponderant amount of water or consisting of silica and water.

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In accomplishing the purposes of this invention, it is usually sufficient to apply to the fibers an amount of silica varying from 0.1 to 3.0% based on the weight of the fibers. However, in some instances it is desirable to use larger amounts, as when a harsh and/or stiff finish is desired, and in such cases it is possible to employ amounts up to 4 or 5%.

The concentration of the colloidal solution or sol used is relatively unimportant, as it is possible to employ a wide variety of solution strengths depending upon the type of apparatus used or the degree of pick-up which is possible in the particular apparatus employed for applying the sol. Generally, however, it is preferable to employ solutions having an SiO_2 concentration between 0.1 and about 5%, although concentrations as high as 30% can be used.

A wide variety of wetting agents may be used in accordance with the invention, including such substances as the sodium salts of alkylated benzene sulfonates such as sodium octyl benzene sulfonate and sodium decyl benzene sulfonate; sodium lauryl sulfate, the sodium salt of methyl stearamide ethionic acid, dioctyl sodium sulfosuccinate and the like. Suitable softening agents include neutral type softeners, such as aqueous emulsions of oils, fats, waxes and fatty acids, including stearic, oleic and palmitic acids or mixtures thereof; and cationic type softeners, such as cetyl dimethyl benzyl ammonium chloride and the quaternary ammonium salts of diethyl aminoethyl oleyl amide hydro-acetate. The wetting agent, if used, should be present in amounts varying from 0.1 to 5% of the colloidal solution, while the softening agent should be added in amounts varying from 0.03 to 3% of the solution.

A further understanding of the invention will be obtained from the following examples:

Example I

A colloidal solution of silica was prepared as follows:

Seventy-three pounds of 66° Bé. H_2SO_4 were diluted with 358 pounds of water and charged to a mixing tank. Four hundred and seventy-two pounds of a sodium silicate solution analyzing 8.9% Na_2O and 29% SiO_2 were diluted with three hundred and seventy-seven pounds of water and added with stirring to the acid solution. The mixture set to a gel a few minutes after the mixing was completed. After 16 hours aging the syneresis liquor was siphoned off and the gel crushed to one-inch lumps. These lumps were washed with a continuous flow of water for 16 hours. The washed gel was then covered with seven hundred and fifty pounds of water containing 0.9 pound of NaOH . After standing 6 hours the excess solution was drained off and a portion of the gel was charged to an autoclave. The gel was heated for three hours, using steam at two hundred and fifteen pounds per square inch absolute pressure in the jacket of the autoclave. The contents of the autoclave were then blown out and the small amount of residual undispersed gel was removed by filtration. The solution so produced contained about 12.5% SiO_2 . This was then diluted with water until it contained only 0.1% SiO_2 , after which sufficient dioctyl sodium sulfosuccinate was added to provide about 0.25% in the solution.

In making sols as illustrated by the foregoing example, the aging step may be omitted entirely but it is preferable to age for at least a few hours. The time the gel remains standing in the caustic

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soda solution may be as little as 30 minutes. Moreover, the time the gel remains in the autoclave may also be varied, depending upon the size and shape of the autoclave used.

A sliver made from one-inch staple raw cotton was immersed in the colloidal solution prepared as described above, and after passing through the solution was run through squeeze rolls adjusted to pick-up a weight of solution equal to the weight of the sliver. The sliver was then dried and made into a yarn having a weight of 9240 yards per pound with a twist of 15 turns per inch. A similar yarn was made of untreated cotton, and a standard 120 yard skein was made from both the treated and untreated yarns. These skeins were conditioned in an atmosphere of 70° F. and 65% relative humidity for 24 hours. The conditioned skeins were then tested for strength on a standard tensile strength testing machine. The skein made from the untreated yarn had a break of 225 pounds, while the skein made from the treated yarn broke at 275 pounds, which represents an increase in tensile strength of 22%.

