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3,094,372 PROCESS OF WET CREASEPROOFING CELLULOSE FABRICS WITH SPECIFIC ALDEHYDE SOLU-TIONS CONTAINING SPECIFIC METAL SALTS AND PRODUCTS PRODUCED THEREBY Gilbert S. Hibbert, Disley, William S. Miller, Ashton-on-Mersey, and Arthur Schofield, Middleton, England, as-signors to The Calico Printers' Association Limited,

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This invention relates to processes for the treatment of cellulosic fabrics and to cellulosic fabrics having improved properties produced by such processes. More par- 15 ticularly, the invention is concerned with processes for finishing textiles comprising cellulosic fibers to impart to the textiles the ability to recover from creases imposed on the textile in laundering, so that upon drying, the textile need not be ironed. Likewise, the invention is particularly concerned with new and improved textiles comprising cellulosic fibers which after laundering recover during drying from any creasing imposed upon the textile while in the wet state so that ironing is not necessary to obtain an acceptable crease-free appearance.

FIELD OF INVENTION

The art of treating textiles, non-woven fabrics and other fibrous webs to impart crease resistance or improved recovery from creasing has become highly de- 30 veloped. Actually, so-called "wash and wear" fabrics and garments have become very popular and are in great demand, although all too often the purchaser finds to his dissatisfaction that the garment he has purchased does not sufficiently resist creasing in the wet state during 35 laundering and must be ironed to obtain a wearable appearance.

During wear, creasing takes place while the garments are in a more or less dry condition, but during laundering, creasing takes place while the garments are in a wet 40 condition. A good deal of attention has been paid to methods of preventing or reducing the tendency of textiles to crease in the dry condition, but less attention has been paid to preventing them from creasing in the wet state. Many processes intended to reduce dry creasing do confer some degree of resistance to creasing in the wet state also, provided that the treated material is handled with some care and is not subjected to the severe creasing conditions involved in normal laundry practice, such as hydro-extracting in a centrifugal machine, mangling, or wringing by hand. Under these more severe conditions, processes intended to improve the dry creaseresistance do not improve the wet crease-resistance of the fabric to such an extent that the need for a final ironing treatment is eliminated, if the fabric is to have an 55 acceptable appearance after washing.

It is possible to form textiles and non-woven fabrics from special fibers or fiber combinations so that the fabrics possess less tendency toward creasing, or better ability to recover from creasing than other fabrics made from more commonly used and generally less expensive fibers or fiber combinations. Such an approach to production of crease-proof textiles, however, is limited, and the textile industry finds it necessary in order to meet the demands of the purchasing public, to have available methods for treating or finishing textiles to further improve the crease-proofness of fabrics, whether made of special fibers

One of the more widely used methods for treating textiles to impant crease resistance involves impregnation of the textiles with synthetic thermosetting resins. Such

treatment of textiles is mentioned in numerous patents, including U.S. 2,121,697, 2,339,203 and 2,416,884. However, the use of such methods in crease-proofing textiles introduces other problems, such as the tendency of the fabrics to acquire an affinity for chlorine during bleaching and laundering operations utilizing chlorine containing laundering agents. This chlorine-retention leads to serious damage to the cloth when it is ironed after laundering.

Another general type of procedure which has been developed for finishing fabrics in order to obtain products with improved dry-crease recovery properties involves the treatment of the textile with aqueous solutions of formaldehyde and an acid catalyst, followed by drying and heat treating. It has been found that although such procedures provide fabrics which have some improvement in wetcrease recovery, in comparison with untreated material, the improvement in the wet-crease recovery is not sufficient to eliminate the need for final ironing after laundering if the fabrics are to have an acceptable appearance. Processes involving treatment of cellulosic materials with an aldehyde in the presence of an acid catalyst to improve dry-crease recovery of fabrics are disclosed, for example in U.S. Patents 1,558,453, 2,118,685, 2,233,402 and 2,530,175.

