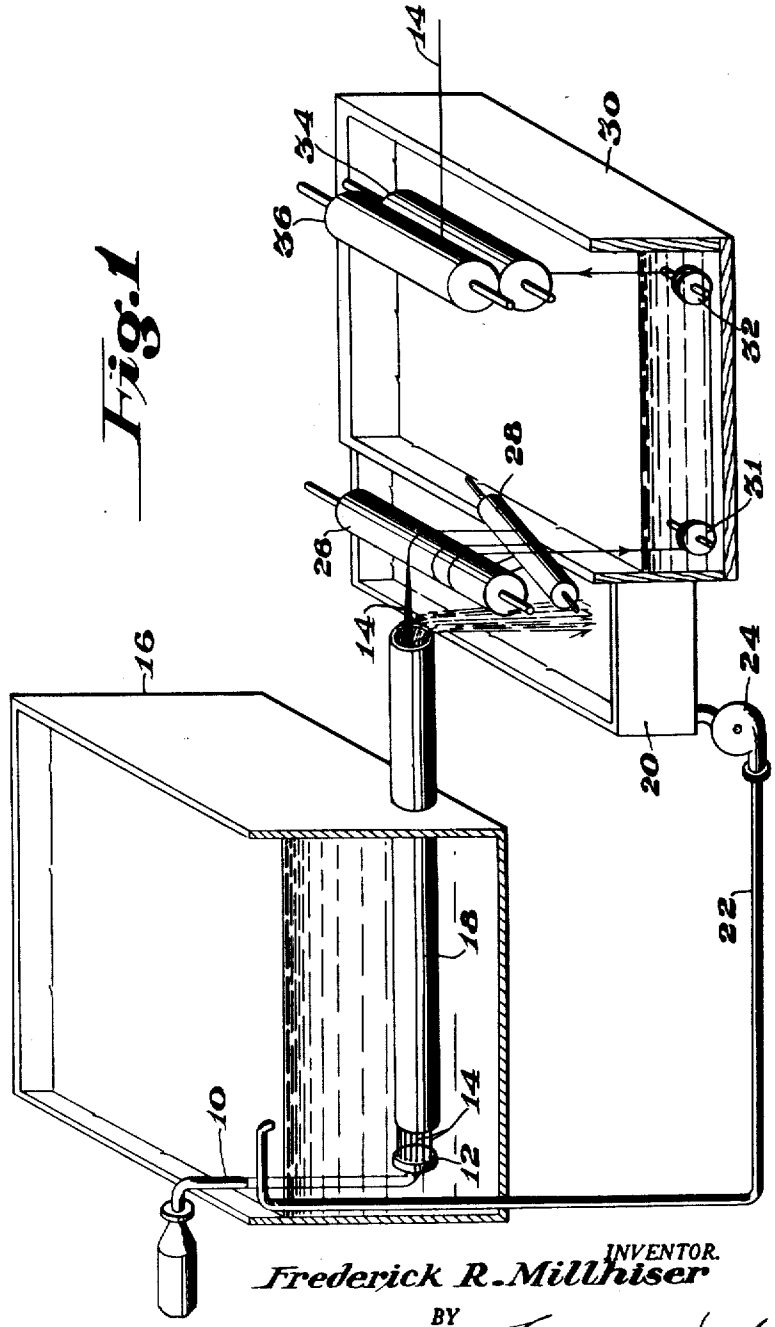
April 20, 1948. F. R. MILLHISER 2,440,057

PRODUCTION OF VISCOSE RAYON

Filed March 15, 1944 3 Sheets-Sheet 1

Fig. 1



INVENTOR.
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April 20, 1948. F. R. MILLHISER 2,440,057

