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METHOD FOR PRODUCING HIGH TENACITY ARTIFICIAL YARN AND CORD

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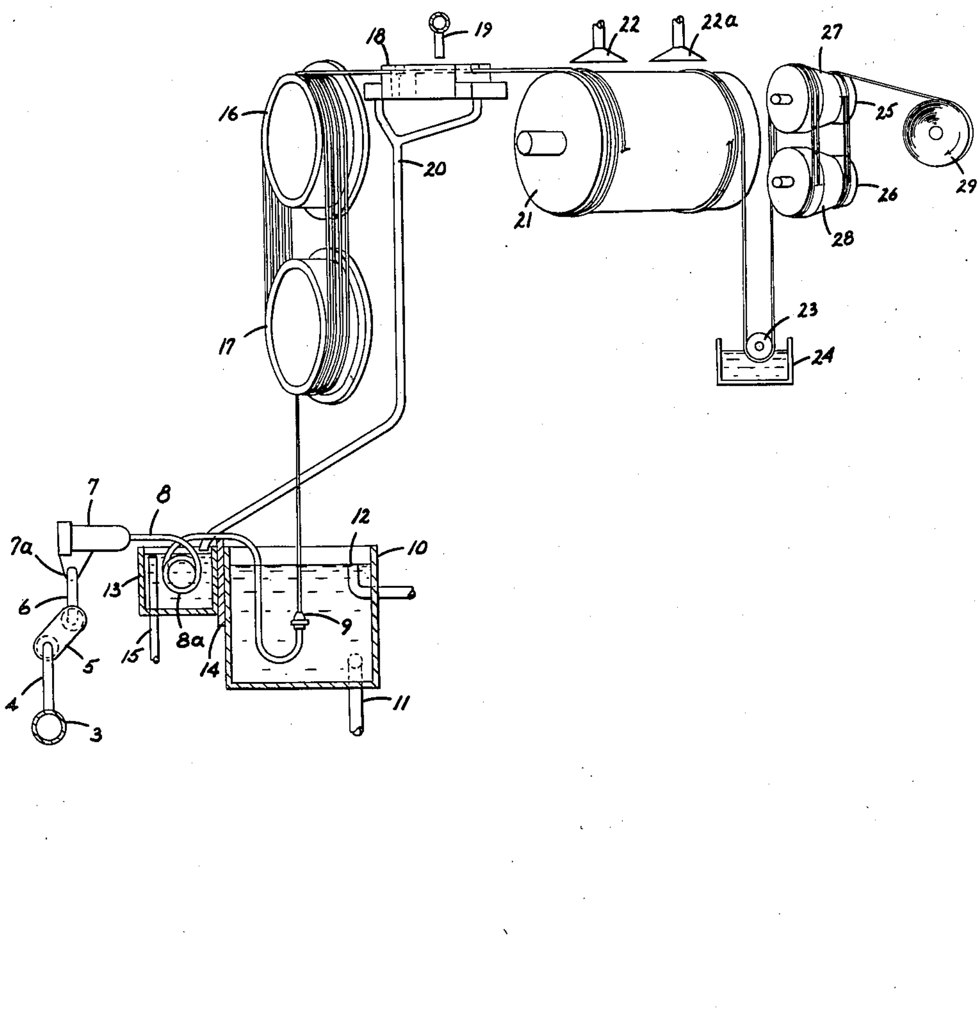

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METHOD FOR PRODUCING HIGH TENACITY ARTIFICIAL YARN AND CORD

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This invention relates to the production of high tenacity artificial filamentary yarns and cord from regenerated cellulose by the viscose process, and has as its object an improved process adapted to produce yarns and cords having increased flexing life.

In accordance with the present invention, it has been found that improved regenerated cellulose yarns and cords having high tenacity and long flexing life can be obtained by spinning a viscose having a temperature of at least 30° C. into a regenerating coagulating bath having a temperature of 40 to 75° C. or higher, stretching the partially regenerated filaments between 40 and 80%, washing out the acid while completing the regeneration of the filaments, relaxing, drying, and collecting by winding and/or twisting.

