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PROCESS FOR IMPROVING CELLULOSE TEXTILE MATERIAL BY ALKALINE TREATMENT

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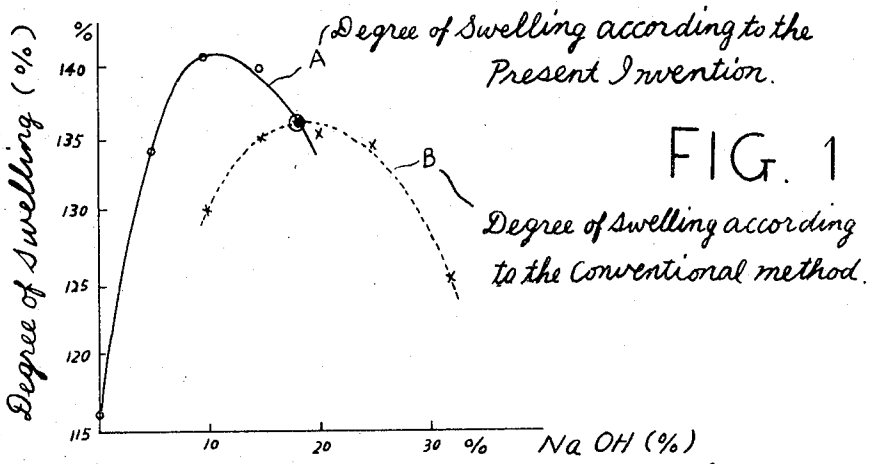


FIG. 1

Swelling Effect of caustic soda Treatment on cellulose Textile Fiber.

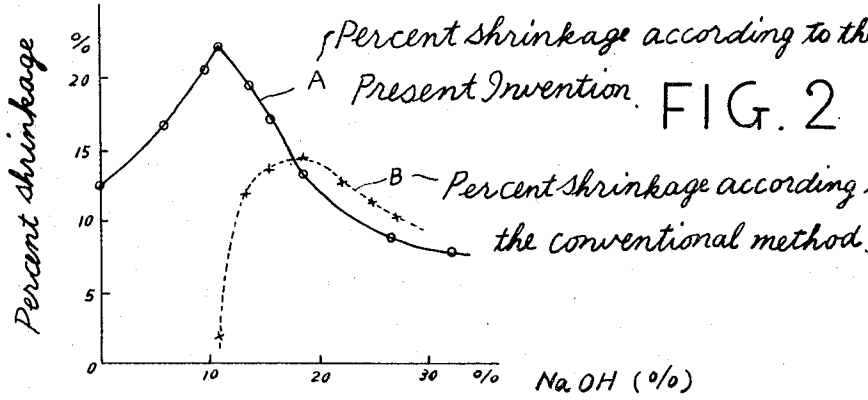


FIG. 2

Percent shrincage of cotton single fiber.

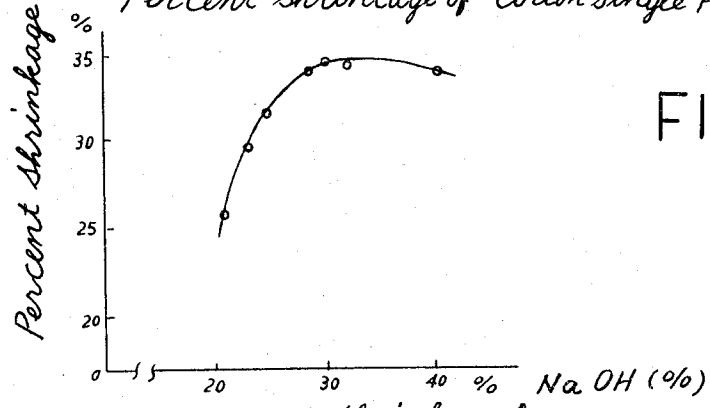


FIG. 3

Percent Shrinkage of yarn.

