

## UNITED STATES PATENT OFFICE

CAMILLE DREYFUS, OF NEW YORK, N. Y., AND WILLIAM WHITEHEAD, OF CUMBERLAND, MARYLAND, ASSIGNORS TO CELANESE CORPORATION OF AMERICA, A CORPORATION OF DELAWARE

## PROCESS OF MAKING ARTIFICIAL FILAMENTS AND PRODUCT THEREOF

No Drawing.

Application filed September 8, 1928. Serial No. 304,805.

This invention relates to the production of artificial filaments, yarns, bristles and the like made of or containing cellulosic material.

An object of our invention is to produce filaments of cellulosic material, which filaments contain lumens or other hollow spaces therein.

Another object of our invention is to produce hollow filaments or bristles from solutions of cellulosic material by first forming a solid skin on the stream of material flowing from the orifices of a spinnerette and then solidifying the interior thereof. Other objects of our invention will appear in the following detailed description.

In the process of making artificial filaments from cellulosic derivatives as now practiced, solutions of cellulosic material are extruded through circular orifices of a spinnerette, either into a precipitating bath of uniform nature as in wet spinning, or into a substantially uniform evaporative atmosphere as in dry spinning, and the filaments thus formed are drawn off at a comparatively high speed. The filaments thus formed, however, have a solid cross section.

We have found that if the spinning solution as it is extruded from the orifices of a spinnerette is subjected to such conditions that it first starts to dry or skin on the surface only, and is then submitted to more drastic conditions so that the interior of the filaments solidify, filaments having hollow spaces therein may be formed. Such hollow filaments are in some instances preferred to the solid filaments, since among other advantages, for a given weight, they have greater covering power than the solid filaments.

In accordance with our invention, we prepare filaments having lumens or hollow spaces therein by the dry spinning or wet spinning of solutions containing cellulosic material in such a manner that the filaments or fibers under formation are first subjected

to an atmosphere or bath that acts merely to skin or dry the surface of the filaments only, and then the filaments are subjected to an atmosphere or bath that causes rapid solidification of the interior of the filaments.

By the process of our invention filaments, yarns, bristles or artificial horse hair, etc., may be made. The filaments formed may be of a very fine denier, on the order of less than 2 to 15 or more denier, which may be associated together to form artificial yarns. This process may be employed for preparing strands or filaments of much heavier denier such as 100, 300 to 1500 denier or more, thus forming stiff bristles or artificial horse hair that may be used in brushes that are adapted for toilet use.

This invention may be applied for making filaments or bristles by the viscose method, wherein a solution of viscose is extruded through spinnerettes into an aqueous bath containing a precipitating agent such as an acid.

This invention is eminently suited for making filaments from organic derivatives of cellulose. Examples of organic derivatives of cellulose are organic esters of cellulose such as cellulose acetate, cellulose formate, cellulose propionate, cellulose butyrate or cellulose ethers such as ethyl cellulose, methyl cellulose or benzyl cellulose. These organic derivatives of cellulose are dissolved in a suitable solvent and the solution extruded through orifices of a spinnerette, either into a bath containing a liquid which causes precipitation of the cellulose derivative from the solution as in wet spinning, or into a drying and evaporative atmosphere as in dry spinning.

The solvents employed for making solutions of the organic derivatives of cellulose may be any suitable ones, examples of which are acetone, acetone and water, methylene chloride and ethyl alcohol, ethylene dichloride, ethylene dichloride and alcohol

(ethyl or methyl), chloroform, or chloroform and ethyl or methyl alcohol, etc.

Where methods of wet spinning are used in carrying out our invention, the solution of the cellulosic material may be extruded first into a bath having a slight precipitating or coagulating effect so that only the exterior surface of the filaments is first solidified, and the filaments then passed into a bath that has a stronger precipitating action to complete the precipitation. Thus in making artificial yarn from viscose, the filaments may be extruded into a bath containing but a slight amount of acid, and then passed into a bath containing a larger percentage of acid.

