

# UNITED STATES PATENT OFFICE.

CHARLES A. ERNST, OF LANSDOWNE, PENNSYLVANIA, ASSIGNOR TO SILAS W. PETTIT, OF PHILADELPHIA, PENNSYLVANIA.

## FORMING OF FILAMENTS OUT OF VISCOSE OR SIMILAR VISCOUS MATERIAL.

No. 896,715.

Specification of Letters Patent.

Patented Aug. 25, 1908.

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*To all whom it may concern:*

Be it known that I, CHARLES A. ERNST, a citizen of the United States, and a resident of Lansdowne, State of Pennsylvania, have  
5 invented certain new and useful Improvements in the Forming of Filaments Out of Viscose or Similar Viscous Material, of which the following is a full and exact description.

My invention relates to the method of  
10 forming filaments out of viscose or similar viscous substances and particularly to the treatment of the viscose itself, and to the production of filaments therefrom, and the object of my invention is to produce a vis-  
15 cose which will immediately coagulate into a filament when ejected from the spinneret into a suitable fixing or coagulating bath.

Further objects of my invention are to hold or retain the cellulose before spinning,  
20 dissolved in a solvent which contains less alkali than it has heretofore been thought practicable to employ.

Other objects of my invention will appear in the specification and claims below.

25 In order to obtain a smooth uniform solution or distention of cellulose sodium xanthate, sometimes called cellulose xanthate, which may be the product obtained by treating soda cellulose with carbon bisulfid  
30 in the manner referred to in my prior patent, No. 863,793, patented August 20, 1907, it has been found advantageous to dissolve the xanthate in a dilute solution of caustic soda. When a comparatively strong solution of  
35 caustic soda is used, a quick and complete dissolving or distention of the xanthate therein is effected, but the presence of any considerable amount of caustic soda, or a similar alkali, in the dissolved cellulose xanthate,  
40 renders the coagulation of the viscose, when ejected into a suitable bath through a spinneret, very difficult and slow. It is difficult, however, to properly dissolve the cellulose xanthate in a solution of caustic soda much  
45 weaker than a 5% solution. I have found that after the sodium cellulose xanthate or cellulose xanthate has been once dissolved in a caustic solution it will remain in solution or distention, even though a comparatively  
50 large amount of the alkaline solvent be neutralized. In other words, I have found that although a suitable amount of caustic soda is necessary to properly dissolve and first form a solution of the xanthate of cellulose

which will spin well, the said xanthate, when  
55 once dissolved, will remain, without coagulation, in a state of solution or distention in a medium which contains a considerably less amount of caustic soda than was originally required to dissolve it. Upon this  
60 principle I have based a method of making a viscose which has the advantage of being a perfect solution attained by the use of a solvent having a comparatively great alkalinity but in which the alkali will not prevent a  
65 rapid coagulation of the viscose in the setting, or neutralizing or coagulating solution.

In carrying out my invention, I dissolve the sodium cellulose xanthate or cellulose xanthate in a caustic soda solution, (as for in-  
70 stance a 5% solution, or stronger,) and after thoroughly mixing the same I obtain a perfectly formed solution or distention of the cellulose xanthate. I then add to the  
75 solution so formed, an acid, or an acid salt, or both to partly neutralize the caustic soda. When so treated the cellulose sodium xanthate will remain in a perfectly clear  
80 solution, or uniform distention for a day or more, according to the proportion of acid used, notwithstanding the fact that it is held  
85 in a medium or solvent which contains less caustic soda than was necessary to effect that uniform and complete solution, or distention of the xanthate. Although the cel-  
90 lulose xanthate solution is in a more unstable condition than it was before the acid was added, it remains in a solution sufficiently long to be conveniently handled and stored before spinning.

I have used acetic acid as an acid and sodium bisulfite as an acid salt for neutralizing the caustic soda, but any acid, or acid salt which does not decompose the xanthate may  
95 be used, as for instance, lactic acid, oxalic acid, sulfurous acid, etc. What I claim, then, is the use of an acid or an acid salt in sufficient quantities to partly neutralize the alkalinity of viscose, and thereby render it  
100 more susceptible of being rapidly and easily coagulated in a weak coagulating or neutralizing bath. It is, of course, not necessary to add the acid immediately after the viscose is made, but it may be added any time before  
105 the spinning takes place.

After the viscose has been formed, in the manner above described, it is ejected into a weak acid fixing or coagulating bath, the ac-



tion of which is to transform the liquid cellulose xanthate into solid filaments of cellulose xanthate. These filaments are wound upon spools as fast as they are formed, in any suitable manner, and they are then subjected to the action of a suitable fixing solution, as for instance a solution of sodium bisulfite, in which they are allowed to stand for a time sufficiently long to completely transform the filaments of coagulated viscose into filaments of cellulose or artificial silk.

