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TREATMENT OF FABRIC CONTAINING CELLULOSE ESTERS

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This invention relates to the treatment of yarns or fabrics containing organic esters of cellulose and particularly cellulose acetate with a bath containing an alkaline agent, whereby the fabric may be dyed with cotton dyes, and other of its properties improved.

An object of our invention is to treat yarns or fabrics containing organic esters of cellulose with an alkaline solution in such a manner that the yarn is saponified on the surface, whereby the fabric may be dyed with cotton dyes in a satisfactory manner.

Another object of our invention is to treat yarns or fabrics containing cellulose acetate with alkali solutions of such concentration and under such conditions that the same may be dyed with dischargeable colors, whereby discharge prints may be produced on such fabrics.

The discharge printing of fabric containing organic derivatives of cellulose and particularly cellulose acetate to produce light figures on dark backgrounds has heretofore offered serious difficulties. This is due to the fact that the dark colors, such as reds, blues and blacks that are available for the dyeing of cellulose acetate fabrics cannot be discharged by pastes containing ordinary color discharging agents. We have discovered that if fabrics containing yarns of cellulose acetate yarns are treated with an alkaline bath under the conditions set forth below, intense saponification takes place on the surface of the yarns, so that a fabric is produced which is capable of being dyed with cotton dyes that may be discharged by printing pastes containing ordinary discharging agents. Moreover the fabric, when treated by our process has further desirable properties, such as having its safe ironing point materially increased.

In accordance with our invention, we treat fabrics or yarns made of or containing organic esters of cellulose with a saponifying bath containing an alkaline solution, either of comparatively low concentration at comparatively high temperatures, or of comparatively high concentration at low tempertures. The fabric may then be dyed with suitable dyes, and if desired, the dyed fabric may then be printed with a discharge paste which contains a reducing agent, with or without colors. The printed fabric may then be steamed and further treated in any suitable manner.

The fabric to be treated by our invention may contain yarns of any suitable organic ester of cellulose such as cellulose acetate, cellulose formate, cellulose propionate or cellulose butyrate. The fabric may consist wholly of yarns made of one or more of these organic esters of cellulose, or it may be a mixed fabric containing yarns of these organic esters of cellulose and the yarns of other fibers. Examples of such mixed fabrics are mixed cellulose acetate and cotton and mixed cellulose acetate and silk.

The alkaline bath used for partially saponifying the cellulose acetate contained in the fabric or yarn may be either of low concentration or of high concentration depending on the temperature of treatment. If an alkaline solution of low concentration is used, higher temperatures may be employed in the process, whereas if alkaline solutions of high concentrations are used low temperatures of treatment are employed. If a solution of intermediate concentration is employed, intermediate temperatures of treatment should be used.

The saponifying bath may contain any suitable alkali or alkaline salt for producing hydroxyl ions in aqueous solutions. Of the alkali suitable, sodium hydroxide, potassium hydroxide or ammonia may be mentioned. Examples of alkaline salts are sodium or potassium carbonates, sodium or potassium phenolate, sodium or potassium sulphite, sodium or potassium silicate, or tri-sodium phosphate. In order to promote intense surface saponification of the yarn containing the organic ester of cellulose buffer salts or modifying agents such as soap, sodium acetate, borax or alcohol, etc. may be added to the saponifying bath.

When alkaline solutions of low hydroxyl concentration are employed, the bath should contain at least 0.2 grams of sodium hydroxide or the equivalent amount of other alkaline substances per liter of solution, when temperatures of 80° C. are employed. While

we do not limit ourselves to the specific concentrations, we have found that solutions having a hydroxyl ion content corresponding to 0.4, 0.5, 0.6, 0.7 or even 1.0 grams of sodium hydroxide per liter, give eminently satisfactory results. The temperature of the bath is maintained at 60° to 90° C. and preferably at about 80° C. However, after saponification of the cellulose acetate textile material has been initiated, the saponifying bath may contain less sodium hydroxide, since saponification then proceeds in a bath containing hydroxyl ions in amounts corresponding to less than 0.2 grams of sodium hydroxide per liter. The saponification may proceed to any desired extent, say 3% or 5 to 10% or more. These percentages represent the percent of weight the cellulose acetate has lost through the saponification treatment.

