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METHOD OF MAKING ARTIFICIAL
FILAMENTS

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This invention relates to the production of artificial filaments containing organic derivatives of cellulose such as yarns, bristles, horse-hair, ribbons, artificial straw and the like hereinafter called "artificial filaments" from solutions containing organic derivatives of cellulose, and relates more particularly to the production of such artificial filaments by a wet spinning process.

An object of our invention is to produce finer filaments and yarns of organic derivatives of cellulose than has been heretofore possible by wet spinning methods. A further object is to produce filaments containing organic derivatives of cellulose, which filaments have more or less continuous lumens or hollow spaces therein. Further objects of our invention will appear from the following detailed description.

Heretofore, artificial filaments have been spun from solutions of organic derivatives of cellulose by a wet spinning process at temperatures approximately that of normal atmospheric temperatures. We have found that if the solutions containing organic derivatives of cellulose are at elevated temperatures just prior to their extrusion through orifices of a spinnerette into a precipitating bath, because of their lower viscosity, the filaments formed may be drawn out to finer sizes or deniers. Moreover, if the precipitating bath is maintained at a temperature above the boiling point of the solvent, hollow filaments are formed.

In accordance with our invention, we form filaments by the extrusion of solutions of organic derivatives of cellulose in a suitable solvent through the orifices of a spinnerette, while the solution is at an elevated temperature, into a precipitating bath that contains a liquid that forms a solution with at least part of the solvent.

Filaments of any suitable size may be formed by our invention. Thus, filaments say of 1 to 10 denier, such as are used in making artificial yarns, may be made by our process. These filaments may be used as such, or they may be associated or twisted together to form yarns that are useful for making woven or knitted fabrics. However, filaments of heavier denier say 20 to 2000 denier or more, may be made by our invention, and these filaments may be used as artificial bristles in the making, for instance, of various brushes, for making braids or for making artificial furs, etc. Obviously from the point of view of fine draw-down, the greatest advantages accrue when fine filaments are made. However, since by our invention hollow filaments may be made, this

invention may be employed advantageously for preparing the heavier bristles.

The organic derivatives of cellulose employed in our process may be any suitable organic ester of cellulose or cellulose ether. Examples of organic esters of cellulose are cellulose acetate, cellulose formate, cellulose propionate and cellulose butyrate, while examples of cellulose ethers are ethyl cellulose, methyl cellulose and benzyl cellulose.

The organic derivatives of cellulose may be dissolved in a suitable solvent or solvent mixture, such as acetone, mixtures of acetone and ethyl or methyl alcohol, mixtures of acetone and water, ethylene dichloride, mixtures of ethylene dichloride with ethyl or methyl alcohol, and mixtures of methylene chloride and ethyl or methyl alcohol.

The bath in which the stream of the solution of organic derivatives of cellulose is extruded contains a liquid that forms a solution with the solvent. This may be water, but we prefer to use an oily liquid or liquids that form solutions with the solvent employed, and where a mixed solvent is used, the oily liquids may form a solution with but one component of the solvent mixture. In either event, the precipitating bath dissolves away the solvent component from the solution of the derivatives of cellulose, thus causing the cellulose derivative to precipitate in the form of a filament. The term oily liquid not only includes such oils as olein, castor oil and the like, but also includes mineral oils, such as kerosene, gasoline, mineral lubricating oils, etc.

The temperature of the solution of the organic derivatives of cellulose may be raised by any suitable means prior to extrusion through the orifices of the spinnerette. This may be done by heating the precipitating bath in which the spinnerette is situated. The solution may, if desired, be heated prior to introduction to the spinnerette, either in a tank or in the pipe-lines leading to the jet, or both, by steam or hot water coils or jackets, by electrical heaters, etc. If desired, precipitating baths of elevated temperature may also be employed in those cases where the spinning solution is preheated by extraneous means.

By the foregoing method, filaments of very fine denier may be formed, since the reduced viscosity due to the elevated temperature permits the drawing out of the filaments to very fine deniers.

If the precipitating bath is maintained at a temperature above the boiling point of the solvent or one of the components of the solvent mixtures, hollow filaments are formed. This process is of particular advantage in the forma-

tion of heavy artificial filaments for use as bristles in brushes and the like. The formation of hollow spaces in the filament is probably due to the fact that owing to the elevated temperature, the surface of the filament becomes hard or tough by precipitation, and upon the sudden evaporation of the solvent in the interior, hollow spaces are formed.

