United States Patent

[11] 3,626,045

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[33] **Great Britain**
[31] **60,676/68**

[54] **PROCESS FOR MAKING TUBULAR FILAMENTS**
4 Claims, 1 Drawing Fig.

[52] **U.S. Cl.** **264/54,**
161/178, 264/188, 264/195
[51] **Int. Cl.** **D01f 3/10**
[50] **Field of Search** 161/178;
264/188, 193, 195, 41–55

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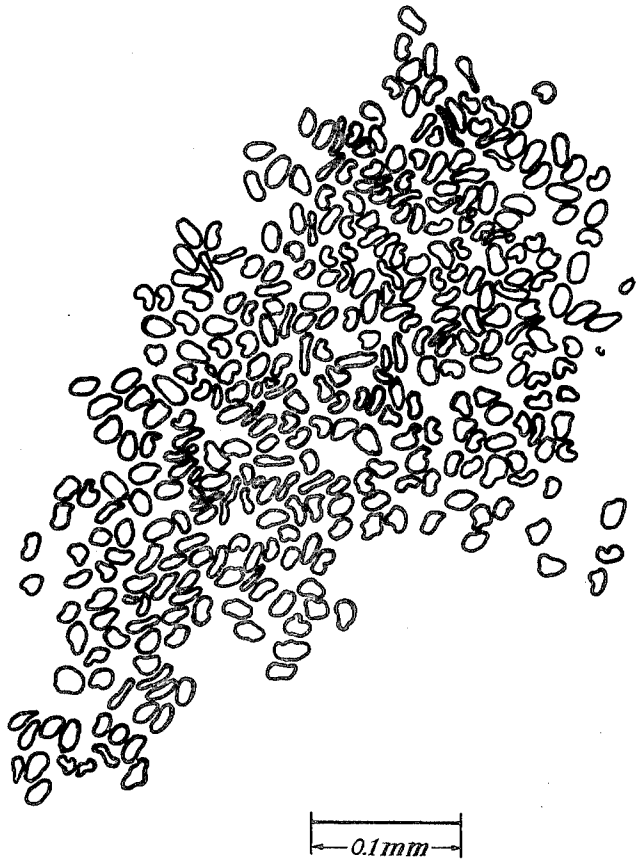
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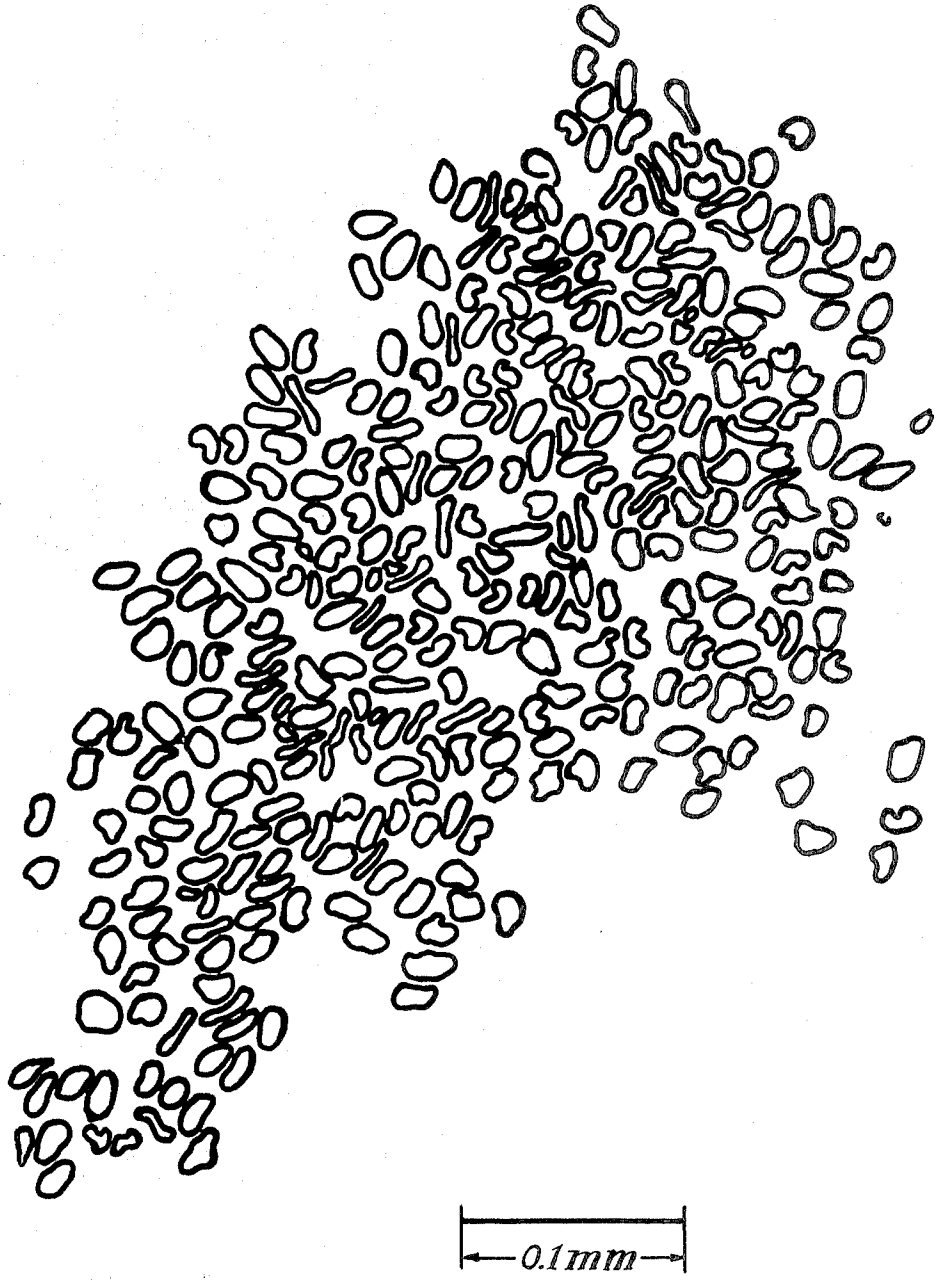
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Attorney—Davis, Hoxie, Faithfull and Hapgood

