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PROCESS FOR PRODUCING CRIMPABLE REGENERATED CELLULOSE FILAMENTS

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This invention relates to the production of regenerated cellulose filaments, fibers, yarns and the like that are capable of being crimped. More particularly, it relates to the production of heavy denier, crimpable structures of regenerated cellulose, which can be crimped in water and, when crimped, display outstanding durability.

Heavy denier fibers and filaments (10 denier per filament and higher), as crimped, are desirable as a replacement for wool in the manufacture of rugs, carpets and upholstery. Prior art processes for preparing heavy denier crampable filaments have not been successful for several reasons. The filaments, when crimped in water, often did not provide a satisfactory crimp level. Where a satisfactory crimp level was obtained, the durability, as reflected by the flex life, was unsatisfactorily low.

The object of this invention is a process for producing heavy denier regenerated cellulose yarns, filaments and fibers that are highly crimpable. A further object is a process for producing such heavy denier regenerated cellulose structures that are highly crimpable in water. Another object is to improve the flex life of these crimpable heavy denier structures without sacrificing crimp level. A further object is to provide a process that will operate at high speeds, i.e., 100 yards per minute or more. Other objects will appear hereinafter.

The objects are accomplished by a process wherein a viscose solution having an alkali content of 5–7%, a cellulose content of 6–7.5%, and a salt index of 3–6, preferably heated to a temperature of 40–80° C., is extruded through the orifices of a spinneret into a coagulating and regenerating bath having a sulfuric acid content of 6.5–9.5%, preferably 8–9%, a sodium sulfate content of 15–30%, preferably 20–25%, a zinc sulfate content of 1.0–2.5% and a temperature between 40 and 60° C. to form filaments, imposing a tension of less than 0.30 gram per denier, preferably less than .25 gram per denier, on said filaments in the coagulating and regenerating bath, washing said filaments without applying any substantial stretch, stretching said filaments 7 to 20% in an aqueous solution of a lubricant, said solution preferably maintained at a temperature of 60 to 100° C. and drying said filaments while retaining the aforementioned stretch.

The yarns produced by the above process are then completely relaxed by suspending them freely in an aqueous liquid bath in the substantial absence of tension to impart crimp. The bath may be a caustic solution or, surprisingly, plain water. When plain water is used, it is preferred that the temperature be raised to 60–100° C. The crimped yarns of 10 denier per filament and higher display as many as 15 crimps per inch or higher and flex lives of over 1500 cycles as measured on a modified Masland flex tester.[1]

It is noteworthy that this process provides a high degree of crimp in heavy denier yarns contrary to statements in U.S. Patent Nos. 2,517,694 and 2,572,936 that "above about 10 deniers per filament crimping is negligible." It is also interesting to note that contrary to U.S. Patent No. 2,515,834 where at least 0.5 gram per denier spinning tension must be imposed on the filaments and a stretch of at least 40% must be imposed during spinning and U.S. Patent No. 2,686,709 where tension approaching the breaking tension must be imposed during spinning, the present invention requires a low spinning tension (less than 0.3 gram per denier) and substantially no spinning stretch to provide heavy denier yarn of high crimpability and excellent flex life.

[1] Textile World 100, 260, 262 (February 1950).

The important aspects of the invention are illustrated in the following examples. In the examples parts are by weight unless otherwise stated. Crimps per inch and crimp shrinkage were measured by observing 10 inch lengths of yarn. Flex life was measured in the Masland flex tester by reciprocally flexing the yarn through an angle of 252° between a pair of wires 0.005 inch in diameter at 115 cycles per minute under a load of 0.05 gram per denier.

EXAMPLE I

A viscose spinning solution containing 7% cellulose and 6% alkali (calculated as sodium hydroxide) was ripened to a salt index of 3.5 (as described by Ott, High Polymers, volume 5, page 837). The solution, at about 20° C. was extruded through a 90 hole spinneret into a coagulating and regenerating bath at 54° C. which contained 8.4% sulfuric acid, 23.7% sodium sulfate, 1.5% zinc sulfate and 4.0% glucose. The filaments were converged into a yarn and passed around rollers in the bath to provide a bath travel of 200 inches. From the bath the yarn was passed over a feed wheel and into a rotating bucket where it was collected as a cake. The spinning speed was approximately 100 yards per minute and the denier of each of the filaments was 10 denier.

The cakes were purified and dried in the conventional manner. The yarns were then slashed which involved stretching in an aqueous solution containing 2% of the sodium salt of a sulfonated vegetable oil and 0.5% glycerin, followed by drying without relaxation. The yarn samples were then placed while substantially free of tension in water at 90° C. for 3 minutes and finally dried again.

