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TEXTILES, FILMS, AND THE LIKE

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This application is a continuation in part of application S. No. 173,870 and S. No. 173,871, both filed November 10, 1937, and contains the subject matter of both said applications and no other subject matter. The invention relates to the treatment of fabrics composed of or containing filaments or fibres of cellulose, regenerated cellulose or an organic derivative of cellulose, and has as its principal object an improved process for increasing the resiliency of such materials.

In treating materials of the kind referred to above with a view to forming within the filaments or fibres a synthetic resin or other water-insoluble elastic substance of very high molecular weight so as to increase the resilience, some proportion of the resin-forming substance or substances applied is almost invariably left as a coating on the outside of the filaments or fibres. When under the action of heat, polymerisation or condensation of the resin-forming substances applied to the materials occurs, a certain amount of the resulting resinous substance is formed outside the filaments. This exterior coating of the filaments has a deleterious effect on the physical properties of the material. We have now found that this effect may be prevented by subjecting the materials to a process adapted to break up the continuity of such exterior coating and preferably to remove the same.

The mechanical treatment may comprise drawing the material past and pressing it against, driven studded rolls, e. g., passing the material through the passes of pairs of rolls, one roll of each pair being armed with studs or projections, the other being resilient so that the studs or projections may sink into its surface, or both being studded, the studs of the one being staggered relative to those of the other. The material is preferably caused to pass in succession through a number of such pairs of rolls. An alternative method is to draw the fabric by means of rolls in contact with a sharp edge, for example the edge of a square steel bar or of a blade which may or may not be flexible. The bars or blade may be caused to move relative to the fabric and one method of applying the treatment of the invention is to pass the fabric in contact with a roll armed with blades. A further method is to subject the fabric to a beating operation, for example by drawing it between a fixed buffer and a reciprocating or swinging member adapted to beat the material against the buffer. Another method is to crumple the fabric and pass it several times in the crumpled state through rolls, the crumpling being effected in a different direction after each passage through the rolls. Whatever the method adopted, it should be such as to break the continuity of the film or coating surrounding the individual filaments of the material without injuring the fabric.

After subjection to the breaking operation the fabric may be treated so as to remove the broken up material of the coating. This may be done, for example, by means of an air-blast or by a brushing operation.

The invention is applicable quite broadly to processes in which a resinous substance is formed in the filaments or fibres of a fabric comprising cellulose, regenerated cellulose or an organic derivative of cellulose by polymerisation or condensation of a substance or substances applied to the materials. The resinous substance formed in the materials must be a solid, relatively elastic water-insoluble substance of very high molecular weight which can readily be formed in the materials under the action of heat or components capable of being introduced into the materials.

Among the synthetic resins special mention may be made of those formed by condensation of aldehydes and ketones, for example acetaldehyde, benzaldehyde, acetone and particularly formaldehyde, with amines or amides, such as urea, thiourea, guanidine, monomethyl, monoethyl, methyl ethyl, phenyl phenyl-ethyl and phenyl-methyl urea dicyandiamide, aniline, methyl-aniline, diphenylamine, benzyl-aniline, α-naphthylamine, para-toluene-sulphonamide and other aromatic sulphonamides and N-alkyl and other derivatives thereof, and phenols, particularly ordinary phenol; from carboxylic acids containing more than one carboxyl group, for example succinic and phthalic acids and alcohols containing more than one hydroxy group, for example glycol, diethylene glycol and glycerol; and from unsaturated compounds, for example acrylic acid, acrolein, and particularly vinyl esters.

The mechanical treatment may be applied to the material after the heating step which results in the formation of the desired resinous substance, or before condensation or polymerisation to form the resinous substance has proceeded to completion. Thus, after application of the substance or substances which are to condense or polymerise in the material to form the desired substance, the materials may be heated to a temperature insufficient to induce the condensation or polymerisation but sufficient to convert the resin-forming substance or substances adhering

to the exterior of the filaments into a substantially dry coating and this coating may be broken up or removed before the condensation or polymerisation stage.

The following examples illustrate the invention:

Example 1

A bath is made up as follows:

In a 40% aqueous solution of formaldehyde an equimolecular proportion of urea is dissolved, 6% of boric acid is added and the solution is boiled until condensation has progressed almost to the point of water-insolubility. This can be ascertained by removing small samples from time to time and boiling further until a sample removed, when further boiled for a few seconds, precipitates on cooling. The solution of the condensation product is then diluted to about quarter strength.