Example II

One hundred and thirty-four and two-tenths pounds of 35.5% hydrochloric acid were diluted with 293 pounds of water and charged to a mixing tank. Four hundred and seventy-two pounds of a sodium silicate solution analyzing 8.9% Na_2O and 29% SiO_2 were diluted with three hundred and seventy-seven pounds of water and added with stirring to the acid solution. The mixture set to a gel a few minutes after the mixing was completed, and the resulting gel was further treated as described in Example I with the formation of a colloidal solution or sol containing about 12.5% SiO_2 . This was then diluted with water until it contained only 0.4% SiO_2 , after which sufficient dioctyl sodium sulfosuccinate was added to provide about 0.25% in the solution.

A sliver made from one-inch staple raw cotton was immersed in the dilute colloidal solution prepared as described above, and after passing through the solution was run through squeeze rolls adjusted for 100% pick-up. The sliver was then dried and made into a yarn having a weight of 9240 yards per pound with a twist of 12 turns per inch. A similar yarn was made of untreated cotton, and a standard 120 yard skein was made from both the treated and untreated yarns. These skeins were conditioned in an atmosphere of 70° F. and 65% relative humidity for 24 hours. The conditioned skeins were then tested for strength on a standard tensile strength testing machine. The skein made from the treated yarn possessed a breaking strength of 320 pounds, while the untreated yarn was very much weaker and entirely unsatisfactory for further processing.

Example III

A silica sol containing about 12.5% silica was prepared as described in Example I. The resulting sol was divided into two portions, one of which was diluted with water to a concentration of about 1% silica and the other to a concentration of about 0.1% silica. To each solution was then added about 0.25% of dioctyl sodium sulfosuccinate. Sliver made from one-inch staple raw cotton was divided into two sections, one section being immersed in the 1% silica solution and the other in the 0.1% silica solution described above. The treated sliver was then separately

passed through squeeze rolls adjusted for 100% pick-up. The sliver was then dried and separately made into yarns having a weight of 18,480 yards per pound with a twist of 21 turns per inch. A similar yarn was made of untreated cotton, and a standard 120 yard skein was made from both the treated and untreated yarns. These skeins were conditioned in an atmosphere of 70° F. and 65% relative humidity for 24 hours. The conditioned skeins were then tested for strength on a standard tensile strength testing machine. The skein made from the yarn treated with 1.0% silica possessed a breaking strength of 130 pounds. The skein made from yarn treated with 0.1% silica possessed a breaking strength of 120 pounds, while the skeins made from the untreated yarn possessed a breaking strength of only 80 pounds.

Example IV

A silica sol containing about 12.5% silica was prepared as described in Example I. The resulting sol was diluted with water until it contained only 0.8% SiO_2 . This was applied at the opener hopper to cut rayon staple by spraying on the fiber an amount of the sol sufficient to deposit 0.2% SiO_2 based on the weight of the fiber. The fiber was then blown to a bin, and subsequently carded and spun in the usual manner. In comparison with untreated fibers the treated fibers carded more uniformly indicating a greater coefficient of friction. They also produced a stronger web and a stronger and more uniform sliver, and resulted in a much stronger and more uniform yarn. The treated staple also blended better than the untreated staple with wool fiber.

Example V

A silica sol containing about 12.5% silica was prepared as described in Example I. The resulting sol was diluted with water until it contained only 0.8% SiO_2 , after which there was added about 2% of pine oil containing a small amount of an emulsifying agent. This was applied at the opener hopper to cut rayon staple by spraying on the fiber an amount of the sol sufficient to deposit 0.2% SiO_2 based on the weight of the fiber. The fiber was then blown to a bin, and subsequently carded and spun in the usual manner. In comparison with untreated fibers the treated fibers carded more uniformly indicating a greater coefficient of friction. They also produced a stronger web and a stronger and more uniform sliver, and resulted in a much stronger and more uniform yarn. The treated staple also blended better than the untreated staple with wool fiber.