In the table below, two yarns prepared according to the invention are compared to a third yarn prepared by a process outside the invention. This latter yarn was spun under a tension of 0.5 gram per denier compared to the yarns produced by the process of this invention which were spun under a tension of 0.21 gram per denier. Although the yarn prepared outside the invention developed adequate crimp, its flex life was less than ½ that of the yarns produced by the present invention.

Table I

Yarn No	1	2	3
Spin Tension (grams/denier)	0.21	0.21	0.5
Slasher Stretch (Percent)	10.2	15.0	9.0
Crimps/inch	10.4	15.0	10.5
Crimp Shrinkage (Percent)	32.0	35.0	25.0
Flex Life (Cycles)	2,600	1,700	800

EXAMPLE II

The yarns of this example were prepared in accordance with the general procedure described in Example I. Yarn Nos. 1 and 2 are those shown in Example I. Yarns 3 and 4 were prepared by applying low spinning tensions as required by the present invention but no slasher stretch was applied. Instead the finish solution was applied prior to the first drying step. The crimp developed when the

yarn was relaxed in water was much too low (yarn No. 3). When the yarn was relaxed in a 4% sodium hydroxide solution at 25° C. for three minutes (yarn No. 4), the crimp developed was higher but still too low for satisfactory use.

Table II

Yarn No.	1	2	3	4
Spinning Tension (grams/denier)	0.21	0.21	0.28	0.28
Slasher Stretch (Percent)	10.2	15.0	0	0
Crimping Solution	Water	Water	Water	NaOH
Crimps/inch	10.4	15.0	2.0	5.0
Crimp Shrinkage (Percent)	32.0	35.0	12.0	19.0

EXAMPLE III

In this example the yarn was stretched about 25% in a water bath at 60° C. immediately after leaving the spinning bath. The yarn was then wound on a bobbin, washed and dried in the conventional manner. The spinning tension was 0.25 gram per denier which is within the process of the present invention. The viscose solution, the spinning bath and the relaxing treatment to develop crimp were those described in Example I.

In the table below the yarn is compared to the yarns of the present invention. The results, especially the comparative flex lives, show the importance of stretching during the slashing operation rather than at any other stage of the process.

Table III

Yarn No.	1	2	3
Spinning Tension	0.21	0.21	0.25
Stretch immediately following spinning (Percent)	0	0	25
Slasher Stretch (Percent)	10.2	15.0	0
Crimps/inch	10.4	15.0	7-8
Flex Life (Cycles)	2,600	1,700	800

EXAMPLE IV

In this example, 18 denier per filament yarns were spun according to the procedure given in Example I. In one case the viscose was spun as it came from the ripening tank at 20° C. In the second case the viscose was heated to 44° C. by means of an oil-bath heat exchanger placed in the line just prior to the spinneret. The spinning tension imposed on both yarns was 0.25 gram per denier and a stretch of 15% was applied during the slashing operation. The resulting yarns were placed free of tension in water at 95° C. for 5 minutes. The results are given in the table below.

Table IV

Yarn No.	1	2
Temperature of Viscose (° C.)	20	44
Crimp Shrinkage (Percent)	33	47

As shown in the above examples there are two primary limitations that are critical in the process of this invention:

(1) The spinning tension must be less than 0.3 gram per denier.

(2) The yarn must be stretched 7 to 20% during the slashing operation.

It is also important that this stretch during slashing is not released during subsequent drying. In other words, the yarn must be dried at constant length.

The viscose spinning solutions for use in this invention may be prepared in the conventional manner by treating wood pulp or cotton linters pulp with a sodium hydroxide solution followed by xanthation with carbon disulfide and dilution with dilute sodium hydroxide solution or water. The only limitations on the viscose solution are those enumerated previously; the ultimate viscose solution must contain 5 to 7% alkali (sodium hydroxide) and 6 to 7.5% cellulose, have a salt index of 3 to 6, and preferably should be heated to 40 to 80° C. prior to spinning. Heating may be accomplished by a small oil-bath heat exchanger placed between the spinning pump and the spinneret as described in Example IV. Heating may also be accomplished by other suitable heating means such as steam, hot water or coagulating bath; or an electrical heating unit may be used.

The coagulating and regenerating bath contains the conventional ingredients. However the amounts used are important. A sulfuric acid content of 6.5 to 9.5%, a sodium sulfate content of 15 to 30%, a zinc sulfate content of 1.0 to 2.5% are vital. The bath may also contain other ingredients such as 3 to 4.5% glucose, etc. if desired.

The required low spinning tension is maintained upon the filaments by using rollers within the bath which offer little resistance to the filaments passing over them. Spinning tubes such as described in U. S. Patent No. 2,440,057 are particularly useful in maintaining spinning tension at the required low level.