A fabric of regenerated cellulose artificial silk is soaked in this bath for about 20 minutes. It is then removed, dried at about 60° C. and passed in turn through the passes between a number of positively driven wooden rollers armed with metallic studs. This breaks up the coating of resinous matter which is present on the materials after the drying step. On emerging from the last set of rollers the material passes between two revolving brushes extending over the whole width of the fabric and arranged so as to remove the disintegrated resinous matter. Suction means may be provided to receive the resinous matter so removed. The material is then transferred to a heated chamber in which it is maintained at a temperature between 120° and 180° C. for about 10 minutes.

Example 2

The process is carried out as in Example 1 except that the material is subjected to the heat treatment immediately after drying and is then subjected to the mechanical treatment.

Example 3

A fabric of regenerated cellulose is soaked in a 5% aqueous solution of urea containing 0.5% of boric acid for 15–20 minutes, dried at 40–50° C. and exposed in a closed chamber to an atmosphere of formaldehyde vapour at a temperature of 100° C. for two hours. The material is then removed, rinsed in warm soapy solution, dried and subjected to the mechanical treatment described in Example 1.

Example 4

A fabric of regenerated cellulose is soaked for 15 minutes in aqueous solution containing 20% of acetone, 2% of urea, 2% of hexamethylene tetramine and .1% of ammonium persulphate. The material is removed from the bath, mangled so that it retains about its own weight of solution, dried at 50–60° C. and subjected to the mechanical treatment described in Example 1 after which it is transferred to a heated chamber where it is maintained at a temperature of 130–140° C. for 1½–2 hours.

Example 5

A fabric of regenerated cellulose is soaked in an aqueous solution containing 2% of phenol and 2% of paraformaldehyde. After immersion in this solution for 15 minutes, the material is removed and subjected in turn to the drying, mechanical treatment and final heat treatment described in Example 1.

Example 6

The process is carried out as described in Example 5 except that the treating bath consists of an aqueous solution containing 2% of phenol, 2% of hexamethylene tetramine and .1% of ammonium persulphate.

Example 7

The process is carried out as in Example 5 except that the bath is an aqueous solution containing 3% of dimethylol urea and .2% of ammonium dihydrogen phosphate. In a similar way materials of natural cellulose can be treated.

In treating materials of cellulose or regenerated cellulose instead of the acid catalysts specified alkaline catalysts can be used, e. g., small quantities of caustic soda or sodium carbonate, particularly in the initial stages of condensation.

Instead of the mechanical treatment specified, the material may be passed through the passes of a series of pairs of rolls, each pair consisting of a studded roll of non-resilient material in contact with a roll covered with rubber or the material may be passed between a fixed buffer and a swinging or reciprocating member adapted to beat the fabric against the buffer. The buffer and the swinging and reciprocating member may be of wood and either or both may be coated with fabric.

Example 8

A fabric of cellulose acetate artificial silk is soaked in a 20% aqueous solution of acetone until highly swollen. A bath is then made up as follows:

In a 10% aqueous solution of formaldehyde an equimolecular proportion of urea is dissolved, 3% of formic acid is added and the solution is boiled until condensation has progressed almost to the point of water-insolubility. This can be ascertained by removing small samples from time to time and boiling further until a sample removed, when further boiled for a few seconds, precipitates on cooling. The solution of the condensation product is then diluted to about half strength.

The fabric is soaked in the bath for about 20 minutes. It is then removed, dried at about 60° C. and subjected to the mechanical treatment of Example 1. Suction means are provided to receive the resinous matter so removed. The material is then transferred to a heated chamber in which it is maintained at a temperature between 120° and 180° C. for about 10 minutes.

Example 9

The process is carried out as in Example 8 except that the material is subjected to the heat treatment immediately after drying and is then subjected to the mechanical treatment.

Example 10

A fabric of cellulose acetate artificial silk, which has been saponified non-superficially until the acetyl number calculated as acetic acid is about 25, is soaked in a 2% aqueous solution of urea containing 0.2% of boric acid for 15–20 minutes, dried at 40–50° C. and exposed in a closed chamber to an atmosphere of formaldehyde vapour at a temperature of 100° C. for two hours. The material is then removed, rinsed in warm soapy solution, dried and subjected to the mechanical treatment described in Example 8.

Example 11

The process is carried out as in Example 10

except that the urea is applied in solution in 20% aqueous acetone.

Example 12

A fabric of cellulose acetate artificial silk is soaked for 15 minutes in aqueous solution containing 20% of acetone, 2% of urea, 2% of hexamethylene tetramine and 0.1% of ammonium persulphate. The material is removed from the bath, mangled so that it retains about its own weight of solution, dried at 50–60° C. and subjected to the mechanical treatment described in Example 1 after which it is transferred to a heated chamber where it is maintained at a temperature of 130–140° C. for 1½–2 hours.

