This invention relates to filaments of fine size and made of organic derivatives of cellulose.

This application is a continuation in part of application S. No. 286,070 filed June 16, 1936, which has issued as Patent No. 1,996,755, and application S. No. 410,247, filed May 12, 1936.

An object of our invention is to make filaments of organic derivatives of cellulose of finer size than has been heretofore possible. Another object of our invention is to make fine filaments by extruding a solution containing an organic derivative of cellulose and a compound of the polyhydric alcohol class, through fine orifices into a precipitating fluid of gaseous medium. Still another object of the invention is the improvement in spinning stability of low concentration spinning solutions. Other objects of our invention will appear from the following detailed description.

The preparation of artificial filaments of very fine size or denier is often highly desirable since yarns or fabrics made from such fine filaments have many excellent properties as to lustre, feel, hand, softness and the like. Heretofore it has not been possible to produce by ordinary methods filaments of organic derivatives of cellulose that are appreciably finer than two deniers. We have found that if the viscosity of the spinning solution is kept high as the concentration of the organic derivative of cellulose therein is decreased, it may be done by incorporating in the spinning solution a compound of the polyhydric alcohol class, it is possible to spin filaments having deniers as low as 0.5 or less. Heretofore it has been impossible to spin into filaments a solution of cellulose acetate wherein the concentration of the cellulose acetate was 20% or less without considerable trouble in spinning stability such as for instance the solution instead of being formed into filaments at the orifices formed drops at low feed rates or formed a spray at high feed rates and for many other reasons. If one attempts to overcome these difficulties by employing a cellulose acetate of very high viscosity without maintaining the concentration below 20% there is developed, if the ordinary methods are employed, a solution of poor spinning stability, and one, the yarn formed from which, is poor in textile properties. We have found that we can use 20% and less concentrations of normal or lower viscosity cellulose acetates in spinning without trouble, like dripping, spraying, etc., if we add to the spinning solution compounds that have the quality of increasing the viscosity of the spinning solution.

In accordance with our invention we prepare filaments of low denier and yarns containing such filaments by extruding a solution, regulated as to viscosity and concentration, containing an organic derivative of cellulose, a suitable solvent and a compound, for instance one of the polyhydric alcohol class, that enters into colloidal solution with the organic derivative of cellulose and solvent, through orifices of suitable size and shape into a fluid causing the solidification of the filaments, while regulating the speed of formation of the filaments and the speed of evaporation or removal of the solvent from the filaments whereby by filaments of fine denier and either flat or bulbous cross section according to desire are formed.

Any suitable organic derivatives of cellulose such as organic esters of cellulose or cellulose ethers may be employed for making the filaments. 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. However, we prefer to employ an acetone soluble cellulose acetate and generally the solutions employed for spinning contain from 15 to 50% by weight of such cellulose acetate depending upon circumstance hereinafter described.

As stated the spinning solution contains for instance a compound of the polyhydric alcohol class. The term "compound of the polyhydric alcohol class" includes not only the polyhydric alcohols but also their derivatives such as substitution products, esters and ethers thereof. Examples of polyhydric alcohols are the glycols such as diethylene glycol, triethylene glycol, or ethylene glycol and also glycerol and diglycerol. Examples of ethers of polyhydric alcohols are the monomethyl ethers of ethylene glycol or diethylene glycol, the monomethyl ethers of ethylene glycol or of diethyleneglycol and the monobutyl ethers of ethylene glycol or of diethylene glycol, as well as the corresponding di-methyl, ethyl or butyl ethers of these glycols. Examples of esters of the polyhydric alcohols are the acetyl ester of diglycerol, diethylene glycol or ethylene glycol. The derivatives of triethylene glycol may also be employed and also the higher viscosity forms of the polyhydric alcohols. The lower esters such as monacetin and diacetin may be employed as thinners in certain cases. These various compounds or mixtures of same may be incorporated in the solutions of the derivatives of cellulose in amounts varying from 2% to 25% of the weight of the organic derivative of cellulose. The amount employed will depend upon
the viscosity of the compound, the viscosity of the organic derivative of cellulose and its concentration and the type and size of the filaments desired.

In making the spinning solution any suitable solvent for the derivative of cellulose may be employed. We prefer to employ acetone or mixtures of acetone and water, acetone and ethyl alcohol, or acetone and methyl alcohol. The proportions of acetone in such mixtures may vary from 75 to 100% of the weight of the solvent mixture employed. However, other solvents or solvent mixtures may be employed, examples of which are mixtures of methylene chloride and ethyl or methyl alcohol and mixtures of ethylene dichloride and methyl or ethyl alcohol.

The spinning jets through which the solution of the derivative of cellulose is extruded may have any suitable number of orifices, say from 10 to 150 or above depending upon the number of filaments in and the denier of the yarn to be made. The yarns may be formed by bringing together at their exit from the spinning cabinet the smaller yarns or groups of filaments from several spinning jets. The orifices of the spinning jets may have any shape although we prefer to use circular orifices. As to the size of the orifices, this may be varied somewhat in accordance with the fineness of the filaments to be formed, thus the orifices may vary according to the usual practice in the manufacture of fine filaments, say from 0.04 to 0.15 mm. diameter. In accordance with this invention however the size of the orifice is not entirely controlling as to the fineness of the filament produced. Thus by maintaining the viscosity of the spinning solution relatively high in proportion to the concentration of the organic derivative of cellulose therein low, very fine filaments may be formed using relatively large size orifices. This results in a great advantage as the spinning jets may be made heavier so that they do not bulge at high-speed and high-pressure spinning conditions. The jets containing the larger size orifices are more readily cleaned and moreover are less liable to become clogged.

