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Published Apr. 27, 1943 Serial No. 379,722

ALIEN PROPERTY CUSTODIAN

PROCESS FOR THE MANUFACTURE OF EFFECT THREADS

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No Drawing. Application filed February 19, 1941

This invention relates to a process for the manufacture of effect threads.

The hitherto known manner of manufacturing effect threads (immune threads) from cotton or other vegetable fibres consisted in subjecting the previously alkalised fibres to the action of aromatic acid chlorides dissolved in indifferent solvents. It has also been proposed to replace the rather expensive aromatic acid chlorides by the cheaper sulphonic acid chlorides. According to a further suggestion the alkalised fibres were to be treated with the sulphonic acid halides of basic aromatic compounds, equally dissolved in indifferent organic solvents. By all these substantially similar treatments effected with solutions of the mentioned aromatic acid halides the cellulose was converted into esters. Therefore the solvents employed could only be "indifferent" ones, i. e. such as would not dissolve or affect in any way the cellulose esters formed. The esterification was achieved in baths, with the aid of the heat formed in the course of the reaction itself and/or with the supply of external heat. The acid halides had to be employed therein in large excess which rendered this manner of working rather expensive since under the reaction conditions required for a proper carrying out of the esterification the aromatic acid halides dissolved are, at least in part, subject to decomposition so that only part of them, if any, can be employed again.

In my prior application Serial No. 295,837 I have disclosed a process which materially cheapens the manufacture of effect threads. This process was based on the recognition of the fact that the esterification can be effected with best results outside the bath, by impregnating the previously alkalised threads with the solution of the acid halide selected, for instance by immersion or drawing through the solution, and effecting the esterification subsequently, i. e. on the impregnated thread itself, by exposing the same, after removal of any excess of solution adhering thereto, for some hours to the action of warm air. In this way considerable savings could be made since a short immersion or drawing through of the threads does not affect the solution.

It has now been found that impeding the evaporation of the solvent from the impregnated thread during the esterification has a very favorable effect on the esterification reaction and also on the quality of the product obtained. For this purpose the impregnated threads are subjected to further treatment in an atmosphere formed by the vapors of the solvent employed. This further treatment may also be effected in an atmosphere which is formed by a gas saturated with the vapors of the solvent. The solvent vapors or the gas saturated with them, such as nitrogen, air enriched with nitrogen, air, etc., is most preferably passed through the reaction space (i. e. the esterification space). The esterification is carried out at a temperature of from about 30 to 120° C depending on the nature of the cellulose comprised in the raw material and the kind of esterifying agent employed. The reaction takes about 1 to 2 hours. To maintain the required temperature the gas stream saturated with the solvent vapors and passed through the esterification space may be preheated. The solvent vapors contained in the gas stream leaving the reaction space may be recovered therefrom in a known manner.

The process may be applied in the same manner to loose fibres, yarns, fabrics and so on, bleached or unbleached, that is, it may be employed in any stage of the working-up of the cotton or other vegetable fibres.

Without limiting the invention to any particular procedure, the following examples are given to illustrate the preferred mode of operation.

Example 1

10 kgs of cotton yarn are uniformly impregnated with a 14% alcoholic NaOH-solution. After having been freed from the excess lye by centrifuging, the yarn is dried at a moderate temperature and thereupon immerged into an esterifying bath comprising 20 kgs of p-toluene sulphonic acid chloride dissolved in 90 litres of carbon tetrachloride. The yarn is then freed from the excess of the esterifying solution by centrifuging and placed into a container which is adapted to be heated and provided with gas in- and outlets. Through this container a stream of air enriched with nitrogen, saturated with carbon tetrachloride vapors and preheated to about 60 to 80° C is passed for 1 to 2 hours. Thereafter the yarn is removed from the container and washed in soap baths having a temperature of about 70 to 80° C until it becomes inodorous.

The solvent vapors are recovered from the air stream in a known manner.

Example 2

10 kgs of cotton yarn are treated as in Example 1 except that as an esterifying bath a solution of 14 kgs of benzoyl chloride in 90 litres of benzol is employed. The yarn is then placed into a container adapted to be heated and provided with a reflux cooler. At the bottom of said container an evaporation bowl containing some benzol is provided so as to maintain a uniform benzol vapor atmosphere during the esterification reaction. Care is being taken to prevent the benzol condensed in the reflux cooler from dripping onto the yarn under treatment and to direct it instead into the evaporation bowl at the bottom of the container. The temperature in the container is kept at about 80 to 120° C.

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