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PRODUCTION OF IMMUNIZED COTTON OR OTHER CELLULOSIC FIBERS

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1 Claim. (Cl. 8—20)

This invention relates to processes for the treatment of cotton or other cellulosic fibers for the purpose of "immunizing" them against the action of direct cotton dyestuffs.

It is well known that the mono-, di- and tri-acetates of cellulose have practically no affinity for the direct cotton dyestuffs. The tri-acetate or acetates having acetyl values between those of di- and tri-acetate are soluble in organic solvents and may be employed in the manufacture of cellulose acetate artificial silk. On the other hand, the cellulose acetates having acetyl values up to and including that corresponding with the di-acetate, or even slightly higher, and produced by the acetylation of cotton without loss of structure, are employed for use as "effect threads" in the manufacture of various mixed fabrics. The present invention is concerned with the manufacture of "effect threads" of this type.

According to the present invention, immunized fibers are produced by the esterification of cotton or other cellulosic fibers with the aid of sulphonic acids as catalysts. As is already indicated above, the esterification will only be partial and may be carried to the point mono- or di-ester stage or to an intermediate stage or to slightly above the di-ester stage. I have found that the sulphonic acids are of great value in obtaining these effect threads without loss of structure of the cotton or other fiber. Some of the sulphonic acids have already been suggested in acetylations to the tri-acetate stage, but they have not come into general use for this purpose. I find that they are admirably adapted for the purpose of manufacturing effect threads.

As examples of suitable sulphonic acids for use in accordance with the present invention the following may be instanced, sulpho acetic acid, ethylene sulphonic acid, methyl sulphonic acid, propyl 1.2 or 1.3 di-sulphonic acid, sulpho succinic acid, ethanol sulphonic acid, benzyl sulphonic acid, benzene sulphonic acid, para-toluene sulphonic acid, phenol sulphonic acids, phenol di-sulphonic acid, sulpho salicylic acid, sulpho phthalic acid, naphthalene sulphonic acids and naphthol sulphonic acids. I prefer to employ the aliphatic sulphonic acids. As acidylating agents the aliphatic acid anhydrides are preferred, for example acetic anhydride, propionic anhydride, butyric anhydride, lauric anhydride and the like.

The esterification of the cotton or other cellulosic fiber may be effected with or without the aid of suitable diluents, for example acetic acid, propionic acid, butyric acid, ether, carbon tetrachloride, benzene, toluene, ligroin and the fractions of petroleum in general, or mixtures thereof. The present catalysts are particularly adapted to produce a moderate action capable of being adequately controlled so that the "effect threads" only are produced and the esterification does not proceed to the tri-ester stage.

The fibers treated in accordance with the present invention may be cotton, jute, linen or any other natural cellulosic fiber or the cellulosic type of artificial silk, such as viscose, cuprammonium and nitro cellulose artificial silks. They may be treated in the form of filaments, yarns, threads or the like either in the loose or in hank form or in the form of other wound packages, or fabrics made of or containing the said fibers may be treated. Local "immunized" effects may be obtained by the local application of the esterifying mixture, as for example by printing methods.

The fiber to be esterified may, if desired, be pretreated before the actual esterification, as for example by a pretreatment with organic acids as described in French specification No. 565,654, in large or small quantities and in the liquid or vapour state (compare U. S. Patent No. 1,831,101). Again a pretreatment with hydrohalide acids as described in U. S. applications Nos. 328,306 filed 24th December, 1928, and 348,981 filed 21st March, 1929, may be applied, though this is less advantageous in the case of the present invention inasmuch as the esterification is to be performed in the presence of sulphonic acids. A further form of pretreatment consists in treating the cotton or other fiber with the catalyst to be employed in the subsequent esterification, such pretreatment being carried out with or without diluents, as for example those listed above for the esterification itself. Furthermore the cotton may be pretreated with the esterifying agent, for example an anhydride, before carrying out the main esterification, such pretreatment being applied with the esterifying agent alone or mixed with a diluent as already described, or with the catalyst or with both. For instance the cotton may be immersed for some time in the complete esterification mixture at a temperature insufficient to cause substantial esterification, and subsequently the temperature may be raised to effect the esterification proper.

As already indicated the ester content of the products produced in accordance with the present invention may vary up to the di-ester stage or slightly above. I find that it is particularly advantageous to carry the esterification to between the mono- and the di-ester stage, for example in the case of an acetylation to an acetyl

value of between 25 and 40%, calculated as acetic acid. The degree of esterification may be controlled within the desired limits in any suitable manner, for example by suitable adjustment of the temperature or time of the esterification or by an adjustment of the quantity of esterifying agent used.

The following example illustrates the present invention but is not to be considered as limiting it in any way:—

Example

100 parts of cotton yarn are immersed in a bath containing 500 to 700 parts of acetic acid, 125 to 225 parts of acetic anhydride and 15 to 20 parts of sulpho acetic acid. The temperature is maintained at about 50° C. for ½ hour, after which the cotton is removed, washed and dried. The acetyl content is about 30%, calculated as acetic acid.

In a similar manner other sulphonic acids, for example those listed above, may be employed. Again instead of employing acetic anhydride other aliphatic anhydrides may be employed, using in general somewhat higher temperatures as the carbon content of the acid anhydride increases. Where as in the case of lauric anhydride the acidylating agent is a solid at ordinary temperatures, it may be melted, if feasible, alone or in admixture with the corresponding acid and the yarn or fabric immersed in the melted bath containing the catalyst, or the acidylating agent with or without the corresponding acid may be dissolved in a suitable diluent, for example a hydrocarbon, and the yarn or fabric then immersed in the bath maintained at a suitable temperature.

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

Process for immunizing cellulosic fibers comprising esterifying the same at a temperature of about 50° C. to a point not substantially exceeding the di-ester stage in the presence of a sulfonic acid.

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