It has also been disclosed in British Patent 462.005 that an increase in recovery from dry-creasing can be imparted to cellulosic fiber containing textiles by treatment in a solution containing formaldehyde, an acid, and a high concentration of a salt having a strong affinity for water, but without a specific swelling effect on the cellulosic fibers. In order to produce this effect, the salt, calcium chloride, is used at a concentration of about 70% of its saturation concentration, since at lower concentrations of salt, the beneficial effects desired to be obtained are lost. The wet-crease recovery of cloth treated in this way is not sufficient to eliminate the need for ironing after laundering if the fabric is to have an acceptable ap-

Many other procedures have been described in the technical literature and patents for improving both wet and dry-crease recovery properties of fibrous webs. Many of these require the use of costly reagents, special handling procedures or involved processing equipment. Consequently, because of the highly competitive nature of the textile industry, and the resulting substantial increase in cost involved with the utilization of many of these suggested or patented finishing operations, they have never been utilized in the commercial production of treated textiles.

OBJECTS

A principal object of this invention is the provision of new processes for improving the wet-crease recovery of cellulosic fabrics. Further objects include:

(1) The provision of new and improved cellulosic fabrics which possess remarkable ability to recover during drying from creases imposed upon the fabric while it is in the wet state.

(2) The provision of processes for treating cellulosic fabrics to create products which have sufficient ability to recover from creases imposed in the wet state to eliminate the need for ironing after laundering for the fabrics to have an acceptable appearance.

(3) The provision of methods for improving the wetcrease recovery properties of cellulosic fibers and fabrics comprising cellulosic fibers which may be carried out without need for special handling equipment or costly reagents, so that the textiles may be finished at a reasonable cost to give products which will be competitive with other textiles.

(4) The provision of methods for improving wetcrease recovery of cellulosic fabrics by a treatment which

does not materially affect the dyeing properties of the fabrics.

Other objects and further scope of applicability of the present invention will become apparent from the detailed description given hereinafter; it should be understood, 5 however, that the detailed description, while indicating preferred embodiments of the invention, is given by way of illustration only, since various changes and modifications within the spirit and scope of the invention will become apparent to those skilled in the art from this de- 10 tailed description.

GENERAL DESCRIPTION

These objects are accomplished according to the present invention by the treatment of cellulosic fabrics by 15 first impregnating the fabric with an aqueous solution of a water-soluble aldehyde, a strong acid, and a salt, then holding the textile in an extended condition completely free of any creases, while impregnated with the solution at a temperature between about 0° and 100° C., and a 20 time sufficient to obtain a substantial wet-crease recovery effect, then completely freeing the textile of any of the treating solution and finally drying the solution-free textile. The acidic aqueous treating solution should have the ability to swell the cellulosic fabric at least about as much as pure water and should contain at least about 50 grams per liter of the water soluble aldehyde, at least about 10 grams per liter of the strong acid, and an amount of the salt between about 50 and 400 grams per liter to impart to the solution a pH of less than 0, par- 30 ticularly a pH between about -0.5 and -0.6 as measured potentiometrically using a quinhydrone electrode.

Salts which may be used in the aqueous treating solutions include alkali metal or alkaline earth metal salts of monobasic strong acids, e.g. CaCl₂, or the alkali metal 35 acid salts of polybasic strong acids, e.g. NaHSO₄.

One of the most suitable ways of holding the textile in an extended condition to permit the treating solution to act upon the textile is to tightly wind the solution saturated material around a cylindrical roller, and then wrap 40 a water-tight covering to prevent drying out during the lying period which, for example, would be 24-48 hours at about 20° C.

It has been discovered that by bringing about a reaction between a water-soluble aldehyde and cellulose fibers in the presence of an acid catalyst under such conditions that the fibers are maintained in a more or less fully swollen condition, the ability of the fabric comprising the cellulose fibers to recover from creases imposed on the fabric in the dry state is not substantially affected, but the ability to recover from creases imposed on a fabric in the wet state is increased to such an extent that a final ironing, after laundering, is not necessary in order for the fabric to have an acceptable and wearable appear-