In one method of carrying out our process by a dry spinning method, the temperature of the solution of the cellulose derivative is kept considerably below the boiling point of the solvent or solvents employed prior to its being extruded through the orifices of the spinnerette into the drying or evaporative atmosphere. Because of the low initial temperature of the spinning solution, the filaments formed travel for a considerable distance in the drying atmosphere before the inner portions thereof become sufficiently hot to cause evaporation of the solvent from the interior at an appreciable rate. The outer portions of the filaments, however, skin or dry very much sooner than the inner portions. As a result of this, when the inner portions dry rapidly and thus probably shrink at the time that the temperature of the interior becomes elevated, hollow filaments are formed.

In another form of carrying out our invention, the solution of the derivative of cellulose is extruded through the spinnerette into an atmosphere which is maintained at a temperature that does not exceed the boiling point of the solvent employed, and the filaments are then caused to pass through a drying atmosphere at a temperature considerably above the boiling point of the solvent. In this manner, a preliminary skin is formed on the filament during its travel through the atmosphere that is maintained at a lower temperature, while the solvent from the interior of the filament is not substantially volatilized. When the filament passes through the second drying atmosphere that is maintained at the higher temperature, the solvent of the interior thereof is evaporated and hollow filaments are thus formed.

Still another method of carrying out our invention is one wherein the solution of the cellulose derivative is caused to pass through the orifices of a spinnerette, first through an atmosphere that is saturated with or contains a substantial quantity of a vapor or of vapors of the solvent evolved from the filaments themselves, and is then passed into a

drying atmosphere substantially above the boiling point of the solvent employed and which is not saturated with such vapors. Because of the presence of the vapor in the first drying zone, the evaporation of the solvent is restricted so that evaporation substantially only on the surface of the filament takes place, thus forming a skin, or else the solution is evaporated to a very high concentration by the time it reaches the rapid drying atmosphere. In either event when the filament is subjected to the more rapid drying atmosphere of the second zone, either because of the skin formed or because of its more viscous nature, the evaporation of the residual solvent in the second zone causes the formation of hollow filaments.

In the dry spinning processes in accordance with our invention, the actual temperature of the spinning solution prior to extrusion as filaments, the distance of travel of filaments through the drying atmosphere, or the temperatures of the drying atmospheres will depend on such factors as the speed of spinning, the concentration of the derivative of cellulose in the spinning solution, and the solvent employed.

In the dry spinning processes here described, it will be observed that in all cases the filaments travel some distance through the drying atmosphere before hollowness is obtained. We have found that in our process the filaments are actually drawn out at or near the spinning jet since the filaments under formation are weaker and more plastic at this point probably because of the fact that they contain more solvent. This drawing out at this point makes possible the formation of hollow filaments of fine denier or size—a result that has not been heretofore possible.

In those steps of our process where drying atmospheres of temperatures materially above the boiling point of the solvents are used, other methods of rapid drying may be employed. Thus the filaments may be brought in contact with rolls or other devices heated by steam, electrical resistances, etc.

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

#### *Example 1*

The following is an example of a process wherein hollow filaments are obtained by keeping the temperatures of the spinning solution below the boiling point of the solvent prior to extrusion through the orifices of the spinnerette. A spinning solution is made by dissolving one part of an acetone soluble cellulose acetate in 4 parts of a solvent mixture consisting of 70 parts by weight of ethylene dichloride and 30 parts by weight of methyl alcohol. This solution is

maintained at a temperature of 40° C. prior to its being extruded through the orifices of a spinnerette into a drying atmosphere maintained at 100° C. The rate of spinning is maintained at 100 meters of length of filament per minute, and the filaments thus formed are drawn and wound in any suitable manner.

#### Example 2

The following is a description of the process wherein the filaments are first passed into an atmosphere maintained at a temperature below the boiling point of the solvent employed and then into a drying atmosphere maintained at a temperature above the boiling point of the solvent. A solution is made by dissolving one part by weight of cellulose acetate of about 54 to 55% acetyl value and insoluble in chloroform in 3 parts of a solvent mixture containing 90 parts by weight of acetone and 10 parts by weight of water. This solution is passed through the orifices of a spinnerette into a first drying zone of one foot in length which drying zone is maintained at a temperature of 50° C. and is then passed into a second drying zone of 5 feet in length maintained at 100° C. The rate of spinning is 120 meters of length of filament per minute.