Example VI

A silica sol containing about 12.5% silica was prepared as described in Example I. The resulting sol was diluted with water and an aqueous mineral oil-lard oil emulsion until it contained only 1.0% SiO_2 . This was applied at the opener picker to wool raw stock by spraying on the fiber an amount of the sol sufficient to deposit 0.3% SiO_2 based on the weight of the fiber. The fiber was then blown to a bin, and subsequently carded and spun in the usual manner. In comparison with untreated fibers, the treated fibers carded more uniformly, indicating a greater coefficient of friction. They also produced a stronger web and a stronger and more uniform sliver, and resulted in a much stronger and more uniform yarn. The treated fiber also blended better than the untreated fiber with cut rayon staple.

Example VII

A silica sol containing about 12.5% silica was prepared as described in Example I. The resulting sol was diluted with water until it contained only 2% SiO_2 . This was separately applied to cut rayon staple as in Example V and wool fibers as in Example VI by separately spraying the stock with the sol. The treated fibers were then blown to a bin, and subsequently blended, carded and spun in the usual manner. In comparison with untreated fibers, the treated fibers blended and carded more uniformly. They also produced a stronger web and a stronger and more uniform sliver, and resulted in a much stronger and more uniform yarn.

Example VIII

A silica sol containing about 12.5% silica was prepared as described in Example I, and then diluted with water until it contained about 0.5% silica. To this was added sufficient sodium decyl benzene sulfonate to provide about 0.5% in the solution. A sliver made from one-inch staple raw cotton was immersed in the dilute colloidal solution prepared as described above, and after passing through the solution was run through squeeze rolls adjusted for 100% pick-up. The sliver was then dried and made into a yarn having a weight of 9240 yards per pound with a twist of 12 turns per inch. A similar yarn was made of untreated cotton and a standard 120 yard skein was made from both the treated and untreated yarns. Upon testing and comparing these skeins on a standard tensile strength testing machine, after first conditioning them in an atmosphere of 70° F. and 65% relative humidity for 24 hours, it was found that the treated yarn possessed a breaking strength considerably higher than the untreated yarn. Moreover, increase in tensile strength was also noted in the treated yarn as compared with yarn treated in a similar manner but without the sulfonate.

In the examples, reference is made to the treatment of fibers of cotton, wool, rayon and mixtures of rayon and wool. It should be understood, however, that equally good results can be obtained with all types of textile fibers, including, in addition to those named above cellulosic fibers, such as cellulose nitrate, cellulose acetate, viscose, cuprammonium rayon and high tensile strength rayon; protein fibers, such as silk and fibers made from the casein in milk; synthetic fibers, such as fibers made of the copolymer of vinyl chloride and vinylidene chloride, the copolymer of adipic acid and hexamethylene diamine, the copolymer of vinyl chloride and vinyl acetate, and glass fibers; and mixtures or blends of any two or more of the above fibers.

It can be seen from the above examples that the sols employed in accordance with this invention may be applied either to the raw stock or equivalent fiber or to the sliver. It is possible however to obtain the unusual and entirely unexpected results of this invention by applying the sol to the roving or at any other stage prior to spinning. When the sol is applied to the fibers as described above, it is possible to reduce the number of turns per inch in the yarn prepared therefrom as a result of the reduced slippage caused by the sol treatment. This in turn permits a considerable speeding up of the spinning process and causes a marked increase in the tensile strength of the yarn. When the sol is applied at an early stage in the processing, numerous other advan-

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tages are made possible in addition to those outlined above. Thus, fibers or raw stock treated in the manner described herein are more readily blended with other types of fibers, and the blended or unblended fibers may be carded more uniformly due to the resulting increase in coefficient of friction of the fiber surfaces. Moreover, as a result of the treatment it is possible to produce a stronger web and a stronger and more uniform sliver than is otherwise possible. It is also noteworthy that the silica applied to the fibers in accordance with this invention does not appreciably dust out during subsequent manipulation of the fibers.