PRODUCTION OF VISCOSE RAYON

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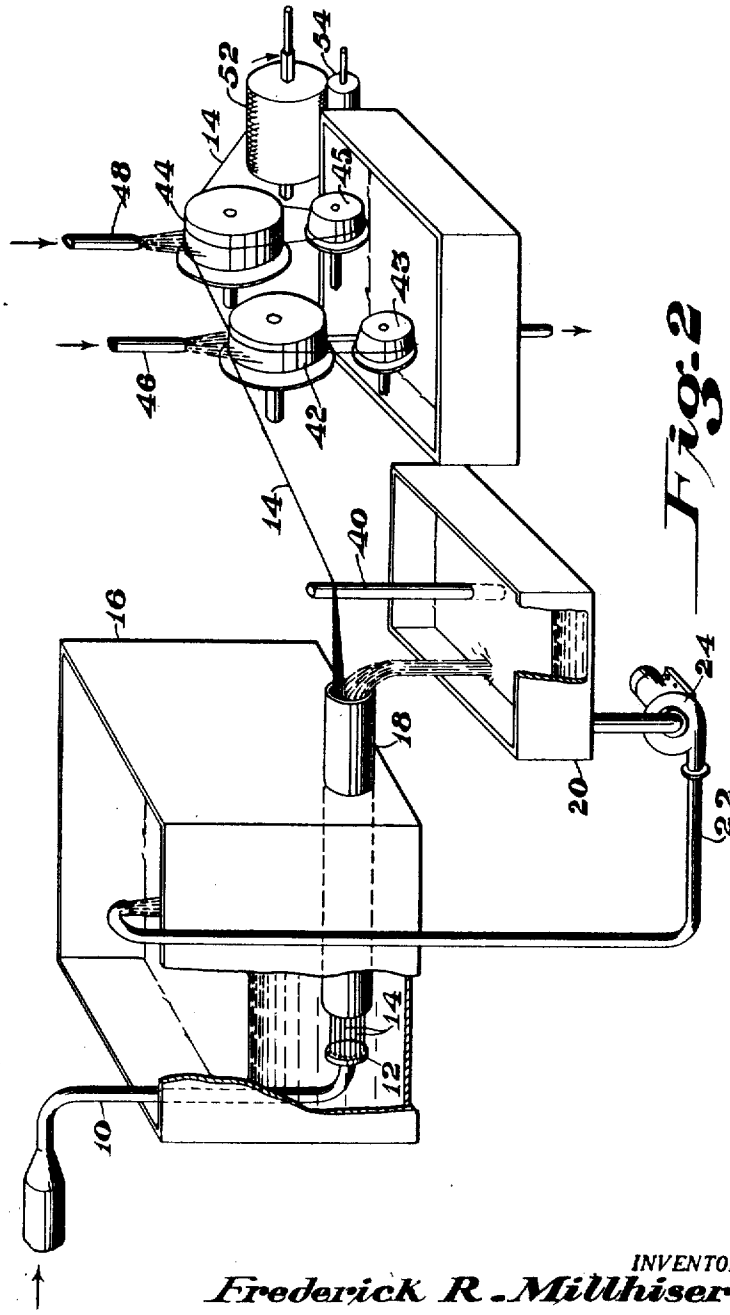


Fig. 2

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April 20, 1948. F. R. MILLHISER 2,440,057