INVENTORS:
TOSHIHIKO SUMINOKURA,
YOJURO KYOGOKU &
KAZUO SEKIGUCHI,
BY
Browne, Schuyler + Beveridge,
ATTORNEYS

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PROCESS FOR IMPROVING CELLULOSE TEXTILE MATERIAL BY ALKALINE TREATMENT

Toshihiko Suminokura, Yojuro Kyogoku, and Kazuo Sekiguchi, Neyagawa, Japan, assignors to Kurashiki Spinning Co., Ltd., Kurashiki, Japan, a corporation of Japan

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ABSTRACT OF THE DISCLOSURE

Cellulose textile material is treated with a solution having a high caustic soda concentration of 25–35% at room temperature in the first step, and thereafter is treated with a solution of a low caustic soda concentration of about 10% at room temperature in the second step, and thereby the cellulose textile material swells much more than conventional one-step treatment with a caustic soda solution.

If the above two-step method is used without tension, the stretchability, moisture adsorption and dyeing capacities of the cellulose textile material will be improved; if wit htension, its luster and strength will be improved.

This invention relates to a process for improving cellulose textile material characterized in that it is successively treated with separate alkaline solution of different concentrations controlled in a special manner.

An object of the present invention is to provide a process for treating cellulose textile material in such a manner as to make it highly stretchable.

Another object is to provide a process for imparting splendid luster to cellulose textile material, and thereby imparting a lustrous silky appearance thereto.

Another object is to provide a process for imparting an increased breaking strength to cellulose textile material.

Another object is to provide a process for imparting an improved moisture adsorption to cellulose textile material.

Another object is to provide a process for imparting an improved dyeing capacity to cellulose textile material.

Still another object is to provide a process for profitably achieving the objects hereinabove described on a commercial basis.

Accompanying drawings are graphs showing the effects of some embodiments of the present invention.

FIG. 1 is a graph showing the lateral swelling of a cotton single fiber in a caustic soda solution according to the process of the present invention, as compared with that of another cotton single fiber treated according to the conventional method.

FIG. 2 is a graph showing the percent shrinkage of cotton single fiber shrunk in a caustic soda solution, as in FIG. 1, according to the process of the present invention, as compared with those of another cotton single fiber treated according to the conventional method.

FIG. 3 is a graph showing comparative percent shrinkage of a series of 28/2s cotton yarn when each of them is treated with a caustic soda solution of a certain concentration different from one another in the first step, and thereafter is also treated with a caustic soda solution of a definite concentration in the second step.

A process for treating cellulose textile fibers with an alkaline solution has been well-known as mercerization since Mercer was granted a patent on his invention in 1843. Also Lowe was granted, in 1889, a patent on his invention regarding a process for washing cellulose textile fibers with tension by means of water after their alkaline treatment. This process has been well-known also as mercerization and generally employed ever since. Lowe's patent relates to a process of mercerization with tension, and has been of much practical use as a process of imparting a silky appearance to cotton, the so-called silket cotton. Quite recently another process, which is usually called slack mercerization has attracted much attention. This process consists in treating cotton textile fibers with an alkaline solution without tension so as to impart stretchability to the cotton textile fibers by making use of their shrinking during the treatment.

Each of these processes depends on the fundamental principle of treating cotton textile fibers with an alkaline solution: slack mercerization providing a stretchable cotton; mercerization with tension, a silket cotton. Both processes make use of the properties of cellulose textile fibers to swell when immersed in an alkaline solution and thereby produce a certain looseness in their molecular structure, especially their molecular orientation and crystallinity.

A process of slack mercerization makes use of the properties of cellulose textile fibers to swell on treating in a lateral direction and shrink in a longitudinal direction; a process of mercerization with tension makes use of an improvement of surface luster of the cellulose textile fibers brought about both by the surface smoothness resulting from swelling on treating and by improved rearrangement of their molecular structure so as to bring about a satisfactory orientation resulting from stretching fibers under above-mentioned loose molecular structure. In order to improve the effects of the treatment, therefore, it is apparent that in either case it is necessary to improve the degree of swelling of the cellulose textile fibers when they are treated with an alkaline solution and thereby causing a looseness in the molecular structure.

Generally, in treating cellulose textile fibers with an alkaline solution, as for example with caustic soda, their maximum degree of swelling will be obtained when the concentration of the caustic soda is about 18%. It cannot be said, however, that even such a maximum degree of swelling is not enough for either imparting stretchability to the fibers as a result of their longitudinal shrinkage or imparting highly silky appearance to the fibers as a result of their improved surface smoothness and molecular orientation.