Example 13

A fabric of cellulose acetate artificial silk is soaked in an aqueous solution containing 2% of phenol, 2% of paraformaldehyde and 0.1% of formic acid. After immersion in this solution for 15 minutes, the material is removed and subjected in turn to the drying, mechanical treatment and final heat treatment described in Example 1.

Example 14

The process is carried out as described in Example 13 except that the treating bath consists of an aqueous solution containing 2% of phenol, 2% of hexamethylene tetramine and 0.1% of ammonium persulphate.

Example 15

The process is carried out as in Example 13 except that the bath is an aqueous solution containing 20% of acetone, 3% of dimethylol urea and 0.2% of ammonium dihydrogen phosphate.

The invention is of particular importance in connection with the treatment of fabrics of regenerated artificial silk formed for example by the viscose, or cuprammonium processes or by the complete saponification of organic esters of cellulose and of fabrics made of or containing cellulose acetate. Fabrics treated according to the invention may contain in addition to filaments, fibres or yarns of organic derivative of cellulose, filaments, fibres or yarns of other materials, e. g., cellulosic or regenerated cellulosic materials, materials of animal origin or artificial materials not derived from cellulose. The invention, moreover, includes the treatment of materials composed of or containing filaments or fibres of yarns of natural cellulose alone or in admixture with other materials.

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

1. Process for increasing the resilience of fabrics having a basis of materials selected from the group consisting of natural cellulose, regenerated cellulose and organic derivatives of cellulose, which comprises impregnating the fabric with resin-forming matter in solution, said resin-forming matter being capable of conversion into a water-insoluble synthetic resin under the influence of heat, drying the fabric, subjecting it to mechanical treatment adapted to break up any film formed by the resin-forming matter on the outside of the fabric, removing the disrupted film and converting the resin-forming matter remaining in the material to a water-insoluble synthetic resin under the influence of heat.

2. Process for increasing the resilience of fabrics having a basis of materials selected from the group consisting of natural cellulose, regenerated cellulose and organic derivatives of cellulose, which comprises impregnating the fabric with resin-forming matter in solution, said resin-forming matter comprising an aldehyde and a substance selected from the class consisting of urea, thio-urea and dicyandiamide, drying the fabric, subjecting it to mechanical treatment adapted to break up any film formed by the resin-forming matter on the outside of the fabric, removing the disrupted film and converting the resin-forming matter remaining in the material to a water-insoluble synthetic resin under the influence of heat.

3. Process for increasing the resilience of fabrics having a basis of materials selected from the group consisting of natural cellulose, regenerated cellulose and organic derivatives of cellulose, which comprises impregnating the fabric with resin-forming matter in solution, said resin-forming matter comprising urea and formaldehyde, drying the fabric, subjecting it to mechanical treatment adapted to break up any film formed by the resin-forming matter on the outside of the fabric, removing the disrupted film and converting the resin-forming matter remaining in the material to a water-insoluble synthetic resin under the influence of heat.

4. Process for increasing the resilience of fabrics having a basis of cellulose acetate, which comprises impregnating the fabric with resin-forming matter in solution, said resin-forming matter being capable of conversion into a water-insoluble synthetic resin under the influence of heat, drying the fabric, subjecting it to mechanical treatment adapted to break up any film formed by the resin-forming matter on the outside of the fabric, removing the disrupted film and converting the resin-forming matter remaining in the material to a water-insoluble synthetic resin under the influence of heat.

5. Process for increasing the resilience of fabrics having a basis of cellulose acetate, which comprises impregnating the fabric with resin-forming matter in solution, said resin-forming matter comprising an aldehyde and a substance selected from the class consisting of urea, thio-urea and dicyandiamide, drying the fabric, subjecting it to mechanical treatment adapted to break up any film formed by the resin-forming matter on the outside of the fabric, removing the disrupted film and converting the resin-forming matter remaining in the material to a water-insoluble synthetic resin under the influence of heat.

6. Process for increasing the resilience of fabrics having a basis of cellulose acetate, which comprises impregnating the fabric with resin-forming matter in solution, said resin-forming matter comprising urea and formaldehyde, drying the fabric, subjecting it to mechanical treatment adapted to break up any film formed by the resin-forming matter on the outside of the fabric, removing the disrupted film and converting the resin-forming matter remaining in the material to a water-insoluble synthetic resin under the influence of heat.

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