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PROCESS OF DRAWING CELLULOSE ACETATE THREADS

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This invention relates to a process of drawing cellulose acetate threads and more particularly to a process of drawing cellulose acetate threads by wet spinning the reaction mixture of the acetylation process.

In wet spinning cellulose acetate, it is customary to prepare an acetone soluble mass which is precipitated after the acetylation process and then dissolved in a suitable solvent. Heretofore, the acetylation reaction mixture was not used for spinning directly into a precipitation liquor as the product formed was undesirable because of insufficient lustre and softness.

We have now found that the acetylation reaction mixture can be used in wet spinning if a chlorine derivative of an aliphatic hydrocarbon, which is insoluble in the precipitate bath, is added to the acetylation reaction mixture, with increased stretching of the thread and without the disadvantage of a white product being formed. This addition can be made before the acetylation process or after, for example, during the hydration process. The hydration process consists in treating the acetylation reaction mixture with an aqueous means for the purpose of changing the cellulose acetate from the chloroform soluble form to the acetone soluble form. According to the condition of the cellulose acetate and the spinning, pure water may be used in the precipitate bath or salts and the like may be added. When the threads begin to precipitate in a plastic condition, it is possible to draw the same at a velocity of over 100 meters per minute through an opening of one tenth (0.1) mm. forming a single glossy thread of about 0.3 den. and a tensile strength of at least 200 g. per 100 den.

According to this invention, we prefer to pass the threads first through a warm precipitate bath and then through a cold precipitate bath. It is also understood that various softening means, such as resins, etc. may be added to the acetylation liquor.

Example I

10 parts of cellulose such as linters are acetylated in the presence of 0.5 to one part sulphuric acid, 25 parts of acetic anhydride and 5 parts of glacial acetic acid in the known manner, and hydrated until the mass is acetone soluble. The sulphuric acid of the acetylation reaction mixture is neutralized and 6 parts of methylene chloride are added. The mass is now spun through a nozzle into water in the known way and the exceedingly fine and soft threads formed are drawn through a precipitate bath of about 30° C., and then through a precipitate bath of about 19 to 20° C.

Example II

10 parts of linters are acetylated as in Example I described. Then 6 parts of acetylene dichloride are added and the reaction mixture hydrated in the known way. The mass is neutralized and spun as in Example I. The temperature of the first precipitate bath is between 40 and 50° C., and of the second precipitate bath 10 to 20° C.

Example III

10 parts of linters are acetylated with one part of acetic acid, 0.5 to one part of sulphuric acid, 15 parts of ethylene chloride and 25 parts of acetic anhydride. The excess anhydride is removed and the mass is hydrated and spun as in Example II.

Example IV

10 parts of linters are acetylated with a softening agent without changing the structure of the cellulose. The excess anhydride and softening agent are removed by centrifugation and the triacetate formed is hydrated in the known manner with 10 parts of methylene chloride, one part of acetic acid and one part of sulphuric acid. The further treatment as in Example I.

We claim as our invention:

1. Process of drawing cellulose acetate threads comprising adding a chlorine substituted aliphatic hydrocarbon containing less than three carbon atoms, which is insoluble in the precipitate bath, to a mixture of cellulose, sulphuric acid, acetic anhydride, and acetic acid, and acetylating, hydrating and wet spinning the resulting mass in a precipitate bath.

2. Process of drawing cellulose acetate

threads comprising adding a chlorine substituted paraffin hydrocarbon containing less than three carbon atoms, which is insoluble in the precipitate bath, to a mixture of cellulose, sulphuric acid, acetic anhydride and acetic acid, and acetylating, hydrating and wet spinning the resulting mass in a precipitate bath.

3. Process of drawing cellulose acetate threads comprising adding a chlorine substituted olefine hydrocarbon containing less than three carbon atoms, which is insoluble in the precipitate bath, to a mixture of cellulose, sulphuric acid, acetic anhydride, acetic acid and acetylating, hydrating and wet spinning the resulting mass in a precipitate bath.

4. Process of drawing cellulose acetate threads comprising adding a chlorine substituted aliphatic hydrocarbon containing less than three carbon atoms, which is insoluble in the precipitate bath, to a mixture of cellulose, sulphuric acid, acetic anhydride, acetic acid, and acetylating, hydrating and wet spinning the resulting mass in a warm and then in a cold precipitate bath.

