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PRODUCTION OF ARTIFICIAL SILK

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My present application has to do with a new process for treating artificial filaments by the dry-spinning process.

One object of my invention is to spin fine cuprammonium filaments into a countercurrent vapor medium and so percipitate or harden them.

Other objects will become apparent from a study of the following specification.

The production of artificial films, filaments, threads, etc., from cuprammonium solutions is usually accomplished by extruding the products into a coagulating bath, for example, into water. One type of apparatus in regular use comprises a cylindrical or conical chamber through which the filaments and coagulating bath pass.

This type of treatment requires a large amount of the coagulating liquid, usually one kilogram of the filaments requires two cubic meters of coagulating bath. The water usually used must be as pure as possible, that is, must be as free as possible from impurities and substances which tend to harden it. Therefore, it is necessary, under the present processes, for large quantities of water to be mechanically and chemically treated. This involves much labor and expense. Further, the water is usually used at a temperature of from 30° to 50° C. This, in itself, involves a large expense.

Because of these expenses the present mode of precipitating artificial filaments from cuprammonium solutions makes the cost of the same too high.

It has already been proposed that the filaments be precipitated or coagulated by passing acidic, or heated gases, or steam through the spinning vessel in the same direction as the filaments. It has also been proposed that the action of this media be employed to stretch the filaments. This process requires the extraction of the ammonia from the spinning solution in order that a quick precipitation of the cellulosic filaments may be possible.

This process has not come into commercial use because, although the use of acidified steam gives a uniform filament, the presence of some ammonia causes them to become sticky. Attempts made to extract all of the ammonia prior to spinning have failed since such treatment tends to gel the solution.

One purpose of my present invention, therefore, is to produce filaments, through the gaseous or dry spinning process which are equal in quality to those obtained through the wet-spinning process.

Another purpose of my invention is to provide a suitable process for extruding cuprammonium solutions into countercurrent heated gases or steam. Should a considerable stretching be necessary it is advisable that heated neutral gases in which steam is present or water has been vaporized, be used.

One advantage arising from my process is that the threads are better precipitated without requiring a complete elimination of the ammonia from the spinning solution. This process also permits the concentration and economical recovery of the ammonia.

All of these advantages are lacking in the known processes where the gases flow in the same direction as the filaments. It would seem plausible that the use of the countercurrent principle with cuprammonium filaments would cause the action of surface friction on the fine filaments to react injuriously. My new process, therefore, has a surprising result in that the countercurrent flow of the gases not only does not injure the filaments but eliminates many disadvantages heretofore arising.

In my process the spinneret is arranged within a closed pipe-like container or chamber, and the large number of filaments drop therethrough under the force of gravity. Opposing their passage through the chamber a gaseous stream containing steam or vaporized water is introduced from below. The presence of this water vapor aids in the precipitation of the filaments. The precipitated filaments pass out from the bottom of the container or chamber. The copper may then be removed from the filaments by treatment on spools or in the form of cakes or skeins.

Example 1.—The spinning solution used contains 12% cellulose, 4% copper, and its ammonia content has been reduced to 5%. This solution is expressed through a spinneret containing 50 orifices of a size of 0.1 mm. into a closed chamber having an inlet below and an outlet above. The filaments are opposed by a countercurrent stream of 70% carbonic acid saturated with steam, the filaments being kept in contact with fresh, warmed carbonic acid vapors of this strength until the precipitation is complete. The filaments pass into an acid bath which closes the lower end of the chamber. This acid tends to bring the copper into a soluble form and complete the hardening of the filaments. Further treatment of the filaments may be according to any of the well-known methods.

Example 2.—A solution containing 8% of cellulose, 2.7% of copper and 4% of ammonia is ex-

pressed through a spinneret containing holes 0.5 mm. in width into a wooden cylinder, while, from below, a current of hot air mixed with water vapor is introduced. The action of this gaseous medium on the filaments is purely physical and the filaments gradually harden while being stretched through means of a mechanical stress applied. The precipitating medium, saturated with ammonia, is removed from the upper part of the container. Further treatment of the filaments may be by any of the well-known processes.

Having now fully set forth my invention as required by the patent statutes, what I desire to claim is:

1. In the manufacture of cuprammonium filaments, the step of precipitating the extruded filaments through the action of a counter current gaseous stream of heated carbonic acid.

2. In the manufacture of cuprammonium filaments, the step of precipitating the extruded filaments through the action of a counter current gaseous stream of heated carbonic acid of a 70% strength.

3. In the manufacture of cuprammonium filaments, the step of precipitating the extruded filaments through the action of a counter stream of heated carbonic acid of a 70% strength containing water vapor.

4. In the manufacture of cuprammonium filaments, the step of precipitating the extruded filaments through the action of a counter current steam of heated carbonic acid of a 70% strength containing steam.

5. In a process for producing cuprammonium filaments, the steps of passing the extruded filaments through a counter current heated gaseous medium and then through an acid bath before they contact with the outer air.

6. In the manufacture of fine denier filaments from cuprammonium solutions, the steps of making up a cuprammonium solution, extruding it through a spinning orifice from 0.1 to 0.5 mm. in diameter, into a counter current heated gaseous stream containing 70% of carbonic acid, and stretching the filaments while in this stream of gas.

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