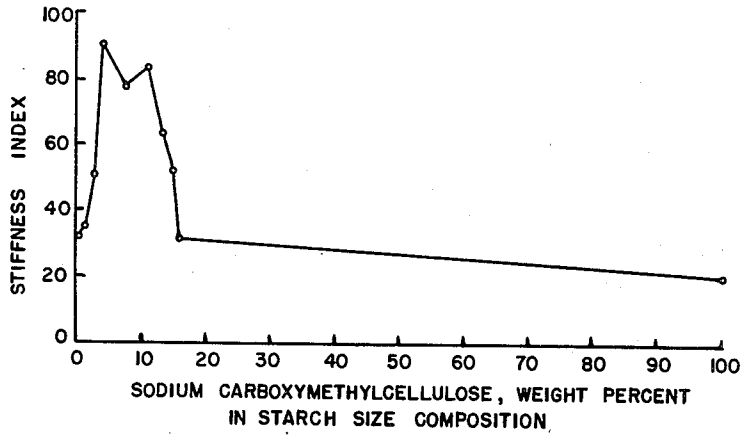
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METHOD OF SIZING TEXTILES AND SIZING COMPOSITION THEREFOR

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# METHOD OF SIZING TEXTILES AND SIZING COMPOSITION THEREFOR

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This invention relates to an improved method of sizing all types of textiles and to the novel sizing composition employed. In a more specific aspect, this invention relates to a method of sizing textiles which employs a sizing composition comprising a water-soluble salt of carboxymethylcellulose and corn starch or rice starch, which composition within a certain range of proportions of ingredients, provides unusually increased stiffening for textiles.

Many textile materials are subjected during processing to a finishing operation in which a film-forming size envelops the yarns and filaments which make up the textile. The film-forming finishes most commonly used in textile processing, as well as in industrial and home laundry operations, are derived from starch. Starch is especially suited for cotton textiles, having been used before the advent of modern finishing to facilitate mechanical processing of cotton and to add body, weight and stiffness to cotton goods.

Stiffness is one of the characteristics of sized textiles which is of importance and it is desirable to achieve the maximum stiffness with the minimum amount of starch so that the finished textile is smooth and has a soft feel, i. e., "boardiness" is avoided in the sized textile.

An object of this invention is, therefore, to provide a new and improved method of sizing textiles.

A further object of this invention is to provide a new and improved sizing composition for textiles.

A still further object of this invention is to provide a sizing composition comprising a starch ingredient which imparts higher stiffening to the sized textile than can be achieved with starch alone without increasing the amount of starch employed.

These and other objects and advantages will be apparent to those skilled in the art from reading the accompanying disclosure.

We have found that an unusual and unexpected increase in stiffness occurs in textiles sized with an aqueous dispersion of a sizing composition comprising a mixture of chemically unmodified corn starch or rice starch and a water-soluble salt of carboxymethylcellulose when said mixture contains the carboxymethylcellulose salt within a critical range of amount. Specifically, we have found that the stiffness of textiles sized with the composition of this invention is very much greater than the stiffness obtained under comparable conditions with either the starch or the carboxymethylcellulose salt alone. Along with the unexpected increase in stiffness, textiles sized with the composition of the invention have other characteristics which are very desirable, such as, a relative lack of stickiness during drying and ironing, a relative lack of staining of colored textiles, and a definitely superior softness, feel and smoothness to the touch as compared to textiles sized with other starch-based sizing compositions. A further desirable characteristic of textiles sized with the composition of the invention is excellent resiliency properties. Textiles so sized have a feel of flexibility, which property prevents the sized textile from being crushed easily when worn or used.



The method of sizing of this invention and the sizing composition of this invention can be used as warp sizing or fabric sizing for all types of textiles, such as cotton, silk, rayon and synthetics.

