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MANUFACTURE OF THREADS, FIBRES, FILA- MENTS AND THE LIKE FROM VISCOSE

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This invention relates to the manufacture of threads, fibres, filaments and the like from viscose.

British patent specification No. 183,882 describes the manufacture of high molecular weight viscose by treating cellulose with caustic alkali, in concentrations of from about 15 percent upwards, in a quantity of not more than 2 molecules per molecule of cellulose, with carbon disulphide employed in a quantity of not more than about half a molecule. The specification states that the viscose can be used for spinning or producing artificial silk, films and the like in the usual way. A suitable coagulating bath is described as containing alkali bicarbonate at moderately raised temperature of, for example, 30-40° C.

The object of the present invention is to produce threads, fibres, filaments and the like (hereinafter referred to as threads) of improved cellulosic products from viscose.

According to the present invention a process for the production of threads of cellulosic products from viscose comprising extruding viscose of gamma value 15 to 45 into an aqueous coagulating solution of sodium bicarbonate of greater than 50 percent saturation at 35-45° C. to form water-soluble threads of sodium cellulose xanthate, removing the water-soluble threads from the coagulating solution and treating these threads with one or more selected reactants to produce threads of stable cellulosic products.

In a preferred form of the invention viscose of gamma value 25 to 40 is spun into a saturated solution of sodium bicarbonate maintained at 40° C.

The freshly formed threads are removed from the coagulating solution and are washed with sodium chloride solution or with sodium bicarbonate solution alone or followed by a mixture of two volumes of acetone with one volume of water and finally with acetone alone. The threads are then air-dried. The acetone may be replaced by ethyl or methyl alcohol.

The solubility of the air-dry threads of sodium cellulose xanthate in cold water depends upon the age of the viscose prior to spinning. Viscose ripened up to an age of about 9 days, that is, having a gamma value of about 15, and spun in accordance with the invention yields threads of sodium cellulose xanthate which are soluble in cold water. Viscose of age over about 9 days yields threads which are insoluble in water but readily dissolve in dilute sodium hydroxide solution.

The preparation of threads of sodium cellulose xanthate is illustrated by the following example:

Example 1

Alkali cellulose formed by steeping wood pulp of 85 percent alpha-cellulose content and 7 percent moisture content in an 18 percent aqueous sodium hydroxide solution for 6 hours, followed by pressing, had the following composition—30 percent cellulose, 15.5 percent sodium hydroxide and 50.5 percent water. The alkali cellulose was then shredded into particles, aged for 38 hours and xanthated with carbon disulphide added in the proportion of 10.2 kgms. of carbon disulphide to 100 kgms. of

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alkali cellulose. The cellulose xanthate thus formed was dissolved in a 4.4 percent aqueous sodium hydroxide solution to form viscose. The viscose comprising 7.5 percent cellulose, 7 percent sodium hydroxide and 2.1 percent sulphur by weight, was stored at 20° C. in a viscose cave. After two days a sample having a gamma value of 38 was extruded from a standard spinneret having 28 holes of 3.5 thousandths of an inch each into a saturated aqueous solution of sodium bicarbonate at 40° C.

The freshly formed threads of sodium cellulose xanthate after a bath travel of 80-90 cms. were removed from the bath, and washed with a warm saturated aqueous solution of sodium bicarbonate. The threads were then washed with a mixture of 2 volumes of acetone to 1 volume of water followed by a wash of acetone alone. Finally the threads were air-dried.

The threads were almost transparent, bright and of a pale greenish colour having a circular cross-section with a smooth surface.

A sample of the viscose was spun on successive days until the viscose had gelled. The threads obtained in each case were tested for solubility in water (assessed after the threads had been steeped in acetone for 2-3 hours and then air-dried) with the following results:

Day:

- 1—Viscose manufactured and delivered to cave.
- 2—Viscose stored in cave at 20° C.
- 3—Water-soluble threads obtained.
- 4—Water-soluble threads obtained.
- 5—Water-soluble threads obtained.
- 6—Water-soluble threads obtained.
- 7—Water-soluble threads obtained.
- 9—Water-soluble threads obtained.
- 11—Threads obtained which were insoluble in water but dissolved in dilute sodium hydroxide.
- 13—Viscose slightly lumpy, spinning difficult; threads obtained were insoluble in water, but dissolved in dilute sodium hydroxide.
- 16—Viscose had gelled.

The tenacities of the air-dried threads were found to be 0.5-1.0 gram per denier for the water-soluble threads and 1.0-1.5 grams per denier for the water-insoluble threads. When the threads were subjected to stretching during spinning or after washing with the acetone-water mixture the tenacity of the air-dried threads increased to 1.0-1.5 grams per denier for the water-soluble threads and 1.5-2.0 grams per denier for the water-insoluble threads.

On storage at 20° C. the water-soluble threads gradually became water-insoluble over a period of days. This change, however, was substantially retarded by storage at 0° C.

The sodium bicarbonate may be replaced by potassium bicarbonate or ammonium bicarbonate with such modification of the spinning conditions as may be necessary in order to produce satisfactory threads, but the threads then obtained will not necessarily consist mainly of sodium cellulose xanthate.

Owing to their unstable nature, threads of sodium cellulose xanthate have little commercial value alone but owing to the fact that they can readily be converted to a variety of stable cellulosic threads by treatment with one or more selected reactants without any substantial change in shape, they are very valuable as intermediate products.

The threads of sodium cellulose xanthate may be converted partly (that is, on the surface only) or completely

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to threads of regenerated cellulose by suitable treatment with, for example, dilute acids, mixtures of alcohol and water, and hot water, the selection of the reagent depending upon the solubility of the threads in water. The smooth circular cross-section of the filaments of sodium cellulose xanthate is retained in the regenerated filaments.