In order to bring about the reaction between the aldehyde and the cellulose, a strong acid catalyst is necessary. With dry processes, it is sufficient to impregnate the cloth with a solution of the aldehyde and a small amount of a strong acid and concentrate the catalyst on the cloth by drying. With wholly wet processes, however, the full 60 concentration of acid catalyst must be present at the start of the operation. This involves the use of high concentrations of strong acids, particularly if the reaction is to be carried out at room temperature or below, and this makes the process difficult to carry out in practice and 65 necessitates the use of special machinery. An important feature of this invention is the discovery that the concentrations of acid used as catalyst can be considerably reduced if inert salts are added to the reaction mixture, the 70 mixture of acid and salt apparently functioning as a stronger catalyst than acid alone, at the same concentration. With a given acid and a given salt, the increase in catalytic effect increases with an increase in concentration of the salt up to the point where the concentra- 75 case.

EXAMPLES

A more complete understanding of the new procedures of this invention and the improved fabrics obtained thereby can be had by reference to the following examples of actual operations in accordance with the invention. In these examples, all parts and percentages are by weight, unless otherwise specified and all pH values are as measured potentiometrically using a quinhydrone electrode in which the usual pH scale, with values of from 0 to 14, is extended to include negative values of pH.

Example 1

A mercerized cotton cloth is impregnated by immersion in an aqueous solution having a pH of about -0.5consisting of 250 g. formaldehyde (40% solution), 150 g. hydrochloric acid (32° Tw.) and 200 g. calcium chloride (anhydrous) per liter. The cloth is withdrawn from the bath of solution and squeezed so that 100 parts of solution are present on each 100 parts of cloth. Immediately after the impregnation, the cloth is wound around a cylindrical roller, care being taken that no creases are introduced into the cloth. The outside of the roll is then covered with a sheet of water-impervious polyethylene film.

The roll of cloth is allowed to lie at 20° C. for 24 hours, after which the roll is unwound and washed in cold water for five minutes, then in an aqueous solution containing 2 g./l. of sodium carbonate at 60° C. for five minutes and finally in cold water for a further five minutes and dried.

Cloth is obtained which appears visually unaltered by the treatment but, after laundering, it is found to dry out substantially free from creases, whereas untreated cloth, after laundering and drying, remains badly creased.

Example 2

A fabric constructed from yarns composed of 33% cotton and 66% polyethylene terephthalate staple, and which has been heat-set and mercerized, is impregnated with an aqueous solution having a pH about -0.6 consisting of 250 g. formaldehyde (40% solution), 100 g. sulphuric acid (98%) and 150 g. sodium chloride per liter, and processed in an identical manner to that of Example 1. The treated cloth, after laundering, dries out substantially free from creases and is markedly better in this respect than cloth which has not been treated.

Example 3

A mercerized cotton cloth is impregnated with an aqueous solution having a pH of -0.5 consisting of 250 g. formaldehyde (40% solution), 150 g. hydrochloric acid (32° Tw.) and 250 g. lithium chloride per liter, and processed as described in Example 1. The treated cloth is printed with a printing color composed of:

100 g. Caledon Brilliant Purple 4RS (I.C.I.)

30 g. glycerine

60 g. sodium carbonate

120 g. sodium formaldehyde sulphoxylate

690 g. dextrin thickener

dried, steamed for ten minutes, oxidized in an aqueous solution of 2 g. potassium dichromate and 5 g. acetic acid per liter for two minutes at 60° C., soaped in a solution of 2 g. of soap per liter for ten minutes at 100° C., rinsed in water and dried. The resulting printed cloth can be laundered by any normal method and it is found to dry out substantially free from creases.

In another case, the lithium chloride in this example is replaced by 200 g. of magnesium chloride. The resulting printed cloth is found to be substantially equivalent in appearance and properties to the cloth of the first

In yet another case, 300 g. of sodium nitrate are used instead of the lithium chloride and comparable results are

Example 4

A mercerized cotton cloth is impregnated with an 5 aqueous solution having a pH of -0.5 consisting of 250 g. formaldehyde (40% solution), 200 g. trichloracetic acid, and 200 g. calcium chloride (anhydrous) per liter, and processed as described in Example 1. The treated cloth is then saturated by immersion in an aqueous solution containing 100 g. dimethylol urea and 10 g. ammonium dihydrogen phosphate per liter, dried, heattreated for five minutes at 150° C., washed off in dilute sodium carbonate solution, and dried. The treated cloth shows a high degree of recovery from creases imparted to it in either the dry or the wet condition.