The saponification of the fabric with alkaline solutions of low concentrations may be carried out in a tank, on the jig, on the winch or in any other suitable device. Often it is desirable to thoroughly wet out the fabric prior to subjecting it to the saponification bath. The addition of soap to the saponifying bath is of great advantage in promoting level saponification of the organic esters of cellulose material, and also the presence of the soap in the saponifying bath tends to help to cause the saponification to be concentrated on the surface of the yarn containing the organic esters of cellulose, which causes desirable dyeing properties. While the proportions of soap to the solution may be varied within wide limits, we have found that the presence of 0.5 grams to 10 grams of soap per liter of solution produces satisfactory results.

In one mode of carrying out our invention with the use of solutions containing alkali in low concentrations, the goods are scoured in an open bath on strings or in any suitable machine such as a jig, with a bath containing olive oil soap for an hour or two. The goods may then be removed from the bath and saponified in a separate bath containing fresh water and alkali, but they are preferably saponified directly in the general scouring bath by the addition of a requisite amount of caustic soda. In one mode of partially saponifying the fabric containing the organic ester of cellulose with an alkaline solution of low concentration, the fabric is immersed in a bath of such large volume that it contains sufficient alkali to cause the desired degree of saponification but which is not of such concentration to exceed the maximum permissible concentration of alkali material at the temperature to be employed. Thus for example, in saponifying a fabric consisting wholly of cellulose acetate of approximately 54% acetyl value, we have found that a bath of up to 120 to 180 times the weight of the fabric can advantageously be used, and that such bath should preferably contain an amount of caustic soda to the extent of 4 to 8% of the weight of the fabric, when a bath temperature of 80° C. is used. Obviously, if the fabric or yarn contains other organic esters of cellulose or an alkaline material other than caustic soda is used, or if the temperature of the bath is other than 80° C., the proportions will be varied accordingly. Likewise it is apparent that when smaller amounts of alkaline material are used the volume of the saponification bath may be correspondingly decreased.

In another mode of carrying out our invention when alkaline solutions of low concentrations are employed, the fabric containing the yarns of cellulose acetate is treated in an alkaline bath of comparatively small volume and containing a relatively small amount of caustic soda. As the saponification proceeds, further quantities of caustic soda are added in small increments, until the desired degree of saponification has taken place. Until the operator has gained sufficient experience, it is preferable to check up the amount of caustic soda in the bath by titration with sulphuric acid to insure that the amount of caustic soda added is not sufficient to cause the saponifying bath to have a concentration exceeding the maximum permissible degree.

When concentrated alkaline solutions are used, the concentration of the alkali solution preferably corresponds to from 5 to 20% of caustic soda by weight. We have found that the use of 10% caustic soda solution is suitable for causing the cellulose acetate fabric to take direct cotton dyes in a satisfactory manner. However, if it is also desired to increase the safe ironing temperature, a 20% caustic soda solution is preferred, since by the use of this solution the safe ironing temperature of a cellulose acetate fabric is increased from 220° C. to 260° C. or above. However, no great advantage from the dyeing point of view is obtained by using a caustic soda solution of greater than 10% concentration. The saponifying bath may also contain such assistants as soap, sodium acetate, etc. The alkali solution may be applied to the fabric containing the organic esters of cellulose in any suitable manner. However, we prefer to apply the caustic soda when applied in comparatively concentrated solution to the fabric by padding the caustic solution onto the fabric and then permitting the same to stand for an appreciable length of time as will be described below. The temperature of treatment is preferably room temperature and should not exceed 35° C. for obtaining the best results.

