

UNITED STATES PATENT OFFICE

2,301,003

METHOD OF PRODUCING RAYON FIBERS
OR FILAMENTS

Walther Zetzsche and Erich Graumann, Premnitz, Germany, assignors, by mesne assignments, to Walther H. Duisberg, New York, N. Y.

No Drawing. Application December 29, 1938, Serial No. 248,240. In Germany January 19, 1938

2 Claims. (Cl. 18—54)

This invention relates to an improved method of producing rayon filaments or fibers, and more particularly filaments or fibers of increased tensile strength.

U. S. Patent No. 2,082,814 to Zetzsche et al. describes a process in which viscose is first shaped with or without stretching in a bath having only a coagulating effect so as to produce a cellulose xanthate thread which is then decomposed in a second bath without tension by the action of a hot liquid or of an acid liquid to produce a cellulose hydrate thread.

There has also been proposed a process in which viscose is coagulated in a salt solution or in alcohol, the coagulated thread is heated to a high temperature for expelling water and finally decomposed in an acid bath.

For the purpose of increasing the tenacity of cellulose hydrate threads it has been proposed to stretch the cellulose hydrate threads, spun in the usual acid spinning bath, in a hot salt bath directly following the spinning bath.

The present invention is based on the observation that the capacity of the threads for being stretched, and thereby the tensile strength obtainable, may be increased by spinning the viscose in a bath having merely a coagulating effect, for example in a salt bath, a feebly acidified salt bath or an alcohol bath, and subjecting the cellulose xanthate threads produced, after they have passed a tensioning device to a strong stretching in a bath which neither dissolves nor decomposes the xanthate threads and is maintained at a temperature above room temperature, preferably between 50° and 85° C., and finally decomposing the xanthate threads in a third bath.

The process of the present invention consists, therefore, in subjecting xanthate threads made in the usual manner with the application of strong preliminary tension to a further strong stretching during or after their passage through a bath maintained at a temperature above room temperature, and only then decomposing them in a further bath. The bath kept at a temperature of 50° to 85° C. and used in stretching the threads preferably consists of a salt solution, for instance a solution of 15 to 25 per cent strength of sodium or ammonium sulfate or of an indifferent liquid, preferably miscible with water, for instance glycerol or glycol. Liquids which are immiscible with water, for instance paraffin oil, are also useful as stretching baths, but they have the disadvantage of soiling the succeeding decomposing bath and of being also

liable to give trouble in the after-treatment of the threads.

The xanthate threads must be decomposed in hot baths, which may be quite or substantially chemically indifferent, or by acid baths, the operation being conducted at raised temperature in the first instance or at a normal temperature in the second instance. The process has the advantage over known processes that the threads subjected to stretching are still fully in a plastic or extensible condition and that for the stretching the enhanced extensibility of the xanthate threads is used in a warm bath. This kind of stretching very favorably affects the properties, particularly the tenacity and extensibility of the finished threads.

The stretched threads may be decomposed without tension, for example after they have first been cut to staple length, or they may be decomposed under tension. When the decomposition occurs without tension, artificial wool and like curly threads or fibers are produced; when the decomposition occurs under tension the finished fibers more closely resemble ordinary artificial silk.

The following examples illustrate the invention:

Example 1

A viscose containing 7.7 per cent of cellulose and 6.5 per cent of alkali is spun to xanthate threads in a coagulating bath comprising 24 per cent of ammonium sulfate and 6 per cent of sodium sulfate. The bundle of filaments is guided over glass rods for producing tension, is drawn through a pair of rollers at 35 meters per minute and passed through a bath containing sodium sulfate solution of 18 per cent strength at 50° C.; when it has left this bath the bundle passes over the second arrangement of tensioning rods to a second pair of rollers having a peripheral speed of 50 meters per minute. The stretched band is now led to a cutting device and the staple fibers are finally decomposed in a boiling hot solution of sodium sulfate containing 29 per cent of Na_2SO_4 so as to produce cellulose hydrate.

Example 2

A viscose containing 8.8 per cent of cellulose and 6.6 per cent of alkali is coagulated in a bath containing 12 per cent of ammonium sulfate and 11 per cent of sodium sulfate. The xanthate threads are passed over a tensioning device, for example a row of staggered glass rods, then through a solution of sodium sulfate of 18 per

cent strength and at 50° C. so as to stretch them as strongly as possible. The stretched bundle of threads is cut to staple and the latter is decomposed in an acid spinning bath containing 13 per cent of H_2SO_4 and 28 per cent of Na_2SO_4 .

Example 3

Xanthate threads spun as described in Example 1 or 2 are stretched as strongly as possible in a sulfate bath heated to 80° C. and then decomposed in a boiling hot solution of 1 per cent strength of sulfuric acid.

By tensioning device is to be understood in the examples a device consisting of rollers or rods in staggered relationship over which the threads are guided with formation of more or less pointed angles. By stretching, on the other hand, is to be understood the drawing of the bundle of threads in the direction of draft between two rollers or pairs of rollers. Remarks concerning stretching and tensioning of the threads in the examples relate to bundles of threads of 14,000 deniers which are made by combining small bundles of threads issuing from spinnerets having 600 perforations of 0.08 mm. When the thickness of the resultant sliver of filaments is varied the tensions and stretchings must be correspondingly changed, as will be readily apparent to one skilled in the art.

We claim:

1. A process of producing rayon fibers of improved tensile strength, which process comprises, spinning viscose into a bath merely capa-

ble of coagulating the cellulose xanthate without decomposing the same, withdrawing the threads formed from said bath at a rate substantially greater than the speed with which said threads issue from the spinneret, leading said threads into a bath consisting of a chemically indifferent non-solvent for the cellulose xanthate, said bath being heated to a temperature of between about 50° and 85° C., stretching the threads thus pre-heated to as near their breaking point as possible without actually breaking them, withdrawing said threads from said last named bath and decomposing the cellulose xanthate to cellulose hydrate.

2. A process of producing rayon fibers of improved tensile strength, which process comprises, spinning viscose into a bath merely capable of coagulating the cellulose xanthate without decomposing the same, withdrawing the threads formed from said bath at a rate substantially greater than the speed with which said threads issue from the spinneret, leading said threads into a bath consisting of a chemically indifferent non-solvent for the cellulose xanthate, said bath being heated to a temperature of between about 50° and 85° C., stretching the threads thus pre-heated to as near their breaking point as possible without actually breaking them, withdrawing and cutting the continuous xanthate threads, and decomposing the cellulose xanthate to cellulose hydrate.

WALTHER ZETZSCHE.
ERICH GRAUMANN.