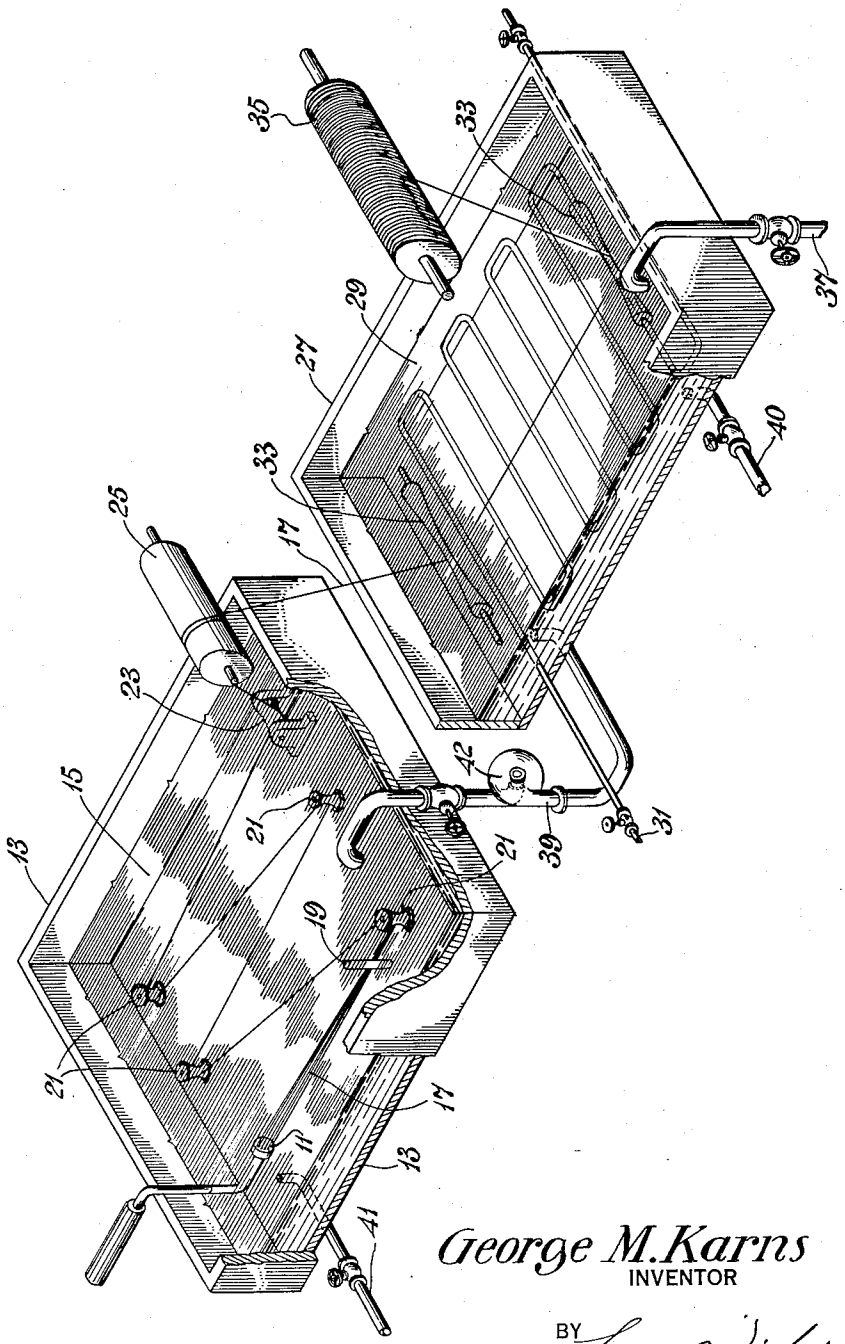
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PRODUCTION OF REGENERATED CELLULOSE THREADS

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PRODUCTION OF REGENERATED CELLULOSE THREADS

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This invention relates to an improved process for spinning high tenacity rayon yarn by the viscose process. It relates more particularly to an improvement in that type of process wherein the freshly precipitated regenerated cellulose yarn is subjected to a stretching operation while in contact with a liquid having an elevated temperature.

In recent years it has been shown that the tenacity of regenerated cellulose filaments and yarns can be greatly increased by stretching, or extending, the freshly precipitated filaments and yarns while contacting them with liquid having an elevated temperature.