Example 5

A mercerized cotton cloth is impregnated as described in Example 1, but after being wound around the cylin- 20 drical roller, the roll of cloth is allowed to lie at 40° C. for four hours, and then washed off as described in Example 1. The improvement in wet crease resistance of the cloth is similar to that obtained by treating for twentyfour hours at 20° C.

In another case, similar results are obtained by allowing the roll of cloth to lie for 72 hours at 10° C. In yet another case, similar results are obtained by allowing the cloth roll to lie for 1½ hours at 60° C.

Example 6

A mercerized cotton cloth is impregnated with an aqueous solution having a pH of -0.5 consisting of 250 g. acetaldehyde, 150 g. hydrochloric acid (32° Tw.), and 100 g. calcium chloride (anhydrous) per liter and proc- 35 essed in an identical manner to that of Example 1. Although the wet crease recovery angle of the treated cloth is less than that of cloth treated with formaldehyde, nevertheless, after laundering, it dries out substantially free from creases.

Example 7

A mercerized cotton cloth is impregnated with a water solution having a pH of -0.6 consisting of 250 g. pyruvic aldehyde (30% solution), 150 g. hydrochloric 45 acid (32° Tw.), and 200 g. calcium chloride (anhydrous) per liter and processed as described in Example 1.

It is found that the effect produced in the cloth is less marked than in the case of the same cloth treated with formaldehyde. However, the cloth is found to dry out, 50 after laundering, substantially free from creases.

In another case, the pyruvic aldehyde is replaced in the operations of this example with 500 g. hydroxy adipaldehyde (25% solution). It is found that the resulting treated cotton cloth is very similar in appearance and 55 crease recovery properties to the cloth made with pyruvic aldehyde.

In yet another case, the pyruvic aldehyde is replaced with 250 g. glyoxal (30% solution) and a comparable treated cotton cloth is obtained.

Example 8

A spun viscose cloth is impregnated by immersion in an aqueous solution having a pH of -0.6 consisting of 500 g. formaldehyde (40% solution), 150 g. hydrochloric 65 acid (32° Tw.), and 200 g. calcium chloride (anhydrous) per liter. The impregnated cloth is squeezed so that 100 parts of solution are present on each 100 parts of cloth. Immediately after the impregnation, the cloth is wound around a cylindrical roller, and the outside of the roll 70 covered with a sheet of water-impervious film. The roll of cloth is allowed to lie at 20° C. for 48 hours. Then, the cloth is unwound from the roll and is washed in cold water for five minutes, then in an aqueous solution containing 2 g./l. of sodium carbonate at 60° C. for five 75 salt, thereby reducing the effective hydrogen-ion concen-

minutes and finally in cold water for a further five minutes and dried. There is obtained a treated cloth which exhibits, in a very pronounced manner, the ability to recover from creases imposed upon it in the wet state.

DETAILED DESCRIPTION

An essential reagent of the new fabric treating procedures of the invention is a water-soluble aldehyde. aldehyde should be sufficiently soluble in water that at least 50 grams per liter of the aldehyde may be dissolved in water or strongly acidic aqueous solutions containing a substantial concentration of salt. Most effective results are obtained in the new procedures using formaldehyde as the water-soluble aldehyde. Commercial formalin, which contains about 37% to 40% active formaldehyde is a good reagent for use in conducting the new fabric treating operations, but other sources of formaldehyde and formaldehyde donors may be used, e.g., paraformaldehyde. Examples of other aldehydes which may be used include glyoxal, ethyl glyoxal, propionaldehyde, isobutyraldehyde, acetaldehyde, pyruvic aldehyde, hydroxy adipaldehyde and comparable water-soluble aldehydes. Mixtures of aldehydes may be used.