PRODUCTION OF VISCOSE RAYON

Filed March 15, 1944 3 Sheets-Sheet 3

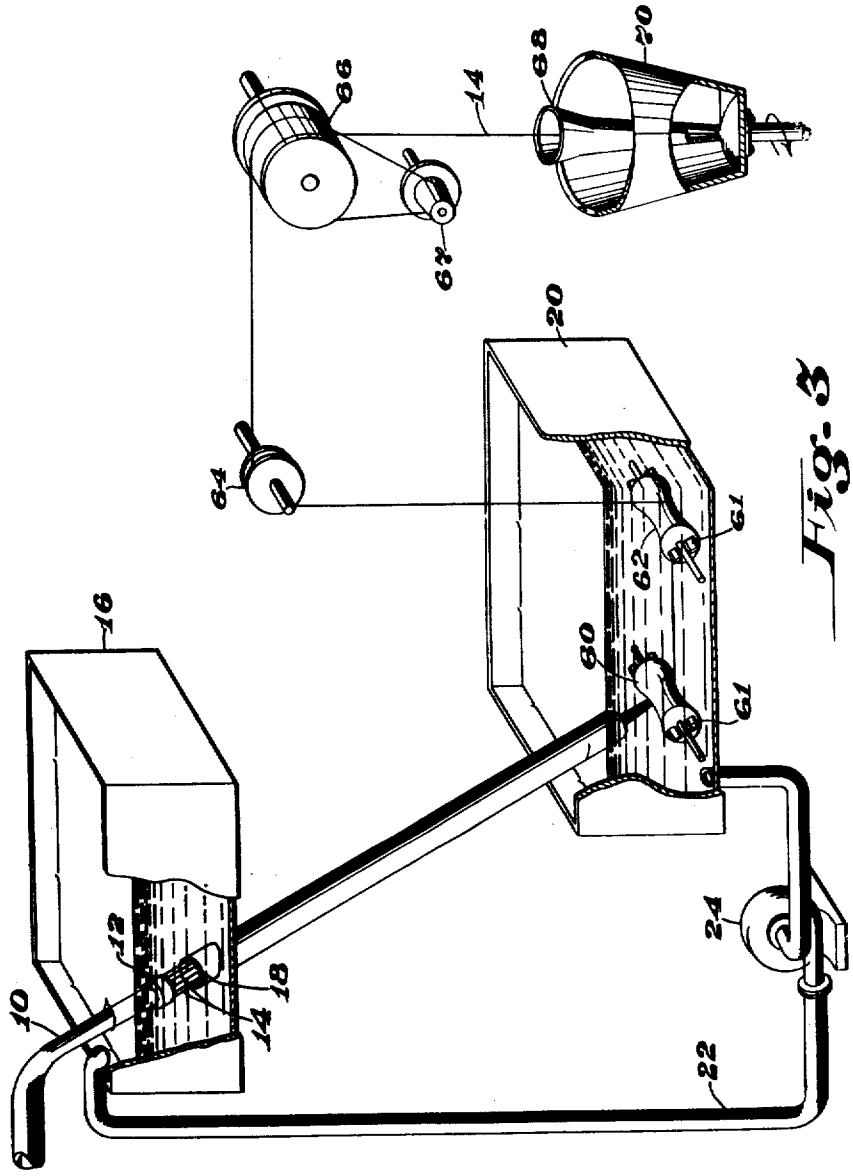


Fig. 3

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Patented Apr. 20, 1948 2,440,057

UNITED STATES PATENT OFFICE

2,440,057

PRODUCTION OF VISCOSE RAYON

Frederick R. Millhiser, Richmond, Va., assignor to E. I. du Pont de Nemours & Company, Wilmington, Del., a corporation of Delaware

Application March 15, 1944, Serial No. 526,512

11 Claims. (Cl. 18—54)

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This invention relates to the production of viscose rayon, and more particularly it relates to a new and improved method for the production of viscose rayon yarn having highly desirable physical characteristics.

It is an object of this invention to provide a new and improved process for the spinning of viscose rayon yarn whereby to produce such yarn having a high silk factor (by "silk factor" is meant dry tenacity in grams per denier x percentage elongation).

It is another object of this invention to provide a new and improved process for the spinning of viscose rayon yarn whereby such yarn may be spun at very high spinning speeds and yet produce a yarn having a high silk factor.

Other objects of the invention will appear hereinafter.

The objects of this invention may be accomplished, in general, by extruding viscose through a spinneret into a coagulating bath, and immediately stretching the resulting filaments adjacent to the spinneret before complete coagulation of the filaments has taken place, then passing the filaments through said bath at a very low tension (between 0.01 and 0.06 gram per denier), and thus without substantially stretching the same until they are capable of withstanding a tension at least 0.5 gram per denier without breaking, and then stretching the filaments at least 25%.

The tension to which freshly precipitated filaments or yarn can be subjected without breaking can be readily determined by means of any of the conventionally used, commercially available tensiometers of the type applicable to moving yarns. A yarn is capable of withstanding a given tension, for example 0.5 gram per denier, when the total load on the yarn (0.5 gram per denier multiplied by the denier of the yarn) can be applied to the spinning yarn and yet not interrupt the continuity of spinning by breaking the yarn. The denier of the yarn is the weight, in grams, of 9000 meters of the yarn at a moisture content of 11% even though the tension measurement is made on the wet yarn in the spinning solution.

The details of the invention will be more clearly apparent by reference to the following detailed description of a few representative embodiments of the invention when taken in connection with the accompanying illustrations, in which:

Fig. 1 is a perspective view, with parts cut away, showing one embodiment of apparatus suitable for use in accordance with this invention.

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Fig. 2 is a diagrammatic view with parts in perspective and parts in elevation showing a slightly modified embodiment of the apparatus.

Fig. 3 is another diagrammatic view with parts in perspective and elevation, showing another embodiment of the apparatus.