The viscose to be spun proceeds from the ripening and filtering equipment which is usually disposed in a separate room, commonly in a basement where a low room temperature from 18 to 22° C. may be maintained. As such viscose proceeds through the lines to the spinning machine in the spinning rooms, it may absorb some heat and rise to a temperature of 25 to 28° C. by the time it reaches the spinnerets of the machine. In order to raise the temperature into the range of at least 30° C., means is provided for heating a portion of the line through which the viscose passes on its way to the spinnerets. While the heating means could be applied to a common system for feeding a number of machines in the spinning room, or to the header associated with each spinning machine, it is preferred for reasons stated hereinafter to provide a system of heating the individual supply lines leading to the spinnerets. For example, electric heating coils, high frequency electrostatic heating devices, or heating baths may be applied to the viscose lines. The individual supply line or rounder may comprise a tortuous channel made by bending a portion of its length into the form of a helical coil, and this rounder may be submerged in the spinning bath to derive its heat from such bath or it may be submerged in some other liquid bath having a sufficiently high temperature to heat the viscose to the proper temperature. When a hot aqueous liquid (at 60 to 100° C.) is used for treating the yarns before, after, or during stretching, the hot liquid can be circulated through the yarn-treating stage and the viscose-heating stage in succession in either order.

It has been found that it is necessary not only to have the viscose at a proper temperature but also to relax the filaments in an amount of the

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order of 3 to 10% after stretching and before or during drying in order to obtain a combination of a high tenacity and exceptionally long flexing life.

Before drying the yarn and after it has been rendered acid-free, a soft finish may be obtained by the application of sodium oleate, polyvinyl alcohol, ethylene oxide, modified sorbitan dipalmitate, or compositions containing such ingredients in suitable proportions. If desired, desulfurization, bleaching, and such procedures may be applied with appropriate intermediate washes. Such treatments should precede the application of a softener when such softener is desired to be applied to the product. Depending upon the use to which the yarn obtained is intended to be put, it may be collected with or without twist. When it is desired to produce tire cord from such yarn, it is generally preferred to collect the individual yarns with a predetermined amount of twist so that a subsequent doubling operation may serve to combine the desired number of individual plies in the final cord with the proper twist in the plies and the cord respectively.

In the following examples, the relative fatigue rating or flexing life is determined on the flexing machine of the U. S. Rubber Company, in which a number of the cords are assembled in parallel relationship into a ribbon or band 30 inches long and the ribbon thus obtained is secured at the opposite end within two jaws, the upper of which is reciprocated ½ inch from the initial setting with the bands taut at a frequency of 500 per minute. A tension device on the lower jaw takes up any slack developed because of permanent elongation during the test. This flexing is carried out at a temperature of about 120° F.

The viscose employed may have from 6 to 8% cellulose, (which may be wood, cotton, linters, or any mixture thereof, preferably from 50 to 100% cotton) 6 to 8% caustic, and 30 to 40% carbon disulfide based on the cellulose in the viscose. The salt test may be from 3 to 6 and the ball fall viscosity before heating may be from 40 to 200 seconds.

The spinning bath may contain from 7 to 12% sulfuric acid, 15 to 25% sodium sulfate, and from 1 to 10% of the sulfate of zinc, magnesium, or iron, or a mixture of the latter sulfates.

The viscose preferably should have a temperature of 45° C. to 60° C. and the bath a temperature of 50 to 65° C. In the preferred instance the difference in temperature between the viscose and spinning bath is not over 10° C. and the optimum difference is about 0° C. to 5° C.

The drawing illustrates a preferred embodiment of equipment in accordance with the present invention. As shown, the single figure represents a schematic view in perspective with parts in section.

As shown, the viscose is fed to the machine through a header **3** to which there are connected a number of branch outlets **4** connected to the individual spinning pumps **5** of positive displacement type, such as ordinary gear pumps. The discharge lines **6** from the individual pumps **5** lead to a candle filter **7** through a swivel joint **7**_a_ which allows swinging of the candle filter **7** and rounder **8** in a counterclockwise direction as viewed in the figure, to remove the spinneret **9** out of the coagulating bath contained within the receptacle **10**, which may be a trough extending the length of the machine or individual receptacles, one for each spinneret. A feed pipe **11** serves to supply the coagulating bath to the receptacle **10** and an overflow pipe **12** displaced from **11** longitudinally of the machine serves to control the level of the bath in the trough **10** and assures circulation of the bath past the spinneret or spinnerets **9**.