The aqueous treating solutions may contain the watersoluble aldehyde in various concentrations. Preferably, however, the aldehyde should be present in a concentration of at least 50 grams per liter. Considerably higher concentrations than this may be used and will be generally limited by the upper limit of solubility of the aldehyde used, or stability of the concentrated aldehyde solution in the presence of the accompanying acid catalyst and modifying salt. Generally, a concentration of aldehyde above about 300 grams per liter of treating solution provides no extra benefit for the required additional amount of aldehyde.

Another essential reagent of the new operations is a catalyst material consisting of a strong acid, i.e., an acid having an ionization constant of at least 0.01. Hydrochloric acid is the preferred acid for use in the new operations, but other acids which may be used include sulfuric acid, phosphoric acid and trichloroacetic acid. Less preferred acids include dichloroacetic acid, phosphorous acid, sulfurous acid, pyrophosphoric acid, naphthalene sulfonic acid and comparable strong acids. Compatible mixtures of acids may be used if desired.

The concentration of the strong acid in the treating solution may be varied. Preferably, the solution should contain at least 10 grams per liter of active acid material. Higher concentrations may be used, but the concentration of acid in conjunction with the inert salt in the solution should be such that swelling of the cellulosic material being treated is not appreciably reduced below its swelling in pure water. Preferably, an upper limit of about 300 grams per liter of active acid material should be observed in forming the new fabric treating solutions.

The increase in the catalytic activity of the acid by the addition of an inert salt is presumably explainable in terms of the increase in activity of the acid by the action of the salt; it is therefore limited to salts formed from strong acids. Any water soluble metal salt of a strong monobasic acid is capable of producing this effect, but with multi-basic acids, only the acid salts can be used, since the neutral salts of such acids are converted in the first place into acid salts, when added to aqueous solutions of strong acids, and the effective hydrogen ion concentration of the solution is reduced. Thus, Na₂SO₄ does not increase the activity of the solution when added to aqueous sulphuric or hydrochloric acid, but NaHSO4 does.

The salt used in forming the treating solutions should be inert with respect to the particular acid being used. Thus, the salt should not interact with the acid, either to form insoluble compounds, to liberate volatile materials or to be transformed from a neutral salt into an acid 7

tration of the solution. Furthermore, the salt should have sufficiently high solubility in the acid solution to provide the mixture with a pH of less than 0 as measured potentiometrically with a quinhydrone electrode. If the limitations upon choice of salt used in forming the treating solutions as described above are observed, it has been found that satisfactory treating solutions are produced, if the concentration of salt is between about 50 and 400 grams per liter of treating solution.

The preferred inert salt used in forming the solutions 10 is calcium chloride, used in concentrations of 100 to 350 grams per liter of solution, but other usable salts include magnesium chloride, sodium chloride, sodium bisulfate, sodium nitrate, sodium trichloroacetate, monosodium phosphate, potassium chloride, lithium chloride, and comparable alkaline metal and alkaline earth metal salts of strong acids. Also usable, but less preferred, are other metal salts such as aluminum chloride, zinc chloride, and the like. Compatible mixtures of salts may be used.

The activity of the acidic treating solution will vary 20 to some extent depending upon the particular acid and inert salt employed in the formation of the solution. As previously indicated, the quantity of salt used in conjunction with the acid employed should be such that the actual degree of swelling obtained in the treatment of the cellulosic fibers is not appreciably less than that which occurs in pure water alone. For treating solutions of this type, the most convenient method of expressing the activities of the mixtures is in terms of their pH values as measured potentiometrically using a quinhydrone electrode. In such measurements of pH values, the usual pH scale, with values of 0 to 14, extends to cover mixtures having greater apparent acidity and includes negative values of pH. In order to be effective in producing the desired crease recovery properties in cellulosic fabrics, treating solutions should have a pH value of below 0. Solutions with a pH as measured by a quinhydrone electrode of between -0.2 and -0.8 are effective and solutions having a pH between about -0.5 and -0.6 are preferred, when working at a temperature of 20° C.