Referring to Fig. 1 of the drawings, reference numeral 10 designates a conduit for the forwarding of a viscose filament-forming composition to spinneret 12. The viscose is extruded through the spinneret 12 to form a plurality of filaments 14 which are passed through a tubular member 18 positioned near the bottom of a coagulating bath tank 16. The bath tank 16 is made of sufficient depth that the head of the liquid in the tank will cause a flow of the bath through the tube 18 at the desired velocity. The yarn, composed of the filaments 14, and the coagulating and regenerating bath thus flow cocurrently through the tube 18. The outlet end of the tube 18 is positioned above a receiving tank 20 from which the coagulating bath liquid may be returned to the bath tank 16 through conduit 22 and pump 24.

The yarn 14 is drawn from the tube 18 by means of feed rollers 26 and 28. The yarn is passed about these rollers with a sufficient number of turns that slippage of the yarn will be eliminated. From rollers 26 and 28 the yarn passes about freely rotatable rollers 31 and 32 which are positioned in a stretching bath contained in tank 30. The stretching bath may be composed of the same composition as the coagulating bath liquid or it may be composed of a similar liquid having lower acidity, or even water. The stretching bath liquid may be heated to any desired temperature under the boiling point so as to impart additional plasticity to the yarn to enable it to be stretched to a greater degree. The yarn is removed from the stretching bath contained in tank 30 by means of squeeze rollers 34 and 36 which are driven at a peripheral speed at least 25% greater than the peripheral speed of feed rollers 26 and 28. These rollers are adapted to clamp the yarn with sufficient force to prevent slippage of the yarn therebetween. The squeeze rollers 34 and 36, and the feed rollers 26 and 28 represent alternative forms of yarn feeding structures which may be used as desired. Feed rollers 26 and 28 may be replaced by squeeze rollers similar to rollers 34 and 36 or alternatively, squeeze rollers 34 and 36 may be replaced by feed rollers similar to rollers 26 and 28. From squeeze rollers 34 and 36, the yarn 14 is collected in any

desired manner, for example, on a bobbin, in a bucket, or on a yarn treating belt.

The tube 18 should be positioned within a distance of one and one-half (1½) inches from the face of the spinneret 12. The head of the coagulating bath above the tube 18 is preferably such as to give a velocity of flow of the bath liquid through the tube slightly less than the speed of the yarn being drawn through the tube. If desired, the speed of the liquid flowing through the tube can be adjusted by means of a cap placed on the outlet end of the tube, said cap having an opening of restricted size. As the yarn speed is made slightly greater than the velocity of the liquid flowing through the tube, there will be at least a sufficient tension on the yarn to insure that the filaments will be held straight and under a very slight tension, i. e., a tension of at least 0.01 gram per denier. On the other hand, the speed of the yarn must not be greatly in excess of the velocity of the liquid since this would entail the dragging of the yarn through the liquid with a substantial tension. The tension on the yarn, as measured, at the outlet end of the tube 18, should not exceed 0.06 gram per denier. By the above construction, the yarn is stretched between the face of the spinneret and the inlet mouth of the tube 18. As soon as the yarn enters the tube 18, it will be moving along with the liquid and at a speed only slightly in excess of the liquid so that there will be substantially no stretch on the yarn within the tube 18. Since the tension on the yarn is between .01 and .06 gram per denier, the stretch on the yarn passing through the tube will be negligible. As will later appear in the specification, it is essential that the yarn be maintained in contact with the bath without substantial stretch from a point where the yarn will no longer be substantially stretched by the initial stretching tension, a tension not to exceed 0.06 gram per denier, (a point generally within 1½ inches from the face of the spinneret) to a point where the yarn is capable of withstanding a tension of at least 0.5 gram per denier without breaking.

Referring to Fig. 2 of the drawings, like reference characters designate like parts as shown in Fig. 1. Reference numeral 40 designates a convergence bar about which the yarn is drawn from the mouth of the tube to feed wheel 42. Feed wheel 42, and cooperating guide roller 43 are operated at such a speed that the yarn passes through tube 18 at a speed slightly in excess of the velocity of the liquid flowing through the tube. From feed wheel 42, the yarn passes to a second feed wheel 44 and cooperating guide roller 45. The second feed wheel 44, as in the case of Fig. 1, is caused to rotate at a peripheral speed at least 25% in excess of the peripheral speed of feed wheel 42. Thus, the yarn will be stretched at least 25% between wheels 42 and 44.