The fibres or filaments formed in the precipitating bath may be wound at a uniform feed rate by any suitable device. Prior to winding, the filament is caused to travel a suitable distance under the precipitating bath to render the same capable of being wound. The distance through which the filament travels prior to being wound varies in accordance with such factors as the speed of spinning, the size of the filaments produced, etc. Thus, if heavy filaments for use as artificial bristles having a weight of say 1000 denier, are to be made, since the precipitation requires a longer time than in the case where filaments of say 5 denier for artificial yarn, are to be made, a longer travel through the precipitating bath, or a lower rate of spinning or both will be required.

We have found that after a comparatively short contact between the precipitating bath and the filament, the surface of the filament is sufficiently precipitated so that the filament thus formed may be wound in layers on bobbins without coalescing. Consequently, instead of employing a long precipitating bath between the spinning jet and the winding device, the winding device may be placed close to the jet but within the precipitating bath and the precipitation of the filaments is completed while on the winding device itself.

In order to further illustrate our invention without limiting the scope thereof, the following specific examples are given.

Example I

A solution containing one part of an acetone soluble cellulose acetate having an acetyl value of 54% in 3 parts of a solvent mixture comprising 95 parts of acetone and 5 parts of water, is employed as a spinning solution. This solution is extruded through a jet having circular orifices of 0.08 mm. diameter into a precipitating bath consisting of kerosene maintained at a temperature of 45° C. The rate of spinning is 50 meters of length of filament per minute. By this process filaments of low denier are obtained. On the other hand if the kerosene were maintained at 25° C., the finest filament that may be spun has a denier of twice that of the filament produced at 45° C.

Example II

A solution containing one part of the cellulose acetate described above and 3 parts of a solvent mixture consisting of 60% of ethylene dichloride and 40% of methyl alcohol is spun in the same manner as that described in Example I. When the kerosene bath is maintained at 50° C., filaments having a low denier may be formed, whereas if the temperature were kept at 25° C., the finest filaments that can be formed have denier twice of that produced at 50° C.

If in this example the kerosene is maintained at a temperature of 65° C., the methyl alcohol, which is practically insoluble in kerosene, evaporates off, and thus hollow filaments are formed.

It is to be understood that the foregoing detailed description is given merely by way of illus-

tration and that many variations may be made therein without departing from the spirit of our invention.

Having described our invention, what we claim and desire to secure by Letters Patent is:

1. Method of forming artificial filaments which comprises extruding a solution of a derivative of cellulose in an organic solvent through an orifice into a substantially stationary precipitating bath, the bath being maintained at a temperature above the boiling point of a substantial part of the solvent in the spinning solution.

2. Method of forming artificial filaments which comprises extruding a solution containing cellulose acetate through an orifice into a substantially stationary precipitating bath, the bath being maintained at a temperature above the boiling point of a substantial part of the solvent in the spinning solution.

3. Method of forming artificial filaments which comprises extruding a solution containing cellulose acetate through an orifice into a substantially stationary precipitating bath containing an oily liquid and that is maintained at a temperature above the boiling point of a substantial part of the solvent in the spinning solution.

4. Method of forming artificial filaments comprising heating a solution containing an organic derivative of cellulose and then extruding the same through an orifice into a precipitating bath containing an oily liquid and that is maintained at a temperature above the boiling point of a substantial part of the solvent in the spinning solution.

5. Method of forming artificial filaments, comprising extruding a solution of an organic derivative of cellulose in a volatile solvent through an orifice into a precipitating bath containing kerosene maintained at a temperature above the boiling point of a constituent of the spinning solution, said constituent having a low solubility in the precipitating bath.

6. Method of forming artificial filaments, comprising extruding a solution of an organic derivative of cellulose in a volatile solvent through an orifice into a precipitating bath maintained at a temperature above the boiling point of a constituent of the spinning solution, said constituent having a low solubility in the precipitating bath.

7. Method of forming artificial filaments, comprising extruding a solution of an organic ester of cellulose in a volatile solvent through an orifice into a precipitating bath maintained at a temperature above the boiling point of a constituent of the spinning solution, said constituent having a low solubility in the precipitating bath.

8. Method of forming artificial filaments, comprising extruding a solution of an organic ester of cellulose in a volatile solvent through an orifice into a precipitating bath containing kerosene at a temperature above the boiling point of a constituent of the spinning solution, said constituent having a low solubility in the precipitating bath.

9. Method of forming artificial filaments, comprising extruding a solution of cellulose acetate in a volatile solvent through an orifice into a precipitating bath containing kerosene maintained at a temperature above the boiling point of a constituent of the spinning solution, said constituent having a low solubility in the precipitating bath.

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