ABSTRACT: A controllable commercial process for producing tubular regenerated cellulose filaments comprises extruding a viscose of a specific composition and containing sodium carbonate into a sulfuric acid spin bath of a specific composition to form filaments followed by stretching of the filaments. Tubular filaments with increased wet modulus and a tendency to crimp are produced by stretching the filaments by an amount approaching their breaking stretch.





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PROCESS FOR MAKING TUBULAR FILAMENTS

This invention relates to a process for making tubular filaments of regenerated cellulose by the viscose process. Many such processes have been proposed in the past but tubular regenerated cellulose filaments have not been produced on a commercial scale.

Collapsed tubular filaments, on the other hand, have been made commercially and a process for making them is described in our British Pat. Spec. No. 945,306. These filaments are essentially different from the filaments with which the present invention is concerned in that their walls have caved in leaving little void space and producing a flat filament having a high width to thickness ratio.

The object of the present invention is a controllable commercial process which is capable of producing regenerated cellulose filaments of which at least 90 percent by number in a cross section of a spun tow are tubular filaments which have not collapsed to the flat state.

According to this invention, a process for making tubular filaments of regenerated cellulose comprises extruding a viscose containing 6.5 to 9.5 percent by weight of cellulose, 5.0 to 7.0 percent by weight of caustic soda, 3.0 to 4.0 percent by weight of sodium carbonate and 0.75 to 2.0 percent by weight of polyethylene glycol based on the weight of cellulose, and having a salt figure of 12 to 18, into a spinning bath containing 8.0 to 10.0 percent by weight of sulfuric acid, 1.0 to 3.0 percent by weight of zinc sulfate, and 20.0 to 26.0 percent by weight of sodium sulfate, and at a temperature of 25° to 45° C., to form filaments, directing the filaments along an immersion path of length 30 cm. to 185 cm., and stretching the filaments by at least 40 percent.

