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TREATING TEXTILE MATERIALS AND ARTICLES MADE THEREFROM

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12 Claims. (Cl. 28—1)

This is a divisional application and relates to the subject-matter which has been divided out from my copending application for patent Serial Number 41,175, filed September 18, 1935.

This invention relates to methods of treating textile materials to improve their appearance and more particularly to methods of treating lustrous textile materials such as yarns, and woven, knitted or other textiles or articles made therefrom in order to reduce their luster. Although the invention is of primary usefulness in the treatment of yarns and fabrics made from viscose, or other forms of regenerated cellulose artificial silk, or cellulose esters or ethers for instance cellulose acetate artificial silk, in other words rayon in general, it also can be used to advantage for reducing the luster of natural silk and mercerized materials.

For some years after the introduction of synthetic textiles the high luster of these materials was considered a desirable feature, but a change in taste caused the brilliantly lustrous yarns to lose favor for most purposes, and now there is a decided preference for yarns and fabrics of dull appearance.

This invention therefore, has as an object the provision of methods of improving the appearance of textiles in accord with present day taste. A further object is to provide treatments for textiles which will give them a dull finish highly resistant to removal by laundering. Other objects and advantages of the invention will be pointed out in the following description or will be apparent from such description. These objects are accomplished by my invention wherein various dulling agents are applied to textile materials, or articles made therefrom, in a medium comprising a dilute aqueous caustic alkali solution of a cellulose derivative of the kind incompletely soluble in aqueous caustic alkali solution under ordinary conditions, but soluble therein at low temperature, and wherein the impregnated textiles are subsequently treated with an acid to desolubilize the coating medium. By the definition, "cellulose derivative of the kind incompletely soluble in aqueous caustic alkali solution under ordinary conditions", is meant a group of low-substituted cellulose derivatives comprising hydroxyalkyl cellulose (glycol ether of cellulose) methyl and ethyl ethers of cellulose and certain inorganic esters of cellulose, e. g. the sulfates containing a low percentage of the ester group. To further clarify the meaning of "low-substituted" as used herein it may be explained that a low-substituted cellulose derivative is one in which comparatively few of the replaceable hydroxyl hydrogens of the cellulose molecule are replaced or substituted by etherifying or esterifying radicals. The adjective "low" does not refer to the molecular weight of the radicals entering the cellulose molecule as substituents, nor to any of their other characteristics, but merely to the proportion of these radicals introduced.

At the present state of development in the art, applicant is unaware of a better way to describe the particular cellulosic compounds in which he is interested than by the definition "low-substituted", (that is, with comparatively few etherifying or esterifying radicals introduced), and by pointing out the striking solubility characteristic. This method, it is thought, sharply defines the compounds under consideration to those experienced in the art.

It is known that these cellulose bodies when specially prepared under conditions to assure low substitution in the cellulose unit have the property of being substantially insoluble in water or aqueous caustic alkali at ordinary temperatures, but are capable of being brought into solution in a dilute aqueous solution of a caustic alkali by chilling (in some cases for a predetermined time) below 0° C. until crystals of ice appear, or else by cooling to a lesser stage (+1 to +3° C.) and using a colloid mill or dispersing agents or soluble colloids to aid solution, as described in my British Patent 410,152. Once dissolved, the temperature of the alkaline solution of cellulose ether or ester can be raised to ordinary or room temperature without causing precipitation or any other bad effect.

In the preferred form of this invention it is desirable that the concentration of caustic alkali should not substantially exceed 5% and the percentage of cellulose derivative dissolved therein should be very low, for example, in the neighborhood of 2 parts per hundred. The proportion of dulling agent to cellulose derivatives may be varied widely.

As dulling agents, there may be employed numerous suitable materials such as zinc oxide, titanium oxide, stannic oxide, tin phosphate, china clay, barium sulphate, antimony oxide and compatible mixtures of these. Materials of this nature may be applied either in suspension as such in the alkaline cellulose derivative solution, or certain of them may be applied in solution in the form of alkali soluble compounds, in which latter case the dulling material is afterward precipitated by suitable means upon the textile mate-

rial or article during the operation of fixing the cellulosic medium.

In the operation of the process of this invention the textile material or article made therefrom is passed through a dilute aqueous caustic soda bath containing the dulling agent and cellulose derivative and after thorough wetting it is passed through pressure rolls to remove the excess liquid. Alternatively, the solution may be applied to one side only of the fabric, in which case it may be advisable not to pass the treated fabric through nip rolls if there is danger of forcing the liquid through the fabric.

The fabric is next treated with an agent to neutralize alkalinity and in that way fix the pigmented cellulosic coating on the textile fibers. This may be accomplished by means of a bath of dilute aqueous acid, for example, sulfuric, phosphoric, hydrochloric, formic or acetic acid. Or, if preferable because of the properties of the dulling agent used, there may be substituted an atmosphere of a mildly acid gas such as carbon dioxide. Thus in the case of titanium oxide, stannic oxide, china clay or barium sulphate either a bath of dilute aqueous acid or an atmosphere of carbon dioxide may be used, whereas in the case of zinc oxide which is more easily soluble in an acid bath, the treatment with carbon dioxide is to be preferred. In this case the zinc oxide will be partly or wholly converted into carbonate.

