

## UNITED STATES PATENT OFFICE

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## SPINNING PROCESS

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This invention relates to the spinning of re-generated cellulose filaments and yarns, and more particularly it relates to the spinning of such filaments and yarns having a surface-delustered effect.

The art has long demanded delustered rayon yarn. Rayon yarn is commonly delustered by the addition of a white pigment, for example, finely divided titanium dioxide, to a filament-forming composition, for example, viscose. The pigment is embodied throughout the yarn spun from such viscose, and serves to diffuse and scatter the light that impinges on the surface of the rayon filament.

It is an object of the present invention to provide a new and improved method for the production of yarns and filaments having a surface-delustered effect due to irregularities in the surface structure thereof.

Other objects of the invention will be apparent from the description that follows.

The above objects are accomplished, for example, by spinning filaments and yarns from a viscose composition containing a quaternary ammonium hydroxide into a coagulating bath having a strong, fast coagulating action and substantially no regenerating action, or only a small or delayed regenerating action, and subsequently regenerating said yarns and filaments.

In accordance with the preferred method of carrying out the invention, the yarn is stretched after coagulation and prior to substantial regeneration thereof. Preferably, the yarn is stretched at least 25% over the spun length thereof. The yarn can, of course, be stretched any amount less than the breaking strength thereof.

It is also preferred, in accordance with the present invention that the yarn be regenerated in a relaxed condition, preferably in a sufficiently relaxed condition to permit substantial shrinkage thereof, i. e. shrinkage while substantially free from tension.

Any conventionally used viscose composition may be employed in the process of the present invention. The viscose may, for example, have a sodium chloride (salt) index within the range of 3.5 to 8. It may have a cellulose content of between 5% and 9%, and it may have a caustic alkali content, calculated as NaOH, of from 4% to 8%.

The viscose composition must contain a quaternary ammonium hydroxide, such as, for example, trimethyl benzyl ammonium hydroxide, triethyl benzyl ammonium hydroxide, tripropyl benzyl ammonium hydroxide, trimethyl phenyl ammoni-

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um hydroxide, triethyl phenyl ammonium hydroxide, tetramethyl ammonium hydroxide, trimethyl ethyl ammonium hydroxide and trimethyl cyclohexyl ammonium hydroxide. The quaternary ammonium hydroxide is preferably added to the viscose in an amount between 1% and 5% of the weight of the viscose, depending upon the cellulose content of the viscose. As a further guide, it is preferred that the said quaternary ammonium hydroxide be present in the viscose in an amount between 15% and 50% of the weight of the cellulose contained in the viscose.

It is essential that the yarns and filaments be spun in a coagulating bath having a strong, fast coagulating action and substantially no regenerating action, or only a slow or delayed regenerating action on the filaments.

The coagulating bath should have a sufficiently fast coagulating action, relative to any regenerating action thereof, so that the yarns and filaments will be substantially completely coagulated before any substantial regeneration takes place. Coagulating baths of this type have a pH value of between 3.0 and 7.0, and therefore may be comprised of neutral and slightly acid solutions. The coagulating baths may contain one or more water-soluble salts, for example, sodium bisulfite, sodium sulfite, sodium sulfate, monosodium phosphate or disodium acid phosphate, ammonium acid phosphate, ammonium sulfate, sodium lactate and ammonium lactate. Preferably, the amount of salt in the coagulating bath should be relatively high, for example, between 25% and 40% of the bath composition.

The yarns and filaments may be finally regenerated in any known manner, for example, by means of an acid bath such as a sulfuric acid bath commonly used in the regeneration of cellulose xanthate structures, by heat, such as by a boiling salt solution, or by a combination of acid and heat.

The following examples are given to illustrate preferred methods for carrying out the present invention, it being understood that the invention is not to be limited to the details set forth in these examples:

*Example I*

0.75 pound of Triton B (a 40% aqueous solution of trimethyl benzyl ammonium hydroxide) was thoroughly mixed with 15.75 pounds of 7% cellulose, 6% sodium hydroxide viscose. After deaerating and ripening to a (sodium chloride) salt index of 5.5, the viscose was extruded at 17 grams per minute through a spinneret containing

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120 holes of 0.0035" diameter into a coagulating bath comprised of 15% sodium bisulfite, 3% sodium sulfite and 10% sodium sulfate (pH of bath 5.2) maintained at a temperature of 55° C. The coagulated xanthate filaments were drawn from the bath at 600" per minute under a tension of 5 grams by a positively driven roller and stretched 125% through a similar bath maintained at a temperature of 100° C., under a tension of 110 grams. The stretched yarns were collected in a relaxed condition and regenerated by boiling 10 minutes in the stretching bath. After washing and drying in a relaxed condition, the yarn was quite dull. Microscopic examination of the longitudinal section showed a rough, finely crenulated surface. When dyed black with a direct dye-stuff such as "Pontamine" Black ENB (Color Index No. 381), this yarn was dull black in appearance.