Various expendients have been proposed previously to improve the degree of swelling in order to overcome the aforementioned difficulties, that is, the difficulties attributable to insufficient swelling. Thus, it has been proposed to scour cellulose textile fibers well in order to remove impurities out of the surfaces of the fibers. It has also been proposed to rub away the primary wall of cotton which restricts swelling. But it is too expensive to scour well; it is impossible to remove the primary wall by polishing it on

a large commercial scale even if it may be possible experimentally. So neither of these expedients has been found to be practical.

As a result of laborious research concerning this matter, however, we have now succeeded in overcoming the aforementioned difficulties. In accordance with the present invention, cellulose textile fibers are first treated with a solution having a high caustic soda concentration of 25–35% at room temperature in the first step, and thereafter are treated with a solution having a low caustic soda concentration of about 10% at room temperature in the second step, and thereby the degree of swelling of the cellulose textile fibers are increased to a marked degree.

If this two-step method is adapted and if cellulose textile fibers are treated without tension, their stretchability, moisture adsorption and dyeing capacities will be improved. Or if the cellulose textile fibers are treated with tension, their luster and strength will be increased. Based on this principle, it is easy to bring into practice an effective treatment of improving the stretchability, luster or other properties of cellulose textile material on a large commercial scale at low cost.

FIG. 1 shows a swelling effect of a cellulose textile fiber on treating by means of a caustic soda solution according to the present invention. The curve B shows, by way of comparison with the present invention, the relationship between the degree of swelling of the cellulose textile fiber by means of a caustic soda solution at room temperature and the concentration of the caustic soda solution according to the conventional method. The swelling of the cellulose textile fiber is greatest when the concentration of the caustic soda solution is about 18%. The degree of swelling at this time is 136%.

After the cellulose textile fiber is treated with a solution having a high caustic soda concentration of 32% in the first step, the fiber is again treated with different solutions having different caustic soda concentration in the second step. The curve A shows the degree of swelling in this case. The maximum degree of swelling is 141% when the concentration of the caustic soda of the second step is 10%. Hence FIG. 1 shows clearly that the degree of swelling in the curve A is much greater than that in the curve B, that is, the present invention increases the degree of swelling much more than the conventional method.

Now consider the action of an alkaline solution on cellulose textile fibers.

Generally, it is assumed that a chemical combination takes place between cellulose textile fibers and a caustic soda solution and forms a molecular complex of cellulose and caustic soda. This is called the "alkali cellulose." It is said that at room temperature alkali cellulose I is produced when the concentrations of caustic soda solutions are 12–22%; alkali cellulose II when the concentrations are 25% or over.

Alkali cellulose I has better hydration capacity and swelling than alkali cellulose II. In order to increase the swelling of cellulose textile fibers, therefore, it is necessary to permit the cellulose textile fibers to become the structure of alkali cellulose I, and to make it absorb much more water so as to increase lateral swelling.

In the present case, cellulose textile fibers are first treated with a solution having a high caustic soda concentration so as to produce alkali cellulose II. By immersing the sample in a caustic soda solution of about 10% concentration, its structure will become similar to that of alkali cellulose I having a great hydration capacity. What is more, concentration difference of caustic soda solutions between inside and outside of the cellulose fibers will rapidly become greater at this very moment, which gives rise to osmotic pressure. These phenomena make the fibers absorb greater quantity of water, increase its hydration capacity still greater and bring about a highly swelling condition. Thus highly hydrated alkali cellulose composed of alkali, water and cellulose is formed. We can see that in the caustic soda treatment of the second step the optimum condition is obtained when the solution has a concentration of 10% at room temperature (FIG. 1).

Now consider the test results of slack mercerization to which a two-step treatment with a caustic soda solution is applied. In FIG. 2 the curve B shows the percent shrinkage of a cotton single fiber treated by the conventional method with respect to caustic soda solution having different concentrations. And the curve A shows the percent shrinkage of a cotton single fiber which was treated, as mentioned above, with a solution having a caustic soda concentration of 31.2% in the first step and then treated with a solution having different concentration in the second step.

A comparison of the curve A with the curve B shows that with the conventional method maximum percent shrinkage was only 13% when the concentration of the caustic soda solution was about 18% while with the method of the present invention maximum percentage was 22%. The method of the present invention was as follows: The cotton single fiber was treated with a solution having a caustic soda concentration of 31.2% in the first step and then was treated again with a solution having a caustic soda concentration of 10%. These were the results of tests conducted on a cotton single fiber. But similar results were obtained with respect to other cellulose fibers.

