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METHOD OF PRODUCING ARTIFICIAL THREADS

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5 Claims. (Cl. 18—54)

This invention relates to regenerated cellulose structures produced by the viscose method and in one of its specific embodiments relates to the production of threads having increased tensile strength both in the wet and dry states.

According to the common practice of producing artificial threads by the viscose method, a viscose solution prepared from cellulose xanthated with about 35% of carbon bisulphide, based on the weight of the cellulose, is spun in a coagulating bath. If the coagulating bath is an acid one, it usually contains from about 8% to 20% of an acid, such as sulphuric. The threads resulting from such a process possess a dry tenacity of approximately 1.50 grams per denier and a wet tenacity of approximately 0.70 grams per denier. As is apparent, these tensile strengths are relatively low and many attempts have been made to improve them. One of the methods devised to produce threads of increased tensile strength consisted of spinning a viscose solution, produced in the usual manner, in an acid bath containing 50% or more of sulphuric acid whereby the threads were rendered capable of being stretched to an increased extent. Another method contemplated reducing the amount af alkali in the viscose to about 5% and spinning such a viscose into an acid bath containing 45% sulphuric acid.

I have found that other changes in the nature of the viscose and in the process used in the preparation thereof have a controlling effect on the concentrations of acid which should be used in the bath and on the tensile strength of the threads produced. More particularly I have found that the amount of carbon bisulphide used in the xanthation has a marked effect on the amount of acid which should be used in the spinning bath to give threads of very high tensile strength. Specifically, I have found that, when less than 25% of carbon bisulphide is used in the xanthation and the viscose produced therefrom spun in an acid bath containing sulphuric acid, not in excess of 50%, threads having greatly increased tensile strength, both wet and dry, are produced.

It is therefore an object of this invention to provide a method of producing artificial threads by the viscose process having increased tensile strength, both in the dry and wet states.

Another object of this invention is to provide a method of producing artificial threads by the viscose process having relatively high tensile strength, wherein not more than 25% of carbon bisulphide, based on the weight of the cellulose, is employed in the xanthation.

A further object of this invention is to provide a method of preparing artificial threads having increased tensile strength which comprises spinning a viscose solution produced from cellulose xanthated with not more than 25%, and preferably 17%, of carbon bisulphide, based on the weight of the cellulose, in an acid bath containing less than 50% and preferably from over 40% to 47% of sulphuric acid.

Other objects will appear from the following description and appended claims.

Essentially this invention comprises spinning a viscose solution prepared from cellulose xanthated with a relatively small quantity of carbon bisulphide in a bath containing a relatively high concentration of acid. The amount of carbon bisulphide employed in the xanthation step does not exceed 25%, and is preferably not less than 10%, by weight based on the cellulose. The bath into which this viscose solution is spun has an acid concentration of less than 50%, and preferably from over 40% to 47%, of a mineral acid such as sulphuric. The bath travel and the rate of spinning may vary within wide limits depending inter alia on the concentration of the acid and/or the quantity of bisulphide used in the xanthation.

In order to more clearly define and explain this invention, the following specific examples are set forth.

Example I.—100 parts of wood pulp of good grade employed for rayon, and preferably a material having a high alpha cellulose content, and containing 6% or 7% of moisture are steeped in a caustic soda solution of 17% to 20% strength at about 18° C. for 1 to 3 hours. The mass is then pressed until the press-weight of the wet alkali cellulose is about 300 to 320 pounds. After it has been shredded and aged at 20° C. for any suitable length of time, such as 50 or 60 hours, the aged alkali cellulose is xanthated according to any well-known manner at any suitable temperature, for instance 25° C., for approximately 2 to 3 hours using 17 parts of carbon bisulphide to 100 parts of cellulose. The resulting cellulose xanthate is formed into a viscose solution by dissolving it in a caustic soda solution at 18° C., the quantity of caustic soda being such that the viscose solution contains 7% of cellulose and 8% of alkali calculated as NaOH. The solution thus produced is filtered and then ripened for 12 to 20 hours at approximately 18° C. After ripening, the solution is finally spun into a bath

containing 45%–50% of acids and preferably 47% sulphuric acid maintained at approximately 10° C.

A thread of 2.5 deniers per filament has been successfully produced when the pump delivery was 10 grams of viscose per minute and the viscose was forced through spinnerets having 40 orifices or holes, each 0.0035 inch in diameter, the thread having a bath travel of from 20 to 30 inches and a take-up speed of 3,000 inches per minute.

