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PRODUCTION OF YARNS OF REGENERATED CELLULOSE HAVING IMPROVED PROPERTIES

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This invention relates to the production of regenerated cellulose yarns having improved properties, and the resulting yarns per se.

Yarns of regenerated collulose which are worked up to provide particularly resistant woven or knitted fabrics or which are used for technical purposes, more especially as tire cords, must be characterized by high bending and abrasion strengths as well as very good tensile strength and elasticity values in order that they may be able to withstand high loads. The lowering of the degree of swelling and also an improvement in the resistance to fatigue are also of great importance.

Various methods have been tried for satisfying these and other technical requirements and various processes have been developed by which it is possible to produce yarns having a strong and pronounced peripheral zone, it being known that such yarns are characterized by good elasticity properties, low swelling factors and high strength values. The abrasion and bending strengths are favorably influenced by spinning viscoses with high gamma numbers and also a high content of carbon bisulphide; however, the last feature in particular is so uneconomical that such a working method cannot be seriously considered for manufacture on a large technical scale. Good results have furthermore been obtained by adding monoamines having at least 4 C-atoms to the viscose and spinning in coagulation baths with a high zinc sulphate content. The amounts of these additives should not be more than 4 millimoles per 100 grams of viscose. These monoamines have also been added in substantially the same amounts to the spinning baths. The spinning baths used when operating by these methods contain zinc sulphate, namely, in amounts of 3 to 25%. However, serious disadvantages may be encountered in carrying out these processes, since the spinning conditions must be maintained within very narrow limits and in addition only a small latitude is allowed for the draw-off speed. For example, if the desired technical results are to be assured, it is not possible to operate with draw-off speeds greater than 20–25 meters/minute.

It has now been found that high-grade yarns having a strong peripheral zone as well as still further improved strength and elasticity properties are produced, the said yarns showing a low swelling factor and a high resistance to fatigue, if to a viscose which is not too ripe there is added a small amount of monoisopropanol amine and the viscose is then spun in baths which have a zinc sulphate content of at least 3.2% and the sulphuric acid concentration of which is the same as, or smaller than, the alkali concentration of the spun viscose. It has been discovered that the difficulties and disadvantages that may arise when spinning viscose with additions of monoamines having at least 4 C-atoms are avoided by using the aforementioned compound, and that it is possible to work with draw-off speeds of 40 to 50 meters/minute while still achieving the desired technical results.

The monoisopropanol amine is added in amounts of 0.06–0.4% by weight, preferably 0.10–0.15% by weight, to the viscose, it being in fact possible to introduce this additive at the time of dissolving the xanthate. The additive can be employed with all viscoses of conventional working compositions, and as such there may be mentioned those having 7–8% of cellulose and 5–7% of NaOH. The spinning ripeness of the viscose should not be below a γ-number of 42. The spinning baths to be used should contain zinc in amounts of at least 3.2%. The sodium sulphate content can be within the usual limits between 14.00% and 20.00%, while the sulphuric acid content should be the same as or somewhat lower than the NaOH content of the viscose. The spinning bath temperature should be between 45° and 70° C. Very good results are obtained with draw-off speeds of 40 to 50 meters/minute. However, these speeds can be still further increased if provision is made, by the use of known means, for causing the spinning bath to flow at a speed which is substantially equal to the yarn speed, at least over the first part of the path of yarn travel beyond the spinning nozzle, so that the resulting reduction in relative speed between the yarn and the spinning bath has the net result that the friction between the thread and the spinning bath is largely reduced. The length of travel in the spinning bath should be at least 30 cm. The thread is then subjected in a second hot and weakly acid bath to a stretching of 80–120%, preferably 50–100%, after which it may be collected on a spool or in a centrifuge or placed on a perforated belt.

The yarns spun by the process according to the present invention show strength values of 400–420 g./100 den. These values can be increased to 430–460 g./100 den. by afterstretching. The yarns are particularly suitable for use as tire cord owing to their high shrinkage and abrasion values, and also owing to an extraordinarily low swelling factor.

In order to indicate still more fully the nature of the present invention, the following examples of typical procedures are set forth, it being understood, however, that this description is presented by way of illustration only, and not as limiting the scope of the invention. Parts are by weight unless indicated otherwise by the context.

