# UNITED STATES PATENT OFFICE

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

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When cellulose acetate artificial silk, especially "dry-spun" cellulose acetate artificial silk made from cellulose acetate solutions of relatively low concentrations, is exposed to 5 the action of certain agencies, such as hot or boiling solutions of acids or acid salts, or hot water, and particularly water at from about 90° C. to the boiling point, or moist steam, the cellulose acetate artificial silk can lose its 10 natural high lustre to a greater or less extent and become converted into a more or less lustreless condition.

The application of processes in which such delustring of cellulose acetate artificial silk 15 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, threads, 20 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 re-25 sulting from such treatment of the said patent are in fact "hollow" filaments, due probably to the action of the delustring and 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 lustre thereto.

Some qualities of cellulose acetate artificial silk are much more resistant than others to delustring agencies, this 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 60 the lustre to cellulose acetate artificial silk which has become partially or entirely de-

The object of the present invention is to provide means for restoring or imparting 65 lustre completely or to any desired degree to partially or entirely delustred cellulose acetate filaments or fibres (hereinafter included in the term filaments) in threads, knitted, woven or other fabrics or other goods contain- 70 ing them. The term delustred as used in the specification and claims includes partially or completely delustred threads, fabrics, etc., of cellulose acetate. The terms material as used in the claims is to be interpreted as including threads, fabrics and other goods.

According to the invention we treat the crinkling agents in releasing in or from the threads, fabrics or other goods consisting of filaments residual traces of solvent present, or containing cellulose acetate artificial silk which is or has become partially or entirely 80 delustred, with aqueous solutions or dilutions (hereinafter referred to as aqueous swellingliquids) of one or more swelling-substances, namely substances which are solvents or swelling agents for cellulose acetate and 85 which are more or less soluble in or are miscible with water. As solvents or swelling agents we may employ acetic acid, phenol, benzyl alcohol, triacetin, cyclohexanone, ammonium thiocyanate or other thiocyanates, or 90 other substances capable of dissolving or swelling cellulose acetates. As regards liquid solvents or swelling agents, it is preferable to employ those having a higher boiling point than water, though we do not limit ourselves 95 in this respect, as we may use solvents or swelling agents or similarly acting substances

having lower boiling points than water.

The solvents or swelling agents will be employed in such dilution that their action is

capable of restoring or imparting the lustre to the desired degree without causing disintegration or permanent deformation of the cellulose acetate filaments or fusing or stick-

5 ing together of the filaments.

In performing the invention we may steep the threads, fabrics or other goods composed of or containing the more or less delustred filaments in a solution or dilution of greater 10 or less strength of one or more of the aforesaid solvents or swelling agents, the duration of the treatment varying according to the degree of lustrelessness of the filaments, the extent to which lustre is to be restored, or im-15 parted, the concentration of the solvents or swelling agents and the temperatures em-

The temperature at which the treatment is carried out will vary with the concentration 20 of the solvent or swelling agent, but it should in no case be sufficiently high to cause the filaments or threads to fuse or stick together or to become permanently deformed; for example the treatment is preferably performed 25 at substantially ordinary temperatures, i. e.

not exceeding about 30°-40° C.

The most suitable strength of solution or dilution to be employed is that which will just fall short of causing the filaments to 30 stick or fuse together or cause permanent deformation after about 10 to 20 hours treatment at substantially ordinary temperatures. Such solutions or dilutions we have found are capable of restoring the lustre almost com-35 pletely in about 1 to 3 or 5 hours at about 20° C. More concentrated solutions or dilutions of the solvents or swelling agents may be employed to relustre in a shorter time, but care should be exercised in their use. 40 for example solutions or dilutions which relustre in 10 to 20 minutes at a given temperature may cause the filaments to fuse or stick together or become permanently deformed if the treatment is prolonged for any substan-45 tial length of time. For practical purposes the solutions or dilutions which relustre in about 3 hours at substantially ordinary temperatures are the most useful, as it is safe to leave the material in such for any time up to 50 about 10 hours without danger of causing the filaments to fuse or stick together or become permanently deformed.

After steeping the goods to be relustred in the before mentioned solutions or dilutions of 55 solvents or swelling agents for the requisite length of time according to the degree of lustre to be restored or imparted, they are removed and dried or allowed to dry preferably without rinsing. The excess solution may 60 with advantage be removed by draining or whizzing (centrifuging) before the goods are allowed to dry. The drying is preferably performed at or about ordinary temperature, i. e. not exceeding about 35°-40° C., but 65 higher temperatures may be used according After soaking for the required length of time 130

to the solution used. The drying may be When dry, rehastened by blowing air. maining traces of solvent may be removed by

blowing, heating or light scouring.