#### Example 3

The following is an example of our process wherein the filaments are first dried in an atmosphere that is saturated with vapors and then into a more rapid drying zone. A spinning solution is made by dissolving one part of a chloroform soluble cellulose acetate in 3 parts of a solvent mixture consisting of 85 parts by weight of methylene chloride and 15 parts by weight of methyl alcohol. The solution is extruded through the orifices of a spinnerette into a first drying zone which is maintained at a temperature of 40° C. and which contains air that is saturated with vapors of the solvents employed. The length of this first drying zone is one foot. The filaments are then passed through a second drying zone containing air that is not saturated with the solvents, vapors and which zone is maintained at 80° C. The length of the second rapid drying zone is 5 feet.

It is to be understood that the foregoing detailed description is given merely by way of illustration and that many variations may be made therein without departing from the spirit of this invention.

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

1. Process of producing yarns, filaments, bristles and the like having hollow spaces therein comprising forming the same from solutions containing cellulosic material under such conditions that elimination of sol-

vent takes place in two stages, the first stage of solvent elimination being a retarded one under conditions which tend to cause surface coagulation only and the second stage being effected under conditions that tend to cause complete coagulation.

2. Process of producing yarns, filaments, bristles and the like having hollow spaces therein comprising forming the same from solutions containing cellulose acetate material under such conditions that elimination of solvent takes place in two stages, the first stage of solvent elimination being a retarded one under conditions which tend to cause surface coagulation only and the second stage being effected under conditions that tend to cause complete coagulation.

3. Process of producing yarns, filaments, bristles and the like having hollow spaces therein comprising passing a solution of organic derivatives of cellulose through orifices of a spinneret into a heated zone under such conditions that a comparatively low rate of solvent elimination takes place which tends to cause surface coagulation only and then into a zone where an increased rate of solvent elimination takes place and wherein complete coagulation is effected.

4. Process of producing yarns, filaments, bristles and the like having hollow spaces therein comprising passing a solution of cellulose acetate through orifices of a spinneret into a heated zone under such conditions that a comparatively low rate of solvent elimination takes place which tends to cause surface coagulation only and then into a zone where an increased rate of solvent elimination takes place and wherein complete coagulation is effected.

5. Process of producing yarns, filaments, bristles and the like having hollow spaces therein comprising forcing a solution of an organic derivative of cellulose in a solvent through the orifices of a spinneret into a drying atmosphere maintained at a temperature below the boiling point of the solvent wherein solidification of the surface only of the filaments takes place and then into a drying atmosphere maintained at a temperature above the boiling point of the solvent employed and wherein completion of solidification is effected.

6. Process of producing yarns, filaments, bristles and the like having hollow spaces therein comprising forcing a solution of cellulose acetate in a solvent through the orifices of a spinneret into a drying atmosphere maintained at a temperature below the boiling point of the solvent wherein solidification of the surface only of the filaments takes place and then into a drying atmosphere maintained at a temperature above the boiling point of the solvent employed and wherein completion of solidification is effected.

7. Yarns, filaments, bristles and the like

containing cellulosic material and having  
hollow spaces therein, which have been  
formed by passing solutions of cellulosic ma-  
terial into a zone under conditions tending  
5 to cause surface coagulation only and then  
into a zone tending to cause complete coagu-  
lation.

8. Yarns, filaments, bristles and the like  
containing cellulose acetate material and  
10 having hollow spaces therein, which have  
been formed by passing solutions of cellulose  
acetate material into a zone under conditions  
tending to cause surface coagulation only  
and then into a zone tending to cause com-  
15 plete coagulation.

In testimony whereof, we have hereunto  
subscribed our names.

CAMILLE DREYFUS.  
WILLIAM WHITEHEAD.

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