Patented Aug. 26, 1930 1,774,184

UNITED STATES PATENT OFFICE

CHARLES WILFRED PALMER AND STEPHEN MILLER FULTON, OF SPONDON, NEAR DERBY, ENGLAND, ASSIGNORS TO CELANESE CORPORATION OF AMERICA, A CORPORATION OF DELAWARE

TREATMENT OF THREADS, FABRICS, OR OTHER MATERIALS COMPOSED OF OR CONTAINING ARTIFICIAL FILAMENTS AND PRODUCT

No Drawing. Application filed November 16, 1925, Serial No. 69,525, and in Great Britain May 26, 1925.

This invention relates to the treatment of threads, knitted, woven or other fabrics or other materials composed of or containing filaments or fibres (hereinafter included in the term filaments) or threads of cellulose acetate, in which the filaments are in a partially or completely delustred condition, and the object of the invention is to provide means whereby the lustre may be restored partially or completely to the partially or completely delustred cellulose acetate filaments in such goods.

When cellulose acetate artificial silk, and particularly "dry-spun" cellulose acetate artificial silk made from cellulose acetate solutions of relatively low concentrations, is subjected to the action of certain agencies such as hot or boiling acid solutions or solutions of acid salts or hot or boiling water, particularly water between about 90° C. and the boiling point, or moist steam, the cellulose acetate artificial silk can lose its natural high lustre to a greater or less extent and become converted to a more or less delustred condition.

The application of processes in which such delustring of cellulose acetate artificial silk occurs, for the production of threads or goods showing the effects of wool, hair or other modified effects, forms the subject of U. S. Patent No. 1,554,801, according to which lustrous cellulose acetate filaments or threads, or goods containing the same, are subjected to the action of agents whereby the cellulose acetate filaments or fibres are delustred to any desired extent and more or less crinkled. The filaments or fibres of cellulose acetate resulting from such treatment of said patent are in fact "hollow" filaments, due probably to the action of the delustring and crinking agents in releasing in or from the filaments residual traces of solvent present in the filaments.

The present invention on the other hand concerns the treatment of cellulose acetate artificial silks which have been deprived wholly or partly of their natural lustre by the action of delustring agencies, for the purpose of restoring or imparting lustre thereto. The term "delustred" as used in the specification and claims includes partially or completely delustred threads, fabrics, etc. of cellulose acetate. The term "materials" as used in the claims is to be interpreted as including threads, fabrics and other goods.

Some qualities of cellulose acetate artificial silk are much more resistant than others to delustring agencies, the resistance varying with the quality of the cellulose acetate of which the threads or filaments are made and also with the concentration of the cellulose acetate solutions used for spinning the same. In particular, threads or filaments made with very high quality cellulose acetates substantially undegraded in the cellulose molecule are especially resistant to delustring agencies.

Further, artificial silks spun from cellulose acetate solutions of high concentrations, e. g. containing from 20 to 25 or 30% or more of cellulose acetate, are not so liable to become delustred as those produced from less concentrated solutions, containing for example under about 10% or up to about 16 or 18% of cellulose acetate.

Delustring may, however, occur even with very resistant qualities of cellulose acetate artificial silk under prolonged or especially intensive action of said agencies.

Hitherto it has not been possible to restore the lustre to cellulose acetate artificial silk which has become partially or entirely delustred.

The object of the present invention is to provide means for restoring or imparting lustre completely or to any desired degree to partially or entirely delustred cellulose acetate filaments or fibres in threads, knitted, woven or other fabrics or other goods containing them.

We have found that by treatment of the threads or other goods composed of or containing such delustred filaments or fibres with solutions of salts,—preferably neutral salts,—or similarly acting agents, like sugars such as cane sugar, the lustre of the cellulose acetate filaments or fibres can be partially or entirely restored, and that any desired degree of relustring may be obtained according to the salt or body employed, the concentration and temperature of the solu-

tion and the duration of the treatment. In practice the conditions of concentration, temperature and duration are adapted together according to the relustring power of the agents used and to the degree to which the lustre is desired to be restored or imparted.

