

Patented Apr. 30, 1940

2,198,660

UNITED STATES PATENT OFFICE

2,198,660

TREATMENT OF TEXTILE AND OTHER MATERIALS

Henry Dreyfus, London, England

No Drawing. Application October 16, 1936, Serial No. 105,927. In Great Britain November 8, 1935

9 Claims. (Cl. 8—132)

This invention relates to improvements in the treatment of textile and other materials and is more particularly concerned with increasing the tensile strength of certain esterified or etherified cellulose products.

In U. S. Letters Patents Nos. 2,142,721, 2,142,722 and 2,142,909 processes are described for increasing the tensile strength of continuous filaments and threads thereof, of spun yarns and of other materials such as ribbons, foils, films or the like of cellulose acetate or other cellulose ester or ether, which consists in subjecting the materials to a stretching operation in the presence of steam or in hot water. Though improvement in the properties can be obtained with dry steam, the steam employed is preferably saturated or wet since the best results are obtained with the steam in this condition. The temperature may, for example, be 110°, 120° or 130° C., or more, and the appropriate pressure may be used either to maintain the steam in a saturated or wet condition or to maintain the water in the liquid state, for example pressures of 10, 20, 30 pounds or more per square inch above atmospheric pressure.

It has now been found that by stretching the materials in steam or hot water as described in the above specifications an improvement in the tensile strength may be obtained also of artificial filaments, yarns, foils, films and similar materials made of cellulose derivatives which are insoluble in organic solvents. Such materials may be obtained by partially saponifying artificial materials made of cellulose derivatives which are soluble in organic solvents or by the esterification or etherification of natural or regenerated cellulose materials. The stretch obtainable in these materials under any particular conditions of steam or hot water treatment varies with the character of the material under treatment, and particularly with the ester or ether content of the material and with the conditions under which the esterification, etherification or saponification has been carried out. For example, regenerated cellulose materials which have been acetylated in suspension (so as not to lose their fibrous form) in presence of hydrolytic agents, for example sulphuric acid, phosphoric acid or strongly acid salts such as bisulphates, zinc chloride, ferric chloride and the like, are more amenable to the stretching treatment than are similar materials which have been acetylated in the presence of catalytic agents such as sodium or potassium acetate or tertiary organic bases which have no substantial hydrolytic action upon cellulose. Similarly, the degree of esterification also has an effect. Acety-

lation to the mono-acetate stage only produces a material which is not very amenable to the stretching treatment in the steam or hot water. When, however, the acetylation is carried further, say to the di-acetate stage or to a stage between the di- and tri-acetates, especially in the presence of hydrolytic agents, a product is produced which is eminently amenable to the stretching treatment in the presence of the steam or in the hot water. Similarly, where a saponified cellulose ester material has been treated, the degree of saponification has a marked effect upon the extent to which such materials can be stretched in the presence of steam or hot water.

Saponification may be carried out to such an extent that the materials are insoluble in organic solvents, or it may be interrupted before this stage is reached and while the materials are still soluble in organic solvents, and the process of the present invention is applicable to the saponified materials whether they are soluble or insoluble in organic solvents. Saponifying the ordinary acetone-soluble cellulose acetate filaments and threads to produce a loss in weight of, say, 5% to 10% on the initial weight of the materials, does not produce any marked change in the extent to which the materials may be stretched. When, however, the saponification is carried further, say to a loss in weight of 10% or 20% or more, the ability to stretch in the steam or hot water progressively decreases.

The process of the present invention is also applicable to the stretching of artificial materials, whether soluble or insoluble in organic solvents, obtained by esterifying or etherifying natural or regenerated cellulose filaments, fibres, foils and other materials while maintaining the materials in their original form, e. g., by acetylating in benzene or other non-solvent liquid.

Stretching in the presence of steam or hot water also improves the properties, and particularly the tensile strength of artificial filaments, yarns, foils, films and similar materials obtained by subjecting to esterification or etherification, for example in the presence of ferric chloride, sodium acetate or zinc chloride, artificial filaments, yarns, foils, films and similar materials having a basis of a cellulose ester or ether, and the processes described in the present application are also applicable to such materials.

By suitably choosing the conditions of acetylation or other esterification or of etherification or of saponification in the manufacture of the initial materials, high degrees of stretch may be obtained, e. g., stretches up to several hundreds per

cent. In general, the higher the stretch required or the greater the rate of stretching required, the higher will be the temperature of the treatment.

The materials to be treated according to the invention may initially have any desired lustre from a high lustre to medium lustre or approximately the lustre of natural silk or even a dull lustre or no lustre at all. Thus, the materials may have a low lustre due to the presence therein of titanium dioxide or other pigments.

The stretching operation may be carried out upon the materials during the course of their travel from one point to another, i. e., upon the running materials. The treatment may be applied to a single thread, e. g., during winding from a cake or cheese or bobbin on to a bobbin, or it may be applied to a number of threads simultaneously. Thus, threads may be stretched while running from one creel of bobbins to another creel. When simultaneously stretching a number of threads, it is advantageous to arrange the threads in parallel alignment in the form of a sheet or, if desired, in the form of two parallel sheets.

Apparatus suitable for the treatment either of threads or yarns or of ribbons, foils, films or the like, is described in U. S. applications S. Nos. 48,084, filed November 4, 1935; 47,798, filed November 1, 1935, and 69,282, filed March 17, 1936.

The following examples illustrate the invention but are not to be regarded as limiting it.