Immersion of the threads of sodium cellulose xanthate in aqueous solutions of salts of metals such as calcium, zinc and thallium permits cation-exchange to take place resulting in threads having modified properties, particularly in respect of their solubility in aqueous media. Threads modified in this way are readily converted to threads of regenerated cellulose by the action of acids.

The threads of sodium cellulose xanthate may be converted partly (that is, on the surface only) or completely to threads of methyl cellulose xanthate by suitable treatment with methyl iodide as is illustrated in the following example:

Example 2

A skein of sodium cellulose xanthate threads prepared in Example 1 was immersed for 30 minutes at 20° C. in a solution consisting of 100 mls. methyl iodide, 3,000 mls. acetone and 1,000 mls. water. The skein was converted substantially to threads of methyl cellulose xanthate and was washed with a mixture of acetone and water and, finally, with water.

The threads of methyl cellulose xanthate may be converted to threads of cellulose thiourethane by treatment with ammonia as illustrated by the following example:

Example 3

The skein of methyl cellulose xanthate threads prepared in Example 2 was immersed for 12 hours at 20° C. in an aqueous solution containing 15 percent by weight of ammonia. The threads of methyl cellulose xanthate were substantially converted to threads of cellulose thiourethane (cellulose xanthamide) and were washed with water and dried.

Cellulose thiourethane threads are particularly useful as effect threads since they are capable of being dyed by wool dyestuffs.

When threads of sodium cellulose xanthate are treated with sodium monochloroacetate, threads of sodium cellulose xanthoacetate are produced as illustrated by the following example:

Example 4

A skein of sodium cellulose xanthate threads prepared in Example 1 was immersed for 12 hours at 20° C. in a solution of sodium monochloroacetate obtained by dissolving 500 grams of monochloroacetic acid in 5,000 mls. water and neutralising the resulting solution to pH 7 with sodium bicarbonate. The threads were substantially converted to threads of sodium cellulose xanthoacetate which were washed with a mixture of acetone and water and finally dried by treatment with acetone.

Sodium cellulose xanthoacetate threads, which are soluble in water, may be converted to threads of cellulose xanthoacetic acid by treatment with a dilute mineral acid as illustrated in the following example:

Example 5

A skein of threads of sodium cellulose xanthoacetate prepared in Example 4 was immersed for 1 hour at 20° C. in normal hydrochloric acid. The threads were substantially converted to threads of cellulose xanthoacetic acid which were washed with water and dried.

Threads of cellulose xanthoacetic acid are insoluble in water but readily dissolve in alkaline solutions such as dilute sodium hydroxide or ammonia solutions. They are particularly useful as "scaffolding threads" in the manufacture of woven and knitted products when the threads can be removed from the fabric by treatment with a suitable alkaline liquid.

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Sodium cellulose xanthoacetate threads may also be converted into threads of cellulose thiourethane by treatment with ammonia as illustrated in the following example:

Example 6

A skein of threads of sodium cellulose xanthoacetate prepared in Example 4 was immersed for 12 hours at 20° C. in an aqueous solution containing 15 percent by weight of ammonia. The threads were substantially converted to threads of cellulose thiourethane.

By using the sodium cellulose xanthate threads as intermediate products it is seen that a variety of useful cellulosic threads may be readily prepared by the action of one or more reagents thus avoiding the development of separate spinning techniques for each particular thread.

What I claim is:

1. A process for the production of threads of cellulose derivatives from viscose which comprises extruding viscose of gamma value 15 to 45 into an aqueous coagulating solution of sodium bicarbonate of greater than 50% saturation, at 35° C. to 45° C. to form water soluble threads of sodium cellulose xanthate, removing the water soluble threads from the coagulating solution and reacting the threads with at least one compound selected from the group consisting of sodium monochloroacetate and methyl iodide.

2. A process for the production of threads consisting at least in part of methyl cellulose xanthate which comprises extruding viscose of gamma value 15 to 45 into an aqueous coagulating solution of sodium bicarbonate of greater than 50% saturation at 35° C. to 45° C. to form water soluble threads of sodium cellulose xanthate, removing the water soluble threads from the coagulating solution and reacting said threads with methyl iodide.

3. A process for the production of threads consisting at least in part of sodium cellulose xanthoacetate which comprises extruding viscose of gamma value 15 to 45 into an aqueous coagulating solution of sodium bicarbonate of greater than 50% saturation at 35° C. to 45° C. to form water soluble threads of sodium cellulose xanthate, removing the water soluble threads from the coagulating solution and reacting said threads with sodium monochloroacetate.

4. A process for the production of threads consisting at least in part of cellulose xanthoacetic acid which comprises extruding viscose of gamma value 15 to 45 into an aqueous coagulating solution of sodium bicarbonate of greater than 50% saturation at 35° C. to 45° C. to form water soluble threads of sodium cellulose xanthate, removing the water soluble threads from the coagulating solution, reacting said threads with sodium monochloroacetate to convert the threads at least in part to sodium cellulose xanthoacetate and then treating the threads so converted with a dilute mineral acid.

5. A process for the production of threads consisting at least in part of cellulose thiourethane which comprises extruding viscose of gamma value 15 to 45 into an aqueous coagulating solution of sodium bicarbonate of greater than 50% saturation at 35° C. to 45° C. to form water soluble threads of sodium cellulose xanthate, removing the water soluble threads from the coagulating solution, reacting said threads with an etherifying agent selected from the group consisting of sodium monochloroacetate and methyl iodide and thereafter reacting the threads with ammonia.

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