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2,946,650

PROCESS FOR THE MANUFACTURE OF VISCOSE RAYON STAPLE

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7 Claims. (Cl. 18—54)

This invention relates to a process for producing rayon staple or filament having a high degree of polymerization (D.P.) from alkali cellulose and this application for patent is a continuation-in-part of my co-pending application Serial No. 650,818, filed April 5, 1957.

The basic process, as described in application Serial No. 650,818, comprises essentially the steps of xanthating alkali cellulose having a high degree of polymerization with a mixture of carbon disulfide and a surface active agent in the presence of a mono-amine, extruding the resulting viscose into a spinning bath containing a weak solution of sulfuric acid and some sodium sulfate and thereafter immersing the thread in a secondary bath containing sulfuric acid. While Turkey red oil was the only surface active agent mentioned in that application, it has been subsequently found that xanthating in a mixture of carbon disulfide, mono-amine and sulfonated oil produces slightly improved properties in the final rayon product.

According to this invention, the alkali cellulose is xanthated with carbon disulfide, a mono-amine and a surface active agent, such as Turkey red oil or sulfonated oil, and the resulting viscose is spun in a spinning bath containing oxalic acid or a mixture of oxalic acid and sulfuric acid. The properties produced in the resulting rayon are improved so as to give a better quality of rayon staple or filament and moreover, the oxalic acid content in the bath facilitates the spinning due to the fact that the gel-thread is more elastic and stronger thus preventing breaking of the thread in the bath and allowing a higher and a greater variation in speed of thread travel through the bath as on stepped godets which are frequently provided for controlling the tension within the bath.

The concentration of the oxalic acid in the spinning bath should preferably be within the range of 6 grams per liter minimum and 50 grams per liter maximum and when a mixture of oxalic acid and sulfuric acid is employed in the bath, it is preferred that the oxalic acid be within the range of 1 to 30 grams per liter and that the sulfuric acid be lower than 15 grams per liter maximum. It may be noted that while in either case the sodium ion accumulates unavoidably in the spinning bath, the maximum allowable amount of sodium ion should be less than 20 grams per liter.

The following examples illustrate actual processes using oxalic acid and a mixture of oxalic acid and sulfuric acid in the production of rayon from viscose which have been laboratory demonstrated.

EXAMPLE I

Wood pulp with D.P. 1200 is steeped in alkali soda solution of 18.8% for 2 hrs. at 20° C., pressed to 2.7 times of original weight of pulp, crushed at 28° C. for 1.5 hrs. Alkali-cellulose thus obtained is xanthated with a mixture of 55% of carbon disulphide, 1% of triethylamine and 2% of a nonionic active agent, i.e. calculated on weight of pulp, at 18–28° C. for 2.5 hrs. After xanthation, viscose, having viscosity of 430 sec. with cel-

lulose content of 4.75% and total alkali content of 3.33% is obtained by dissolving in diluted soda solution. This viscose is extruded into spinning bath containing oxalic acid of 10.7 grams per liter and thereafter the gel-thread is stretched step by step on a five-stepped godet (ratio of consecutive steps is 1:2:2.5:3:3.2) immersed in the spinning bath. The thread is successively treated in a regenerating bath containing sulfuric acid of 15 gr. per liter at room temperature without tension. The final spinning velocity is 25 m./min. Characteristics of the thread thus produced are shown in case 1 in the table shown below.

EXAMPLE II

The same viscose produced by the method described in Example I is spun in the spinning bath with a mixture of oxalic acid of 6 gr. per liter and sulfuric acid of 3 gr. per liter under stretching on a 4-stepped godet with ratio of 1:2:2.5:3, and in the second bath of 15 gr. per liter of sulfuric acid at 85° C., the thread is again stretched 10% more and regenerated perfectly. Characteristics of the thread as produced by the process in this example are shown in the following table as case 2.

Table

	Denier	Dry Strength, g./d.	Wet Strength, g./d.	Dry elongation (per-cent)	Wet elongation (per-cent)	Knot Strength, g./d.
Case 1.....	0.88	5.51	4.23	12.9	15.8	2.88
Case 2.....	0.90	5.87	4.60	11.5	12.4	2.48
Case of Serial #650,818.....	0.90	5.38	4.07	12.2	14.1	2.70

Some modifications may be made in the process as above described without departing from the principle and scope of invention as defined in the following claims.

What is claimed is:

1. A process for producing a regenerated cellulose rayon staple or filament having a high degree of polymerization, comprising the steps of introducing alkali cellulose into a mixture of carbon disulfide, Turkey red oil and triethylamine, extruding the resulting viscose into a spinning bath containing a solution of oxalic acid and sodium ions which accumulate unavoidably during spinning and thereafter immersing the thread in a secondary bath containing a weak solution of sulfuric acid.

2. A process for producing a regenerated cellulose rayon staple or filament having a high degree of polymerization, comprising the steps of introducing alkali cellulose into a mixture of carbon disulfide, Turkey red oil and triethylamine, extruding the resulting viscose into a spinning bath containing a solution of a mixture of oxalic acid and sulfuric acid and sodium ions which accumulate unavoidably during spinning and thereafter immersing the thread in a secondary bath containing a weak solution of sulfuric acid.

3. A process for producing a regenerated cellulose rayon staple or filament as claimed in claim 1 in which the amount of oxalic acid in the spinning bath is substantially within the range of 6 to 50 grams per liter.

4. A process for producing a regenerated cellulose rayon staple or filament as claimed in claim 2 in which the amount of oxalic acid in the spinning bath is substantially within the range of 6 to 50 grams per liter.

5. A process for producing a regenerated cellulose rayon staple or filament as claimed in claim 2 in which the said spinning bath contains oxalic acid substantially within the range of 1 gram per liter to 30 grams per liter and sulfuric acid substantially within the range of 3 grams per liter to 15 grams per liter.

6. A process for producing a regenerated cellulose

rayon staple or filament as claimed in claim 5 in which the sulfuric acid content of the secondary bath is about 15 grams per liter and the temperature of the bath is about 85° C.

7. A process for producing a regenerated cellulose rayon staple or filament as claimed in claim 1 in which the content of sulfuric acid in the secondary bath is approximately 15 grams per liter and the secondary bath is substantially at room temperature.

References Cited in the file of this patent

UNITED STATES PATENTS

1,683,199	Lilienfeld	Sept. 4, 1928
2,647,114	Torke et al.	July 28, 1953
2,663,704	Yehling	Dec. 22, 1953