Solutions containing 250 g./l. of formaldehyde (40%) with a pH of -0.5 to -0.6 can be produced by the following varied combinations of strong acids and salts of strong acids:

50 g./l. hydrochloric acid (32° Tw.), 300 g./l. calcium chloride

150 g./l. hydrochloric acid (32° Tw.), 100 g./l. calcium chloride

100 g./l. hydrochloric acid (32° Tw.), 200 g./l. magnesium chloride

100 g./l. hydrochloric acid (32° Tw.), 200 g./l. lithium chloride

100 g./l. sulphuric acid (98%), 150 g./l. sodium chloride

100 g./l. trichloroacetic acid, 300 g./l. calcium chloride

The effectiveness of the process, as regards its ability to impart an improved recovery from wet creasing, is best determined directly by measuring the angle of recovery after creasing the material under standard conditions. It has been found that the angle of recovery increases progressively as the amount of aldehyde fixed on the cloth increases. The relationship between the angle of recovery and the amount of aldehyde fixed is not, however, linear; at first, the angle of recovery increases rapidly with increase in the amount of aldehyde fixed, but after a certain point, further improvement in the angle of recovery is only obtained by considerable increases in the amount of aldehyde fixed. The optimum amount of formaldehyde required to be fixed on cellulosic fibers appears to be about 0.6% and this amount gives angles of recovery from wet creasing of 140° or better. The amount of aldehyde fixed on the fabric may be determined by the increase in weight of the fabric before and offer and

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treatment as measured by weighing the dried fabric after conditioning at controlled humidity.

The impregnation of the cellulosic fabric with the treating solution and the period of holding the impregnated fabric in an extended condition free of creases in order to permit reaction to occur between the cellulosic fibers and the treating solution, can be carried out at various temperatures within the range of about 0° C. to 100° C., although best results are obtained from a viewpoint of control and final fabric properties employing temperatures for impregnation and subsequent reaction within the range of about 10° C. to 60° C. The time of treatment varies as an inverse function of the temperature, shorter times being required, the higher the temperature, and vice versa. It is most convenient to carry out the process at or near room temperature (18-22° C.) and at this temperature the time of treatment of the solution impregnated cloth in the extended condition is preferably of the order of 24-48 hours. Operation at other conditions of time, temperature and pH values of the solution can be used if desired. Thus, pH values of the solution lower than -0.5 to -0.6 can be used if it is desired to work at temperatures lower than 20° C., and/or times shorter than 24 hours, and conversely, pH values higher than -0.5to -0.6 can be used if it is more convenient to work at temperatures higher than 20° C., and/or times longer than 48 hours. It will be possible for the operator to easily determine the optimum conditions of time, temperature and pH values for any special treating solution by observing the limits as outlined above and using the results obtained at a pH value of between -0.5 to -0.6at a temperature of about 20° C. and a time of treatment between about 24 and 48 hours as a guide.

The impregnation of the cellulosic fabric can be carried out by any known method, such as padding, spraying, dipping alone or followed by hydro-extraction. The fabric may be treated when it is saturated with the treating solution, i.e., when it contains all of the treating solution which the fabric is capable of holding in an extended condition after removal from the treating bath. However, the process can be operated with the fabric impregnated to an extent less than complete saturation. Satisfactory results have been obtained with cloth which has been impregnated with about 40 to 150 parts by weight of solution per 100 parts of the dry weight of the fabric.

The term "cellulosic fabrics" as employed in the specification and the accompanying claims, means fabrics made from a substantial percentage of cotton, linen, viscose rayon or comparable cellulosic fibers or mixtures of such fibers. The fabrics may be woven or knit textiles, nonwoven fabrics or comparable fibrous webs. Furthermore, the fabrics may be made of mixtures of cellulosic fibers and non-cellulosic fibers and such mixture of fibers can occur as spun mixed-fiber yarn or as monofilaments or single-fiber yarn knitted or woven in varying amounts in the formation of the fabric. Non-cellulosic fibers may be natural or synthetic yarns or monofilaments made of materials which are not decomposed by the treating solutions under the conditions of treatment as specified. Particularly good results have been obtained in treating fabrics composed of mixtures of cellulosic fibers with noncellulosic fibers such as cellulose acetate, polyethylene terephthalate and polyvinylidene chloride. Many noncellulosic fibers are substantially inert toward the aldehyde treatment as herein described, but with mixture fabrics containing cellulosic fibers, especially in an amount of 25% or more of the total fabric content, the overall improvement in wet-crease recovery can be sufficient to enable the fabric to be laundered without need for subsequent ironing in order to possess a satisfactory wearable appearance.