United States Letters Patent No. 1,901,007 discloses the process for the production of high tenacity (dry tenacity of 2.85 grams per denier) regenerated cellulose fibers, which process comprises the passing of freshly produced acid-moist fiber bunches through a trough having a length of 2 to 12 meters, preferably 5 meters, which trough is filled with water kept at a temperature of about 70° to 100° C., and simultaneously stretching the fibers by subjecting them to a high tension of, for instance, 0.5 to 0.8 kilograms per square millimeter. The speed with which the fiber bunch is guided through the bath may vary between 40 to 60 meters per minute depending upon the length of the water bath.

United States Letters Patent No. 1,996,989 discloses a similar process for the production of high tenacity regenerated cellulose filaments and yarns. The process of this patent comprises the drawing of the filament bundle immediately after it passes out of the acid precipitating liquid through a hot, flowing water bath having an elevated temperature, for example 70° C., and simultaneously subjecting the filaments to strong tension. It is essential in accordance with the process of this patent to remove the acids, salts and coagulation products of decomposition from the filaments directly after the precipitation thereof.

It is an object of the present invention to provide an improved process for the production of high tenacity regenerated cellulose rayon from viscose.

It is a further object of the present invention to provide a process for the production of high tenacity regenerated cellulose rayon from viscose, which process represents an improvement in efficiency and decreased filament degradation over previously known processes.

Other objects of the invention will appear hereinafter.

In accordance with the present invention, the filaments or yarns, which are formed by the extrusion of a viscose solution into a coagulating and regenerating bath, after substantially complete regeneration, are stretched while passing through a hot (60° C. to boiling temperature) sulfuric acid bath for a distance not to exceed 45 inches based on spinning speeds of at least 3000 and up to 8000 or more inches per minute.

It has been discovered that very material advances in economy and material reduction in yarn degradation will be obtained if the freshly precipitated yarns and filaments are stretched in a hot sulfuric acid bath, and if the contact with said hot sulfuric acid bath be confined to a distance of 45 inches or less, based on spinning speeds of 3000 to 8000 inches per minute.

On the basis of spinning speeds of the order of 3000 to 8000 inches per minute the 45-inch maximum contact of the yarns or filaments with hot sulfuric acid during the stretching operation is critical, from a standpoint of yarn degradation, when compared with the prior art processes in which it was considered necessary to contact the threads in hot liquids for a distance of 2 to 12 meters (78 to 460 inches) at speeds considerably less than 3000 inches per minute.

The procedure and apparatus by means of which the process of the present invention may be carried out will be clearly apparent from the following description when taken in connection with the accompanying illustration.

The accompanying illustration is a diagrammatic perspective view of one form of apparatus suitable for use in accordance with the present invention.

Referring to the illustration, reference numeral 11 designated a spinneret which is positioned in tank 13 containing a coagulating and regenerating bath 15. A viscose solution is extruded through spinneret 11 into bath 15 for the formation of a multifilament yarn 17. The yarn 17 is passed about pin 19 which functions as a convergence guide to bring the several extruded filaments together into a single yarn. The yarn is then passed about a plurality of roller guides 21, thence around guide 23. After passing around guide 23 the yarn is drawn from the coagulating and regenerating bath by means of transfer roller 25. The yarn may be looped one or more times about the transfer roller 25 to prevent slippage of the yarn thereon. The yarn is then passed around rollers 33 which are submerged in a hot sulfuric acid bath 29 in tank 27. The yarn is drawn from the hot sulfuric

acid bath 29 and passed onto a collecting bobbin 35. The hot acid bath 29 in tank 27 is maintained at the proper elevated temperature, 60° C. to boiling temperature, in any desired manner, for example, by means of steam coils 31 positioned in the bottom of the tank. The composition of the acid bath 29 may be maintained substantially constant by the continuous, or periodic addition of a replenishing solution of predetermined composition through conduit 37. A portion of the hot acid bath from tank 27 may be continuously or periodically pumped into the coagulating and regenerating bath 15 by means of pump 42 and conduit 39, or the hot acid bath may be drained through conduit 40. Spent coagulating and regenerating liquid may be passed from the coagulating tank 13 through conduit 41.

It is to be understood that the illustrated and above-described apparatus is disclosed for illustrative purposes only. Obviously various different forms and modifications of apparatus can be employed equally well.