If desired, the yarn being stretched between feed wheels 42 and 44 may be supplied with a coagulating fluid or a stretching fluid at room temperature, or at a temperature considerably above room temperature. This liquid may be applied by means of jets 46 and 48 positioned respectively above feed wheels 42 and 44. The yarn 14 passing from feed wheel 44 is wound on bobbin 52 which may be rotated by means of a surface drive roller 54.

Referring to Fig. 3 of the drawings, in which like reference characters again designate like parts as shown in Figs. 1 and 2. The yarn passing from tube 18, which in this instance is positioned at a relatively sharp downward angle from tank 16 to tank 20, is passed about vane guide rollers 60 and 62. These rollers are provided with vanes 61 of a sufficient size to offer a material resistance to rotation when positioned within the bath contained in tank 20. The yarn is passed from vane guide rollers 62 about freely rotatable grooved roller 64, and then about feed wheel 66 and guide roller 67 which draws the yarn from the tube 18 about the vane guide rollers 60 and 62. The speed of feed wheel 66 is so adjusted that the tension on the yarn between the outlet end of tube 18 and vane guide roller 60 will be between 0.01 and 0.06 gram per denier. It will be readily apparent that the yarn between vane guide roller 60 and feed wheel 66 will be progressively tensioned to stretch the same. This is due to the pull on the yarn against the resistance to rotation of rollers 60 and 62.

From feed wheel 66 the yarn 14 is passed into reciprocating funnel 68 which in turn guides the yarn onto the inside of rotating bucket 70 in a known manner.

The viscose filament-forming composition is stretched in the coagulating bath at a tension not to exceed 0.06 gram per denier until said stretching tension will no longer impart a substantial stretch to the freshly formed filaments. Generally this stretch will take place within approximately the first 1½ inches of yarn travel from the spinneret. By stretching the yarn in this region, the filaments are attenuated before coagulation is completed, i. e., the yarn is still largely in a highly tender or even liquid state. The yarn must be stretched at this point sufficiently to insure the formation of straight filaments, however, it may be stretched to any degree short of severing the filaments. The filaments in this region, because they are largely in a highly tender or liquid state, will take a high degree of stretch without subjecting them to a tension exceeding 0.06 gram per denier, without micellar orientation, and without impairment of properties of the yarn, and thus permit the spinning of viscose rayon yarn in the coagulating bath at very high speeds, for example, speeds of the order of 4,000 to 10,000 inches per minute.

After the stretching of the filaments adjacent the spinneret, they must be maintained in contact with the coagulating bath under very little tension and stretch, i. e., at a tension of between 0.01 and 0.06 gram per denier, until the filaments are capable of withstanding a tension of at least 0.5 gram per denier without breaking, after which they may be subjected to a considerable additional stretch. When spinning in the coagulating bath at a speed of about 5,000 inches per minute, a 200 denier yarn will have to be held within the above narrow range of low tensions for a distance of approximately 15 inches. A higher denier yarn will have to be held within said range of tensions for a slightly longer distance, and at higher spinning speeds, the yarn will have to be held within said range of tensions proportionately longer.

After the filaments are capable of withstanding a tension of at least 0.5 gram per denier without breaking, they are subjected to a stretch of at least 25% and preferably at least 50%. This does not mean that the filaments need be tensioned to at least 0.5 gram per denier; it is only necessary that they be set-up sufficiently to be capable of withstanding a tension of at least 0.5 gram per denier. The filaments may obtain this second stretch in the coagulating and/or regenerating

bath, or they may be removed from the coagulating bath to a stretching bath which may be composed of water having a temperature between 60° C. and 100° C., or a low acid bath (having a lower acidity than the coagulating bath) heated to between 60° C. and the boiling point thereof. The filaments may, in fact, be stretched in any fluid medium liquid or gaseous, which will not deteriorate or degrade the same.

The coagulating bath may have any composition which will coagulate the viscose filament-forming composition. Preferably, however, this bath should be of the so-called Mueller type containing from 6% to 13% H_2SO_4, from 15% to 30% inorganic salts such as for example, Na_2SO_4, $ZnSO_4$ and/or $FeSO_4$. Particularly desirable results have been obtained by the use of a coagulating and regenerating bath containing between 6% and 12% H_2SO_4, between 18% and 28% Na_2SO_4, between 0.5% and 15% $ZnSO_4$ and 0% and 10% $FeSO_4$. By the use of such a bath in combination with the above-described process steps, it is possible to obtain an exceptionally desirable viscose rayon yarn. The presence of both $ZnSO_4$ and $FeSO_4$ in the bath appears to impart a higher loop strength and fatigue resistance to the yarn than by the use of either $ZnSO_4$ or $FeSO_4$ alone.