By following the methods of this invention, it is possible to produce from treated or slip resistant fibers, yarns having the same or greater tensile strength than the untreated yarn even though the number of turns per inch of the treated yarn is substantially less than the number of turns per inch required to give maximum strength to the untreated yarn. This is illustrated by the following tests on cotton yarn prepared from $1\frac{1}{8}$ inch staple cotton by passing card sliver below rolls through a vat containing the silica sol and a wetting agent. The treated sliver was passed through a pair of squeeze rolls as hereinbefore described, dried and spun into yarns of different turns or twists per inch. The following data show tensile strength values of yarns of different twists per inch and different yarn number prepared from both treated and untreated fibers.

Number of Twists per inch	Strength in Pounds of Standard 120 Yard Skeins of 11's Yarns			
	Un- treated	Fiber Treat- ed With 1% Silica Sol	Fiber Treat- ed With 2% Silica Sol	Fiber Treat- ed With 3% Silica Sol
18.2.....		210	230	230
17.4.....	220	225	240	235
15.7.....	245	260	265	260
14.1.....	240	275	285	275
13.2.....	235	280	295	285
12.4.....	215	280	305	300
11.6.....	200	275	315	310
10.7.....	170	265	310	320
9.9.....	135	255	290	325
8.3.....	55			

Number of Twists per inch	Strength in Pounds of Standard 120 Yard Skeins of 22's Yarns			
	Un- treated	Fiber Treat- ed With 1% Silica Sol	Fiber Treat- ed With 2% Silica Sol	Fiber Treat- ed With 3% Silica Sol
18.2.....	85	105		
17.4.....	90	105	120	125
15.7.....	95	110	130	135
14.1.....	95	115	137	140
13.2.....	90	115	140	143
12.4.....	80	115	140	145
11.6.....	65	110	135	145
10.7.....	55	105	130	140
9.9.....	35	100		138

In each of the slivers treated the squeeze rolls were adjusted to pick up a weight of solution equal to the weight of sliver, which deposited in the fiber 1, 2 and 3% of silica respectively, based on the dry weight of the fiber.

This increase in tensile strength of yarns or fabrics, prepared from fibers treated in accordance with our invention, can also be obtained in the case of yarns or fabrics made of animal, vegetable and synthetic fibers, or mixtures thereof, of the types hereinbefore described. Also, similar improved results are obtained when

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the fibers are made into yarns having greater or smaller yarn numbers than the ones used in the above tabulations, for example, yarn numbers of 8's, 33's, 66's and the like.

The treatment of yarns and fabrics with the hereindescribed silica sols is further described in a co-pending application of ours entitled "The treatment of textile materials," Serial No. 588,422, filed April 14, 1945.

The sols described herein may be applied to the fibers by spraying, immersion, wiping or the like, either with or without the assistance of padding or squeeze rolls or other types of extracting equipment. As a wide choice may be made in the strength of the solution used, it is possible to use all types of apparatus in applying the sols. Thus, the desired amount of silica may be readily applied to the fibers by adjusting the concentration of the solution in accordance with the amount of pick-up possible with the particular apparatus employed. The per cent pick-up referred to herein is a measure of the amount of solution by weight picked up or retained by the fibers. For example, 100% pick-up means that the fibers have picked up an amount by weight of the soil or solution equal to the weight of the fibers.

The preferred sols for the purposes of this invention usually have, as initially prepared, a pH ranging from about 7 to 10. It is possible, however, to employ the sols at a somewhat lower pH, if desired, as for example where an acid reacting sol is desired. Thus, the sols may be applied to the fibers within the range of 4 to 10 pH, depending on requirements and the most suitable pH for the particular fibers being treated.

By examination in the electron microscope, it has been observed that the colloidal particles in these preferred sols have a generally spherical shape. Moreover, they vary in size from about 40 to 80 millimicrons in diameter with the average particle in most instances about 60 millimicrons in diameter.