The number and type of roller guides 21 in the coagulating and regenerating bath will be determined by the spinning speed, by the size of the threads or filaments being spun, by the tension to be imposed on the yarn, and by the composition of the coagulating and regenerating bath liquid. It is necessary that a sufficiently long regenerating bath travel for the yarn be provided to substantially complete the regeneration of the yarn to regenerated cellulose. If the regenerating bath tank is comparatively short, sufficient roller guides must be provided to accomplish this result. The transfer roller 25 may be made of such size and provided with such a surface that the thread or yarn need not be looped around the same to prevent slippage. On the other hand, the transfer roller may comprise a pair of cooperating squeeze rollers or a pair of spaced rollers about which the yarn is repeatedly passed to prevent yarn slippage and arranged so as to prevent the passing threads from overlapping and chafing each other. The transfer roller may be rotated by the pull exerted thereon by the yarn, or the roller may be positively driven in any desired manner. The collecting bobbin may be of any type well known in the art. The collecting bobbin will, of course, be provided with necessary traverse guides in a manner well known in the art. If desired, the yarn may be drawn through the hot acid bath 29 by means of a feed roll or the like and collected in a rotating bucket in accordance with the conventional bucket process for the production of rayon yarn. In any event, the collecting bobbin, or the feed roller when using the bucket process, will be rotated at a peripheral speed sufficiently higher than the peripheral speed of transfer roller 29 to impart a stretch of at least 10% to the yarn as it passes through the hot acid bath 29. The stretch may be 25%, 50% or 100% or more, depending upon the physical characteristics to be imparted to the yarn and how much stretch the yarn will stand without breaking.

The spent coagulating and regenerating liquid may be discarded, or it may be renovated, as desired.

For convenience the invention will be described in terms of a spinning process as depicted in the drawing where the yarn is collected on a spin-bobbin.

To avoid confusion the term "spinning speed" will be understood to refer to the peripheral velocity of the spin-bobbin in the bobbin process or the feed wheel speed in the bucket process, and the term "spinning tension" is the tension on the yarn, measured at a point just ahead of the spin-bobbin or its equivalent, when the spinning process is in actual operation.

To further illustrate the principles of the invention, the following examples are given, but it is to be understood that the invention is not necessarily limited thereby. In all of these examples viscose prepared from cotton linters and containing approximately 7% cellulose and 6% sodium hydroxide is used. Obviously viscose having different characteristics can be employed in the practice of the invention depending upon the type of yarn to be produced. Particularly good results have been obtained by the use of a viscose spinning solution which has been ripened to a point where it has an index materially greater than 4.0, for example an index of 4.5 to 6.0, or even greater, as determined by the method of Reinthaler and Rowe, "Artificial silk," 1928, page 69.

Example I

Viscose is passed through a spinneret containing 200 holes, with an average viscose delivery of 37.9 grams per minute, into an aqueous spinning bath containing 9% sulfuric acid, 19% sodium sulfate, 4% glucose and 0.85% zinc sulfate, said bath being maintained at 45°±1° C. The extruded filaments are guided through this bath by a plurality of roller guides selected to impose tension on the yarn during a bath travel of 200 inches, so that as the yarn is withdrawn from the bath over a positively driven transfer roll having a peripheral velocity of 3000 inches per minute, the tension will be 108 grams. From the transfer roller the yarn is led into a secondary bath, which consists of an aqueous solution containing 2% sulfuric acid, maintained at 90°±2° C., through which it is guided by a plurality of roller guides for a bath travel of 22 inches, whence it is collected on a spin-bobbin at a spinning speed of 4100 inches per minute with a spin tension of 270 grams. The finished yarn, after being washed and dried on the bobbin in conventional fashion has a denier of about 275, a dry tenacity of 3.66 grams per denier, and a wet tenacity of 1.84 grams per denier with dry and wet elongations of 5.5% and 12.7% respectively.

Example II

Viscose is passed through a spinneret containing 800 holes, with an average viscose delivery of 169.2 grams per minute into aqueous spinning and secondary baths having the same compositions and temperatures, respectively, as described in Example I. In this case, however, the spinning bath travel is 200 inches while the secondary bath travel is 11 inches and the roller guides in the two baths are selected so that at a spinning speed of 4500 inches per minute the spin tension will be 1100 grams when the yarn driven transfer roller runs at a peripheral speed of about 3550 inches per minute. The finished yarn, after washing and drying on the bobbin in conventional fashion and after twisting to 3 turns per inch, has a denier of about 1100, a dry tenacity of 3.7 grams per denier and a wet tenacity of 2.01 grams per denier with dry and wet elongations of 6.1% and 12.5% respectively.

