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YARN PRODUCTION

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This invention relates to the production of regenerated cellulose yarn from viscose compositions. More particularly, it relates to an improved process for the spinning of viscose rayon yarn in a coagulating and regenerating bath containing a high concentration (from 8% to 18%) of zinc sulfate.

The art has long recognized the virtue of coagulating-regenerating baths containing a high concentration of zinc sulfate for use in the spinning of viscose rayon yarn. Yarn produced by the use of such spinning baths has very desirable physical characteristics. However, the extrusion, or spinning, of viscose, of the type universally used in the production of viscose rayon and containing 6% to 8% caustic (calculated as NaOH), into an acid spinning bath, containing an appreciable quantity of zinc sulfate, is accompanied by the very objectionable phenomenon of "spinneret cratering." As the concentration of zinc in the coagulating bath increases, so also does spinneret cratering increase. For example, when viscose containing 6% alkali and 7% cellulose is spun into a bath containing 8% sulfuric acid, 11% sodium sulfate, 4% glucose, and 15% zinc sulfate at 60° C., about 24% of the orifice area of the spinneret is clogged, incrustated or cratered in four hours. It can readily be seen that under such conditions, sustained spinning of a uniform denier yarn is impossible.

"Spinneret cratering" is a term commonly applied to the formation of incrustations and depositions which accumulate on the spinneret face and in the spinneret orifices during the production of regenerated cellulose yarn from viscose by the wet spinning process. Under some conditions, especially with spinning baths containing high percentages of zinc sulfate, these incrustations are apparently caused, to a large extent, by the deposition of acid soluble zinc sulfide at the face of the spinneret.

The art has long been faced with the problem of effectively reducing the craters or depositions in the minute orifices of the spinneret. Such deposits disrupt the flow of viscose thus causing broken filaments or filaments of uneven denier to be formed. Numerous additions to the coagulating bath and to the viscose have been proposed to reduce spinneret cratering and thereby permit uninterrupted and uniform spinning. In most cases, the proposed addenda are expensive. Moreover, the addition of such cratering inhibitors to the viscose or to the coagulating and regenerating bath usually involves additional labor and control difficulties.

It is therefore an object of this invention to provide an improved process for the spinning of viscose rayon with a coagulating-regenerating bath containing a high concentration of zinc sulfate (8% to 18% by weight).

It is a further object of this invention to provide a process for the spinning of viscose rayon with a coagulating-regenerating bath containing a high concentration of zinc sulfate (8% to 18% by weight) which process will not be subject to excessive spinneret cratering.

Other objects of the invention will appear hereinafter.

The objects of this invention may be accomplished, in general, by spinning a low caustic viscose (3% to 5.5% caustic calculated as NaOH) into a coagulating and regenerating bath containing a high concentration of zinc sulfate (8% to 18% by weight of zinc sulfate).

It is also essential, for the purpose of obtaining good spinning conditions and optimum physical characteristics of the resulting yarn, that the sulfuric acid content of the coagulating bath be held within certain limits correlated with the caustic content of the viscose and the zinc sulfate content of the coagulating bath. The sulfuric acid content of the coagulating bath should therefore be within plus or minus 0.6% of that determined by the following equation:

$$A=0.75C-0.1S+3.8$$

where A is the percentage concentration by weight of the sulfuric acid, C is the percentage concentration by weight of the NaOH content of the viscose, and S is the percentage concentration by weight of the zinc sulfate in the coagulating bath. For example, if a 7% cellulose, 4% NaOH viscose is to be spun into a bath comprised of 10% zinc sulfate, by the use of the above equation the acid concentration will be found to be 5.8% H_2SO_4 . The sulfuric acid concentration in the coagulating bath may then vary between 5.8% minus 0.6% i. e., 5.2%, or 5.8% plus 0.6% i. e., 6.4%. Under such conditions, the spinning of the viscose yarn may be carried out with excellent spinning continuity and the resulting yarn will have excellent physical characteristics. Furthermore, the spinning may be carried out for long periods of time without encountering excessive cratering of the spinnerets. The following table illustrates a number of viscose compositions containing varying percentages of caustic and coagulating baths containing varying percentages of $ZnSO_4$, together with the corre-

lated H_2SO_4 concentration to be used in accordance with the present invention. All figures in the table are percentages by weight.

Viscose		Coagulating bath composition				
Cellulose	NaOH	Calculated H_2SO_4	Satisfactory limits H_2SO_4	Na_2SO_4	Glucose	ZnSO_4
7.0	5.5	6.9	6.3-7.5	10	4	10
7.0	5.5	6.1	5.5-6.7	2	4	18
7.0	4.0	5.8	5.2-6.4	11	0	10
7.0	3.5	5.4	4.8-6.0	10	4	10
7.0	3.5	4.6	4.0-5.2	2	4	18

The temperature of the coagulating and regenerating bath has been found to influence spinneret cratering to a certain extent. Spinneret cratering is most effectively reduced when the temperature of the bath is maintained substantially above 40°C ., for example, if the bath is maintained at about 60°C ., or higher. Although lower bath temperatures, such as $40^\circ\text{--}50^\circ\text{C}$., may be employed satisfactorily when the concentration of the zinc sulfate is in the neighborhood of 10%, at high concentrations of zinc sulfate such as 18%, it is generally advisable to use a bath temperature of 60°C . or even as much as 75°C .