Example 1

Alkali cellulose composed of linters or wood cellulose with a high α-cellulose content is sulphidised for five hours with 38% carbon bisulphide and the xanthate obtained is dissolved to give a viscose having 7.4% of cellulose and 5% of NaOH. During the dissolving operation 0.1% of monoisopropanol amine is added to the viscose. The viscose is then filtered in the usual way, deaerated and finally ripened, and is spun with a viscosity of 60 seconds by the falling-ball method and with a γ-number of 44. The spinning bath has a composition of 4.8% of H_2SO_4, 4% of $ZnSO_4$ and 17% of Na_2SO_4, and a temperature of 60° C. The yarn has 1000 filaments with an individual titre of 1.65 den. and is guided over a distance of 65 cm. in the spinning bath and is withdrawn by a roller at a speed of 21 meters/minute. The yarn then travels through a second weakly acid bath which has a temperature of about 90° C. and is withdrawn at the end of said second bath by a second roller revolving sufficiently fast to give a yarn draw-off speed of 42 meters/minute, so that the yarn is stretched by 100%. From the second roller the yarn runs into a centrifuge rotating at 4600 r.p.m. and is given a twist therein of 120 turns per meter. After being washed and finished, the yarn is finally stretched by 8% and dried. The finished yarn has tensile strengths of 4.4 g./den. (dry) and 3 g./den. (wet), and elongation values of 14% (dry) and 27% (wet).

Example II

A viscose corresponding to that described in Example I is spun at a temperature of 60° C. into a bath having the following composition: 5% of H_2SO_4, 4% of $ZnSO_4$ and 16% of Na_2SO_4. A spinneret with 120 orifices is used; the yarns have a titre of 60 den. After being stretched by 100% in a second hot bath, the yarn is collected in a centrifuge. It has a tensile strength of 4.2 g./den. with an elongation of 15%.

Upon testing the abrasion strength of yarns produced according to the present invention in comparison to that of other yarns a substantial improvement is clearly evident. The tests are carried out with a yarn loaded with 30 g. (and having a total titre of 1650 den.) passing over a roller rotating at a speed of 100 r.p.m. Whereas a standard staple fibre yarn is worn through after 600 revolutions of the roller, a cotton yarn after 7000 revolutions, and a yarn of the best quality intended for tire cord after 80,000 revolutions, it is found that the yarn produced according to the present invention withstands 250,000 revolutions of the roller, i.e., a more than threefold improvement over the next best yarn tested.

While specific examples embodying the present invention have been described above, it will be apparent that many changes and modifications may be made in the methods of procedure and in ingredients recited without departing from the true spirit of the invention. It will therefore be understood that the examples cited and the particular proportions, ingredients and methods set forth above are intended to be illustrative only, and are not intended to limit the scope of the invention.

What is claimed is:

1. In a method for the manufacture of regenerated cellulose yarns from viscose, the step which comprises adding to viscose a small amount of mono-isopropanol amine.

2. In a method for the manufacture of regenerated cellulose yarns from viscose, the step which comprises adding to viscose approximately 0.06–0.4% by weight of mono-isopropanol amine.

3. In a method for the manufacture of regenerated cellulose yarn from viscose, the step which comprises adding to viscose approximately 0.10–0.15% by weight of mono-isopropanol amine.

4. The method of producing regenerated cellulose yarns from viscose comprising spinning viscose into an aqueous spinning bath containing sulphuric acid and zinc sulphate, the viscose containing a small amount of mono-isopropanol amine.

5. The method of producing regenerated cellulose yarn from viscose comprising spinning viscose into an aqueous spinning bath containing sulphuric acid and at least 3.2% zinc sulphate, the viscose containing approximately 0.06–0.4% by weight of mono-isopropanol amine.

6. The method of producing regenerated cellulose yarn from viscose comprising spinning viscose into an aqueous spinning bath containing sulphuric acid and at least 3.2% zinc sulphate, the viscose containing approximately 0.06–0.4% by weight of mono-isopropanol amine and the concentration of sulphuric acid being at most the same as the concentration.

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