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UNITED STATES PATENT OFFICE.

HARRY S. MORK, OF BROOKLINE, AND CHARLES F. COFFIN, JR., OF WATERTOWN, MASSACHUSETTS, ASSIGNORS, BY MESNE ASSIGNMENTS, TO LUSTRON COMPANY, INC., OF BOSTON, MASSACHUSETTS, A CORPORATION OF MASSACHUSETTS.

MANUFACTURE OF CELLULOSE-ACETATE ARTIFICIAL SILK.

No Drawing.

Application filed March 22, 1923. Serial No. 626,872.

To all whom it may concern:

Be it known that we, (1) HARRY S. MORK, and (2) CHARLES F. COFFIN, JR., citizens of the United States, residing at (1) Brookline, 5 (2) Watertown, in the counties of (1) Norfolk, (2) Middlesex, and State of (1) and (2) Massachusetts, have invented certain new and useful Improvements in the Manufacture of Cellulose-Acetate Artificial Silk, 10 of which the following is a specification.

This invention relates specifically to improvements in the manufacture of cellulose acetate artificial silk as described in United States Patent No. 1,107,222, patented August 11, 1914, to Harry S. Mork. 15

The said patent describes a process of making cellulose acetate artificial silk comprising dissolving the cellulose acetate in a mixture of tetrachlorethane and an alcohol, 20 and squirting the resulting solution through fine orifices into a coagulating or setting bath composed of one or more aliphatic hydrocarbons, preferably petroleum hydrocarbons, or a mixture of aliphatic hydrocarbons and tetrachlorethane. 25

We have found that better coagulation can be secured, and consequently more uniform spinning conditions can be maintained, if there is incorporated in the original cellulose acetate solution a limited proportion 30 of an aliphatic hydrocarbon or mixtures of aliphatic hydrocarbons. The effect of such addition is to bring the spinning solution considerably nearer to its coagulating point, and thereby to improve the coagulation in the second bath. The aliphatic hydrocarbons to be added may be the same as those used in the coagulating bath, but as this latter usually comprises a mixture of various hydrocarbons boiling over a considerable 40 temperature range, we regard it as preferable, though not essential, to add to the original solution hydrocarbons corresponding to the lower boiling constituents of the hydrocarbon mixture used in the coagulating bath. These lower-boiling hydrocarbons will dry out of the silk more rapidly. We do not however wish to restrict ourselves to the use of a hydrocarbon identical with a 50 component of the coagulating bath, or a mixture of hydrocarbons corresponding to the mixture employed in the coagulating bath.

When the coagulation bath becomes heav-

ily charged with tetrachlorethane it can be 55 distilled to recover the tetrachlorethane and the distillation may be conducted so that the tetrachlorethane distilled off will contain some hydrocarbons. Such a distillate can be used to provide all or part of the tetrachlorethane and hydrocarbon necessary 60 for the preparation of the spinning solution.

No definite percentage can be prescribed for the addition of the aliphatic hydrocarbon to the cellulose acetate solution, since the 65 quantity to be added will depend upon the precise composition of the solvents used in the preparation of this solution, the concentration of the solution, and the character of the hydrocarbons to be added. In 70 usual practice, however, the tetrachlorethane largely predominates in the cellulose acetate solution, while the hydrocarbon predominates at least initially in the coagulating bath. 75

Following is a specific example in accordance with our invention, it being understood that the invention is not limited to the particular materials or proportions recited therein by way of illustration: 80

306 grams of fibrous cellulose acetate prepared in accordance with the United States Patent 854,374, patented May 21, 1907, to Harry S. Mork, are dissolved in a mixture 85 of 1120 c. c. of tetrachlorethane, 420 c. c. of denatured alcohol and 160 c. c. of 150° fire test kerosene. The solution is thoroughly agitated, then filtered and squirted under pressure through a suitable "spinneret" into a coagulating bath composed of 9 parts by 90 volume of kerosene and 1 part by volume of tetrachlorethane.

Another method of preparing a spinning solution is as follows:

306 grams of cellulose acetate are dissolved 95 in a mixture of 1280 c. c. of tetrachlorethane distillate and 420 c. c. of denatured alcohol. The distillate may contain 87½% of tetrachlorethane and 12½% kerosene by volume. 100

It is not essential that the hydrocarbon be present in the solvent mixture when the cellulose acetate is dissolved because the cellulose acetate may first be dissolved in a mixture of commercially pure tetrachlorethane and a suitable alcohol and the hydrocarbon be subsequently mixed into the solution. 105

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To the solutions as above described may be further added other solvents and non-solvents of the cellulose acetate such as chloroform, acetone, benzol, toluol, xylol, ethyl-methyl ketone, ethyl acetate, and carbon tetrachloride, for the purpose of altering the coagulation properties of the solution or the character of the artificial silk produced therefrom. Similar results are obtained by the addition of aliphatic hydrocarbons or mixtures thereof or mixtures of aliphatic hydrocarbons with other solvents and non-solvents of cellulose acetate to solutions of cellulose acetate in solvents other than tetrachlorethane or tetrachlorethane and alcohol hereinbefore described. Other solvents for the cellulose acetate which may be used are for example chlorinated hydrocarbons of lower boiling point than tetrachlorethane, dichlormethane, chloroform, dichlorethane, trichlorethane etc., either alone or in conjunction with alcohol.

We claim:

1. As a novel composition of matter, a coagulable solution comprising cellulose acetate, tetrachlorethane, and an aliphatic hydrocarbon.
2. As a novel composition of matter, a

coagulable solution comprising cellulose acetate, tetrachlorethane, an aliphatic hydrocarbon and an alcohol.

3. Process of making cellulose acetate artificial silk comprising preparing a solution of cellulose acetate in a solvent containing tetrachlorethane and an aliphatic hydrocarbon, and coagulating said solution in a bath likewise containing an aliphatic hydrocarbon.

4. Process according to claim 3 in which the cellulose acetate solvent comprises tetrachlorethane, an alcohol, and an aliphatic hydrocarbon.

5. As a novel composition of matter, a coagulable solution comprising cellulose acetate, a solvent therefor, and an aliphatic hydrocarbon.

6. Process of making cellulose acetate artificial silk comprising preparing a solution comprising cellulose acetate, a cellulose acetate solvent and an aliphatic hydrocarbon, and coagulating said solution in a bath comprising an aliphatic hydrocarbon.

In testimony whereof, we affix our signatures.

HARRY S. MORK.
CHARLES F. COFFIN, JR.