In the above description, the only means shown, for imparting a preliminary stretch to the filaments while they are incompletely coagulated and then maintaining the filaments in contact with the coagulating bath without imparting any substantial stretch thereto until they are capable of withstanding at least 0.5 gram per denier tension, comprises a tubular member through which the bath is passed cocurrently with the yarn. Any other means, for example a mechanical positively operated means, for accomplishing this same result may be used equally well, however, the cocurrent bath-flow tube means is considered to be the most convenient and practical.

By the practice of the present invention, viscose rayon yarn having an exceptionally high silk factor can be produced at spinning speeds far greater than possible heretofore. Spinning speeds in the coagulating bath of 4,000 to 10,000 or more inches per minute, with the production of commercially satisfactory yarn, are easily producible by following the practice of this invention. When it is considered that the spun yarn can then be stretched 25% to 100% in a stretching bath to raise the spinning speed at the take-up bobbin, bucket or belt to 5,000 to 20,000 inches per minute, the importance of this invention becomes obvious. The process of the present invention is not, however, to be limited to the above-mentioned high spinning speeds.

In previously known processes for the spinning of viscose rayon yarns, i. e., the spinning processes where the yarn is stretched throughout the coagulation thereof, the cross-section of the filaments changes from a relatively rounded, finely crenulated shape to an irregular, coarsely crenulated shape as the spinning speed is increased. By the process of the present invention, however, even at very high spinning speeds the cross-section of the filaments have the same relatively rounded, finely crenulated shape as obtained from the conventionally known spinning processes operating at low speeds of 2,000 to 3,500 inches per minute.

From the results obtained by the process of the present invention it appears exceedingly important, particularly when spinning at high speeds, that the preliminary stretch imparted to the yarn be applied sufficiently near the spinneret face that a tension of less than 0.06 gram per denier will impart a very substantial stretch to the yarn, this will usually be within a distance of 1½ inches from the spinneret face, and that the tension on the yarn be kept at a very minimum, i. e., between 0.01 and 0.06 gram per denier, between the point where the initial tension no longer substantially stretches the filaments and the point at which the filaments are capable of withstanding a tension of at least 0.5 gram per denier without breaking. The filaments can then be stretched from 25% to any degree below the breaking strength of the filaments to produce highly desirable yarns of excellent physical characteristics.

The following example, in which all parts are by weight unless otherwise specified, illustrates the process of this invention. It is, of course, to be understood that this example is only illustrative and is not intended to limit the scope of the invention.

Example I

A viscose solution prepared from equal portions of wood pulp and cotton linters and containing 7% cellulose and 6% caustic and ripened to a salt index of 3.75 was extruded through a spinneret into a bath containing 11.5% H_2SO_4, 21% Na_2SO_4 and 0.7% $ZnSO_4$ and maintained at a temperature of 55° C., to form a 188 denier, 40 filament yarn. A tubular member, comprising a hollow glass tube 47 inches long and having an inner diameter of $\frac{11}{32}$ inch was mounted in the bath with its opening $\frac{5}{8}$ inch from the face of the spinneret, the tube extending downwardly to a second bath contained in a lower trough, a vertical distance of 45 inches separating the surfaces of the two baths and causing a bath flow of 5060 inches per minute through the tube. On leaving the spinneret, the freshly formed yarn was directed into and through the tubular member, the yarn being subsequently removed from the lower bath and passed about a feed wheel having a peripheral speed of 5250 inches per minute, the total tension on the yarn during its travel through the bath and to the feed wheel being of the order of 6 grams. The yarn was then passed to and about a second feed wheel possessing a peripheral speed of 6560 inches per minute, the yarn being stretched to form a yarn having a denier of 150, the yarn being subjected to a tension of 33 grams while passing between the two feed wheels. The yarn was then collected, purified and dried. It possessed a tenacity of 1.79 grams per denier and an elongation of 25.7% (silk factor of 46). It was not brittle and its filaments, which were lustrous, possessed a round, crenulated cross-section.

