

# UNITED STATES PATENT OFFICE

1,983,349

## TEXTILE MATERIAL AND METHOD OF MAKING THE SAME

Camille Dreyfus, New York, N. Y.

No Drawing. Application September 29, 1930,  
Serial No. 485,324

8 Claims. (Cl. 118—69)

This invention relates to the preparation of artificial filaments or threads made of organic derivatives of cellulose which are in better form for spinning.

5 An object of my invention is to prepare filaments or threads of organic derivatives of cellulose by incorporating a finely divided material therein so that comparatively short lengths or staples of the same may be successfully associated together to form "spun" yarn. Other objects of my invention will appear from the following detailed description.

The formation of "spun" yarn from comparatively short lengths or staples of filaments or threads composed of organic derivatives of cellulose presents serious difficulty. This is apparently due to the fact that the surfaces of such filaments or threads are quite smooth and therefore such filaments do not adhere to each other or intertwine sufficiently to form commercially satisfactory "spun" yarn. I have found that if such filaments or threads made for example of cellulose acetate have a finely divided material which is insoluble in the base material of the filaments or threads incorporated therein, their surfaces become sufficiently altered so that they more readily adhere to each other, and they then may be successfully associated together in a spinning operation to form commercially useful "spun" yarn.

30 In accordance with my invention I incorporate into filaments, threads or yarns made of organic derivatives of cellulose finely divided material that is not soluble in the organic derivative of cellulose. The filaments or threads so formed, while in the form of short lengths, staples or chappé, may then be subjected to a spinning operation, whereby the same are associated together to form "spun" yarn.

40 The filaments, threads or yarns which are made or treated in accordance with my invention may be made of any suitable organic derivative of cellulose such as organic esters of cellulose and cellulose ethers. Examples of such 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.

50 The filaments or yarns containing the organic derivative of cellulose may be prepared by dissolving the organic derivative of cellulose in a volatile solvent such as acetone and extruding such solution into an evaporative atmosphere, as in dry spinning, or into a precipitating bath as in wet spinning. While the greatest advantages of my invention accrue from the spinning of such fila-

ments or threads solely, they may be associated with fibres of other materials such as cotton, natural silk, wool or reconstituted cellulose and then spun.

As stated I incorporate into the filaments or yarns a finely divided material that is insoluble in the organic derivative of cellulose and is preferably insoluble in the spinning solution employed. This insoluble substance may be of inorganic or organic nature. Examples of inorganic materials are titanium dioxide, antimony oxide, the sulfate of barium, lead or calcium, barium phosphate, zinc oxide, the carbonate of zinc, calcium or magnesium, aluminum oxide, silicon dioxide, the borate of barium or calcium, tin oxide, tin phosphate as is formed in the weighting of natural silk, or silicates such as china clay or other clays, talc or mica. Examples of organic materials are diacetyl benzidine, diacetyl tolidine, dibenzoyl benzidine, naphthyl urea, anthraquinone, anthracene or suitable synthetic or natural resins. The organic material preferably is of a high melting point. Often it is advantageous to employ a mixture of inorganic and organic materials. It is preferable to choose such insoluble compound as does not dissolve in or react with the spinning solution or solvent therein or reagents with which the material comes in contact during its formation or in subsequent treatments, such as dyeing, to form undesired compounds.

The amount of insoluble material incorporated in the filaments, threads or yarns is such as to obtain the desired effect. Generally the amount of insoluble material employed will vary from less than 0.25 to 10% or more of the weight of the organic derivative of cellulose contained in the filaments or yarn. The exact amount depends upon the nature of the insoluble material and the nature of the spinning process to which the staple fibres are subjected.

100 In order to obtain the best results, the insoluble material should preferably be in finely divided form say from less than 0.1 to 15 microns in diameter. This fine size may be obtained by grinding the insoluble material either with water, an oil such as diethylene glycol or olive oil, or part of the spinning solution or the solvent used in the spinning solution in a ball mill and/or colloid mill.

110 In one form of my invention the finely divided insoluble material is added to the organic derivative of cellulose and/or to the dope or spinning solution containing the organic derivative

of cellulose to which may also be added diethylene glycol or oils such as heavy white mineral oil, olive oil, castor oil, Turkey red oil or fatty acids such as oleic acid. The spinning solution containing the pigment is then preferably thoroughly mixed and subjected to the usual filtration through a series of filters before spinning. However the addition of the pigment to the spinning solution at any stage of the filtration or even after filtration is not excluded.

In another form of my invention, the finely divided insoluble material is incorporated in the filaments, threads or yarns by treating the same after their formation, while in substantially continuous lengths in the form of hanks or other suitable packages, or after it has been cut into staple fibres, with a suspension of the finely divided insoluble material in the presence of a swelling agent or penetrating agent for the organic derivative of cellulose of which such filaments are composed and which causes the finely divided insoluble material to become disseminated throughout the filaments. Thus if the filaments are made of cellulose acetate, the swelling agent employed may be an aqueous solution of acetone, acetic acid, diacetone alcohol, thiocyanates of sodium, potassium or calcium etc., care being taken that the swelling agent chosen does not react deleteriously with the insoluble material. Less advantageously the use of the swelling agent may be omitted, when very finely divided materials such as titanium oxide are used.