The sizing composition for textiles of this invention comprises from 1.5 to 15.0 weight percent of a water-soluble salt of carboxymethylcellulose and either chemically unmodified corn starch or rice starch. We have found that, when the amount of the water-soluble salt of carboxymethylcellulose is within 1.5 to 15.0 weight percent of the mixture with chemically unmodified corn or rice starch, there is a most surprising increase in stiffness in a textile sized with an aqueous dispersion of the composition. The stiffness is much greater than that obtained with a comparable amount of the starch or carboxymethylcellulose salt alone. The critical nature of the amount of the water-soluble carboxymethylcellulose salt in the composition of the invention is shown by the accompanying graph wherein stiffness is plotted against varying proportions of starch and carboxymethylcellulose salt in the size composition.

The starch ingredient employed can be either chemically unmodified corn starch or chemically unmodified rice starch. The corn starch can be a pearl or powdered corn starch and can be cooked or uncooked. Rice starch should be uncooked since we have found that the unusual increase in stiffness in sized textiles does not occur when cooked rice starch is used in the composition of the invention. The unusual increase in stiffness did not occur when chemically modified starch, i. e., soluble, acid treated, starch, or when thin boiling, oxidized, starch was used. Furthermore, the unusual increase in stiffness did not occur when other starches, such as potato starch, wheat starch, 50–50 mixtures of wheat and corn starch were used together with a water-soluble salt of carboxymethylcellulose, with the latter present in amounts within the weight percent range which is the essence of the present invention.

Any water-soluble salt of carboxymethylcellulose can be used in the composition of the invention, although we have found that the unusual increase in stiffness does not occur when such cellulose ethers as hydroxyethylcellulose and methylcellulose were used in the composition of the invention. Alkali metal salts of carboxymethylcellulose, i. e., the sodium, potassium, lithium, cesium, and rubidium salts, can be used in the composition of the invention. We have found that other water-soluble salts of carboxymethylcellulose can be used in the composition of the invention also, such as the ammonium salt; alkylamine salts, such as the diethylamine, diisopropylamine, triethylamine, and diisobutylamine salts; alkylenediamine salts, such as the ethylenediamine, 1,2-propylenediamine and hexamethylenediamine salts; alkanolamine salts, such as the ethanolamine salt; alkylarylamine salts, such as the benzyldimethylamine salt; quaternary alkylarylammonium salts, such as the benzyltrimethylammonium salt; the guanidine salt, and the like. Mixtures of such salts can also be used. As was indicated above, the salt of carboxymethylcellulose must be at least somewhat water-soluble and the salts used herein were derivatives of a carboxymethylcellulose having about a 0.6 to 0.7 degree of substitution, i. e., from 0.6 to 0.7 glycollic acid groups per anhydroglucose unit. Water-soluble salts of carboxymethylcellulose have from about a 0.3 to 1.5 degree of substitution and any salt of a carboxymethylcellulose within this range of degrees of substitution can be used in the composition of the invention.

As indicated above, the sizing composition of this invention is based on the discovery of the unusual increase in stiffness in textiles sized with aqueous dispersions of mixtures of chemically unmodified corn or rice starch and

a water-soluble salt of carboxymethylcellulose, wherein the latter is present in the mixture in the range of amounts of from 1.5 to 15.0 weight percent. We have found that such a composition containing less than 1.5 weight percent of the carboxymethylcellulose salt does not provide the increase in stiffness, and we have found that such a composition containing over 15.0 weight percent of the water-soluble salt of carboxymethylcellulose does not provide the increase in stiffness. This is clearly shown in the attached graph. It will be apparent from a review of the examples herein and from the attached graph, as well, that the largest increase in stiffness was obtained when the proportion of the water-soluble salt of carboxymethylcellulose was from about 3.0 to 13.0 weight percent, and therefore, this represents a preferred embodiment of the composition of the invention.