The above indications are only given by 70 way of illustrating some practical forms of execution, and are not to be taken as constituting any limitation of the broad underlying feature of restoring lustre partially or completely to wholly or partially delustred cellu- 75 lose acetate filaments or threads or other goods containing them, by treatment with solutions or dilutions of solvents or swelling agents.

Example 1

More or less lustreless filaments or threads of cellulose acetate or goods composed of or containing such threads or filaments may be relustred by steeping in an aqueous solution of acetic acid containing about 15% to 25% by weight acetic acid. The time necessary to effect relustring may vary from about 30 minutes to about 12 hours, according to the degree of lustrelessness to be cured or the extent to which it is desired to relustre, and to the temperature and concentration of the bath. Treatment for about one hour with a 25% (by weight) solution of acetic acid in water at about 20° C. is in most cases sufficient to restore the lustre to the goods, but if the goods 95 are tightly twisted, or tightly woven, longer treatment may be necessary; for example a solution which effects relustring in normal cases in about 30 minutes may take up to about 2 hours to effect relustring of tightly twisted or tightly woven goods. After soaking, the goods are removed and excess solution may be drained or whizzed off and the goods may be spread out to dry without rinsing. When dry, the last traces of acid may be removed by rinsing and the goods soaped and finished as desired, for example by a light scouring at about 40° C.

Example 2

110 More or less lustreless goods may be soaked in an aqueous solution of phenol, for example a 1.5% solution, at a suitable temperature, for example about 20° C., whereby the goods are gradually relustred. The duration of the 115 treatment may vary according to the degree of lustrelessness to be cured or the extent to which it is desired to relustre, and to the temperature and concentration of the phenol solution. It is complete when a test sample 120 shows that lustre has been restored to the desired degree. For instance and as an example, practically lustreless goods may be fully relustred completely or practically completely by treatment with a 1.5% solution of 125 phenol in water for about 3 hours at about 15°-20° C. Where the goods treated are tightly woven or tightly twisted, longer treatment may be necessary than with others.

the goods are removed and dried, preferably without rinsing, the drying being performed in a manner similar to that given in Example 1. When dry residual or remaining solvent may be driven off by heating, with or without blowing.

# Example 3

An aqueous solution of benzyl alcohol may 10 be employed as the relustring agent, for instance an approximately saturated aqueous solution (about  $1-1\frac{1}{2}\%$ ) of benzyl alcohol. The duration of the soaking or steeping in the solution will vary with the degree of lustre-15 lessness to be cured or the extent to which it is desired to relustre and to the temperature and concentration of the solution. It is complete when a test sample shows that lustre has been restored to the desired extent. Usu-20 ally treatment for about three hours at about 20°-30° C. in a saturated aqueous solution of benzyl alcohol will be sufficient to restore the lustre completely or practically so to dull goods. Where, however, the goods are tightly 25 twisted or tightly woven, longer treatment may be necessary. After soaking for the re-quired length of time the goods are removed and dried, preferably without rinsing, in the manner indicated in Example 2.

### Example 4

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In using a solution of triacetin for the purpose of the invention we may steep the goods in an aqueous solution, for example a satu-35 rated (about 1%) solution of triacetin at a suitable temperature, for instance about 20°-30° C. The duration of the treatment will vary according to the degree of lustrelessness to be cured or the extent to which it is 40 desired to relustre and to the temperature and concentration of the solution. It is complete when a test sample shows that lustre has been restored to the desired extent. Usually treatment for about three hours at 20°-30° C. in 45 a saturated solution aqueous of triacetin will be sufficient to restore the lustre completely or practically so to dull goods. With fightly twisted or tightly woven goods longer treatment may be necessary. After soaking, the 50. goods are removed and dried as in Examples 2 and 3.

#### Example 5

In using a solution of cyclohexanone, we may for instance employ a 5%-7% aqueous solution thereof at about 20° C. The duration of the steeping will vary with the degree of lustrelessness to be cured or the extent of relustring desired. Usually a 5%-7% solution of cyclohexanone in water at about 20° C. will restore the lustre to dull goods in about one to five hours, though, where the goods are tightly twisted or woven, longer treatment may be required. After steeping for the required time, the goods are removed and dried as in Examples 2, 3 and 4.

# Example 6

Threads or other goods consisting of or containing delustred filaments of cellulose acetate are steeped in a suitable aqueous solution of ammonium thiocyanate or other thiocyanate, for example a solution containing 200–250 grams of ammonium thiocyanate per litre. By treating the goods in this solution for about 1 hour at ordinary temperature, the lustre can be restored. The goods may then be removed from the bath and the excess solution be removed by whizzing or draining. The goods are then preferably spread out and dried at ordinary or only moderately raised temperatures, not over about 35° C. When dry the goods may be rinsed, soaped and finished as desired.