In carrying out the invention the relustring is preferably performed with more or less heated or hot or boiling solutions, but we do not limit ourselves in this respect.

In particular we have found that solutions of ammonium sulphate, sodium sulphate, potassium sulphate, aluminium sulphate, chromates of potassium, sodium, ammonium or aluminium, chlorides of ammonium, sodium, potassium and calcium and like neutral salts are especially useful for the present invention in concentrations of, for example, about 1 to 5% or more, though we do not limit ourselves to any particular concentrations.

The following examples are given to illustrate how the invention may be carried out, it being understood that they are given only as examples and in no way as limitative, and that the concentrations and other conditions may be varied widely.

Example 1

Cellulose acetate artificial silk in fabric or other form which has become partially or entirely delustred, for instance by steaming for about 10 minutes, is boiled with a 2% to 3% solution of ammonium sulphate in water or a 1% to 6% solution of sodium sulphate in water, whereby the relustring is gradually effected. The normal lustre can be substantially restored by about 10–40 minutes boiling or longer at lower temperatures; the treatment may be stopped at any desired intermediate stage. The goods may be rinsed in water to remove the salt after removal from the bath.

Example 2

Cellulose acetate artificial silk in fabric or other form, which has become partially or completely delustred, for instance by boiling with water for about 10 minutes, may be relustred by boiling with an aqueous solution of 3% to 5% strength of ammonium or sodium sulphate. The relustring takes place gradually and can be interrupted at any desired degree; the lustre can be restored substantially to normal by about 15 to 60 minutes boiling in the solution, or the treatment may be stopped at any desired intermediate stage. The goods may be rinsed free from salt after the removal from the solution.

Example 3

Cellulose acetate artificial silk in fabric or other form which has been partially or completely delustred for instance by treatment with boiling water for about 5 minutes or with water at about 95° C. for about 10 minutes may be relustred gradually to any desired extent by heating or boiling with a 5%–10% cane sugar solution in water; with higher concentrations, lower temperatures may be employed. The lustre can be restored substantially to normal by about 10–20 minutes boiling, or the treatment may be stopped at any desired intermediate stage. The goods may be rinsed free from sugar after removal from the solution.

The threads or other goods composed of or containing the delustred filaments may with advantage be treated with one or more solvents or swelling agents, especially solutions of one or more solvents or swelling agents, at ordinary or only moderately raised temperatures prior to treatment with the solutions before mentioned, especially if the latter are intended to be applied hot or boiling. This is especially of advantage when treating badly delustred material, for instance "mixed" threads fabrics or goods of cellulose acetate and wool in which the celulose acetate filaments or threads have become badly delustred in finishing or other processes involving exposure for considerable time to moist steam or boiling solutions. Any substance which is a solvent or swelling agent for cellulose acetate may be usefully employed; those which we have found especially useful for the purpose comprise ammonium sulphate, ammonium thiocyanate, alcohol, and water.

By way of example, goods which have been badly delustred for example by boiling with water for one or two hours or exposure to moist steam for considerable time, may be treated in a solution of a swelling agent at a temperature preferably not exceeding about 35°–40° for about 1 hour to about 12 hours, after which the goods are removed and treated, with or without rinsing, with the hot or boiling solutions above referred to in the manner hereinbefore described.

The following example is given by way of illustration, it being understood it is only illustrative and in no way limitative.

Example 4

Cellulose acetate artificial silk in any form which has become badly delustred, for example through boiling with water for one or two hours, is soaked for about 12 hours in water at 20°–32°C. or a 5% ammonium sulphate solution in water at 20°–30° C. or a 10% ammonium thiocyanate solution in water at 20°–30° C. or a 60% (by volume) solution of alcohol in water at 20°–30° C. After soaking, the goods are removed (rinsed if desired) and then boiled with a 1% to 4% solution of ammonium sulphate, sodium sulphate or other neutral salt in water for about 1 to 5 hours or more according to the degree of more or less complete delustring to be

cured or the extent to which the lustre is to be restored. After boiling for the required length of time the goods may be removed and rinsed free of salt.