Example III

Viscose is passed through a spinneret containing 120 holes, with an average viscose delivery

of 34.3 grams per minute, into an aqueous spinning bath containing 9% sulfuric acid, 19% sodium sulfate and 0.85% zinc sulfate at a temperature of 45°±1° C. The extruded filaments are guided through the spinning bath by a plurality of roller guides selected to impose some tension on the yarn during a bath travel of 130 inches. The yarn is then led out of the spinning bath and over a freely rotating transfer roller into a secondary bath of composition identical with that of the spinning bath, the secondary bath being maintained at 95°±2° C. The yarn is guided through the secondary bath by a plurality of roller guides selected to impose some tension on the yarn while allowing a bath travel of 11 inches, whereupon the yarn is led to the spin-bobbin. The roller guides are selected so that at a spinning speed of 3600 inches per minute the spin tension will be 265 grams when the peripheral speed of the transfer roller is 2960 inches per minute. The finished yarn, washed on the bobbin and twister dried in the manner described in U. S. Patent No. 2,130,247, has a denier of 275, a dry tenacity of 3.35 grams per denier and a wet tenacity of 2.03 grams per denier with dry and wet elongations of 7.1% and 11.9% respectively.

Example IV

A yarn spun according to Example III except for the use of spinning and secondary baths containing 11% sulfuric acid, 26% sodium sulfate and 0.85% zinc sulfate has a denier of 275, a dry tenacity of 3.44 grams per denier and a wet tenacity of 2.06 grams per denier with dry and wet elongations of 7.4% and 11.7% respectively.

The following example illustrates the application of the invention to a bucket spinning process. In this example the apparatus as depicted in Figure 1 can be employed if the spin-bobbin 35 is replaced by a conventional feed wheel and bucket collecting apparatus.

Example V

Viscose is passed through a spinneret containing 120 holes, with an average viscose delivery of 31.2 grams per minute, into an aqueous spinning bath containing 11% sulfuric acid, 23% sodium sulfate, 4% glucose and 0.7% zinc sulfate, said bath being maintained at 45°±1° C. The extruded filaments are guided through the bath by a plurality of roller guides selected to impose a tension on the yarn, during a bath travel of 130 inches. The yarn is then led over a freely rotating transfer roller into a secondary bath containing 2% sulfuric acid, which bath is maintained at 92°±2° C. The bath travel in said secondary bath is 20 inches. From the secondary bath the yarn is led to a positively driven feed wheel, from which it is passed into a centrifugal collecting bucket rotating at 10,000 R. P. M. The spinning speed (feed wheel speed) is 3600 inches per minute and the roller guides in the baths are selected so that a spin tension of 265 grams is obtained when the transfer roll runs at a peripheral speed of 2710 inches per minute. The finished yarn, washed and dried in the cake without bleaching, has a denier of 275, a dry tenacity of 3.08 grams per denier, and a wet tenacity of 1.86 grams per denier with dry and wet elongations of 13.9% and 16.5% respectively.

By reference to the above examples, it will be seen that yarn of exceptionally high tenacity can be produced with great ease even though very high spinning speeds are used. The quality of the yarn is vastly superior to similar yarn produced in prior art processes employing slower speeds and long stretching bath travel. These improvements are made possible by passing the yarn through a secondary heated bath, allowing the yarn to remain in the secondary bath only long enough (less than 45 inches) to bring the yarn and its adherent spinning bath to the temperature of the secondary bath and stretching the yarn while it is passing through the hot bath. The amount of stretch given the yarn affects in large measure the tenacity of the final product; however, the stretch must be imposed on the yarn during a comparatively short travel in the stretching bath so as to prevent yarn degradation. If material yarn degradation takes place the tenacity of the yarn will be substantially lowered.

The composition of the hot acid stretching bath may be the same as that of the coagulating and regenerating bath with the attendant advantages of simplified bath make-up systems, use of hot bath overflow to maintain spinning bath temperature and composition, economical use ultimately as spinning bath, and the like. However, if high salt concentrations are used, the tendency toward crystallization in the hot acid stretching bath is increased so that it is frequently preferable to use a different hot bath composition. To this end then, compositions containing 2% to 13% sulfuric acid, 0 to 30% sodium sulfate and with or without zinc sulfate or glucose may be used. Inasmuch as the higher temperature of the secondary bath is conducive to carbonization of glucose, a glucose-free bath may be preferred.