In contrast to this, a yarn spun in a somewhat similar manner but subjected to a tension of 10 grams during its passage from the spinneret to the first feed wheel and thereafter stretched to its final speed by the application of a tension of 44 grams possessed a tenacity of only 1.69 grams per denier and an elongation of only 15.8% (a silk factor of 26.7).

Since it is not readily determinable at which point in the coagulating and regenerating bath, or in the stretching bath, the filaments become completely coagulated and/or regenerated, the term "spinning speed," as used throughout the specification and claims, unless limited specifically to the spinning speed in a particular locus,

refers to the speed of the yarn at the take-up bobbin, or other collecting device.

By the process of the present invention it is possible to spin a viscose rayon yarn at exceedingly high spinning speeds (speeds from 5,000 to 10,000 or even up to 20,000 inches per minute), with the resulting yarn having physical characteristics which are fully equal to that produced by previously known, conventionally used processes at relatively low spinning speeds (speeds of the order of 3,500 inches per minute).

Since it is obvious that many changes and modifications can be made in the above described details without departing from the nature and spirit of the invention, it is to be understood that the invention is not to be limited to the details described herein except as set forth in the appended claims.

I claim:

1. A process for spinning viscose rayon yarn which comprises extruding viscose through a spinneret into a coagulating bath to produce a continuous filament yarn, stretching the resulting yarn at a tension not to exceed 0.06 gram per denier until said tension will no longer impart a substantial stretch to the yarn, causing coagulating bath to flow in the direction of movement of the yarn, maintaining said yarn in cocurrent contact with said coagulating bath at a tension of between 0.01 and 0.06 gram per denier until it will withstand a tension of at least 0.5 gram per denier, with the yarn travelling at a speed slightly in excess of the speed of the cocurrent coagulating bath liquid, and then stretching said yarn.

2. A process for spinning viscose rayon yarn which comprises extruding viscose through a spinneret into a coagulating bath to produce a continuous filament yarn, stretching the resulting yarn at a tension not to exceed 0.06 gram per denier until said tension will no longer impart a substantial stretch to the yarn, causing coagulating bath to flow in the direction of movement of the yarn, maintaining said yarn in cocurrent contact with said coagulating bath at a tension of between 0.01 and 0.06 gram per denier until it will withstand a tension of at least 0.5 gram per denier, with the yarn travelling at a speed slightly in excess of the speed of the cocurrent coagulating bath liquid, and then stretching said yarn at least 25%.

3. A process for spinning viscose rayon yarn which comprises extruding viscose through a spinneret into a coagulating bath to produce a continuous filament yarn, stretching the resulting yarn at a tension not to exceed 0.06 gram per denier until said tension will no longer impart a substantial stretch to the yarn, causing coagulating bath to flow in the direction of movement of the yarn, maintaining said yarn in cocurrent contact with said coagulating bath at a tension of between 0.01 and 0.06 gram per denier until it will withstand a tension of at least 0.5 gram per denier, with the yarn travelling at a speed slightly in excess of the speed of the cocurrent coagulating bath liquid, and then stretching said yarn at least 25% while in contact with said coagulating bath.

4. A process for spinning viscose rayon yarn which comprises extruding viscose through a spinneret into a coagulating bath to produce a continuous filament yarn, stretching the resulting yarn at a tension not to exceed 0.06 gram per denier until said tension will no longer impart a substantial stretch to the yarn, causing coagulating bath to flow in the direction of movement of the yarn, maintaining said yarn in cocurrent contact with said coagulating bath at a tension of between 0.01 and 0.06 gram per denier until it will withstand a tension of at least 0.5 gram per denier, with the yarn travelling at a speed slightly in excess of the speed of the cocurrent coagulating bath liquid and then stretching said yarn at least 25% while in contact with water having a temperature between 60° C. and 100° C.

5. A process for spinning viscose rayon yarn which comprises extruding viscose through a spinneret into an acid coagulating and regenerating bath to produce a continuous filament yarn, stretching the resulting yarn at a tension not to exceed 0.06 gram per denier until said tension will no longer impart a substantial stretch to the yarn, causing coagulating bath to flow in the direction of movement of the yarn, maintaining said yarn in cocurrent contact with said coagulating bath at a tension of between 0.01 and 0.06 gram per denier until it will withstand a tension of at least 0.5 gram per denier, with the yarn travelling at a speed slightly in excess of the speed of the cocurrent coagulating bath liquid and then stretching said yarn at least 25% while in contact with an acid bath having a lower acidity than said coagulating bath.

