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MANUFACTURE OF ARTIFICIAL SILK

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4 Claims. (Cl. 18—54)

It is already well known to spin viscose solutions into a conventional, primary, acid spin bath containing various salts, and to stretch the threads, thus obtained, in a hot secondary bath. British Patent #467,500, for example, discloses the beneficial effect of the primary spin bath upon the ultimate physical characteristics of spun viscose threads, and it recommends the use of primary spin baths with a high zinc content, i. e., baths containing 4% of zinc sulphate (about 50 g. of $ZnSO_4$ per liter of bath). According to this process, it is possible to produce a 120 den. thread with 60 filaments in a warm spin bath containing about 9.5% of sulphuric acid, 20% of sodium sulphate and 4% of zinc sulphate and to stretch the thread 87% after a bath travel of about 20 cm. in said primary bath in a secondary bath maintained at a temperature of at least 60° C. and preferably of about 95° C., this secondary bath containing only small quantities of the bath ingredients present in the primary bath. The high zinc content of the primary bath is evidently responsible for the good physical properties of viscose threads produced therein. However, it has been found in actual practice that spin baths having a high zinc content cause rapid clogging of spinneret orifices through which the viscose solution is extruded into such baths.

It is, therefore, a primary object of our invention to provide a zinc-bearing spin bath which substantially prevents the clogging of spinneret orifices without reducing the physical characteristics of viscose threads formed therein in comparison with spin baths heretofore known in the art.

Other objects of our invention will become apparent to those skilled in the art from a study of the following specification.

Many theories have been advanced to explain the role which zinc sulphate and other soluble zinc salts play in the aforementioned spinning processes. It has been said that the favorable results obtained with zinc sulphate are due to its retarding effect upon the decomposition of the thread at the beginning of the stretching step. However, our experiments have shown that this retarding effect of zinc sulphate cannot fully explain the beneficial results obtained therewith since ammonium sulphate baths, as is well known, neither cause the formation of a decomposed thread nor a favorable result. For this reason, it is necessary to presume that zinc sulphate causes very specific effects besides retarding the coagulation and/or decomposition of viscose threads. It is very likely that the zinc viscose (xanthate) formed in the primary spin bath causes a retardation of the final decomposition of the thread and that the thread and/or this compound ought not be completely decomposed upon entering the fixing and/or stretching bath. The rate of decomposition of this intermediate compound is however, as our experiments showed, only retarded when the sulphuric acid concentration of the primary spin bath is reduced and preferably brought to a minimum. Since a reduction of the sulphuric acid content of the primary bath causes a reduction of its coagulating and/or precipitating power, it is necessary to compensate for this deficiency by adding other suitable ingredients thereto. Thus, we have found that the zinc sulphate and sulphuric acid content of the primary bath may be reduced provided larger amounts of magnesium sulphate are added thereto. Additions of 150 g., 200 g., 250 g. and more of magnesium sulphate per liter of spin bath have given excellent results. Instead of using a primary bath containing 125 g. of sulphuric acid, 260 g. of sodium sulphate and 52 g. of zinc sulphate per liter, it is possible to use spin baths having the following compositions:

Bath	Sulphuric acid	Sodium sulphate	Magnesium sulphate	Zinc sulphate
	Grams per liter	*Grams per liter*	*Grams per liter*	*Grams per liter*
No. 1	115	260	200	30
No. 2	115	200	250	20
No. 3	110	200	250	16

These novel spin baths substantially inhibit clogging of spinneret orifices. This is especially true when the zinc sulphate content is reduced to about 30 grams per liter of bath, or below this value. The stretchability of threads is at least equal to that one in baths having a higher zinc content while the physical characteristics of the finished threads are maintained. This result is entirely unexpected since magnesium sulphate has heretofore been regarded as a chemical which reduces the stretchability of viscose threads spun in its presence. Indeed, further experiments have proven that magnesium sulphate is not capable of completely replacing zinc sulphate in primary spin baths. For this reason magnesium sulphate makes it possible to reduce the sulphuric acid content of primary spin baths and to cause indirectly a retardation of the decomposition of zinc viscose. For this reason it is

also possible to reduce the zinc sulphate concentrations of the primary spin baths.

We claim:

1. The process of producing threads of regenerated cellulose which comprises first spinning a viscose solution through the orifices of a spinneret into a primary, zinc-bearing, aqueous spin bath to form an incompletely decomposed viscose thread therein and subsequently stretching and decomposing said thread in a hot, secondary, aqueous spin bath to form a thread of regenerated cellulose, said primary spin bath containing about 16 to 30 grams of zinc sulphate, about 110 to 115 grams of sulphuric acid, about 150 to 250 grams of magnesium sulphate and about 200 to 260 grams of sodium sulphate per liter of bath, and said secondary spin bath having a temperature of at least 60° C. and containing less sodium sulphate, zinc sulphate, sulphuric acid and magnesium sulphate per liter of bath than said primary bath, whereby incrustation of said spinneret is substantially prevented.

2. The process of producing threads of regenerated cellulose which comprises first spinning a viscose solution through the orifices of a spinneret into a primary, zinc-bearing, aqueous spin bath to form an incompletely decomposed viscose thread therein and subsequently stretching and decomposing said thread in a hot, secondary, aqueous spin bath to form a thread of regenerated cellulose, said primary spin bath containing about 260 grams of sodium sulphate, about 30 grams of zinc sulphate, about 115 grams of sulphuric acid and about 200 grams of magnesium sulphate per liter of spin bath, and said secondary spin bath having a temperature of at least 60° C. and containing less sodium sulphate, zinc sulphate, sulphuric acid and magnesium sulphate per liter of bath than said primary bath, whereby incrustation of said spinneret is substantially prevented.

3. The process of producing threads of regenerated cellulose which comprises first spinning a viscose solution through the orifices of a spinneret into a primary, zinc-bearing, aqueous spin bath to form an incompletely decomposed viscose thread therein and subsequently stretching and decomposing said thread in a hot, secondary, aqueous spin bath to form a thread of regenerated cellulose, said primary spin bath containing about 200 grams of sodium sulphate, about 20 grams of zinc sulphate, about 115 grams of sulphuric acid and about 250 grams of magnesium sulphate per liter of spin bath, and said secondary spin bath having a temperature of at least 60° C. and containing less sodium sulphate, zinc sulphate, sulphuric acid and magnesium sulphate per liter of bath than said primary bath, whereby incrustation of said spinneret is substantially prevented.

4. The process of producing threads of regenerated cellulose which comprises first spinning a viscose solution through the orifices of a spinneret into a primary, zinc-bearing, aqueous spin bath to form an incompletely decomposed viscose thread therein and subsequently stretching and decomposing said thread in a hot, secondary, aqueous spin bath to form a thread of regenerated cellulose, said primary spin bath containing about 200 grams of sodium sulphate, about 16 grams of zinc sulphate, about 110 grams of sulphuric acid and about 250 grams of magnesium sulphate per liter of spin bath, and said secondary spin bath having a temperature of at least 60° C. and containing less sodium sulphate, zinc sulphate, sulphuric acid and magnesium sulphate per liter of bath than said primary bath, whereby incrustation of said spinneret is substantially prevented.

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