When the dulling material is applied in the form of an alkaline solution, the acid chosen for fixing the cellulose derivative must be such as to produce an insoluble precipitate of pigment; for example, when sodium stannate is employed, dilute phosphoric acid may properly be used as precipitating agent.

The acid treated material is passed into a faintly alkaline washing bath and then is washed with water until the wash waters are neutral.

The material is next dried and further finished in known manner.

If desired, the alkaline suspension or solution containing the dissolved cellulose derivatives and the dulling agent may also contain wetting agents such as sulfonic acids of high molecular weight or softening materials such as the product sold under the registered trade mark, "Cirrasol S A" (a mixture of cetyl alcohol and cetyl sodium sulfonate).

This invention is illustrated but not limited by the following examples, in which the parts are by weight.

Example 1

A glycol cellulose ether of the kind incompletely soluble in caustic soda of any concentration but capable of solution on chilling in 5% sodium hydroxide solution until crystals of ice appear is brought into solution in sodium hydroxide, by mixing 2 parts of the glycol cellulose with 100 parts of 4.5% sodium hydroxide solution in the presence of 1% calculated on weight of glycol cellulose, of the dispersing agent sold under the registered trade mark "Perminal" (an isopropylated naphthalene sulfonate) according to the method described in British Patent No. 410,152. 6 parts of finely ground zinc oxide are then introduced into the solution, and the whole is stirred. 2 parts of the paste sold under the registered trade mark "Cirrasol S A" extended with 12 parts of water is then added to the bath. A length of woven viscose fabric is then passed rapidly through the bath so the period of contact is about 2 seconds, and subsequently passes through the nip rolls to rubber covered rollers and thereafter into a chamber containing an excess of carbon dioxide until the glycol cellulose has been coagulated. The cloth is then washed, dried, and broken.

Example 2

A length of knitted cellulose acetate fabric is passed into a bath made up as in Example 1, containing titanium dioxide instead of zinc oxide. After passing through the nip rolls, the fabric is passed rapidly through a bath containing 3% sulphuric acid, after which it is passed through a neutralizing bath of very dilute ammonia, and is then washed with water, and dried.

Where desired, the de-lustered textiles or articles made therefrom can be dyed with suitable dyes, and it will be understood that when the material is to be dyed it is usually necessary that the dulling agent should be white.

Lustrous textile materials are effectively delustered by the comparatively simple, inexpensive non-hazardous procedure of this invention, and the dull appearance is remarkably permanent even when the treated materials are submitted to frequent laundering.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that I do not limit myself to the specific embodiments thereof except as defined in the appended claims.

I claim:

1. In a delustring process, the steps which comprise impregnating lustrous textile materials and articles made therefrom with a pigment-carrying dilute aqueous caustic alkali solution of a low-substituted cellulose derivative of incomplete solubility in dilute caustic alkali solution at room temperature but completely soluble therein at temperatures below 10° C., and thereafter neutralizing alkalinity of the treated article with acid.

2. A process according to claim 1 in which the pigment used is zinc oxide.

3. A process according to claim 1 in which the pigment used is titanium dioxide.

4. A process according to claim 1 in which the pigment used is barium sulfate.

5. In a delustring process, the steps which comprise impregnating lustrous textile materials and articles made therefrom with an aqueous solution containing a dulling pigment, and by weight less than 10% of caustic alkali and less than 5% of a low-substituted cellulose derivative of incomplete solubility in dilute caustic alkali solution at room temperature but completely soluble therein at temperatures below 10° C., and thereafter neutralizing alkalinity of the treated article with acid.

6. In a delustring process, the steps which comprise impregnating lustrous artificial and natural silks and articles made therefrom with a pigmented dilute aqueous caustic alkali solution of a low-substituted cellulose ether of incomplete solubility in dilute caustic alkali solution at room temperature but completely soluble therein at temperatures below 10° C., and thereafter neutralizing alkalinity of the treated article with acid.

7. A process as claimed in claim 6 in which the impregnating solution also contains wetting and dispersing agents.

8. A process according to claim 6 in which the

impregnating solution also contains a mixture of cetyl alcohol and cetyl sodium sulfonate.

9. In a delustring process, the steps which comprise impregnating lustrous artificial and natural silks and articles made therefrom with a pigment-carrying dilute aqueous caustic alkali solution of a low-substituted alkyl cellulose of incomplete solubility in dilute caustic alkali solution at room temperature but completely soluble therein at temperatures below 10° C., and thereafter neutralizing alkalinity of the treated article with acid.

10. A process according to claim 9 in which the alkyl cellulose is methyl cellulose.

11. A process according to claim 9 in which the alkyl cellulose is ethyl cellulose.

12. In a delustring process, the steps which comprise impregnating lustrous artificial and natural silks and articles made therefrom with a pigment-carrying dilute aqueous caustic alkali solution of a low-substituted glycol ether of cellulose of incomplete solubility in dilute caustic alkali solution at room temperature but completely soluble therein at temperatures below 10° C., and thereafter neutralizing alkalinity of the treated article with acid.

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