6. A process for spinning viscose rayon yarn which comprises extruding viscose through a spinneret into a coagulating and regenerating bath to produce a continuous filament yarn, stretching the resulting yarn at a tension not to exceed 0.06 gram per denier until said tension will no longer impart a substantial stretch to the yarn, said stretching being carried out with the stretched yarn having a linear speed in said bath of at least 4000 inches per minute, causing coagulating bath to flow in the direction of movement of the yarn, maintaining the yarn in cocurrent contact with said coagulating bath at a tension of between 0.01 and 0.06 gram per denier until it will withstand a tension of at least 0.5 gram per denier, with the yarn travelling at a speed slightly in excess of the speed of the cocurrent coagulating bath liquid and then stretching said yarn at least 25%.

7. A process for spinning viscose rayon yarn which comprises extruding viscose through a spinneret into an acid coagulating and regenerating bath to produce a continuous filament yarn, stretching the resulting yarn at a tension not to exceed 0.06 gram per denier until said tension will no longer impart a substantial stretch to the yarn, said stretching being carried out with the stretched yarn having a linear speed in said bath of between 4000 and 10,000 inches per minute, causing coagulating bath to flow in the direction of movement of the yarn, maintaining the yarn in cocurrent contact with said coagulating bath at a tension of between 0.01 and 0.06 gram per denier until it will withstand a tension of at least 0.5 gram per denier, with the yarn travelling at a speed slightly in excess of the speed of the cocurrent coagulating bath liquid and then stretching said yarn at least 25%.

8. A process as defined in claim 8 in which the second named stretching of the yarn is carried out in the presence of the coagulating bath.

9. A process as defined in claim 8 in which the second named stretching of the yarn is carried out in water having a temperature of between 60° C. and 100° C.

10. A process as defined in claim 8 in which

the second named stretching of the yarn is carried out in an acid bath having a lower acidity than the coagulating bath, and having a temperature between 60° C. and boiling.

11. A process for spinning viscose rayon yarn which comprises extending viscose through a spinneret into a coagulating bath to produce a continuous filament yarn, stretching the resulting yarn at a tension not to exceed 0.06 gram per denier until said tension will no longer impart a substantial stretch to the yarn, causing coagulating bath to flow in the direction of movement of the yarn, maintaining said yarn in cocurrent contact with said coagulating bath liquid at a tension not exceeding 0.06 gram per denier until it will withstand a tension of at least 0.5 gram per denier, with the yarn travelling at a speed slightly in excess of the speed of the cocurrent coagulating bath liquid, and then stretching said yarn.

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REFERENCES CITED

The following references are of record in the file of this patent:

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Number	Name	Date
2,036,313	Zimmermann et al.	Apr. 7, 1936
2,182,762	Mengeringhausen et al.	Dec. 5, 1939
2,267,055	Tippetts	Dec. 23, 1941
2,307,863	Soukup	Jan. 12, 1943
2,309,072	Burkholder	Jan. 19, 1943
2,328,307	Thurmond	Aug. 31, 1943
2,340,377	Graumann et al.	Feb. 1, 1944
2,346,696	Moritz et al.	Apr. 18, 1944
2,348,415	Polak	May 9, 1944

Certificate of Correction

Patent No. 2,440,057. April 20, 1948.

FREDERICK R. MILLHISER

It is hereby certified that errors appear in the printed specification of the above numbered patent requiring correction as follows: Column 8, lines 68, 71 and 75, for claim reference numeral "8" read 7; column 9, line 6, claim 11, for the word "extending" read *extruding*; and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 8th day of June, A. D. 1948.

SEAL]

THOMAS F. MURPHY,
Assistant Commissioner of Patents.

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11. A process for spinning viscose rayon yarn which comprises extending viscose through a spinneret into a coagulating bath to produce a continuous filament yarn, stretching the resulting yarn at a tension not to exceed 0.06 gram per denier until said tension will no longer impart a substantial stretch to the yarn, causing coagulating bath to flow in the direction of movement of the yarn, maintaining said yarn in cocurrent contact with said coagulating bath liquid at a tension not exceeding 0.06 gram per denier until it will withstand a tension of at least 0.5 gram per denier, with the yarn travelling at a speed slightly in excess of the speed of the cocurrent coagulating bath liquid, and then stretching said yarn.

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