In still another form of my invention, the filaments, yarns or threads are treated after their formation with a solution of a soluble metal salt in the presence of a swelling agent and subsequently treated with a solution of a salt or acid which forms an insoluble compound with the metal of the first salt. Thus the filaments or threads may be treated with a solution of barium thiocyanate, which acts as a swelling agent, and then treated with a solution of the soluble sulfate or sulfuric acid, whereupon the insoluble barium sulfate is precipitated within the filaments or threads.

In order to impart further improved spinning properties, the filaments or threads either while in continuous form or after they have been cut into short lengths or staples may be treated with a solution of magnesium chloride or other deliquescent or hygroscopic material, as is more fully described in my companion application No. 485,323 filed September 29, 1930.

The continuous filaments or threads are cut into staples or chappé of suitable length which may range from 0.75" to 20" or more in length.

The short lengths of filaments or threads may be subjected to any suitable spinning operation, such as is used for the spinning of natural silk, cotton or wool fibres to form threads by any of the well known systems such as the "cotton", the French, the "worsted", the "wool", the "spun silk", the Bradford system, etc. This spinning operation also includes the preliminary treatments necessary to present the filaments or fibres in the form required for the actual operation of twisting them into yarn.

Because of the presence of the finely divided insoluble material, the surface and/or contour of the filaments of organic derivative of cellulose are so altered that they more readily intertwine or adhere to each other so that they may be spun successfully. By my process, "spun" yarn formed solely from chappé or relatively short

lengths or staples of artificial filaments made of organic derivatives of cellulose such as cellulose acetate can be successfully manufactured on a commercial scale.

In order further to illustrate my invention but without being limited thereto, the following specific example is given.

#### Example

One (1) part by weight of an acetone soluble cellulose acetate is dissolved in three (3) parts by weight of acetone and to this there are added from five thousandths (0.005) to five hundredths (0.05) parts by weight of an insoluble material such as tin oxide, antimony oxide, titanium dioxide, etc. dispersed in a small quantity of water, acetone or a solution of cellulose acetate in acetone. This dispersion may be obtained by grinding the insoluble material in a colloid mill or ball mill until the particles have the desired size. The mass is thoroughly mixed and then filtered. The filtered solution is extruded through the orifices of a spinneret into a drying evaporative atmosphere. The filaments so formed are then cut, preferably as they leave the spinning machine, into staple fibres of about 1" in length. The staple fibres are then subjected to a spinning operation which includes opening, filling, dressing, spreading, drawing, roving and spinning.

If desired the finely ground insoluble material may be added to the solution of the cellulose acetate after it has been filtered or at any stage of the filtration. Likewise, if desired, an oil may be incorporated in the spinning solution.

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 my invention.

Having described my invention, what I desire to secure by Letters Patent is:

1. The method of forming "spun" yarn comprising extruding a solution of organic derivatives of cellulose containing finely divided insoluble material through orifices into a setting medium, treating the filaments thus formed with a hygroscopic substance, cutting the filaments as formed into comparatively short lengths and then associating said short lengths of said filaments together by twisting to form yarn.

2. The method of forming "spun" yarn comprising extruding a solution of cellulose acetate containing finely divided insoluble material through orifices into a setting medium, treating the filaments thus formed with a hygroscopic substance, cutting the filaments as formed into comparatively short lengths and then associating said short lengths of said filaments together by twisting to form yarn.

3. The method of forming "spun" yarn comprising extruding a solution of organic derivatives of cellulose containing finely divided insoluble material through orifices into a setting medium, treating the filaments thus formed with magnesium chloride, cutting the filaments as formed into comparatively short lengths and then associating said short lengths of said filaments together by twisting to form yarn.

4. The method of forming "spun" yarn comprising extruding a solution of cellulose acetate containing finely divided insoluble material through orifices into a setting medium, treating the filaments thus formed with magnesium chloride, cutting the filaments as formed into comparatively short lengths and then associating

said short lengths of said filaments together by twisting to form yarn.

5 5. Staple fibres consisting of short lengths of filaments of organic derivatives of cellulose containing finely divided insoluble material distributed therein and also having a hygroscopic substance on at least the surface thereof.

10 6. Staple fibres consisting of short lengths of filaments of organic derivatives of cellulose containing finely divided insoluble material distributed therein and also having magnesium chloride on at least the surface thereof.

7. Staple fibres consisting of short lengths of filaments of cellulose acetate containing finely divided insoluble material distributed therein and also having a hygroscopic substance on at least the surface thereof.

8. Staple fibres consisting of short lengths of filaments of cellulose acetate containing finely divided insoluble material distributed therein and also having magnesium chloride on at least the surface thereof.

CAMILLE DREYFUS.

15	90
20	95
25	100
30	105
35	110
40	115
45	120
50	125
55	130
60	135
65	140
70	145
75	150