appears to be about 0.6% and this amount gives angles of recovery from wet creasing of 140° or better. The amount of aldehyde fixed on the fabric may be determined by the increase in weight of the fabric before and after 75 procedures mentioned above may be carried out in stand-

ard textile processing equipment. Likewise, hydro-extracting, retention of the fabric in an extended condition while impregnated with the treating solution, washing and drying may be carried out on standard textile handling apparatus. The retention of the solution-impregnated fabric in an extended condition completely free of creases can, for example, be carried out by keeping the fabric stretched on a frame or by festooning, but the most satisfactory method appears to be to tightly wind the impregnated fabric around a cylindrical roller, while maintaining 10 the fabric completely free of creases.

CONCLUSION

In the foregoing specification, there have been described new processes for improving the wet-crease recovery 15 of cellulosic fabrics in which a reaction between an aldehyde and cellulosic fibers takes place entirely in the wet state under such conditions that the textile is swollen to an extent at least about as great as the fibers swell in water alone. This treatment is effected by the use of 20 specified mixtures of water-soluble aldehydes with strong acids and metal salts of strong acids under controlled con-

ditions of temperature, pH and the like.

The described procedures produce cellulosic fabrics having new and improved properties of wet-crease recovery. The treated fabrics are visually unchanged from the appearance of the untreated goods and the dyeing properties of the fabrics are substantially unaltered, so that the treated fabrics can be dyed, printed and finished by normal methods used in the textile finishing industry. One type of finishing operation which may be usefully combined with the new treating procedures of this invention is the finishing of the treating fabrics with synthetic thermosetting resins known to be useful in improving the dry-crease recovery of cellulosic fabrics. As is known, synthetic thermosetting resins are not particularly effective in improving the wet-crease recovery of cellulosic fabrics, and this is particularly troublesome in connection with finishing of cellulosic fabrics with thermosetting resins which are subject to chlorine-retention, since ironing following laundering with chlorine-containing bleaches or the like will cause deterioration of the fabric. By treating according to the processes herein described prior to resin finishing, it is thus possible to obtain a fabric having a high degree of crease recovery, both in the wet and dry states, and more extensive use of the less expensive thermosetting resins which are subject to chlorine-retention can be made in the finishing of textiles. In addition, fabrics treated in accordance with this invention may be given mechanical finishes by the usual methods of embossing, glazing or compressive shrinking.

Having provided a complete description of the invention in such manner as to distinguish it from other inventions and from what is old, and having provided a description of the best mode contemplated of carrying out the invention, the scope of patent protection to be granted the invention is defined by the following claims.

cellulose fibers to impart to the fabric the ability to recover from creases imposed on the fabric in the wet state sufficient to substantially reduce the need for ironing after laundering which comprises impregnating said fabric with at least about 40 parts by weight per 100 parts of the dry weight of the fabric of an aqueous solution of a water-soluble aldehyde selected from the group consisting of formaldehyde, glyoxal, hydroxy adipaldehyde, and pyruvic aldehyde, an acid having an ionization constant of at least 0.01 selected from the group consisting of sulfuric, phosphoric, trichloracetic and hydrochloric acids and a water-soluble metal salt selected from the group consisting of calcium chloride, magnesium chloride, sodium chloride, sodium bisulfate, sodium nitrate, sodium trichloracetate, monosodium phosphate, potas- 75 solution-free fabric. 10

sium chloride and lithium chloride, said solution containing between about 50 to 300 g./l. of said aldehyde, between about 10 to 300 g./l. of said acid and an amount between 50 and 400 g./l. of said salt, the concentration of said acid in conjunction with said salt being such as to impart to the solution a pH between about -0.2 and -0.8, and a swelling of the cellulose fibers of the fabric at least about as great as the swelling in pure water, said concentrations being in g./l. of solution, and causing a reaction to occur between the aldehyde and the cellulose fibers of said fabric while holding the fabric in an extended condition completely free of any creases in the wet state swollen with said solution by so maintaining the swollen fabric at a temperature between about 0° and 100° C. for a time corresponding at the temperature used to between about 24 to 48 hours at 20° C., said time being an inverse function of the temperature until a wet crease-proofing effect is obtained, then completely freeing the fabric of all said solution and drying the solution-