The rapidity and degree of relustring of the cellulose acetate filaments in the present invention may be varied by varying any of the working conditions, e. g. concentration of the solutions of the salts, sugar or other similarly acting relustring agents, duration of relustring treatment and temperature of said treatment. Also when a pretreatment with solutions of solvents or swelling agents is employed, the concentration of the solutions of swelling agents, temperature and duration of this treatment or any of these may be varied as required.

The present invention affords valuable technical advantages in that it allows of restoring or imparting lustre to any desired degree to goods consisting of or containing cellulose acetate artificial silk which has become dulled or delustred as the result of various processes or treatments to which such goods are commonly submitted in industry, for example, dyeing, crêping, scouring and so forth. Especially the invention affords valuable technical advantages with regard to "mixed" yarns, fabrics or goods containing cellulose acetate artificial silk associated with wool or any other fibres. In many processes it is necessary to subject such mixed goods to conditions which cause partial or entire delustring of the cellulose acetate portion. Thus in dyeing the wool portion of a mixed yarn or fabric of cellulose acetate artificial silk and wool, it is necessary to subject the goods to the action of hot or boiling dyebaths, often for considerable time, whereby the cellulose acetate portion of the goods becomes wholly or partially delustred. Again in "crabbing" and "steaming" mixed fabrics containing wool and cellulose acetate artificial silk, it is necessary to subject the fabric at the stretch to the action of boiling water and with moist steam respectively, which may cause the cellulose acetate portion of the goods to become wholly or partially delustred. The processes of the present invention enable the more or less delustred cellulose acetate portion of such mixed goods to be relustred completely or to any desired degree.

It will be seen that the invention provides means not only of restoring the lustre entirely or practically so to delustred cellulose acetate filaments, etc. but also of restoring or imparting the lustre to any desired degree, which is of advantage in many technical aspects. For example it is thus possible to restore or impart the lustre to a modified degree resembling that of natural silk or other fibres or to any other desired degree for other special effects. The term imparting lustre, therefore, is used in the claims to include the various degrees of restoration of lustre obtainable by this process.

What we claim and desire to secure by Letters Patent is:—

1. A process for imparting lustre to delustred cellulose acetate filaments in materials containing the same which comprises treating the material with an aqueous solution of a neutral salt.

2. A process for imparting lustre to delustred cellulose acetate filaments in materials containing the same which comprises treating the material with an aqueous solution of a neutral sulphate.

3. A process for imparting lustre to delustred cellulose acetate filaments in materials containing the same which comprises treating the material with an aqueous solution of ammonium sulphate.

4. A process for imparting lustre to delustred cellulose acetate filaments in materials containing the same which comprises boiling the material with an aqueous solution of a neutral salt.

5. A process for imparting lustre to delustred cellulose acetate filaments in materials containing the same which comprises boiling the material with an aqueous solution of ammonium sulphate.

6. A process for imparting lustre to delustred cellulose acetate filaments in materials containing the same which comprises treating the material with an aqueous solution of a swelling agent for cellulose acetate and afterwards heating with an aqueous solution of a neutral salt.

7. A process for imparting lustre to delustred cellulose acetate filaments in materials containing the same which comprises treating the material at a temperature not exceeding about 35–40° C. with an aqueous solution of a swelling agent, and afterwards at a higher temperature with an aqueous solution of a neutral salt.

8. A process for imparting lustre to delustred cellulose acetate filaments in materials containing the same which comprises treating the material at about ordinary temperatures with an aqueous solution of a thiocyanate and afterwards boiling with an aqueous solution of a neutral salt.

9. As new products, materials comprising relustred filaments of cellulose acetate.

In testimony whereof we have hereunto subscribed our names.

CHARLES WILFRED PALMER.
STEPHEN MILLER FULTON.