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SPINNING ASSISTANTS

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No Drawing. Original application May 28, 1942, Serial No. 444,903. Divided and this application January 12, 1946, Serial No. 640,981

3 Claims. (Cl. 18—54)

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This invention relates to the manufacture of films, filaments and the like by extruding solutions of material to be formed into a coagulating or precipitating medium. This application is a division of my copending application Ser. No. 444,903, filed May 28, 1942, now abandoned.

This invention concerns a novel process of forming artificial filaments, films and related shaped articles by extruding solutions of the material to be formed, such as viscose, into an aqueous coagulating or precipitating bath and it also concerns a novel coagulating or precipitating bath for carrying out the process.

It is an object of the invention to improve the conditions of extrusion and of coagulation or precipitation during the formation of filaments, films and the like to insure that products of uniformly high quality are obtained. Other objects of the invention will be apparent from the description hereinafter.

The process and spinning bath of this invention may be employed in the formation of filaments, films and the like from various solutions, such as those viscose, or related cellulosic derivatives, and of proteinous material, such as casein, but it will be described hereinafter with relation to the spinning of viscose.

In accordance with this invention, a solution of the material from which the filaments and the like are to be formed is extruded through an orifice or a plurality thereof of the appropriate cross-sectional shape into a coagulating or precipitating bath containing dissolved therein a condensation product of ethylene oxide with a polyhydric alcohol selected from the group consisting of sugars and those having the formula RCH_2OH where R is selected from the group consisting of $-(-CHOH-)_nH$ and inner ether anhydrides thereof where n is an integral number from 2 to 6, which product contains at least nine ethylene oxide units per molecule of alcohol. The compounds are derived from the following representative polyhydric alcohols: glycerol, erythritols, pentahydric alcohols, hexahydric alcohols such as mannitol, sorbitol, dulcitol, heptahydric alcohols, as well as such related compounds as the sugars, for example glucose, sucrose, and the like, and anhydro derivatives of the polyhydric alcohols, such as mannitan, sorbitan, mannide, and sorbide. All of these condensation products are water soluble and non-ionogenic. Concentrations of the condensation product within the spinning bath may range from as low as 0.001% to 0.01% up to the limit of solubility of the product therein.

Spinning in the presence of these ethylene oxide products as spinning assistants improves the quality of the products obtained, insuring uniformity and reducing to a minimum any tendency

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of the filaments to break during spinning. This tendency of the filaments to break during spinning is particularly accentuated at high spinning speeds, such as of the order of from 2500 to 8000 inches per minute, and is particularly pronounced when attempts are made to spin viscose containing a high proportion of combined carbon disulphide, particularly at the above-mentioned high speeds. By high carbon disulfide content is meant a viscose containing more than 35% combined CS_2 based on the alpha cellulose, such as between about 36 to 45% higher. The spinning of high tenacity yarns from viscose, such as from viscose containing a high content of combined carbon disulphide, at high speed is in general accompanied by considerable breakage difficulties, which result in what is termed "slugging." The ends of the broken filaments tend to gather upon the adjacent unbroken portions of yarn and give rise to trouble during spinning, especially during the collecting of the filamentary material in centrifugal spinning buckets, where such partially broken yarn frequently becomes wrapped around the tip of the funnel guide which directs the filamentary material into the spinning bucket. Besides the spinning difficulties which accompany the filament breakage and slugging, the yarn obtained when such breakage occurs is of definitely inferior quality. These spinning difficulties are not to be attributed to clogging since they occur even when the spinneret orifices are entirely free of clogging, though any clogging tends to aggravate them.

An additional advantageous effect distinct from that just described is accomplished by spinning in the presence of these ethylene oxide products, namely, the tendency to form solid deposits of irregular shape within the orifices is minimized. For this purpose, however, only those containing a great number of ethylene oxide units are entirely satisfactory. For example, it has been found necessary that the product contain at least on the order of about 50 ethylene oxide units in order to obtain completely satisfactory results of this character, though those containing fewer of these units do show some slight improvement. When this clogging of some of the orifices is complete, it reduces the total number of filaments in the bundle issuing from a given spinneret and tends to make the remaining filaments somewhat larger in diameter than would be the case where no clogging of any of the orifices of the spinneret occurs. Where a number of the holes or orifices in the spinneret are only partially clogged, the result is a mixture of filaments of various sizes issuing from the spinneret. In addition, the filamentary bundle issuing from a given spinneret which is subject to clogging varies in uniformity along its length

depending upon the fluctuation in the extent of clogging in individual orifices, the rapidity of clogging, as well as the completeness of clogging of any of the orifices in the spinneret. The result is a filamentary yarn having considerable non-uniformity within a given cross-section as well as along its length. Other compounds have been suggested for this purpose, such as cation-active compounds and other ethylene oxide derivatives of compounds containing a long chain hydrophobic group. As compared with these other compounds, those of this invention have the advantage of being less rapidly exhausted from the bath under spinning conditions. Generally, at a given concentration of the assistant in the spinning bath, roughly ⅛ as much of the compounds of this invention is consumed during spinning as would be consumed of the compounds heretofore employed. Consumption as used here is meant to include the effects of instability, de-composition, and exhaustion.