The textile sizing composition of this invention is used as an aqueous dispersion and the synergistic increase in stiffness in the sized textile is obtained at any concentration of the composition in water that is used in the art of finishing textiles. Stiffer sized textiles are obtained when larger amounts of the composition of the invention are used than are obtained when smaller amounts are used. It should be remembered that one of the important advantages of the composition of the invention is that a smaller amount of the composition is required to provide a desired stiffness in the resulting sized textile than is required with starch alone.

The concentration of the size composition of the invention, as actually used, can vary widely according to the type of textile or fabric being treated and according to the individual taste of the user of the sized textile. The composition of the invention can be used whenever a textile is to be sized, such as in warp sizing, as well as in commercial and home laundry operations. The composition is ideally suited for commercial laundry operations, where the sizing of cotton fabric is the most important use. In such an operation, the concentration is usually based on the amount of water employed, and an exemplary sizing system is one using 100 pounds of fabric in 40 gallons of water. In this system, the size composition of the invention is used at from 0.037 to 0.056 weight percent concentration, based on the water, for light sizing, such as for curtains and sheets. From 0.15 to 0.19 weight percent of the composition based on the water is used for medium sizing and from 0.30 to 0.45 weight percent is used for heavy sizing. It is emphasized that these specific concentrations of the total size composition are merely examples and should in no way limit the scope of the present invention. Greater concentrations can be used to obtain greater stiffness in the resulting sized textile.

The following examples are supplied to illustrate the invention and should not be used to unduly restrict the scope of the invention as it has been heretofore described.

## EXAMPLE 1

Size compositions were made according to the present invention of chemically unmodified, powdered corn starch and sodium carboxymethylcellulose. The powdered corn starch used was Buffalo powdered corn starch, Corn Products Refining Co., and contained 8.9 percent moisture. The viscosity of 68 grams of the starch in 1000 ml. of soft water at 180° F., after standing for one and one-half hours at room temperature, was 30 centipoises, using a Brookfield Viscometer, Serial No. 1811 with a No. 2 spindle at 60 revolutions per minute. The percent of the powdered corn starch retained on standard screens was 0 on 100 mesh, 2.0 through 100 mesh on 200 mesh, 10.6 through 200 mesh on 325 mesh, and 87.4 through 325 mesh screens.

The size compositions contained varying proportions of the two ingredients as are set forth below in Table 1, ranging from 98.0 weight percent powdered corn starch, 1.3 weight percent sodium carboxymethylcellulose and 0.7 weight percent inert materials, to 76.0 weight percent powdered corn starch, 15.3 weight percent sodium carboxymethylcellulose and 8.7 weight percent inert materials. A size consisting of 100 weight percent of the starch and a size consisting of 100 weight percent of a commercial sodium carboxymethylcellulose containing 36.1 weight percent of inert materials were used in the test, also.

A quantity of the size compositions was added to 500 ml. of demineralized water at 76° F. so as to provide a 0.39 weight percent concentration of the compositions in water, and the water-size mixtures were stirred until the size compositions were completely dispersed in the water.

For comparison purposes, a quantity of the powdered corn starch and a quantity of the commercial sodium carboxymethylcellulose separately were added to 500 ml. of the water at 76° F. in an amount to provide the same concentration as provided by the size composition, 0.39 weight percent, and were stirred so as to obtain complete dispersions in each case.

A 10-inch by 18-inch panel of unfinished Indian Head cotton muslin was placed in each of the water dispersions thus obtained and was stirred therein for 8 minutes. The muslin panels were then hydroextracted for 10 seconds in a laundry extractor having a 25-lb. capacity, 20-inch diameter basket and operating at 1725 revolutions per minute. Each of the panels had a quantity of water removed by the hydroextracting step so that each hydroextracted panel weighed about twice its dry weight.

The hydroextracted panels were then pressed for 30 seconds at 350° F. on a steam heated laundry press. A three-quarter-inch by four-inch strip with the longer axis parallel to the fill threads was immediately cut from each of the pressed panels and each of the strips was measured for its stiffness.

