

## UNITED STATES PATENT OFFICE

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## TREATMENT OF TEXTILE MATERIALS

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This invention relates to processes for effecting a reduction in the lustre of cellulose ester and ether materials.

In U. S. Patent No. 1,554,801, is described the delustring of fibres, threads, fabrics or the like made of or containing cellulose acetate or other esters of cellulose, by the action of hot or boiling water or steam or solutions of certain substances. This method of reducing the lustre of cellulose ester and ether materials, and in particular textile materials made of or containing filaments of acetone-soluble cellulose acetate, has been employed on a very extensive scale. The reduction in lustre has generally been effected by subjecting the materials to the action of an aqueous soap solution at temperatures approaching the boiling point. Usually, it is necessary, in order to obtain the desired degree of delustre, to carry out the treatment with the soap solution for a considerable time, for example from  $\frac{1}{2}$ –4 hours or even more, the time depending for example upon the character of the fabric or other material under treatment and the temperature.

It is not generally practicable, therefore, to carry out the delustring operation in a continuous manner, for example on a jig or other machine in which the fabric is passed through the hot soap solution or the like. Such machines cannot be economically employed to maintain fabrics in contact with liquids for periods and at temperatures of the order mentioned. It is obviously particularly desirable that a method should be devised which will enable the delustring of the materials to be carried out in continuous manner.

Experiment has shown that although the delustring action of hot aqueous media on cellulose ester or ether materials may be accelerated by means of certain substances, the desired degree of acceleration necessary to enable continuous working to be employed can usually only be obtained by the use of relatively high concentrations of the accelerators. The use of such high concentrations in a treatment bath is very undesirable, if only on account of the expense.

I have now found that in effecting the uniform delustring of cellulose ester or ether materials, very satisfactory results may be obtained by first applying to or incorporating in the material a delustring accelerator and thereafter subjecting the material to the action of hot aqueous media (that is, hot aqueous liquids or moist steam). By operating in this way it has been found possible to reduce the necessary time of contact between the materials and the delustring media to an extraordinary extent. For example,

the time of contact may be of the order of 30 seconds. My discovery thus enables for the first time the continuous delustring of cellulose ester or ether materials to be effected in a commercially satisfactory manner. A time of contact between the material and the delustring bath of the order of 30 seconds is very easily obtainable in machines in common use in the dyeing and finishing industry for the continuous treatment of textile fabrics with liquids. Moreover, such times of contact may be obtained without sacrificing the high output which can be maintained by such machines.

The invention is of particular value in connection with the treatment of materials made of or containing filaments of acetone-soluble cellulose acetate and will be more particularly described with reference to such materials.

The preliminary incorporation of the delustring accelerators in the materials may be effected in any desired manner. For instance, the accelerator may be incorporated in spinning solutions from which the artificial filaments of cellulose acetate are produced, or it may be incorporated in the filaments at any other desired stage of their manufacture. Most conveniently, the delustring accelerators are applied to the materials to be delustered in the form of solutions, suspensions or dispersions. Simple aqueous solutions may for instance be employed where the accelerator is sufficiently soluble in water. From a practical point of view, however, most satisfactory results are obtained with the aid of more or less water-insoluble accelerators, the latter being applied to the materials in the form of aqueous suspensions or dispersions. Such dispersions may be prepared in any desired manner, for example by means of soap, Turkey red oil or other dispersing agent, for example any of the dispersing agents described in U. S. Patents Nos. 1,618,413, 1,618,414, 1,694,413, 1,690,481, 1,803,008, 1,840,572, 1,716,721, 1,928,647, and 1,959,352.

Other methods of producing dispersions of the delustring accelerators may however be employed, for example mechanical methods. Thus the compounds may be mechanically dispersed in water in the presence of the protective colloid, for example waste sulphite cellulose liquor, with or without a solvent for the compound, e. g. benzene, xylene or other hydrocarbon.

More particularly in the case of delustring accelerators which are solid at ordinary temperatures, it has been found particularly advantageous to assist the dispersion of the accelerators

by means of a liquid solvent therefor, for example a hydrocarbon, e. g., xylene, or a halogenated hydrocarbon, in the manner described for instance in connection with the dispersion of dyestuffs in U. S. Patents Nos. 1,690,481 and 1,803,008.