The following examples are illustrative of the invention:

Example 1

A viscose solution was extruded into a spinning bath comprising from 8 to 12% sulphuric acid, about 20% sodium sulphate, about 5% zinc sulphate, and 0.01% of a condensation product of ethylene oxide with mannitol containing 100 units of ethylene oxide in the molecule.

Example 2

A viscose solution was extruded into a spinning bath containing from 7 to 9% sulphuric acid, about 14% sodium sulphate, about 1% zinc sulphate, about 5% glucose, and 0.01% of a condensation product of ethylene oxide with mannitol containing 100 units of ethylene oxide in the molecule.

Example 3

A viscose solution was extruded into a spinning bath having the composition of Example 2 except that a concentration of 0.002% of a condensation product of ethylene oxide with mannitol containing 300 units of ethylene oxide was substituted for the product employed in Example 2.

Example 4

A viscose solution was extruded into the spinning bath of Example 1 in which the ethylene oxide product of that example was substituted by 0.1% of a condensation product of ethylene oxide with glycerol having 9 units of ethylene oxide in the molecule.

Example 5

A viscose solution was extruded at a speed of about 2000 inches per minute into a spinning bath having the composition of Example 1 with the exception that an ethylene oxide condensation product with mannitol having 18 ethylene oxide units was substituted for that in the bath of Example 1. The product was employed in 0.05% concentration.

Example 6

A viscose solution was extruded into a spinning bath corresponding to the composition of that in Example 1 with the exception that a condensation product of ethylene oxide with glycerol having 50 units of ethylene oxide in the molecule was substituted for the product in the bath of Example 1. The concentration of the product in the bath was maintained at 0.007% during spinning.

Example 7

A viscose solution was extruded to form filaments at a speed of 2500 inches per minute into a spinning bath corresponding to that of Example 1 with the exception that a condensation product of ethylene oxide with glycerol having 9 units of ethylene oxide in the molecule was substituted for the product in the bath of Example 1, the product being maintained in the bath at a concentration of 0.1%.

Example 8

Viscose solutions having various proportions of CS_2 in the range from 35% to 45% were extruded under the conditions of Example 7 except that the spinning speeds were changed to 2500 to 8000 inches per minute.

Example 9

A viscose solution was extruded into a spinning bath having the composition of Example 2 except that a condensation product of ethylene oxide with glucose containing 50 units of ethylene oxide in the molecule was substituted for the product of Example 2.

Example 10

A viscose solution was extruded into a spinning bath having the composition of Example 2 except that a condensation product of ethylene oxide with sucrose containing 50 units of ethylene oxide in the molecule was substituted for the product of Example 2.

While preferred embodiments of the invention have been disclosed, it is to be understood that changes and variations may be made without departing from the spirit and scope of the invention as defined by the appended claims.

What I claim is:

1. In the method of producing filaments, films and the like, the step of extruding a solution of viscose through a jet into an aqueous precipitating medium containing a condensation product of ethylene oxide with glycerol containing at least 9 ethylene oxide units per molecule.

2. In the method of producing filaments, films and the like, the step of extruding a solution of viscose through a jet into an aqueous precipitating medium containing a condensation product of ethylene oxide with glycerol containing at least 50 ethylene oxide units per molecule.

3. An aqueous acid medium for precipitating viscose extruded in the form of filaments, films and the like, said medium containing a condensation product of glycerol with ethylene oxide containing at least 9 ethylene oxide units per molecule.

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REFERENCES CITED

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

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Certificate of Correction

Patent No. 2,489,310 November 29, 1949

SANFORD A. MOSS, JR.

It is hereby certified that errors appear in the printed specification of the above numbered patent requiring correction as follows:

Column 1, line 24, before the word "viscose" insert *of*; column 2, line 12, before "higher" insert *or*;

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 23rd day of May, A. D. 1950.

[SEAL]

THOMAS F. MURPHY,
Assistant Commissioner of Patents.