A receptacle **13** is provided alongside receptacle **10** and may be insulated therefrom by a layer **14** of material of low heat-conductivity. Like **10**, the receptacle **13** may be individual for each rounder or it may be a trough running the length of the machine. An overflow pipe **15** is provided for controlling the level of heated liquid in the container **13**. The rounder **8** is formed with a portion **8**_a_ thereof in the form of a helical coil to provide for heat transfer from the bath in container **13** to the viscose in the rounder **8** before it is fed to the spinneret **9**.

Filaments passing from the coagulating bath in the receptacle **10** proceed upwardly to a thread-advancing device which may comprise a pair of canted rotors **16** and **17** about which a predetermined number of wraps of the yarn are wound to provide further regeneration prior to stretching. The yarn leaves the upper rotor **16** and passes through container **18** for a hot aqueous liquid continuously supplied thereto from a pipe **19**. The aqueous medium may be hot water, hot dilute acids or salts, and may for example contain from 1 to 5% sulfuric acid, 1 to 3% sodium sulfate, and $\frac{1}{10}$ to 1½% of zinc sulfate. As shown, the overflow pipe **20** in the container **18** discharges into the container **13** for heating the viscose in the tortuous channel **8**_a_. A thread-advancing reel or equivalent thread-advancing device **21** rotates at a peripheral speed from 40 to 80% higher than the peripheral speed of rotors **16** and **17**, to provide the stretch needed at this stage. On the reel **21**, a washing liquid, such as cold or hot water, cold or hot dilute acid or salt solutions, such as a solution containing from 0.1 to 0.5% sulfuric acid, is supplied from a spray or shower head **22**, and additional wash water may be sprayed on the reel by spraying head **22**_a_, and the yarn departing from reel **21** passes under a pulley or rotor **23** immersed in a bath of a soft finish in a container **24** and then it proceeds around a thread-advancing device for drying comprising a pair of rotors **25** and **26** which are driven at a peripheral speed at the yarn-receiving portion somewhat less than the peripheral speed of discharge from reel **21**. The surfaces of the rotors **25** and **26** may have intermediate tapered portions **27** and **28** respectively to allow further shrinkage of the thread during the drying as it reaches this portion of the device. This taper may amount to 3 to 10%. If all of the shrinkage is carried out on the tapered portion of the rotors **25** and **26**, their peripheral speed at the yarn-receiving end may be the same as the peripheral speed of the reel **21** and all shrinkage may be performed on the drums. On the other hand, the differential in the speed between the drums **25**, **26** and reel **21** may be sufficient to take care of all the shrinkage desired, in which case the drums **25** and **26** may be cylindrical throughout their length. Generally, it is preferred to have a portion of the shrinkage occur before the drying drums **25** and **26** are reached, and a portion of the shrinkage occurs on an intermediate portion of the dryer drum where the moisture content has been reduced to about 150% based on the weight of the yarn.

After the yarn departs from the drums or otherwise heated rollers **25** and **26**, it is collected by winding or by winding and twisting at **29**.

Example I

A viscose containing 7.8% cellulose, 6.6% caustic, 36% carbon disulfide (CS_2 based on the alpha cellulose content of the pulp) and having a salt point of 5 as determined with sodium chloride was spun into a bath containing 10% sulfuric acid, 4.5% zinc sulfate and 21% sodium sulfate at 50° C. to produce a 1650 denier, 980 filament yarn. The rounder comprised a 7-foot long stainless steel helical coil immersed in hot water at 60° C. The viscose was heated to 45° C. and the immersion travel of the filaments in the bath was 12 inches at a spinning speed of 107 meters per minute. A 16-yard storage was used on the rotors **16** and **17** of the drawing and immediately thereafter the yarn was stretched 65%. Hot water at about 90° C. was used in trough **18**. After washing to a neutral condition, the yarn passed through a soft finish and was allowed to relax a total of 8%, about 5% on the way to the drying stage and about 3% on a tapered portion of the drying drums. The yarn produced had a dry tenacity of 3.63 grams per denier, a dry elongation of 11.9%, a wet tenacity of 2.28 grams per denier, a wet elongation of 22.0% and a fatigue rating of 135.

The same viscose spun into the same bath and under the same conditions, except that the viscose was not heated but had a temperature of 27° C. as it issued from the spinneret, yielded a yarn having a dry tenacity of 3.65 grams per denier, a dry elongation of 10.8%, a wet tenacity of 2.21 grams per denier, a wet elongation of 20.7, and a fatigue rating of 114. When this same procedure is carried out without relaxation and without heating of the viscose, a fatigue rating of 47 to 60 is generally obtained.