5. Process of drawing cellulose acetate threads comprising adding a chlorine substituted paraffin hydrocarbon containing less than three carbon atoms, which is insoluble in the precipitate bath, to a mixture of cellulose, sulphuric acid, acetic anhydride, and acetic acid and acetylating, hydrating and wet spinning the resulting mass in a warm and then in a cold precipitate bath.

6. Process of drawing cellulose acetate threads comprising adding a chlorine substituted olefine hydrocarbon containing less than three carbon atoms, which is insoluble in the precipitate bath, to a mixture of cellulose, sulphuric acid, acetic anhydride, acetic acid and acetylating, hydrating and wet spinning the resulting mass in a warm and then in a cold precipitate bath.

7. Process of drawing cellulose acetate threads comprising adding a chlorine substituted aliphatic hydrocarbon containing less than three carbon atoms, which is insoluble in the precipitate bath, to a mixture of cellulose, sulphuric acid, acetic anhydride, acetic acid, and acetylating, hydrating and wet spinning the resulting mass in a warm precipitate bath of not above 50° C. and then in a cold of not below 10° C.

8. Process of drawing cellulose acetate threads comprising adding a chlorine substituted paraffin hydrocarbon containing less than three carbon atoms, which is insoluble in the precipitate bath, to a mixture of cellulose, sulphuric acid, acetic anhydride and acetic acid, and acetylating, hydrating and wet spinning the resulting mass in a warm precipitate bath of not above 50° C. and then in a cold of not below 10° C.

9. Process of drawing cellulose acetate threads comprising adding a chlorine substituted olefine hydrocarbon containing less than three carbon atoms, which is insoluble in the precipitate bath, to a mixture of cellulose, sulphuric acid, acetic anhydride, acetic acid and acetylating, hydrating and wet spinning the resulting mass in a warm precipitate bath of not above 50° C. and then in a cold of not below 10° C.

10. Process of drawing cellulose acetate threads comprising adding a member selected from the group consisting of methylene chloride, ethylene chloride or acetylene dichloride to a mixture of cellulose, sulphuric acid, acetic anhydride and acetic acid, and acetylating, hydrating and wet spinning the resulting mass in a warm precipitate bath and then in a cold precipitate bath.

11. Process of drawing cellulose acetate threads comprising acetylating cellulose, adding a member selected from the group consisting of methylene chloride, ethylene chloride or acetylene dichloride to the acetylation reaction mixture and hydrating and wet spinning the resulting mass in a precipitate bath at between 30 to 50° C. and then in a precipitate bath at between 10 to 20° C.

12. Process of drawing cellulose acetate threads, comprising acetylating cellulose without changing its structure, adding a member selected from the group consisting of methylene chloride, ethylene chloride, or acetylene dichloride to the acetylation reaction mixture and hydrating and wet spinning the resulting mass in a precipitate bath at between 30 to 50° C. and then in a precipitate bath at between 10 to 20° C.

13. Process of drawing cellulose acetate threads comprising acetylating a mixture of cellulose, sulphuric acid, acetic anhydride, and acetic acid, hydrating the acetylation reaction mixture, adding a member selected from the group consisting of methylene chloride, ethylene chloride, or acetylene dichloride and wet spinning the resulting mass in a precipitate bath of 30 to 50° C. and then in a precipitate bath at between 10 to 20° C.

14. Process of drawing cellulose acetate threads comprising wet spinning in a precipitate bath an acetone soluble acetylation reaction mixture containing a chlorine substituted aliphatic hydrocarbon containing less than three carbon atoms, which is insoluble in the precipitate bath.

15. Process of drawing cellulose acetate threads comprising wet spinning in a precipitate bath a hydrated acetylation reaction mixture containing a chlorine substituted aliphatic hydrocarbon containing less than three carbon atoms, which is insoluble in the precipitate bath.

16. Process of drawing cellulose acetate threads comprising wet spinning in a precipitate bath a hydrated acetylation reaction mixture containing a chlorine substituted

paraffin hydrocarbon containing less than three carbon atoms, which is insoluble in the precipitate bath.

17. Process of drawing cellulose acetate threads comprising wet spinning in a precipitate bath a hydrated acetylation reaction mixture containing a chlorine substituted olefine hydrocarbon containing less than three carbon atoms, which is insoluble in the precipitate bath.

Signed at Munich, State of Bavaria, Germany, this 2nd day of November A. D. 1931.

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