While I have described one manner in which my invention may be carried out, I do not wish to be construed as limited to the exact process above set forth, for I may fully treat the xanthate of cellulose in the manner described in my previous patent, No. 863,793, August 20, 1907, before adding thereto the acid or acid salts. Thus after dissolving the xanthate of cellulose in a solution of caustic soda, I may add thereto a neutral salt such as sodium sulfite, the action of which is to check the further reaction of the carbon bisulfid upon the soda cellulose which enables me to store the viscose for a comparatively long period of time; then shortly before spinning, I may add thereto a sufficient quantity of the acid or the acid salt or both, to neutralize a part of the alkalinity of the solution and facilitate the rapid spinning of the filaments of viscose.

It is further obvious that, so far as my improved process goes, it is immaterial when the caustic soda is added to the viscose, for the viscose may be rendered strongly alkaline by using an excess of caustic soda during the mercerizing of the cellulose base. When thus produced, of course, the viscose would dissolve in a weaker solution of caustic soda than a 5% solution, but the excess of caustic soda would, similarly, bring the alkalinity of the solution up to the same degree as it would if the process were carried out in the manner I have previously described. In either manner the cellulose would be held suspended in a solution having a greater alkalinity than is desirable for spinning purposes and in carrying out my invention, I add to this alkaline solution a sufficient quantity of an acid to neutralize or partly neutralize the alkalinity of the same.

Having thus fully described my invention, what I claim and desire to protect by Letters Patent of the United States is:

1. The method of forming filaments out of viscose or similar viscous substance, which consists in dissolving the xanthate of cellulose in a solvent which is not neutral, partially neutralizing the reaction of the said solvent until the xanthate is retained in a more unstable, but nevertheless, perfect solution, and then forming a filament out of the viscose so formed by ejecting it in a minute stream into a coagulating medium.

2. The method of forming filaments out of viscose, which consists in dissolving cellulose xanthate in an alkaline solvent, then neutralizing a part of the alkalinity of said solvent without precipitating said xanthate and then spinning the same in a suitable neutralizing solution.

3. The method of forming filaments out of viscose, which consists in dissolving cellulose xanthate in caustic soda, then adding an acid to the viscose so formed to neutralize a part of the caustic soda, and then spinning the same in a weak acid bath.

4. The method of forming filaments out of viscose, which consists in distending the xanthate of cellulose in a comparatively strong alkaline solution, then neutralizing the greater part of the alkalinity of said solvent until the xanthate is held in a solution weaker than that which was originally required to produce it, and then spinning the viscose so formed by ejecting it in filaments in a weak neutralizing solution.

5. The method of forming filaments out of viscose, which consists in dissolving cellulose xanthate in a substantially 5% solution of caustic soda, neutralizing the greater part of the alkalinity of said solvent until the xanthate is in a solution weaker than was required originally to produce the same, and then spinning the viscose so formed by ejecting it into a weak acid bath.

6. The method of forming filaments out of viscose, or similar viscous substance, the step which consists in dissolving cellulose xanthate in an alkaline solvent, and then neutralizing a part of the alkalinity of the said solvent without precipitating the said xanthate.

7. The method of forming filaments out of viscose, which consists in dissolving cellulose xanthate in an alkaline solvent, adding thereto a neutral salt, then neutralizing a part of the alkalinity of said solvent without precipitating said xanthate, and then spinning the same in a suitable neutralizing solution.

8. The method of forming filaments out of viscose, which consists in dissolving cellulose xanthate in an alkaline solvent, then neutralizing a part of the alkalinity without precipitating said xanthate and then ejecting it in a minute stream into a coagulating medium.

9. The method of forming filaments out of viscose or similar viscous substances, which consists in dissolving the xanthate of cellulose in an involatile solvent which is not neutral in its reaction, partially neutralizing the reaction of said solvent without substantially affecting the chemical constituency of the said xanthate and then forming a filament out of said viscose by ejecting it in a minute stream into a coagulating medium.

10. In the method of forming filaments



out of viscose or similar viscous substances,  
the step which consists in dissolving the cellulose base in a solution which is not neutral  
and then partially neutralizing the reaction  
5 of the resulting solution without precipitating said base.

In witness whereof I have hereunto set my

hand this seventeenth day of October, A. D. 1907.

CHARLES A. ERNST.

Witnesses:

ALSTON B. MOULTON;  
ALEXANDER PARK.