Example II

Viscose was spun under the same conditions as in Example I and was heated to a temperature of 45° C., except that the storage on the rotors **16** and **17** was reduced to 2 yards. The resulting yarn had a dry tenacity of 3.43 grams per denier, a dry elongation of 12.8%, a wet tenacity of 2.21, a wet elongation of 20.5, and a fatigue rating of 180. This shows the great improvement obtainable by stretching filaments in a less completely regenerated condition.

The following table lists additional examples using the same conditions as Example II except the shrinkage or relaxation on the way to the drying stage was reduced to 4% and the temperature was varied as indicated.

Example	Viscose Temp., ° C.	Denier	Dry Tensile, g./d.	Dry Ext., Percent	Wet Tensile, g./d.	Wet Ext., Percent	Fatigue Rating
III	30	1,659	3.57	12.5	2.30	21.6	137
IV	40	1,668	3.63	13.5	2.32	22.2	154
V	50	1,666	3.70	13.6	2.33	23.0	199
VI	60	1,671	3.63	13.4	2.26	23.2	187
VII	70	1,671	3.69	13.6	2.30	22.5	183

A comparison of the results shown in the preceding illustrative examples shows the exceptionally good flexing life and how this has been increased by the present invention without any sacrifice in the tenacity of the yarn. In general, it has been found that the heating of the viscose just before spinning gets rid of so-called "native" cellulose, so that a more uniform filament is obtained. Apparently this reduction or complete elimination of native cellulose renders the substance of the filament more amenable to the subsequent processes of stretching and relaxation so that the final product has remarkably improved flexing life without any decrease in tenacity. The reason for such improvement is not clearly understood, but it is clear that stretching should be performed on the filaments while still undergoing regeneration, and preferably before the regeneration has been ¼ to ½ completed.

The product of the present invention can be used for the production of filament yarns or staple fiber yarns where high tenacity regenerated cellulose filaments or fibers are desired. It is particularly advantageous in the production of cords used for reinforcing rubber goods, such as tires, belts, and the like.

It is to be understood that changes and variations may be made without departing from the spirit and scope of the present invention, as defined in the appended claims.

We claim:

1. A process of producing high tenacity regenerated cellulose filaments having a long flex-life comprising the steps of extruding viscose having a temperature of 30° C. to about 70° C. into an acid regenerating and coagulating bath having a temperature of at least 40° C., stretching the filaments 40 to 80% after partial regeneration but before complete regeneration, washing the filaments and completing the regeneration, relaxing the filaments 3 to 10%, and drying them.

2. The process of claim 1 in which the temperatures of the viscose and bath are no more than 10° C. apart.

3. The process of claim 1 in which the temperatures of the viscose and bath are no more than 5° C. apart.

4. The process of claim 1 in which part of the relaxation is effected immediately before drying and the remainder during drying.

5. A process in accordance with claim 1 in which the filaments are subjected to a hot aqueous liquid having a temperature of 60° C. to 100° C. during stretching.

6. A process in accordance with claim 5 in which the filaments are stretched approximately 65%.

7. A process in accordance with claim 5 in which the hot aqueous liquid is applied to heat the viscose passing to the spinneret.

8. A process of producing high tenacity regenerated cellulose filaments having a long flex-life comprising the steps of extruding viscose having a temperature of 45 to 60° C. into an acid regenerating and coagulating bath having a temperature of 40° to 75° C., stretching the filaments 40 to 80% after partial regeneration but before complete regeneration, washing the filaments and completing the regeneration, relaxing the filaments 3 to 10%, and drying them.

9. A process of producing high tenacity regenerated cellulose filaments having a long flex-life comprising the steps of extruding viscose having a temperature of 30° C. to about 70° C. into an acid regenerating and coagulating bath having a temperature of at least 40° C., stretching the filaments 40 to 80% after partial regeneration but before regeneration is half completed, washing the filaments and completing the regeneration, relaxing the filaments 3 to 10%, and drying them.

10. A process of producing high tenacity regenerated cellulose filaments having a long flex-life comprising the steps of extruding viscose having a temperature of 45 to 60° C. into an acid regenerating and coagulating bath having a temperature of 40° to 75° C., stretching the filaments 40 to 80% after partial regeneration but before regeneration is half completed, washing the filaments and completing the regeneration, relaxing the filaments 3 to 10%, and drying them.

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