The apparatus employed to measure the stiffness of each of the test strips was made as a modification of the stiffness testing procedure and apparatus disclosed by W. T. Schreiber and W. L. Stafford, Industrial and Engineering Chemistry, volume 14, No. 3, analytical edition, March 15, 1942, pages 227–231, and depended upon the deformation of the supported test strip bent under its own weight.

The stiffness testing apparatus consisted of a wooden block 4 inches long, 3 inches high and three-quarters of an inch thick having a metal strip attached at one end of the block 1 inch from the bottom of the block. The metal strip measured one-half inch wide, 5 inches long, and one thirty-second inch thick and arched upwardly from its point of attachment to the end of the block over a 90 degree arc so that the metal strip formed a quarter of a circle with a 2-inch radius. A scale divided into millimeters was mounted on the concave face of the metal strip covering the range of 0 to 80 millimeters.

The stiffness measurements were made with each test strip of sized cloth prepared as described above by placing the test strip on the upper anterior surface of the wooden block so that one end of the test strip projected 2 inches beyond the upper end of the block over the upwardly arching metal strip. The stiffness measurement was defined as the distance on the arc of the metal strip in millimeters that the cloth bent or drooped under its own weight when supported in the described manner over the end of the block. Thus, a lower distance in millimeters for a test indicates a higher stiffness in the sized test strip of cloth, and a higher distance in millimeters indicates a lower stiffness in the sized test strip of cloth.

The results of the foregoing tests are set forth in Table 1. In order that the results obtained could be illustrated by plotting increased stiffness with larger numbers and decreased stiffness with smaller numbers, as the stiffness varied with the composition of the size employed, the reciprocal of the distance in millimeters which each test strip drooped was calculated and multi-

plied by 1000 to give the "Stiffness Index" which is plotted in the accompanying graph.

*Table 1*

| Run No. | Corn Starch,[1] Parts | Carbose RD [2] | | Distance Test Strip Drooped, Millimeters | Stiffness Index (1000×1 divided by distance test strip drooped in millimeters) |
|---|---|---|---|---|---|
| | | Parts | Weight Percent Sodium Carboxymethylcellulose | | |
| 1 | 100.0 | | | 30 | 33 |
| 2 | 98.0 | 2.0 | 1.3 | 27 | 37 |
| 3 | 96.0 | 4.0 | 2.6 | 19 | 52 |
| 4 | 94.0 | 6.0 | 3.8 | 11 | 90 |
| 5 | 88.0 | 12.0 | 7.7 | 13 | 77 |
| 6 | 83.5 | 16.5 | 10.5 | 12 | 83 |
| 7 | 79.0 | 21.0 | 13.4 | 16 | 62 |
| 8 | 77.5 | 22.5 | 14.4 | 27 | 52 |
| 9 | 76.0 | 24.0 | 15.3 | 32 | 31 |
| 10 | | 100.0 | 100.0 | 50 | 20 |

[1] Buffalo powdered corn starch, chemically unmodified, Corn Products Refining Co.
[2] Sodium carboxymethylcellulose, Wyandotte Chemicals Corporation, containing 63.9 weight percent active agent and the balance inert materials.

It will be apparent from the data in Table 1 and the accompanying graph in which this data is plotted that a truly unusual increase in stiffness occurs when the amount of the water-soluble salt of carboxymethylcellulose is within the range of 1.5 to 15.0 weight percent. The applicants do not have a ready explanation for this phenomenon, but the practical advantages which accrue therefrom and which have been described heretofore can scarcely be overemphasized.

## EXAMPLE 2

The cotton fabric described in Example 1 was sized with aqueous dispersions of three size compositions at 76° F., according to the present invention, which consisted of the chemically unmodified powdered corn starch employed in Example 1 and (1) Carbose D, Wyandotte Chemicals Corporation's brand of sodium carboxymethylcellulose containing 67 weight percent active agent, 16–18 weight percent sodium chloride, 3–4 weight percent water, and the balance sodium glycollate, (2) purified Carbose D, i. e., sodium carboxymethylcellulose, being Carbose D from which the sodium chloride and sodium glycollate were removed, and (3) Carbose 53 which consisted of 75 weight percent Carbose D and 25 weight percent sodium chloride. The concentration of compositions (1) and (3) was 0.39 weight percent, and the concentration of composition 2(*a*) was 0.37 weight percent and 2(*b*) was 0.36 weight percent. The sizing procedure and stiffness test employed were those described in Example 1 and the results of these tests are set forth below in Table 2.