The invention includes the tubular filaments produced by this process.

The viscose used in the process of the invention may have a concentration of carbon disulfide of 33 to 50 percent by weight based on the weight of cellulose and a ball-fall viscosity of 30 to 180 seconds at 18° C.

The stretching operation preferably is carried out in a hot aqueous bath containing sulfuric acid, preferably a bath at about 95° C. and containing about 2 percent by weight of sulfuric acid. It has been found that fibers with an increased wet modulus and a tendency to crimp, with only slightly reduced tensile properties, can be obtained by stretching the filaments in the stretch bath by an amount approaching the breaking stretch of the filaments, preferably to about 10 percent below break. In this way we have obtained tubular filaments having an initial wet modulus of up to 10.0 at 2.0 percent extension and up to 12.0 at 5.0 percent extension.

Staple fibers produced from the filaments of the invention may be handled with ease on cotton spinning machinery. The resulting yarns are of an increased bulk compared with yarn made from solid, viscose rayon filaments and fabrics made from the yarns have a handle similar to cotton and a greater absorbency.

The invention is illustrated by the following example in which the percentages are be weight.

EXAMPLE

A viscose containing 7.0 percent cellulose, 5.5 percent caustic soda, 45 percent carbon disulfide based on the weight of cellulose, 4.0 percent sodium carbonate, and 1.0 percent polyethylene glycol, and with a salt figure of 15 and a ball-fall viscosity of 50 seconds at 18° C., was extruded into a spin bath containing 9.3 percent sulfuric acid, 2.0 percent zinc sulfate and 23 percent sodium sulfate, to form filaments of 1.5 denier.

The temperature of the spin bath was 30° C. and the immersion path length was 122 cm.

The filaments were stretched in an aqueous stretch bath containing 2.0 percent sulfuric acid and at a temperature of 95° C. to a total of 85 percent stretch.

The average properties of the filaments were as follows:

Air dry tenacity	2.6 g. per denier
Air dry extensibility	11.0 percent
Wet extensibility	11.0 percent
Wet tenacity	1.7 g. per denier
Initial Wet Modulus at 2.0 percent extension	7.6
Initial Wet Modulus at 5.0 percent extension	9.5
Water imbibition	130
Crimp	4-5 waves per cm.

The accompanying drawing is a highly magnified cross-sectional view of the filament tow produced by the process of the example.

What is claimed is:

1. A process for making tubular filaments of regenerated cellulose comprising extruding a viscose containing 6.5 to 9.5 percent by weight of cellulose, 5.0 to 7.0 percent by weight of caustic soda, 3.0 to 4.0 percent by weight of sodium carbonate and 0.75 to 2.0 percent by weight of polyethylene glycol based on the weight of cellulose, and having a salt figure of 12 to 18, into a spinning bath containing 8.0 to 10.0 percent by weight of cellulose, of sulfuric acid, 1.0 to 3.0 percent by weight of zinc sulfate, and 20.0 to 26.0 percent by weight of sodium sulfate and at a temperature of 25° to 45° C., to form filaments, directing the filaments along an immersion path of length 30 cm. to 185 cm., and stretching the filaments by at least 40 percent.

2. A process as claimed in claim 1 in which the filaments are stretched in a hot, aqueous bath containing sulfuric acid.

3. A process as claimed in claim 1 in which the filaments are stretched by an amount approaching the stretch at which the filaments break.

4. A process as claimed in claim 3 in which the filaments are stretched by an amount which is about 10 percent below the stretch at which the filaments break.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,626,045 Dated December 7, 1971

Inventor(s) CALVIN R. WOODINGS

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Col. 2, line 15, "cm." should read -- cms. --;

Claim 1, line 39, delete "of cellulose," ;

line 43, "30 cm. to 185 cm." should be -- 30 cms. to 185 cms. --.

Signed and sealed this 25th day of July 1972.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents