Patented Mar. 27, 1934 1,952,604

UNITED STATES PATENT OFFICE

1,952,604

VISCOSE AND PROCESS OF PRODUCING SAME

Harold Henry Parker, Kenmore, N. Y., assignor to Du Pont Rayon Company, New York, N. Y., a corporation of Delaware

No Drawing. Application November 27, 1931, Serial No. 577,708

18 Claims. (Cl. 106—40)

This invention relates to viscose solutions suitable for casting and spinning and substantially free from partially insoluble materials. More particularly, it relates to a process of producing a viscose solution of the type mentioned.

In the manufacture of viscose for spinning or casting purposes, it is customary to treat alkali cellulose with carbon bisulphide. This treatment produces a xanthated cellulose which is more or less soluble in an aqueous caustic soda solution depending inter alia upon the nature of the pulp, the ratio of carbon bisulphide, and the concentration of the caustic soda solution. In practice, these factors are adjusted to give a viscose solution as free as possible from undissolved fiber and other extraneous foreign materials. However, no matter what precautions are taken, there is usually present in the viscose a comparatively small amount, for example, 0.1% to 0.5% by weight (based on the viscose) of undesirable substances, such as extraneous foreign material, fibrous particles of cellulose which are unattacked during the preparation of the viscose solution because of being protected by some substance such as a film of oil or grease, and semi-solid gelatinous masses of cellulose, the latter hereinafter termed "partially insoluble material".

In the process of spinning a viscose solution, the solution is filtered to a very high degree in order to remove any foreign material present therein. Usually, the filtration satisfactorily removes the extraneous foreign material and the unattacked fibrous particles of cellulose, but, due to the slimy, gelatinous character of the partially insoluble material, the latter penetrates and slides through the filtering medium. As a consequence, the spinning solution is not homogeneous and the spinnerets clog during the spining process and the rate of production necessarily decreases. Even if the spinnerets do not completely clog, one or more of the holes therein become clogged to a point of being "slow holes", whereby the quality of the yarn produced is materially reduced. The resulting thread possesses irregular and non-uniform physical characteristics, such as non-uniformity and irregularity in dyeing, tensile strength, elongation, etc.

When a viscose solution is made from a cellulose xanthate prepared with a relatively small amount of bisulphide per unit of cellulose, the partially insoluble material is present to a greater degree than in viscose solutions in the preparation of which a large quantity of carbon bisulphide per unit of cellulose is used. Therefore, the abovementioned defects are increased to a corresponding degree.

I have found that I can overcome the abovementioned attendant defects by preparing a substantially homogeneous viscose solution suitable for casting and spinning purposes and substantially free from the partially insoluble material by adding thereto, either after or during the preparation thereof, a substance, such as, for example, a small quantity of a xanthate prepared with a higher ratio of carbon bisulphide per unit of cellulose than the xanthate in the viscose solution, which modifies the partially insoluble materials present therein to produce a substantially homogeneous solution, and inhibits said materials from clogging the spinneret holes.

It is, therefore, an object of this invention to prepare a viscose solution suitable for casting and spinning and substantially free from partially insoluble materials.

Another object of this invention is to prepare a viscose solution suitable for casting and spinning and substantially free from partially insoluble materials which comprises preparing a viscose solution in the usual manner and adding thereto, preferably at some stage in the manufacture thereof, a substance, for example, a small quantity of an alkaline solution, such as, a viscose solution made from a cellulose xanthate prepared with a higher ratio of carbon bisulphide per unit of cellulose than the cellulose xanthate employed in making the original viscose solution, which modifies the partially insoluble materials present therein to form a substantially homogeneous solution and whereby the clogging of the spinneret holes is appreciably minimized.

Other objects will become apparent from the following description and appended claims.

In accordance with an illustrative embodiment of this invention, alkali cellulose is xanthated with carbon bisulphide and the resultant xanthate dissolved in an aqueous caustic solution to give a viscose solution containing, for example, 7% cellulose and 6% alkali, the latter being calculated as NaOH. This solution contains the undesirable foreign materials above described and, therefore, in accordance with the principles of this invention, there is added a substance which overcomes and eliminates the deleterious results of the said partially insoluble materials by some sort of reaction therewith. The precise mechanism or reaction produced is not understood. It may be that the added substance reacts with, peptizes and/or renders the

partially insoluble material present soluble in the solution.

In the preferred method the modifying substance is added at some stage in the preparation of the viscose solution. For example, it may be added during the mixing of the cellulose xanthate and caustic alkali solution.