*Table 2*

| Run No. | Size Composition | | Weight Percent Sodium Carboxymethylcellulose | Distance Test Strip Drooped, Millimeters |
|---|---|---|---|---|
| | Powdered Corn Starch, Parts by Weight | Carboxymethylcellulose Salt, Parts by Weight | | |
| 1(*a*) | 94 | 6 (Carbose D) | 4.0 | 16 |
| 1(*b*) | 88 | 12 (Carbose D) | 8.0 | 13 |
| 1(*c*) | 79 | 21 (Carbose D) | 14.1 | 19 |
| 1(*d*) | 67 | 33 (Carbose D) | 22.1 | 32 |
| 2(*a*) | 88 | 8.2 (Purified Carbose D) | 8.5 | 11 |
| 2(*b*) | 79 | 14.1 (Purified Carbose D) | 15.1 | 15 |
| 3(*a*) | 94 | 6 (Carbose 53) | 3.1 | 12 |
| 3(*b*) | 67 | 33 (Carbose 53) | 16.6 | 21 |

## EXAMPLE 3

The cotton fabric described in Example 1 was sized with aqueous dispersions of size compositions at 76° F. and 0.39 weight percent concentration, according to the present invention, containing (1) chemically unmodified, uncooked rice starch, and (2) chemically unmodified, cooked Buffalo powdered corn starch, each in admixture with Carbose RD in the proportion of 88 parts by weight of the starch and 12 parts by weight of Carbose RD. The rice starch used was a powdered rice starch and contained 9.3 percent moisture. The viscosity of 68 grams of the starch in 1000 ml. of soft water at 180° F., after standing at room temperature for one and one-half hours, was 42 centipoises, using a Brookfield Viscometer, Serial No. 1811 with a No. 2 spindle at 60 revolutions per minute. The percent of the rice starch retained on standard screens was 0 on 100 mesh, 5.0 through 100 mesh on 200 mesh, 5.0 through 200 mesh on 325 mesh, and 90.0 percent through 325 mesh screens.

The sizing procedure and stiffness test employed were those described in Example 1. With composition (1) containing uncooked rice starch, the test strip drooped 12 millimeters. A control test with the uncooked rice starch alone was carried out and the test strip drooped 18 millimeters. With composition (2) containing cooked Buffalo powdered corn starch, the test strip drooped 19 millimeters and in a control test with the cooked corn starch alone the test strip drooped 27 millimeters.

## EXAMPLE 4

A series of runs was carried out employing various water-soluble salts of carboxymethylcellulose together with chemically unmodified, Buffalo powdered corn starch in the size compositions of the invention. The sizing procedure and stiffness testing procedure were those described in Example 1. The carboxymethylcellulose salts employed were essentially pure and each test was carried out with 0.38 weight percent of the size composition at 76° F. Each size composition contained 88 parts by weight of the starch ingredient and 8.2 parts by weight of the carboxymethylcellulose salt which provided size compositions having a comparable weight percent of the carboxymethylcellulose salt to run No. 5 in Table 1, Example 1, wherein the size composition contained 88 parts by weight of the starch and 12 parts by weight of Carbose RD. Also, the stiffness test was carried out on the cotton sized with a 0.26 weight percent aqueous dispersion of the pure salt of carboxymethylcellulose itself, for comparison, which amount of the pure salt is about the same as the amount of sodium carboxymethylcellulose in Carbose RD used in Example 1, run No. 10. The results of these runs are set forth below in Table 3.