The concentrations of the solutions or dispersions of delustring accelerators employed may vary within wide limits, depending on the activity of the accelerator and upon the degree of delustre required. Good results may be obtained with the aid of dispersions containing 4 to 10 per cent. of the delustring accelerator. The preparations containing the delustring accelerators, especially when in the form of dispersions of insoluble bodies, are often advantageously applied to the materials at relatively high temperatures, for example 50–85° C. or even higher in order to secure uniform penetration of the material and consequent uniformity of delustre. This is more particularly the case when the delustring accelerator has a relatively low solvent, swelling or plasticizing action on the cellulose ester or ether treated.

The actual application of the solutions or dispersions of the delustring accelerators to the materials may be effected in any convenient manner. I prefer, however, to apply the delustring accelerators by padding or other mechanical impregnation methods, that is methods which enable a predetermined proportion of liquid containing the delustring accelerator to be left in the materials when they are subjected to the delustring treatment proper. A padding mangle for example is conveniently employed, as a machine of this type enables the material to be impregnated thoroughly with the liquid containing a delustring accelerator and yet, by adjustment of the degree of expression, the amount of liquor left on or in the materials may be adjusted within fine limits.

The materials impregnated with the solutions or dispersions of the delustring accelerators are preferably allowed to stand for a time prior to subjecting them to the action of hot aqueous baths or moist steam for the purpose of effecting delustring. The materials may for instance be rolled up as they pass from the padding machine and allowed to stand for the desired period, which may be, for example, from 12–24 hours or more, the precise time depending upon the character of the materials under treatment. Thus, a relatively heavy material such as a heavy crêpe may be with advantage allowed to stand for a longer period than a relatively light material.

The materials impregnated with or containing the delustring accelerators are preferably maintained in a damp state up to the time they are subjected to the action of the hot aqueous delustring media.

The subsequent treatment with hot aqueous media may be effected in any desired manner, for example by steeping the materials therein. The greatest advantages of the new process accrue however, when this treatment is effected continuously, for example by passing the material through a hot aqueous bath or through a chamber supplied with moist steam. As already indicated, such treatment is, in the case of liquid media, very advantageously effected upon a machine of the jig type. The time of passage of the material through the hot aqueous liquid will of course vary according to the degree of delustre

required and according to the activity and proportion of the delustring accelerator which has been applied to the materials.

The delustring proper may be well effected by means of plain hot water as the delustring agent. If desired, however, the treatment liquid may contain any desired additions. Thus, soap may be added thereto, though I have found that such additions are not generally essential.

If desired the baths containing the delustring accelerators, or the liquids employed for the delustring of materials preliminarily treated with delustring accelerators, may contain salts or other substances adapted to modify the delustring action of the bath, in the manner described for instance in U. S. Patent No. 1,808,061. By this means a predetermined degree of delustre may more accurately be obtained.

Various delustring accelerators may be employed. Of especial value are substances which are solvents or swelling agents for cellulose acetate or which are soluble therein. Special mention may be made of compounds or relatively high boiling point, having a plasticizing action on the cellulose acetate, for example compounds such as have boiling points above 200° C., e. g. above 250° C.

The following are examples of compounds or classes of compounds which may be utilized:—

Organic bases, for example aromatic amines, e. g. aniline, chlor-anilines, N-alkyl anilines and other aniline substitution products, also heterocyclic bases, for example pyridine or quinoline.

Phenolic bodies, for example phenols, e. g. ortho-oxy-diphenyl, naphthols and their nuclear substitution products, e. g. chlor-phenols and chlornaphthols.

Terpene alcohols, for example terpeneols, borneols and menthols.

Hydrocarbons, for example naphthalene, diphenyl and other aromatic hydrocarbons and hydrogenated aromatic hydrocarbons, for example tetrahydronaphthalene or decahydronaphthalene, or terpene hydrocarbons, for example pinene or products containing terpene hydrocarbons, e. g. turpentine oil or pine oil.

Halogenated hydrocarbons, for example, para-dichlor-benzene or other chlor-benzenes, chlor-naphthalenes and tetrachlorethane.

Organic acids, for example acetic acid.

Alcoholic bodies, for example cyclohexanol and diethylene glycol or other glycols or polyglycols.

Ethers, for example phenol alkyl ethers or their substitution products, e. g. anisol, phenetole, nitro-anisols, nitro-phenetoles, dibenzyl ether and diphenyl ether.

Ketones, for example aromatic ketones, e. g. benzophenone and naphthyl phenyl ketones.