The substance which is added, as previously mentioned, is of such a character that it modifies the partially insoluble materials present therein to produce a substantially homogeneous solution whereby the spinneret holes are not clogged and yarns of uniform physicals produced As one illustrative specific example of a substance which has given satisfactory results may be mentioned a viscose solution, preferably made from a cellulose xanthate which has been prepared with a higher ratio of carbon bisulphide per unit of cellulose than the xanthate employed in the making of the viscose solution. Satisfactory results have been secured when the added viscose was formed of a xanthate produced with at least 10 parts of carbon bisulphide to 100 parts of cellulose more than the xanthate used in making the solution to which the former was added.

The quantity of substance which is added may vary within wide limits, partly depending on the nature of the original viscose solution. Excellent results have been secured when a relatively small amount, for example, 1%, 5%, 10% and even more, of a viscose solution produced from a xanthate prepared with a larger proportion of carbon bisulphide was added to a viscose solution produced from a xanthate prepared with a lesser proportion of carbon bisulphide.

The aforementioned procedure permits the production of a more usable viscose solution than heretofore wherein the cellulose xanthate is prepared with a relatively small amount of carbon bisulphide. By adding an appropriate amount of a viscose solution containing a highly xanthated xanthate, there is produced a viscose solution which contains a cellulosic compound relatively easy to regenerate and which is substantially homogeneous. When this type of viscose is spun, after filtration in the usual manner, in suitable coagulating baths to form threads, there is produced a filament of exceptionally great strength and substantially uniform physicals.

In order to more clearly explain the nature of the instant invention, there is hereafter set forth several illustrative specific embodiments thereof. It is to be understood that the invention is not restricted to the precise proportions of ingredients and sequence of steps set forth:

Example 1.—Alkali cellulose prepared in any way now in common use in the art is xanthated with 17 parts of carbon bisulphide to 100 parts of cellulose. The xanthate so obtained is mixed with a caustic alkali solution for one hour at 18° C. to produce a viscose solution of desired concentration, such as one containing 7% cellulose and 6% alkali, the latter being calculated as NaOH. There is then added to this solution 1% by weight of a viscose containing 7% cellulose and 6% alkali (calculated as NaOH) and prepared from a xanthate wherein 37 parts of carbon bisulphide to 100 parts of cellulose is utilized. The mass is mixed for approximately two hours at 18° C. After proper aging, the solution is filtered and spun.

Example 2.—A viscose solution prepared as set forth in Example 1 from a cellulose xanthate prepared with 14 parts of carbon bisulphide to 100 parts of cellulose and having a concentration of 7% cellulose and 6% alkali (calculated as NaOH) is very difficult to filter and to spin. By adding thereto 6% of a viscose solution having a concentration of 7% cellulose and 6% alkali (calculated as NaOH) and made from a cellulose xanthate prepared from 37 parts of carbon bisulphide to 100 parts of cellulose, and mixing, a very satisfactory spinning solution is prepared.

Since it is obvious that various changes and modifications may be made in the above description without departing from the nature and spirit thereof, this invention is not restricted thereto except as set forth in the appended claims.

I claim:

1. A process of preparing a viscose solution suitable for casting and spinning, which comprises preparing a viscose solution and incorporating therein a minor portion of a viscose solution made from a cellulose xanthate prepared with a higher ratio of carbon bisulphide per unit of cellulose than the cellulose xanthate used in the preparation of the first-mentioned viscose solution, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

2. A process of preparing a viscose solution suitable for casting and spinning, which comprises preparing a viscose solution and incorporating therein a minor portion of a viscose solution made from a cellulose xanthate prepared with at least 10 parts more carbon bisulphide per 100 parts of cellulose than was employed in making the cellulose xanthate used in the preparation of the first-mentioned viscose solution, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

3. A process of making a viscose solution suitable for coating or spinning which comprises preparing a viscose solution from a cellulose xanthate in which 17 parts of carbon bisulphide were used to 100 parts of cellulose and incorporating therein 1% by weight of a viscose solution which was prepared from a cellulose xanthate in which 37 parts of carbon bisulphide per 100 parts of cellulose were used, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

4. A process of making a viscose solution suitable for coating or spinning which comprises preparing a viscose solution from a cellulose xanthate in which 14 parts of carbon bisulphide were used to 100 parts of cellulose and incorporating therein 6% by weight of a viscose solution which was prepared from a cellulose xanthate in which 37 parts of carbon bisulphide per 100 parts of cellulose were used, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