Suitable moderate tension secured in any of the well-known ways is applied to the threads during their travel in the bath. The coagulated and regenerated threads, after passing through the acid bath, are treated in any suitable manner to arrest the action of the acid. For example, the coagulated and regenerated threads are passed from the acid bath into a bath containing water, in which additional tension may be applied to the partially washed threads. The threads are then accumulated in or on a collection device which may also be treated with suitable washing liquid.

Example II.—100 parts of cotton linters containing 5% to 6% of moisture are steeped in a caustic soda solution of 17% to 20% strength at about 18° C. for 1 to 3 hours. The mass is then pressed until the press weight of the wet alkali cellulose is about 310 to 330 lbs. After it has been shredded and aged at 20° for 80 to 90 hours, the aged alkali cellulose is xanthated according to any well-known manner at any suitable temperature, for instance 25° C. for approximately 2 to 3 hours using 21 parts of carbon bisulphide to 100 parts of cellulose. The resulting cellulose xanthate is formed into a viscose solution by dissolving it into a caustic soda solution at 18°, the quantity of caustic soda being such that the viscose solution contains 7% of cellulose and 8% of alkali calculated as NaOH. The solution thus produced is filtered and ripened for 5 to 10 hours at approximately 18°. After ripening, the solution is finally spun into a bath containing 42% sulphuric acid and 15% acetic acid at 10° C.

The spinning conditions are the same as previously described under Example I:

The use of acetic acid in combination with the sulphuric acid permits the use of lower concentrations of sulphuric acid without losing the effectiveness of a greater concentration of sulphuric acid.

Example III.—Aged alkali cellulose, prepared from high grade wood pulp as set forth in Example I, is xanthated with 14 parts of carbon bisulphide to 100 parts of cellulose as described in the aforementioned Example I. A viscose solution is then prepared as in Example I. After ripening, the solution is spun in a bath at approximately 10° C. containing 44% of sulphuric acid saturated with sodium sulphate. The thread is further treated as set forth in Example I.

It is evident that certain changes can be made in the above examples without departing from the spirit of the invention. For example, a suitable grade of cotton linters or other source of cellulose may be used in place of the wood pulp which has been specifically mentioned. The time of aging of the alkali cellulose is subject to some change but will preferably tend to be for a rather long period in view of the relatively small amount of carbon bisulphide used. The percentage of the cellulose in the viscose may also be varied over the ranges which are normally used in this art, and this invention is not to be restricted to the precise percentage of cellulose which is disclosed in the specific examples. The coagulating bath within the range which has been described may be modified by the addition of any of the well-known materials which are customarily used in coagulating baths, such as inorganic sulphate, glucose and the like. There may also be used with the sulphuric acid either in the presence or absence of the inorganic sulphate such organic acids as acetic acid, which permits the use of smaller quantities of sulphuric acid while still retaining the effectiveness of higher quantities.

The threads produced according to this method have a dry tenacity of at least 3 grams per denier and a wet tenacity of at least 1.8 grams per denier.

By the term "cellulose" as used in this specification and claims is meant the total material in the viscose recoverable as film or thread by coagulation in an acid bath followed by washing and drying.

Though the invention is specifically described above in the manufacture of artificial thread, the invention is not restricted thereto. It is equally applicable for the manufacture of any regenerated cellulose structures including films.

Since it is obvious that various other changes may be made in the specific details set forth above, this invention is not restricted thereto except as set forth in the appended claims.

I claim:

1. The process of producing regenerated cellulose structures which comprises introducing a viscose solution containing 8% of alkali calculated as NaOH prepared from a cellulose xanthated with from 10% to 25% of carbon bisulphide, based on the weight of the cellulose, in a bath containing from over 40% to less than 50% sulphuric acid.

2. The process of producing artificial threads of high tensile strength which comprises spinning a viscose solution containing 8% of alkali calculated as NaOH prepared from a cellulose xanthated with from 10% to 25% carbon bisulphide, based on the weight of the cellulose, in a bath containing from over 40% to less than 50% sulphuric acid.

3. The process of producing artificial threads of high tensile strength which comprises spinning a viscose solution containing 8% of alkali calculated as NaOH prepared from a cellulose xanthated with 17% carbon bisulphide, based on the weight of the cellulose, in a bath containing 47% sulphuric acid.

4. The process of producing artificial threads of high tensile strength which comprises spinning a viscose solution containing 8% of alkali calculated as NaOH prepared from a cellulose xanthated with 21% of carbon bisulphide, based on the weight of the cellulose, in a bath containing 42% sulphuric acid and 15% acetic acid.

5. The process of producing artificial threads of high tensile strength which comprises spinning a viscose solution containing 8% of alkali calculated as NaOH prepared from a cellulose xanthated with 14% carbon bisulphide, based on the weight of the cellulose, in a bath containing sodium sulphate and 44% sulphuric acid.

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