Generally, it is said that the shrinking of cellulose textile fibers by means of a caustic soda treatment without tension is caused by the shrinking of the fibers in a direction of their lengths resulting from the swelling of the fibers in a direction of their widths. In the case of a yarn, however, its percent shrinkage becomes greater because of the combined effects of swelling and shrinking of a single fiber treated and of the twisting of the fiber and of the yarn. In the case of a fabric, its shrinking is caused by aforementioned conditions interacting one another in a complicated manner.

Now consider the results of tests on cotton yarn. A 28/2s cotton yarn was treated with different solutions having different caustic soda concentrations in the first step and thereafter the yarn thus treated was treated with 10% caustic soda solution. In such a treatment it was found that when the concentration of the caustic soda solution was about 30% in the first step, the percent shrinkage of the yarn was most effective, which was about 34% as may be seen from FIG. 3.

What we have described above is a case in which the shrinking was effected without tension. Now we will describe a case in which the process of the present invention was applied to mercerization with tension.

As has been described before, cellulose textile fibers were treated with 31.2% caustic soda solution in the first step and were immersed in about 10% caustic soda solution to swell the fibers to the fullest extent and treated with tension (or treated with tension while the fibers are in a swollen condition). Then it was neutralized, washed with water, and dried.

As may be seen from the examples to be described hereinafter, the yarn or the fabric so obtained had a better luster, stronger strength, better dyeing and absorption capacities than the corresponding yarn or fabric treated by the conventional method.

As has been described above, in treating cellulose textile fibers with an alkaline solution, the present invention provides a process to treat the fibers with a solution having such a caustic soda concentration as to form alkali cellulose II in the first step, and then to treat them again with a solution having a caustic soda concentration of 10% in the second step so as to swell the fibers to the fullest extent.

By so doing, the process of the present invention is capable of improving a slack mercerization effect by increasing the percent shrinkage of the fibers in a treatment without tension, and also is capable of improving a mercerization effect by increasing the luster of the fibers in a treatment with tension.

What is more, in the case of slack mercerization, dyeing and adsorption capacities of the fibers are also improved; in the case of mercerization with tension, the breaking strength, dyeing and adsorption capacities of the fibers are also increased. A treating effect of cellulose textile material can be markedly improved by a process which can be easily brought to practice on a commercial basis. So the present invention has an important bearing on the utilization of the natural fibers, and so has an excellent effect.

Cellulose textile material may be treated according to the process of the present invention in the form of fibers, yarn or fabrics.

Now some embodiments of the present invention will be described in detail.

EXAMPLE 1

A 28/2s scoured cotton yarn was hanked with a diameter of 50 cm. The yarn was treated with 30% caustic soda solution without tension at room temperature for 3 minutes, and the caustic soda solution was lightly removed centrifugally from the surface of the yarn.

Next, the yarn was treated again with 10% caustic soda solution for 3 minutes, and the caustic soda solution was lightly removed centrifugally, neutralized in 0.2% acetic acid solution, washed with water, and dried. Then the yarn thus obtained was shrunk to a hank with a diameter of 31 cm.

Then, the yarn was treated with 3% melamine formaldehyde precondensate (0.3% acid catalyst) solution, removed said solution centrifugally from the surface thereof, pre-dried at 60° C., and cured at 150° C. for 3 minutes. The resultant yarn had much better stretchability and much improved adsorption and dyeing capacities. stretchability than the corresponding yarn or fabric treatby the process of the present invention is better in its stretch-ability than the corresponding yarn or fabric treated by the conventional method, as shown in FIGS. 1 and 2. Furthermore, its dyeing and adsorption capacities are also excellent.

EXAMPLE 2

A 30/2s scoured ramie yarn (in cotton counts) was used. The ramie yarn was treated in the same manner as described in Example 1. Percent shrinkage of the ramie yarn was 50%, which was greater than that of the cotton yarn. As compared with the cotton yarn, the ramie yarn was better in its stretchability. What is more, the breaking strength, adsorption and dyeing capacities of the ramie yarn were also excellent.