*Table 3*

| Run No. | Salt of Carboxymethylcellulose Used | Distance Test Strip Drooped, Millimeters | |
|---|---|---|---|
| | | Buffalo Powdered Corn Starch Plus Salt of Carboxymethylcellulose | Salt of Carboxymethylcellulose Alone |
| 1 | Ammonium Carboxymethylcellulose. | 14 | 44 |
| 2 | Triethylamine Carboxymethylcellulose. | 11 | 39 |
| 3 | Diisobutylamine Carboxymethylcellulose. | 12 | 35 |
| 4 | Ethanolamine Carboxymethylcellulose. | 16 | 44 |
| 5 | Benzyldimethylamine Carboxymethylcellulose. | 14 | 41 |

It will be apparent that the objectives of this application have been accomplished in that an improved method of sizing textiles has been provided wherein a novel size composition is employed which comprises chemically unmodified corn or rice starch and from 1.5 to 15.0 weight percent of a water-soluble salt of carboxymethylcellulose. The unusual result of the use of the method

of the invention is a synergistic increase in stiffening in the sized textile as compared to the stiffening obtained on a comparable basis with either the starch or carboxymethylcellulose ingredient alone.

We claim:

1. A sizing composition for textiles consisting essentially of a starch selected from the group consisting of chemically unmodified corn starch and chemically unmodified and uncooked rice starch, and from 1.5 to 15.0 weight percent of a water-soluble salt of carboxymethylcellulose.

2. A sizing composition for textiles according to claim 1, wherein said salt of carboxymethylcellulose is an alkali metal salt.

3. A sizing composition for textiles according to claim 1 wherein said salt of carboxymethylcellulose is the sodium salt.

4. A sizing composition for textiles consisting essentially of a starch selected from the group consisting of chemically unmodified corn starch and chemically unmodified and uncooked rice starch, and from 1.5 to 15.0 weight percent of a water-soluble salt of carboxymethylcellulose selected from the group consisting of alkali metal, alkylamine, alkylarylamine, alkanolamine salts, and mixtures thereof.

5. A sizing composition for textiles according to claim 4 wherein said salt of carboxymethylcellulose is the triethylamine salt.

6. A sizing composition for textiles according to claim 4 wherein said salt of carboxymethylcellulose is the diisobutylamine salt.

7. A sizing composition for textiles according to claim 4 wherein said salt of carboxymethylcellulose is the benzyldimethylamine salt.

8. A sizing composition for textiles according to claim 4 wherein said salt of carboxymethylcellulose is the ethanolamine salt.

9. A sizing composition for textiles consisting essentially of chemically unmodified corn starch and from 1.5 to 15.0 weight percent of a water-soluble alkali metal salt of carboxymethylcellulose.



10. A sizing composition for textiles consisting essentially of chemically unmodified and uncooked rice starch and from 1.5 to 15.0 weight percent of a water-soluble alkali metal salt of carboxymethylcellulose.

11. A sizing composition for textiles consisting essentially of a starch selected from the group consisting of chemically unmodified corn starch and chemically unmodified and uncooked rice starch, and from 3.0 to 13.0 weight percent of a water-soluble salt of carboxymethylcellulose.

12. A method of sizing textiles, which comprises, treating a textile with a size composition consisting essentially of a starch selected from the group consisting of chemically unmodified corn starch and chemically unmodified and uncooked rice starch, and from 1.5 to 15.0 weight percent of a water-soluble salt of carboxymethylcellulose.

13. A method of sizing textiles, which comprises, forming an aqueous dispersion of a size composition consisting essentially of a starch selected from the group consisting of chemically unmodified corn starch and chemically unmodified and uncooked rice starch, and from 1.5 to 15.0 weight percent of a water-soluble alkali metal salt of carboxymethylcellulose, and contacting the textile with said size composition.

14. A method in accordance with claim 13 wherein said water-soluble salt of carboxymethylcellulose is the sodium salt.

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