2. A process for the treatment of fabrics formed of cellulose fibers to impart to the fabric the ability to recover from creases imposed on the fabric in the wet state sufficient to substantially reduce the need for ironing after laundering which comprises impregnating said fabric with between about 40 and 150 parts by weight per 100 parts of the dry weight of the fabric of an aqueous solution of 50 to 300 g./l. of formaldehyde, 10 to 300 g./l. of hydrochloric acid, and an amount of calcium chloride between 50 and 400 g./l., the concentration of the acid in conjunction with the calcium chloride being such as to impart to the solution a pH of between -0.5 and -0.6 and a swelling of the fabric at least about as great as the swelling in pure water, said concentrations being in g./l. of solution, and causing a reaction to occur between formaldehyde in said solution and the cellulose fibers of said fabric while holding the fabric in an extended condition completely free of any creases in the wet state swollen with said solution by so maintaining the swollen fabric at a temperature between about 0° and 100° C. for a time corresponding at the temperature used to between about 24 to 48 hours at 20° C., said time being an inverse function of the temperature until a wet crease-proofing effect is obtained, then completely freeing the fabric of all said solution and drying the solutionfree fabric.

3. A process as claimed in claim 2 wherein said calcium chloride is present in an amount between 100 and 350 g./l. of said solution.

4. A process for the treatment of fabrics formed of cellulose fibers to impart to the fabric the ability to recover from creases imposed on the fabric in the wet state to substantially reduce the need for ironing after laundering which comprises impregnating said fabric with between about 40 and 150 parts by weight per 100 parts of the dry weight of the fabric of an aqueous solution of 50 to 300 g./l. of formaldehyde, 10 to 300 g./l. of hydrochloric acid, and an amount of alkali metal chloride between about 50 and 400 g./l., the concentration 1. A process for the treatment of fabrics formed of 60 of the acid in conjunction with the alkali metal chloride being such as to impart to the solution a pH of between about -0.2 and -0.8, and a swelling of the fabric at least about as great as the swelling in pure water, said concentrations being in g./l. of solution, and causing a 65 reaction to occur between formaldehyde in said solution and the cellulose fibers of said fabric while holding the fabric in an extended condition free of any creases in the wet state swollen with said solution by so maintaining the swollen fabric at a temperature of between about 0° and 100° C. and a time corresponding at the temperature used to between 24 to 48 hours at 20° C., said time being an inverse function of the temperature until a wet crease-proofing effect is obtained, then completely freeing the fabric of all said solution and drying the 11

5. A process for the treatment of fabrics formed of cellulose fibers to impart to the fabric the ability to recover from creases imposed on the fabric in the wet state to substantially reduce the need for ironing after laundering which comprises impregnating said fabric with between about 40 and 150 parts by weight per 100 parts of the dry weight of the fabric of an aqueous solution having a pH about -0.6 consisting essentially of about 250 g. formaldehyde (40% solution), about 100 g. of sulfuric acid (98%) and about 150 g. sodium chloride 10 per liter of solution, holding the fabric in an extended condition completely free of any creases and causing a reaction to occur between the formaldehyde in the solution and the cellulose fibers of the fabric while in the swollen condition created by impregnation with said solu- 15 tion by so maintaining the swollen fabric at a temperature of about 20° C. for about 24 to 48 hours until a wet

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crease-proofing effect is obtained, then completely freeing the fabric of all said solution and drying the solution-free fabric.

6. A cellulosic fabric which has the ability to recover from creases imposed on the fabric in the wet state sufficient to substantially reduce the need for ironing after laundering as prepared by the process defined in claim 1.

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