EXAMPLE 3

In the first step a 28/2s pure cotton yarn was first treated with 30% caustic soda solution at room temperature for 3 minutes and the caustic soda solution was removed centrifugally from the surface thereof in accordance with the process of the present invention. In the second step, the cotton yarn was treated with 10% caustic soda solution with tension while making it swell under wet condition. Such a tension was applied for 3 minutes at 30° C. to the cotton yarn as to make it 5% longer than the length of the yarn before its treatment. Then the yarn was neutralized with 0.2% acetic acid solution, and washed with water.

For the sake of comparison, a yarn was treated with 15% caustic soda solution; another yarn, in 18% caustic soda solution; and both yarns were treated with tension by the conventional mercerization with tension. Then, 5 inspectors experienced in luster testing were asked to arrange the above-mentioned 3 kinds of yarns in the order

of satisfactory luster according to their judgment by visual sensation. Table 1 shows such results.

TABLE 1

Methods of treatments	Swelling on treating, percent	Order of judgment by different inspectors*				
		A	B	C	D	E
Treated with 30% caustic soda solution, and then treated with 10% caustic soda solution with tension	141	1	1	2	1	1
Treated with 18% caustic soda solution with tension	136	2	2	1	2	3
Treated with 15% caustic soda solution with tension	135	3	3	3	3	2

*The numeral 1 indicates the best quality; 2, the next best quality; and 3, the worst quality.

As may be seen from Table 1, the treatment in accordance with the process of the present invention will give much more excellent luster to cellulose textile material than the treatment in accordance with the conventional method. As has been described before, this is due to the fact that, by the swelling of the cellulose textile material to the fullest extent, molecules are re-arranged easily and satisfactorily while it is being treated with tension.

Table 2 shows the test results of the breaking strength and elongation of yarns treated separately according to three kinds of treatments.

TABLE 2

Methods of treatments	Breaking strength, kgs.	Elongation, percent
Treated with 30% caustic soda solution and then treated with 10% caustic soda solution with tension	13.6	7.0
Treated with 18% caustic soda solution with tension	12.2	7.0
Treated with 15% caustic soda solution with tension	11.7	7.0

As shown in Table 2, a mercerized cellulose textile material treated in accordance with the process of the present invention was better in its breaking strength than the corresponding product treated by the conventional method. This is due to the following reasons: When the cellulose textile material is treated according to the process of the present invention, the fibers swell so well that their defects are much diminished and that their stress is distributed more or less uniformly. What is more, as the fibers are treated under tension when they are in a fully swollen condition, molecules are re-arranged, easily and fully, in a direction of fiber axis. Furthermore, the cellulose textile material treated according to the process of the present invention has excellent dyeing and adsorption capacities. And so it may be said that from any viewpoint it makes an important contribution to the textile field as a process of mercerization. Having described the general nature of the invention and having illustrated its operation and advantages by specific examples, the invention is particularly pointed out and claimed in the appended claims.

We claim:

1. A process for improving cellulosic textile material comprising a first step of treating the cellulosic material without tension with a solution containing 25 to 35% caustic soda at room temperature for about 1 to 10 minutes to thereby produce alkali cellulose II, thereafter treating the cellulosic textile material with a solution containing about 10% caustic soda at room temperature for about 1 to 10 minutes to thereby produce alkali cellulose I, neutralizing the textile material, washing the textile material with water and thereafter drying the textile material.

2. A process according to claim **1** wherein the textile material is placed in tension when treated with the solution containing about 10% caustic soda.

3. A process for improving natural cellulose textile materials comprising a first step of treating natural cellu-

losic material without tension with a solution containing 25 to 35% caustic soda at a temperature of 15 to 30° C. for 1 to 10 minutes, removing excess caustic solution from the textile and thereafter treating the cellulose textile material without tension with a 25–35% caustic soda solution at a temperature of 15 to 30° C. for 1 to 10 minutes, removing the excess caustic soda solution from the cellulose textile material, neutralizing the textile material, washing the textile material and thereafter drying the textile material.

4. A process according to claim **3** wherein the natural cellulosic material is held under tension and then treated with a solution containing about 10% caustic soda at a temperature of 15 to 30° C. for 1 to 10 minutes.

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GEORGE F. LESMES, Primary Examiner

J. R. MILLER, Assistant Examiner

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