Example 1

Cellulose acetate yarn having an acetyl content of about 56 to 58% obtained by the acetylation of an ordinary commercial cellulose acetate yarn in the presence, for example, of ferric chloride, sodium acetate or zinc chloride as catalyst is stretched while passing through wet steam at a temperature of 150 to 155° C.

Example 2

Cellulose acetate yarn which has been saponified with aqueous methylamine until its acetyl content has been reduced to about 45 to 48% is stretched while passing through hot water at a temperature of 140 to 150° C.

Example 3

Cellulose acetate yarn which is insoluble in organic solvents and has been obtained by the acetylation of regenerated cellulose yarn in the presence of, for example, zinc chloride as catalyst, and which contains 1½ to 2 acetyl groups per C₆H₁₀O₅ molecule is stretched while passing through wet steam at a temperature of 155 to 160° C.

The yarn treated according to each of the above examples has an improved tensile strength.

Stretched cellulose derivative materials may be subjected to any desired after-treatment processes. Thus, the stretched materials may be treated with shrinking agents to modify their properties, and particularly to improve their extensibility. Suitable processes for this purpose are described in U. S. application S. No. 611,240 filed May 13, 1932. In particular reference is made to the latent solvents referred to in that specification, for example a mixture of methylene chloride and benzene.

Again, stretched cellulose ester materials may be subjected to processes of saponification. The saponification may be such as to lead to a relatively small loss in weight, for example sufficient

to give the materials an affinity for cotton dye-stuffs, or may be complete or substantially complete, i. e., so as to eliminate all or substantially all of the ester content. The saponification may be effected under such conditions of tension that shrinkage takes place during or immediately after the saponification, this being advantageous from the point of view of the extensibility of the products.

Such further treatments may be carried out as operations separate from the stretching or may be carried out continuously with stretching. Thus, for example, the invention includes a continuous operation which involves first stretching and then shrinking.

Having described my invention what I desire to secure by Letters Patent is:

1. Process for improving the tenacity of artificial materials, which comprises subjecting artificial filaments, yarns, foils, films and similar materials, made of soluble organic derivatives of cellulose which have been rendered insoluble in organic solvents by a change in the number of free hydroxy groups in the organic derivative of cellulose molecule, to a stretching operation in the presence of an agent selected from the group consisting of wet steam and hot water at a temperature above 100° C.

2. Process for improving the tenacity of artificial materials, which comprises subjecting artificial filaments, yarns, foils, films and similar materials, made of soluble organic derivatives of cellulose which have been rendered insoluble in organic solvents by a change in the number of free hydroxy groups in the organic derivative of cellulose molecule, to a stretching operation in the presence of an agent selected from the group consisting of wet steam and hot water at a temperature above 120° C.

3. Process for improving the tenacity of artificial materials, which comprises subjecting artificial filaments, yarns, foils, films and similar materials, insoluble in organic solvents, obtained by the partial saponification of filaments, yarns, foils, films and similar materials made of organic esters of cellulose which are soluble in organic solvents, to a stretching operation in the presence of an agent selected from the group consisting of wet steam and hot water at a temperature above 100° C.

4. Process for improving the tenacity of artificial materials, which comprises subjecting to a stretching operation, in the presence of an agent selected from the group consisting of wet steam and hot water at a temperature above 100° C., artificial filaments, yarns, foils, films and similar materials obtained by reacting soluble organic derivative of cellulose filaments, yarns, foils, films and similar materials with an esterifying agent until they are insoluble in organic solvents.

5. Process for improving the tenacity of artificial materials, which comprises subjecting to a stretching operation, in the presence of an agent selected from the group consisting of wet steam and hot water at a temperature above 100° C., artificial filaments, yarns, foils, films and similar materials obtained by reacting soluble organic derivative of cellulose filaments, yarns, foils, films and similar materials with an etherifying agent until they are insoluble in organic solvents.

6. Process for improving the tenacity of artificial materials, which comprises subjecting to a stretching operation, in the presence of an

1 3 2

2,198,660

3

agent selected from the group consisting of wet steam and hot water at a temperature above 120° C., artificial filaments, yarns, foils, films and similar materials obtained by reacting soluble organic derivative of cellulose filaments, yarns, foils, films and similar materials with an esterifying agent until they are insoluble in organic solvents.

7. Process for improving the tenacity of artificial materials, which comprises subjecting to a stretching operation, in the presence of an agent selected from the group consisting of wet steam and hot water at a temperature above 100° C., artificial filaments, yarns, foils, films and similar materials obtained by reacting insoluble cellulose filaments, yarns, foils, films and similar materials with an esterifying agent, while retaining their insolubility and maintaining the materials in their original form.

8. Process for improving the tenacity of

artificial materials, which comprises subjecting artificial filaments, yarns, foils, films and similar materials made of aliphatic acid esters of cellulose which are insoluble in organic solvents to a stretching operation in the presence of an agent selected from the group consisting of wet steam and hot water at a temperature above 100° C.

9. Process for improving the tenacity of artificial materials, which comprises subjecting to a stretching operation, in the presence of an agent selected from the group consisting of wet steam and hot water at a temperature above 100° C., artificial filaments, yarns, foils, films and similar materials obtained by reacting soluble organic derivative of cellulose filaments, yarns, foils, films and similar materials with an esterifying agent containing an aliphatic acid radicle until they are insoluble in organic solvents.

HENRY DREYFUS. 20