The unusual advantages obtained as a result of the application of the sols described herein are believed to be due to the sub-microscopic roughness imparted by the deposit of exceedingly small particles of silica in the form of an inorganic resinous film. The silica is in any case polymerized or partially polymerized, and produces films having exceptional properties as applied to the fibers.

This application is a continuation-in-part of our co-pending application, Serial No. 588,421, filed April 14, 1945, which is a continuation-in-part of our co-pending application, Serial No. 546,752, filed July 26, 1944.

What is claimed is:

1. The method of treating unspun textile fibers to increase their slip resistance and prepare them for spinning which comprises applying to said fibers at some stage of their processing prior to spinning a colloidal aqueous solution of silica consisting of silica sol free of silica gel in an amount sufficient to supply from 0.1 to 5% of silica based on the weight of the fibers, and allowing the fibers thus treated to dry before allowing said solution to convert to a gel.
2. The method of treating unspun textile fibers to increase their slip resistance and prepare them for spinning which comprises applying to said fibers at some stage of their processing prior to spinning a colloidal aqueous solution of silica consisting of silica sol free of silica gel in an

amount sufficient to supply from 0.1 to 5% of silica based on the weight of the fibers, said solution having a concentration of silica between 0.1 and 30%, and thereafter drying the fibers before allowing said solution to convert to a gel.

3. The method substantially as described in claim 1, but further characterized in that the colloidal solution employed is an aquasol.

4. The method substantially as described in claim 1, but further characterized in that the colloidal solution employed is an organo-aquasol.

5. The method of treating unspun textile fibers to increase their slip resistance and prepare them for spinning which comprises separately applying to two different kinds of fibers at some stage of their processing prior to spinning a colloidal aqueous solution of silica consisting of silica sol free of silica gel in an amount sufficient to supply from 0.1 to 5% of silica based on the weight of the fibers and then drying and blending said fibers before allowing said solution to convert to a gel.

6. The method of preparing fibers for spinning which comprises applying to unprocessed fibers a colloidal aqueous solution of silica consisting of silica sol free of silica gel in an amount sufficient to supply from 0.1 to 5% of silica based on the weight of the fibers, drying the fibers thus treated before allowing said solution to convert to a gel, and then subjecting them to operations normally employed prior to spinning.

7. The method of preparing fibers for spinning which comprises preparing sliver from said fibers, applying to said sliver a colloidal aqueous solution of silica consisting of silica sol free of silica gel in an amount sufficient to supply from 0.1 to 5% of silica based on the weight of the sliver, drying the treated sliver before allowing said solution to convert to a gel, and then subjecting the sliver to operations normally employed prior to spinning.

8. The method of spinning fibers which comprises applying to said fibers at some stage of their processing prior to spinning a colloidal aqueous solution of silica consisting of silica sol free of silica gel in an amount sufficient to supply from 0.1 to 5% of silica based on the weight of the fibers and sufficient to impart slip-proof properties thereto, drying the treated fibers before allowing said solution to convert to a gel, and then spinning the resulting fibers.

9. The method substantially as described in claim 1, but further characterized in that the

colloidal solution has added thereto a small amount of a wetting agent.

10. The method substantially as described in claim 1, but further characterized in that the colloidal solution has added thereto a small amount of a softening agent.

11. The method of treating unspun textile fibers to increase their slip resistance and prepare them for spinning which comprises applying to said fibers at some stage of their processing prior to spinning a stable colloidal aqueous solution of silica consisting of silica sol free of silica gel and having a concentration of silica between 0.1 and 30%, said silica being present in the form of essentially spherical colloidal particles of a size range between 40 and 80 millimicrons in diameter, said solution being applied in an amount sufficient to supply from 0.1 to 5% of silica based on the weight of said fibers, and thereafter drying the fibers before allowing said solution to convert to a gel.

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