Carboxylic amides containing more than 6 carbon atoms, e. g. more than 8 carbon atoms, for example (a) acetylated or other acylated aromatic amines, e. g. acetanilide or (b) amides or substituted amides such as aryl-, aralkyl-, alkyl- or oxyalkyl-amides of aromatic or hydro-aromatic acids or aliphatic acids containing more than 6 carbon atoms, particularly fatty acids containing more than 10 carbon atoms, e. g. benzamide, phthalimide, benzanilide, diacetyl-p-phenylenediamine, diacetyl benzidine, oleic amide or stearic amide.

Amides of sulphonic acids, for example amides or substituted amides (such as alkyl-, oxyalkyl-, aralkyl- or aryl-amides) of benzene, toluene, xylene or naphthalene sulphonic acids, e. g. p-tol-

uene sulphonamide or its N-methyl or N-ethyl derivatives, xylene mono-methyl-sulphonamides or mixtures thereof, and di-p-toluenesulphobenzidine.

5 Camphors, menthols, terpeneols and other hydro-aromatic hydroxy, ketonic or other compounds of relatively high boiling point.

Esters, for example (a) esters of phthalic acid, benzoic acid, naphthenic acids or other aromatic  
10 or hydro-aromatic mono- or poly-carboxylic acids with phenols, aliphatic alcohols, or aromatic alcohols, e. g. benzylbenzoate, dimethyl phthalate, or diethyl phthalate or dibutyl phthalate; (b) esters of aliphatic acids, especially esters  
15 containing 6 or more carbon atoms, e. g. methyl, ethyl or butyl oleate or stearate or diethyl- or dibutyl- tartrate or other esters of hydroxy aliphatic acids; (c) esters of phosphoric acid, e. g. triphenyl or tricresyl phosphate or other aromatic  
20 esters; (d) esters of hydroxy acids, e. g. diethyl or dibutyl tartrate.

In general compounds which do not contain hydroxy or amino groups directly united to aromatic nuclei are to be preferred to such compounds as do contain such groups, in view of their lower tendency to colour the material in the course of the delustring process.

If desired or requisite the materials which have been delustered in accordance with the process  
10 of the present invention may be subjected to a treatment for the removal of any delustring accelerator or other body which may remain therein. Removal of delustring accelerator may for  
15 instance be conveniently effected in many instances by subjecting the fabric to a steaming treatment. Naphthalene for instance is very readily removed by such treatment. When  
steaming is employed for the removal of delustring accelerator or other substance on the materials, it may partially restore the lustre of the materials. In carrying out the delustring operation proper, therefore, due allowance should be  
0 made for any relustring of the material which may take place during the aforesaid steaming treatment.

As explained previously, the invention is of particular value in connection with the delustring of materials made of or containing acetone sol-

uble cellulose acetate. It may, however, be applied in connection with the delustring of other cellulose esters or ethers with hot aqueous liquids or moist steam.

The invention is illustrated but not limited by the following example.

#### EXAMPLE

##### *To delustre cellulose acetate woven fabric*

100 parts of diphenyl is dispersed by heating  
10 with 100 parts of xylene and 600 parts of Turkey red oil, and is diluted with good stirring to 1,000 parts with boiling water. Cellulose acetate woven fabric is padded in this dispersion at a  
15 temperature of 85-90° C. and allowed to remain in this state overnight. The fabric is then passed through an open tank of almost boiling water (98° C.), the time taken for this being 30 seconds, then through a bath of warm water, to remove  
20 excess of delustring agent, and is then plaited into a box. The fabric is then dried, or dyed, or otherwise treated as requisite.

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

1. Process for reducing the lustre of textile materials of organic derivatives of cellulose, which comprises uniformly impregnating the materials with diphenyl, and thereafter subjecting the impregnated materials to the action of a hot aqueous medium.

2. Process for reducing the lustre of textile materials of organic derivatives of cellulose, which comprises uniformly impregnating the materials with an aqueous dispersion of diphenyl, and thereafter subjecting the impregnated materials to the action of a hot aqueous medium.

3. Process for reducing the lustre of cellulose acetate textile materials, which comprises uniformly impregnating the materials with diphenyl, and thereafter subjecting the impregnated materials to the action of a hot aqueous medium.

4. Process for reducing the lustre of cellulose acetate textile materials, which comprises uniformly impregnating the materials with an aqueous dispersion of diphenyl, and thereafter subjecting the impregnated materials to the action of a hot aqueous medium.

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