The fabric or yarn containing the organic ester of cellulose when treated with a saponifying solution by any of the above described

methods is intensely saponified on the surface of the yarn. By "intense surface saponification" is meant the concentration of saponification of the cellulose ester material on the surface of the yarn, while the interior of the yarn is very little affected and, therefore, consists essentially of cellulose esters. This is indicated by the fact that when cellulose acetate fabrics are treated in accordance with the above described methods, the solubility of the fabric in acetone is relatively high, and the fabric may be dyed with cotton dyes because of the fact that the surface of the yarn is essentially reconstituted cellulose or approximately so.

The fabric, after it has been treated with the saponifying bath by any of the above methods, as above indicated, is capable of being dyed by both dyestuffs that have an affinity for cellulose acetate but not for cotton, such as are known as SRA dyes, i. e. generally the water-insoluble dyes of the nitrodiarylamine, azo, aminoanthraquinone or other series applied in the form of aqueous colloidal solutions or dispersions prepared with the aid of sulphoricinoleic acid or other dispersing agent and also dyes that have an affinity for cotton, but not for cellulose acetate, such as direct cotton dyes, because of the intense surface saponification of the yarn. The direct cotton colors available for dyeing this material are within all ranges of colors. These cotton dyes include blues, blacks and reds, which may be discharged by ordinary discharge printing pastes, and therefore discharge prints of dark backgrounds may be produced on this fabric,—a result heretofore not readily attaintable on fabrics containing cellulose acetate yarn.

The fabric, so treated and after having been dyed with direct cotton dyes, may be printed with a discharge paste which contains a reducing agent, and which may or may not contain colors for printing the discharge portions. After printing with the discharge paste, the fabric may be steamed and given other treatments as is well understood in the art.

The discharge paste may be applied in any suitable manner, such as by means of engraved or embossed rolls, plates or blocks. The paste may also be applied through stencils by means of brushing or spraying. The discharge paste may contain any suitable reducing agent such as sodium hydrosulphite, formaldehyde zinc hydrosulphite, or the substances known as "protolin" which is sulphoxylate of zinc formaldehyde, etc. For obtaining a white background the sodium hydrosulphite or formaldehyde hydrosulphite are preferred, whereas if a colored discharge is desired the protolin is preferred.

In order further to illustrate our invention the following specific examples are given.

Example I

The following is an example of a method of treating fabrics containing cellulose acetate yarn with an alkali solution of comparatively low concentration at high temperatures in order to render the same capable of being dyed with cotton dyes.

A woven fabric consisting wholly of cellulose acetate yarn is plaited and strung, and is then immersed in a bath in which the ratio of water to fabric is between 130 to 1 to 160 to 1. This bath contains soap to the extent of one gram per liter of bath and the bath temperature is maintained at 80° C. The fabric is treated in this soap bath for three-quarters of an hour.

The fabric is then raised out of the bath, caustic soda in an amount equal to 6% of the weight of the fabric is added to the bath, and after the solution is homogeneous, the fabric is then immersed in the bath and is treated therein for approximately three hours while the bath temperature is maintained at 80° C. The fabric is then lifted, washed off in water at 60° C. and then may be dyed in the manner set forth below.

Example II

The following is an example of a method of partially saponifying a fabric with an alkaline solution of comparatively high concentration and at low temperature.

A fabric consisting wholly of cellulose acetate yarn is padded through rolls that are covered with several thicknesses of heavy cotton lapping or other absorbent material, using a bath containing, by weight, 100 parts of water and 10 parts of caustic soda. The caustic soda solution is at room temperature, and the fabric is given two runs through the pad. The nip on the pad is arranged so that the fabric picks up an amount of solution equal to its own weight. After the second run on the pad, the roll of fabric is allowed to run 15 minutes, and the solution is then very lightly extracted on the roll. The fabric with the remaining solution is allowed to stand overnight at room temperature. In carrying out this process, it is important to give due heed to the strength of the caustic soda solution and to the temperature of treatment, which should preferably not exceed 35° C. Likewise it is of importance that the fabric should be batched with a minimum amount of tension, and that the caustic soda solution should be allowed to remain on the fabric for a period comparable to that of overnight.