In order to form the filaments, the solution of the derivative of cellulose is extruded through one or more jets into a drum or into a stream of air, as in dry spinning, or into a precipitating or coagulating liquid bath as in wet spinning.

The filaments as they are formed may be drawn out or stretched in order to reduce their size or denier. The drawing out is preferably performed within the spinning machine, while the filaments are passing from the orifices where they are formed to a feed roller rotating at a greater peripheral speed than the rate at which the spinning solution is extruded, while the stretching is carried out outside the spinning cabinet and may be done by any suitable mechanical means. Thus the filaments may be passed around rollers rotating at progressively increasing peripheral speed and thus stretched. Although very fine filaments may thus be formed stretching is not necessary to form filaments as fine as 0.5 denier by the present invention. By the present invention the fine filaments are produced by a regulation of speed of formation, concentration of the organic derivative of cellulose in the spinning solution and the viscosities of the spinning solution and the organic derivative of cellulose thereof.

By varying these factors, according to our invention, very fine size filaments may be made having deniers as low as 0.5 or less. These filaments after being formed may be further reduced in size by saponification.

For the purpose of further describing the invention specific reference is made to the spinning of filaments of cellulose acetate being understood that the other organic ester and ethers function similarly under similar conditions. A solution of cellulose acetate at constant normal concentration with a low viscosity cellulose acetate, having a viscosity of say 5 to 15, is naturally reduced viscosity and when this low viscosity acetate is spun into filaments it produces filaments that are somewhat flat. However it is possible to offset, if desired, some of the reduction in bulbofulness in the filaments by an increase in feed rate and/or a fast removal of solvent vapor around the natal point of the filaments.

If a normal viscosity cellulose acetate having a viscosity of say 16 to 30, is formed into solution of less than normal concentration say less than 25% the viscosity again falls below normal and since below that concentration of the cellulose acetate is the cause of the low viscosity of the spinning solution. The filaments produced are flat which, however, may be partially corrected by employing forced flow of air to more quickly remove the solvent.

The difficulties and flat filaments produced by employing either lower viscosity cellulose acetate or reduced concentration may be overcome by raising the viscosity of the spinning solution to normal consistency by adding a high viscosity diluent such as the polyhydric alcohols etc. The cross section of the filaments may then be controlled producing flat or bulbous filaments as desired by variation in the temperature of the solvent removal medium. In either of the above cases with the addition of the viscous diluent there is substantially a normal amount of solvent used.

Low viscosity cellulose acetate, low concentration of cellulose acetate or both in a spinning solution may successfully be spun by regulating the viscosity of the spinning solution by the viscous diluent. By this method very fine denier filaments may be formed. After formation of the filaments the solvents may be removed therefrom by simple scouring means which may be done in a hank washing machine, while being wound into a package on a fabric washing machine after formation into a fabric or by other known expedients at any of the processing steps from spinning to the finished fabric.

The fine filaments produced are eminently suitable for formation of short lengths or staples adapted to be associated together by a spinning process, such as the cotton, wool, worsted system, to form "spun" yarn.

In order to further illustrate our invention, put without being limited thereto, the following example is given.

**Example**

A spinning solution is prepared from a cellulose acetate having a viscosity of 20. This solution contains 20% cellulose acetate and 80% of acetone solution containing 5% of water. To this solution is added diethylene glycol in amount of 10% of the weight of the cellulose acetate present and the same is spun into filaments by a dry spinning process employing jets of a size 0.07 mm. or less. The filaments produced may have a denier of less than 1 denier and contain a normal amount of residual solvent. By varying either
the temperature of the evaporative medium in spinning cabinet between the limits of 68° to 74° C. or the feed rate or both there are produced yarns consisting wholly of filaments of simple flat cross-section using the lower limits, or yarns consisting wholly near bulbous filaments using the upper limits.

The value of the viscosity given herein is determined by comparing the viscosity of a solution of 6 parts of cellulose acetate and 100 parts by weight of acetone in an Ostwald viscosimeter with pure glycerine at 20° C., the viscosity of the glycerine being designated as 100.

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 our invention.

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

1. Method of forming fine filaments comprising forming a solution of cellulose acetate in a suitable solvent, said cellulose acetate having a viscosity between 5 and 15 units as defined, incorporating in the solution a quantity of a compound of the polyhydric alcohol class which is a non-solvent for the cellulose acetate to increase the viscosity of the solution and extruding the resulting solution through orifices into a medium that causes solidification of the filaments.

2. Method of forming fine filaments comprising forming a solution of cellulose acetate in a suitable solvent, said cellulose acetate having a viscosity between 5 and 15 units as defined, incorporating in the solution a quantity of a compound of the polyhydric alcohol class which is a non-solvent for the cellulose acetate to increase the viscosity of the solution, extruding said resulting solution through orifices into a medium that causes solidification of the filaments and stretching the filaments thus formed.

3. Method of forming fine filaments comprising forming a solution of concentration of at most 20% of cellulose acetate in a suitable solvent, said cellulose acetate having a viscosity between 5 and 15 units as defined, incorporating in the solution a quantity of a compound of the polyhydric alcohol class which is a non-solvent for the cellulose acetate to increase the viscosity of the solution, extruding said resulting solution through orifices into a medium that causes solidification of the filaments and stretching the filaments thus formed.

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