The temperature of the spinning bath is usually selected in the range of 40°–50° C., preferably 45°–50° C., as generally accepted in the art. The temperature of the secondary bath is selected in the range of 60° C. to boiling temperature, and usually 90°–100° C. This preferred range generally yields higher breaking tensions, that is, the tension to which a thread can be subjected before breaking. For example, using a spinning bath of composition and temperature set forth in Example I and a spinning bath travel of 130 inches, if the hot bath is at 60° C., the breaking tension is about 1 gram per denier, while it is about 1.15 grams per denier at 80° C., and 1.2 grams per denier at 90° C.

By following the process of the instant invention it is possible to spin high tenacity yarns of very fine filament denier and particularly to spin such fine denier yarns at economical spinning speeds. The reason for this lies in the fact that the yarn denier can be reduced by a stretching operation in which much greater stretch can be secured than when stretching in the spinning bath is relied upon. Thus, for example, it is practical to spin a yarn of 0.5 to 1 denier per filament at high speed while still keeping the yarn speed in the spinning bath low enough to prevent entrance of the bath into the spinneret, which will result in plugged spinneret holes, and while using a spinneret of larger hole size than has been possible heretofore in the prior art processes. The spinning of such very fine denier yarns from viscose is described with greater particularity in the copending application of Tippetts, Serial No. 278,426 filed of even date herewith.

The invention is particularly useful if high tenacity yarns destined for conversion into twisted cords of high strength and quality, such as are

used in rubber tire constructions and the like, are to be made. In such instances, it is preferable to employ spinning baths containing relatively high proportions of salt. Thus a bath containing 9% sulfuric acid should have 23% or more of sodium sulfate while a bath containing 11% sulfuric acid should have at least 25% sodium sulfate present. To such baths an addition of about 0.85% zinc sulfate is preferable while small amounts of glucose may be used if desired, as previously described. It has been found that a bath containing 11% sulfuric acid, 26% sodium sulfate and 0.85% zinc sulfate can be used satisfactorily as both spinning bath and secondary hot bath for the production of yarns suitable for tire cord manufacture.

Since it is obvious that many changes and modifications can be made in the details of the invention above described, it is to be understood that the invention is not to be limited except as set forth in the appended claims.

I claim:

1. In a process for the spinning of regenerated cellulose yarns and filaments, the steps comprising extruding a viscose solution into a coagulating and regenerating bath of the dilute acid type (of the order of 9% to 11% acid) to form said yarns and filaments, removing said yarns and filaments from said bath and passing them for a distance not to exceed 45 inches, based on a spinning speed of at least 3000 inches per minute, through a 2% to 13% sulfuric acid bath having a temperature of 60° C. to boiling temperature, and stretching said yarns and filaments at least 10% during their passage through said hot acid bath.

2. In a process for the spinning of regenerated cellulose yarns and filaments, the steps comprising extruding a viscose solution into a coagulating and regenerating bath of the dilute acid type (of the order of 9% to 11% acid) to form said yarns and filaments, maintaining said yarn in contact with said coagulating and regenerating bath for a sufficiently long period to substantially completely regenerate the same, removing said yarns and filaments from said bath and passing them for a distance not to exceed 45 inches, based on a spinning speed of 3000 to 8000 inches per minute, through a 2% to 13% sulfuric acid bath having a temperature of 60° C. to boiling temperature, and stretching said yarns and filaments at least 10% during their passage through said hot acid bath.

3. In a process for the spinning of regenerated cellulose yarns and filaments, the steps comprising extruding a viscose solution into a coagulating and regenerating bath of the dilute acid type (of the order of 9% to 11% acid) to form said yarns and filaments, maintaining said yarn in contact with said coagulating and regenerating bath for a sufficiently long period to substantially completely regenerate the same, removing said yarns and filaments from said bath and passing them for a distance not to exceed 45 inches, based on a spinning speed of 3000 to 8000 inches per minute, through a 2% to 13% sulfuric acid bath having a temperature of 90° C. to boiling temperature, and stretching said yarns and filaments at least 10% during their passage through said hot acid bath.

4. In a process for the spinning of regenerated cellulose yarns and filaments, the steps comprising extruding a viscose solution into a coagulating and regenerating bath of the dilute sulfuric acid type (of the order of 9% to 11% acid) to form said yarns and filaments, maintaining said yarn in contact with said coagulating and regenerating bath for a sufficiently long period to substantially completely regenerate the same, removing said yarns and filaments from said bath and passing them for a distance not to exceed 45 inches, based on a spinning speed of 3000 to 8000 inches per minute, through a sulfuric acid bath of a composition essentially the same as said coagulating and regenerating bath and having a temperature of 60° C. to boiling temperature, and stretching said yarns and filaments at least 10% during their passage through said hot acid bath.

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