This effect of temperature on coagulating baths containing a high concentration of zinc sulfate is quite surprising in view of the previously known fact that low temperatures were found to decrease spinneret cratering in processes involving coagulating baths having low percentages of zinc sulfate. It has been known heretofore that coagulating baths containing, for example, 0.7% zinc sulfate are subject to serious spinneret cratering when the bath temperature is maintained at 55°C ., and the lowering, in such instances, of the bath temperature will reduce the spinneret cratering.

The following examples illustrate preferred embodiments of the present invention, it being understood that the details set forth in these examples are merely illustrative and not limitative of the invention.

Example I

A viscose composition comprising 7% cellulose and 4% NaOH is spun into a coagulating-regenerating bath having the following composition: 6% sulfuric acid, 11% sodium sulfate and 10% zinc sulfate. The bath is maintained at a temperature of 60°C . The yarn is spun at a conventional spinning speed of 3500 inches per minute. The spinning may be carried out for extended periods of time without objectionable spinneret cratering. The resulting yarn has very desirable physical characteristics.

Example II

A viscose similar to that described in Example I is spun into a coagulating-regenerating bath having the same composition as that described in Example I. The yarn is spun at a spinning speed of 7000 inches per minute. Such speed of spinning is not feasible in a bath containing no zinc sulfate or relatively low percentages of zinc sulfate. The spinning conditions are substantially as good as those set forth in Example I, and the resulting yarns have excellent physical characteristics.

Although, in accordance with the preferred embodiments of the invention, viscose containing 7% cellulose and 4% caustic is used for the pro-

duction of viscose rayon in a spinning bath having a high zinc concentration, the cellulose content of the viscose may vary between 5%–12% by weight, preferably between 6%–8%, and the alkali content may vary between 3%–5% by weight. Preferably, the caustic content of the viscose should be as low as possible and yet obtain optimum spinnability with any given bath. The viscose may also contain wetting agents, dyes or pigments, or any substance customarily added to the viscose compositions. The coagulating and regenerating bath may contain from 1%–20% sodium sulfate and from 0%–5% glucose. The sulfuric acid content of the spinning bath is preferably adjusted to the zinc sulfate content of the bath and the alkali content of the viscose in accordance with the above formula. The zinc sulfate in the coagulating bath may vary between 8% and 18% by weight; however, the greatest advantages of the invention are forthcoming in spinning baths containing over 10% zinc sulfate. Spinning baths containing over 10% zinc sulfate are useful in the production of yarn having exceptionally desirable physical properties; however, heretofore, spinning baths containing such high concentrations of zinc sulfate could not be readily used due to the tendency of spinneret cratering. If desired, the coagulating bath may contain other agents tending to inhibit cratering, for example polymerized ethylene oxide (Carbowax 4000) which may be used in amounts from 0.001% to 1% based on the total weight of the bath. Such additional crater inhibiting materials are not necessary in a bath to attain the objects of the present invention.

The present invention provides a process for the production of regenerated cellulose yarn with spinning baths containing high concentrations of zinc sulfate in which the spinneret cratering difficulties are no longer excessive as compared with previously known processes involving the use of high zinc concentrations in the spinning bath. By following the process of the present invention, spinning may be carried out at least three times as long as previously known processes involving spinning baths having high concentrations of zinc sulfate. This reduction in spinneret cratering is accomplished in a simple economical manner with a minimum of operational difficulty. In accordance with the process of the present invention, yarns having improved physical characteristics are obtained at normal spinning speeds. Furthermore, by the use of very materially higher spinning speeds, yarns having very satisfactory physical characteristics can be produced. Moreover, by the practice of this invention the addition of spinneret cratering inhibiting agents may be eliminated. Furthermore, the use of viscose with a low caustic content results in a saving in the operating expenses over previously known spinning processes.

Since it is obvious that many changes and modifications can be made in the above described details without departing from the nature and spirit of the invention, it is to be understood that the invention is not limited to the above details except as set forth in the appended claims.

I claim:

1. The process of spinning regenerated cellulose filaments from viscose which comprises extruding viscose containing 3% to 5.5% caustic (calculated as NaOH) into a sulfuric acid coagulating and regenerating bath containing from 8% to 18% zinc sulfate, the sulfuric acid con-

tent of the bath being within plus or minus 0.6% of that determined by the equation:

$$A=0.75C-0.1S+3.8$$

wherein A represents the percentage concentration by weight of the sulfuric acid, C represents the percentage concentration of the caustic (cal-

culated as NaOH) in the viscose and S represents the percentage concentration by weight of the zinc sulfate.

2. The process as defined in claim 1 in which the coagulating and regenerating bath is maintained at a temperature of at least 60° C.

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