#### Example II

1.54 pounds of Triton B (a 40% aqueous solution of trimethyl benzyl ammonium hydroxide) were thoroughly mixed with 30.8 pounds of 7% cellulose, 6% sodium hydroxide viscose. After deaerating and ripening to a (sodium chloride) salt index of 5.4, the viscose was extruded at 17.1 grams per minute through a spinneret containing 120 holes of 0.0035" diameter into a coagulating bath comprised of 18.3% monosodium phosphate and 10% sodium sulfate (pH of bath 4.9) maintained at a temperature of 55° C. The coagulated xanthate gel filaments were drawn from the bath at 600" per minute under a tension of 2.5 grams by a positively driven roller and stretched through a bath containing 16.8% monosodium phosphate and 2.0% sodium sulfate (pH of bath 4.9) maintained at a temperature of 100° C., under a tension of 100 grams. The stretched yarn was collected in a relaxed condition and regenerated by boiling 30 minutes in the spinning bath. After washing and drying in a relaxed condition, the yarn was dull in appearance. When dyed black with a direct dyestuff such as "Pontamine" Black EBN (Color Index No. 581), this yarn was dull black.

The production of surface delustered rayon yarn in accordance with the present invention is particularly desirable in the production of yarns which are desired to be dyed a dull black color. Rayon which is commonly delustered by the addition of a pigment to the viscose and which is dyed with a black dye will normally reflect light sufficiently to make the yarn look bright or lustrous. This is probably due to the fact that yarn dyed black will absorb so much of the light impinged thereon that the amount of light reflected will be sufficient to make the yarn have a bright appearance. Yarn produced in accordance with the present invention, when dyed a black color, will exhibit a dull black appearance.

Obviously, if desired, a yarn may be produced by the addition of a pigment to the viscose in addition to carrying out the process of the present invention to produce on the yarn the above mentioned surface delustered effect. Thus, although dark pigments, e. g. carbon black, when used in viscose in relatively small amounts, will not adequately produce a matt surface effect, nevertheless this defect can be overcome, while still using said dark pigment, if the yarn is made in accordance with the process of this invention.

The use of alkali metal phosphate-containing baths having a pH of 3.0 to 7.0, including the

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coagulating bath of Example II, for the coagulation, under low tension, of filaments from viscose, with stretching prior to regeneration, followed by regeneration, is covered in the copending application of Francis Bernard Cramer and William Frederick Underwood, Serial No. 484,764, filed April 27, 1943. The use of such phosphate baths for the low tension coagulation of filaments from viscose followed by regeneration under low tension, is covered in the copending application of Rudolph Woodell, Serial No. 561,635, filed November 2, 1944, as a division of Rudolph Woodell, Serial No. 509,280, filed November 6, 1943.

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 above described details except as set forth in the appended claims.

#### I claim:

1. The process of producing low luster regenerated cellulose filaments and yarns which comprises spinning filaments and yarns by extruding a viscose composition containing between 1% and 5% by weight of a quaternary ammonium hydroxide into a coagulating bath comprising an aqueous solution of a water-soluble salt and having a sufficiently fast coagulating action, relative to any regenerating action thereof, to substantially completely coagulate said filaments prior to any substantial regeneration thereof, and subsequently regenerating said yarns and filaments.

2. The process of producing low luster regenerated cellulose filaments and yarns which comprises spinning filaments and yarns by extruding a viscose composition containing between 1% and 5% by weight of a quaternary ammonium hydroxide into an aqueous coagulating bath having a pH value between 3.0 and 7.0, and subsequently regenerating said yarns and filaments.

3. The process of producing low luster regenerated cellulose filaments and yarns which comprises spinning filaments and yarns by extruding a viscose composition containing between 1% and 5% by weight of a quaternary ammonium hydroxide into an aqueous coagulating bath having a pH value between 3.0 and 7.0, and having dissolved therein a water-soluble salt, and subsequently regenerating said yarns and filaments.

4. The process of producing low luster regenerated cellulose filaments and yarns which comprises spinning filaments and yarns by extruding a viscose composition containing between 1% and 5% by weight of a quaternary ammonium hydroxide into an aqueous coagulating bath having a pH value between 3.0 and 7.0, and having dissolved therein 25% to 40% of a water-soluble salt, and subsequently regenerating said yarns and filaments.

5. The process of producing low luster regenerated cellulose filaments and yarns which comprises spinning filaments and yarns by extruding a viscose composition containing between 1% and 5% by weight of a quaternary ammonium hydroxide into a coagulating bath comprising an aqueous solution of a water-soluble salt and having a sufficiently fast coagulating action relative to any regenerating action thereof, to substantially completely coagulate said filaments prior to any substantial regeneration thereof, stretching said yarns and filaments at least 25% over the spun length thereof, and subsequently regenerating said yarns and filaments.

6. The process of producing low luster regen-

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erated cellulose filaments and yarns which comprises spinning filaments and yarns by extruding a viscose composition containing between 1% and 5% by weight of a quaternary ammonium hydroxide into a coagulating bath comprising an aqueous solution of a water-soluble salt and having a sufficiently fast coagulating action, relative to any regenerating action thereof, to substantially completely coagulate said filaments prior to any substantial regeneration thereof, and subsequently regenerating said yarns and filaments while sufficiently relaxed to permit substantially complete shrinking thereof.

7. The process of producing low luster regenerated cellulose filaments and yarns which com-

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prises spinning filaments and yarns by extruding a viscose composition containing between 1% and 5% by weight of a quaternary ammonium hydroxide into a coagulating bath comprising an aqueous solution of a water-soluble salt and having a sufficiently fast coagulating action, relative to any regenerating action thereof, to substantially completely coagulate said filaments prior to any substantial regeneration thereof, stretching said yarns and filaments at least 25% over the spun length thereof, and subsequently regenerating said yarns and filaments while sufficiently relaxed to permit substantially complete shrinking thereof.

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