The yarn when removed from the bath is seldom completely regenerated, i.e. some cellulose xanthate still remains unconverted to regenerated cellulose. In prior processes the yarn was stretched while in this condition. However, for the successful results of the present invention the yarn is not stretched at this point but is first completely regenerated, washed and dried. Then as completely regenerated cellulose yarn, it undergoes the slashing operation previously described wherein it is stretched from 7 to 20% in a lubricating solution and subsequently dried at constant length.

The slashing operation as shown in the examples involves passing the yarn through a solution containing a lubricant. A mixture of the sodium salt of a sulfonated vegetable oil and glycerin has been shown in the examples as the lubricant. However, other compounds such as alkali or amino soaps, sulfonated mineral oils, fatty alcohol sulfates, in general any of the compounds commonly used as lubricating finishes for yarns may be used as lubricants with equal success.

The process offers the advantages of simplicity and economy. The restrictions, although important, require no substantial alteration of the conventional viscose process. Spinning speeds of 25 yards per minute to 200 yards per minute may be used. It is noteworthy that the examples were performed at a speed of 100 yards per minute, a relatively high and economical speed in viscose rayon manufacture. A further advantage of the process lies in the level of spinning tension required. Low spinning tension serves to minimize the number of yarn breaks formerly encountered when the high spinning tensions of prior art processes for forming crimpable filaments were used.

The process makes it possible to produce heavy denier (deniers of 10 and higher) filaments capable of being crimped either as yarns or after being made into fabrics. Crimping is accomplished by suspending the yarns or fabrics made from the yarns substantially free of tension in an aqueous liquid bath. An advantage of the present invention is that the aqueous liquid bath can be water, preferably at a temperature of 60–100° C. Aqueous caustic may be used with no such limitation on the temperature (0–100° C.) but is not essential for obtaining a satisfactory crimp level combined with a high flex life in the ultimate yarn. The yarns made by the present invention are useful in manufacturing carpets, rugs and upholstery. They are also useful for other industrial applications such as artificial down or hair substitutes.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that the invention is not limited except as defined in the appended claims.

What is claimed is:

1. A process for producing crimpable regenerated cellulose filaments of excellent flex life comprising extrud-

ing through the orifices of a spinneret a viscose solution having an alkali content of 5–7%, a cellulose content of 6–7.5%, and a salt index of 3–6 into a coagulating and regenerating bath having a sulfuric acid content of 6.5–9.5%, a sodium sulfate content of 15–30%, a zinc sulfate content of 1.0–2.5% and a temperature of 40–60° C. to form filaments, imposing a tension of less than 0.3 gram per denier on said filaments in the coagulating and regenerating bath; washing said filaments without applying any substantial stretch to the filaments; stretching said filaments 7–20% in an aqueous lubricating solution; and drying said filaments while substantially retaining the aforementioned stretch.

2. A process as in claim 1 wherein the tension imposed on the filaments in the coagulating and regenerating bath is less than 0.25 gram per denier.

3. A process as in claim 1 wherein the viscose solution is heated to a temperature of 40–80° C. prior to extrusion.

4. A process as in claim 1 wherein the aqueous lubricating solution is at a temperature of 60 to 100° C.

5. A process for producing crimpable regenerated cellulose filaments of excellent flex life comprising extruding through the orifices of a spinneret a viscose solution having an alkali content of 5–7%, a cellulose content of 6–7.5%, and a salt index of 3–6 into a coagulating and regenerating bath having a sulfuric acid content of 6.5–9.5%, a sodium sulfate content of 15–30%, a zinc sulfate content of 1.0–2.5% and a temperature of 40–60° C. to form filaments, imposing a tension of less than 0.3 gram per denier on said filaments in the coagulating and regenerating bath; collecting said filaments at a speed of

at least 100 yards per minute; washing and drying said filaments without applying any substantial stretch on the filaments; passing said filaments through an aqueous lubricating solution; stretching said filaments 7–20% in said aqueous lubricating solution and drying said filaments while substantially retaining the aforementioned stretch.

6. A process for producing crimped regenerated cellulose filaments of excellent flex life comprising extruding through the orifices of a spinneret a viscose solution having an alkali content of 5–7%, a cellulose content of 6–7.5%, and a salt index of 3–6 into a coagulating and regenerating bath having a sulfuric acid content of 6.5–9.5%, a sodium sulfate content of 15–30%, a zinc sulfate content of 1.0–2.5% and a temperature of 40–60° C. to form filaments, imposing a tension of less than 0.3 gram per denier on said filaments in the coagulating and regenerating bath; washing said filaments without applying any substantial stretch to the filaments; stretching said filaments 7–20% in an aqueous lubricating solution; drying said filaments while substantially retaining the aforementioned stretch and suspending said filaments substantially free of tension in an aqueous liquid bath.

7. A process as in claim 6 wherein the aqueous liquid bath is water at a temperature of 60° to 100° C.

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