After standing overnight, the fabric is rinsed in water and is ready for dyeing.

The fabric that has been treated by either of the specific methods described in Examples I and II may then be dyed with direct cotton dyes or with basic cotton dyes.

Of the direct cotton dyes that may be used, the following are examples of those suitable for a light fastness equal to 75 hours of the fadeometer.

National solantine blue 4–GL
Pontamine fast black CW (Color Index 539)
Pontamine yellow CH (Color Index 365)
Newport direct fast red F (Color Index 419),
Amanil black BOL (American Aniline Prod. Co.) (Color Index No. 493)
Newport light fast blue SFF
Chlorantine fast blue RL (Ciba) see Color Index Supp. page 31.
Chlorantine fast blue 8–GL (Ciba) see Color Index Supp. page 31

If a diazotized and developed dye is desired; Diaminogene extra (General Dyestuffs Corp.) (Color Index No. 317) or diazo fast green BL (General Dyestuffs Corp.) (Color Index No. 532) may be applied to the fabric and the same then diazotized and developed with beta naphthol.

For obtaining a navy color of good fastness to light (75 hours fadeometer) the following is an example of a mixture that may be used:

National solantine blue 4–GL
Pontamine black CW (Color Index 539)
Pontamine yellow CH (Color Index 365)
Newport direct fast red F (Color Index 419)

For a navy of modern fastness to light the following mixture of dyes may for instance be used.

Diazine black H extra (Color Index No. 401)
Chlorazol sky blue FF (Color Index No. 518)
Pontamine yellow CH (Color Index No. 365)
Newport fast scarlet 4–BA (Color Index No. 326)

For obtaining a black with good fastness to light pontamine black CW is an example of a dyestuff that may be used.

If it is desired to obtain discharge printing effects, the fabric, after it has been dyed with any of the above mentioned dyestuffs, may be printed with a discharge paste containing

	Parts
Gum tragacanth 1/12	100
Protolin	40
Water	40

This paste is most suitable for obtaining colored discharges. If a white discharge is desired the protolin is replaced by a hydrosulphite, such as zinc hydrosulphite or sodium hydrosulphite and any suitable desired color is added to the paste. The fabric after printing is dried and steamed and then finished in any desired manner.

It is to be understood that the foregoing detailed description is given merely by way of illustration and that many variations may be made therein without departing from the spirit of this invention.

Having described our invention what we claim and desire to secure by Letters Patent is:

1. Method of treating textile materials containing organic esters of cellulose comprising treating the same with an alkaline solution under such conditions that intense surface saponification of the organic ester of cellulose material takes place and then dyeing the so treated textile material with dispersed relatively water insoluble dyestuffs.

2. Method of treating textile materials containing cellulose acetate comprising treating the same with an alkaline solution under such conditions that intense surface saponification of the cellulose acetate material takes place and then dyeing the so treated textile material with dispersed relatively water insoluble dyestuffs.

3. Method of treating fabrics containing cellulose acetate comprising treating the fabric with an alkaline solution of low concentration and at comparatively high temperature whereby intense surface saponification of the cellulose acetate takes place and then dyeing the same with dispersed relatively water insoluble dyestuffs.

4. Method of coloring materials containing fibres of superficially saponified organic esters of cellulose comprising applying thereto dispersed relatively insoluble dyestuffs.

5. Method of coloring materials containing fibres of superficially saponified cellulose acetate comprising applying thereto dispersed relatively insoluble dyestuffs.

6. A fabric containing yarns of organic esters of cellulose that are intensely saponified on the surface thereof, said fabric being colored with dispersed relatively insoluble dyestuffs.

7. A fabric containing yarns of cellulose acetate that are intensely saponified on the surface thereof, such fabric being colored with dispersed relatively insoluble dyestuffs.

In testimony whereof, we have hereunto subscribed our names.

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