It will be understood that in practising the invention the treatment with the relustring agents may be stopped at any desired point according to the degree of lustre required in the goods.

It will further be understood that the restoring or imparting of the lustre to the cellulose acetate filaments in the present invention may be effected to any desired degree and that the rapidity and degree of relustring may be varied by varying any of the working conditions, e. g. concentration of the solutions or dilutions of the solvents or swelling agents, duration of relustring treatment and temperature of said treatment.

The present invention affords valuable technical advantages in that it allows of re- 100 storing 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 105 goods are commonly submitted in industry, for example dyeing, creping, 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 other fibres. Thus in many processes or treatments it is necessary to subject the mixed goods to conditions which may cause partial or complete delustring of the cellulose 115 acetate portion. For example, in dyeing mixed goods containing wool and cellulose acetate, the high temperatures of the baths necessary to dye the wool portion may cause the cellulose acetate to be largely or entirely 120 delustred. Further, in "crabbing" "steaming" mixed fabrics of wool and cellulose acetate, it is necessary to treat the fabric at the stretch with boiling water and with moist steam respectively, which may cause 125 the cellulose acetate artificial silk portion to become partially or completely delustred. The process of the present invention enables the cellulose acetate portion of such delustred goods to be relustred which has not hereto- 130

may be restored to any desired degree.

means not only of restoring the lustre entirely, 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 im-10 part 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 15 restoration of lustre obtainable by this process.

What we claim and desire to secure by Let-

ters Patent is:

1. A process for imparting lustre to de-20 lustred cellulose acetate filaments in materials containing the same which comprises treating said material with a liquid comprising water and a swelling substance for cellulose acetate, and afterwards, without ma-25 terial reduction of the ratio of swelling substance to water in the liquid retained by the filaments, treating the material for the removal of water by evaporation.

2. A process for imparting lustre to de-30 lustred cellulose actetate filaments in materials containing the same which comprises treating said material with a liquid comprising water and a swelling substance for cellulose acetate, and then without rinsing 35 treating the material for the removal of

water by evaporation.

3. A process for imparting lustre to delustred cellulose acetate filaments in materials containing the same which comprises 40 treating said material with a liquid comprising water and a solvent for cellulose acetate, said solvent having a higher boiling point than water, and afterwards, without material reduction of the ratio of the solvent 45 to water in the liquid retained by the filaments, treating the material for the removal of the water by evaporation.

4. A process for imparting lustre to delustered cellulose acetate in material con-50 taining the same which comprises impregnating said material with a swelling substance for cellulose acetate and afterwards drying the material while still containing the swell-

ing substance.

5. A process for imparting lustre to delustered cellulose acetate in material containing the same comprising treating said material with a solution of a swelling substance for cellulose acetate and afterwards, without material reduction of the ratio of

the solvent in the liquid retained by the material, drying the material.

6. A process for imparting lustre to delustered cellulose acetate in material con-65 taining the same comprising treating said

fore been possible, and moreover the lustre material with an aqueous liquid containing a swelling substance for cellulose acetate, said It will be seen that the invention provides swelling substance having a boiling point above 100° C., and afterwards drying the ma-5 or practically so, to delustred cellulose acetate terial while still containing the swelling substance.

7. A process for imparting lustre to delustered cellulose acetate in material containing the same, comprising treating said material with an aqueous solution of acetic 75 acid and then drying the material in presence

of the acetic acid.

8. A process for imparting lustre to delustered cellulose acetate in material containing the same, which comprises treating said material at temperatures not exceeding 30-40° C. with an aqueous solution containing about 15-25% of acetic acid and then drying the material while still containing acetic acid.

9. A process for imparting lustre to delustered cellulose acetate in material containing the same, which comprises treating said material with a solution of a solvent for cellulose acetate of a concentration less 90 than that capable of causing permanent deformation of the cellulose acetate, and afterwards drying the material in presence of said solvent.

10. A process for imparting lustre to de- 95 lustered cellulose acetate in material containing the same, comprising treating said material with an aqueous solution of a solvent for cellulose acetate of a concentration less than that capable of causing permanent def- 100 ormation of the cellulose acetate, said solvent having a boiling point about 100° C., and afterwards drying the material while still containing said solvent.

11. A process for imparting lustre to de- 105 lustered cellulose acetate in mixed material containing the same associated with other fibres, which comprises treating said material with a solution of a swelling substance for cellulose acetate and afterwards drying 110 the material while still containing said swell-

ing substance.

In testimony whereof we have hereunto subscribed our names.

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