5. A process of preparing a viscose solution suitable for casting or spinning, which comprises preparing a viscose solution and incorporating therein a minor portion of a cellulose xanthate prepared with a higher ratio of carbon bisulphide per unit of cellulose than was used in the preparation of the xanthate for the viscose solution, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

6. A process of preparing a viscose solution suitable for casting or spinning which comprises preparing a viscose solution and incorporating therein a minor portion of a cellulose xanthate prepared with at least 10 parts more carbon bisulphide per 100 parts of cellulose than was em-

ployed in making the cellulose xanthate used in the preparation of the viscose solution, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

7. A method of preparing a viscose solution suitable for casting and spinning which comprises preparing a viscose solution and incorporating therein 1%–10% of a viscose solution made from a cellulose xanthate prepared with a higher ratio of carbon bisulphide per unit of cellulose than the cellulose xanthate used in the preparation of the first-mentioned viscose solution, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

8. A method of preparing a viscose solution suitable for casting and spinning which comprises preparing a viscose solution and incorporating therein 6% of a viscose solution made from a cellulose xanthate prepared with a higher ratio of carbon bisulphide per unit of cellulose than the cellulose xanthate used in the preparation of the first-mentioned viscose solution, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

9. A method of preparing a viscose solution suitable for casting and spinning which comprises preparing a viscose solution and incorporating therein 1% of a viscose solution made from a cellulose xanthate prepared with a higher ratio of carbon bisulphide per unit of cellulose than the cellulose xanthate used in the preparation of the first-mentioned viscose solution, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

10. A process of preparing a viscose solution suitable for casting and spinning which comprises preparing a viscose solution and incorporating therein 1%–10% of a viscose solution made from a cellulose xanthate prepared with at least 10 parts more carbon bisulphide per 100 parts of cellulose than was employed in making the cellulose xanthate used in the preparation of the first-mentioned viscose solution, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

11. A process of preparing a viscose solution suitable for casting and spinning which comprises preparing a viscose solution and incorporating therein 6% of a viscose solution made from a cellulose xanthate prepared with at least 10 parts more carbon bisulphide per 100 parts of cellulose than was employed in making the cellulose xanthate used in the preparation of the first-mentioned viscose solution, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

12. A process of preparing a viscose solution suitable for casting and spinning which comprises preparing a viscose solution and incorporating therein 1% of a viscose solution made from a cellulose xanthate prepared with at least 10 parts more carbon bisulphide per 100 parts of cellulose than was employed in making the cellulose xanthate used in the preparation of the first-mentioned viscose solution, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

13. A process of preparing a viscose solution suitable for casting or spinning which comprises preparing a viscose solution and incorporating therein 1%–10% of a cellulose xanthate prepared with a higher ratio of carbon bisulphide per unit of cellulose than was in the preparation of the xanthate for the viscose solution, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

14. A process of preparing a viscose solution suitable for casting or spinning which comprises preparing a viscose solution and incorporating therein 6% of a cellulose xanthate prepared with a higher ratio of carbon bisulphide per unit of cellulose than was used in the preparation of the xanthate for the viscose solution, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

15. A process of preparing a viscose solution suitable for casting or spinning which comprises preparing a viscose solution and incorporating therein 1% of a cellulose xanthate prepared with a higher ratio of carbon bisulphide per unit of cellulose than was used in the preparation of the xanthate for the viscose solution, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

16. A process of preparing a viscose solution suitable for casting or spinning which comprises preparing a viscose solution and incorporating therein 1%–10% of a cellulose xanthate prepared with at least 10 parts more carbon bisulphide per 100 parts of cellulose than was employed in making the cellulose xanthate used in the preparation of the viscose solution, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

17. A process of preparing a viscose solution suitable for casting or spinning which comprises preparing a viscose solution and incorporating therein 6% of a cellulose xanthate prepared with at least 10 parts more carbon bisulphide per 100 parts of cellulose than was emloyed in making the cellulose xanthate used in the preparation of the viscose solution, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

18. A process of preparing a viscose solution suitable for casting or spinning which comprises preparing a viscose solution and incorporating therein 1% of a cellulose xanthate prepared with at least 10 parts more carbon bisulphide per 100 parts of cellulose than was employed in making the cellulose xanthate used in the preparation of the viscose solution, whereby the final solution is rendered homogeneous and substantially free from partially insoluble materials.

HAROLD HENRY PARKER.