

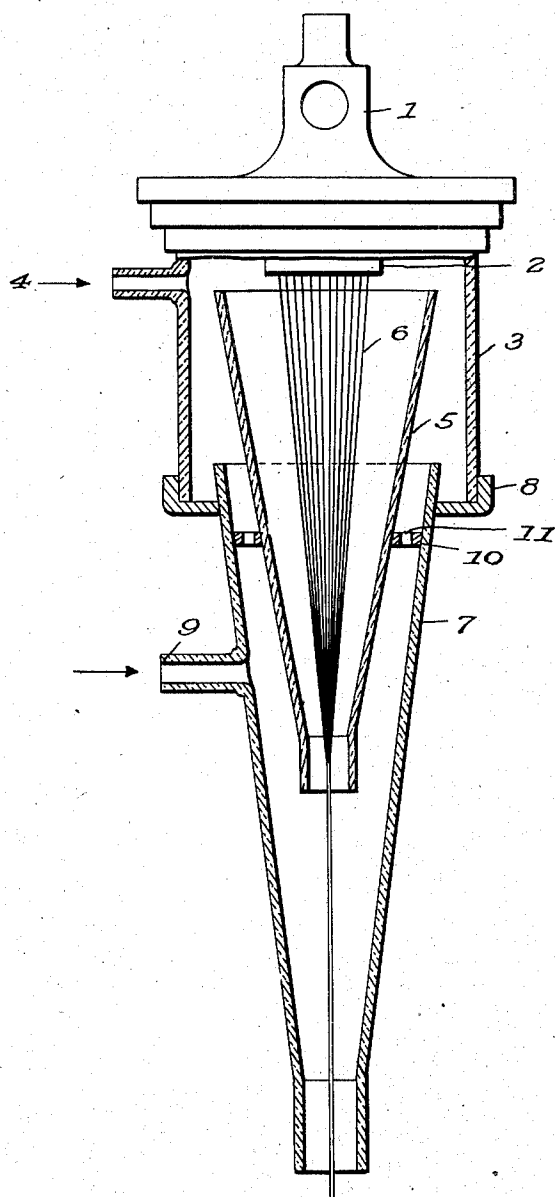
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PROCESS OF SPINNING ARTIFICIAL SILK

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PROCESS OF SPINNING ARTIFICIAL SILK

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The present invention relates to the manufacture of regenerated cellulose in the form of threads, etc., from cuprammonium cellulose solutions in accordance with the stretch spinning process.

In the manufacture of artificial threads, such as artificial silk, staple fibre and the like from cuprammonium cellulose solutions the threads are conventionally extruded through spinning nozzles, having openings of approximately from 0.8 to 1 mm. diameter, into funnels which are continuously supplied with soft wash water to coagulate the same. Hitherto it has only been possible to spin these threads at comparatively low spinning speeds and in most instances this speed has not exceeded 30 meters per minute. Furthermore, to spin at those speeds it has been necessary to employ comparatively large quantities of wash water for a satisfactory coagulation of the threads. Thus about 750 cc. of water are used per minute to spin a cuprammonium cellulose thread of about 120 denier. Additional disadvantages of the conventional cuprammonium spinning process are high costs of softening the wash water, the maintenance of this water before being used in the funnels at a temperature of about 35° C., and the precipitation of copper hydroxide, etc., in the lower part of the spinning funnel, these precipitations not only impairing the transparency of the funnel and in turn the control of the spinning process per se but also injuring the formed thread at the lower portion of the spinning funnel.

It is therefore the primary object of our invention to provide a process which greatly reduces the amounts of wash water necessary for the coagulation of threads produced from cuprammonium cellulose solutions.

Another object of our invention relates to a process permitting a lowering of the temperature of the wash water.

A third object is the provision of a process which prevents the precipitation of copper salts, etc., in the lower part of the spinning funnel.

A fourth object has to do with a process allowing acceleration of the spinning speeds heretofore used in the art, this acceleration resulting in a greater economy of the cuprammonium process.

These and other objects will become apparent to those skilled in the art from a study of the following specification. The drawing illustrates an apparatus capable of carrying out the present method.

Specifically in the drawing, numeral 1 denotes

a cuprammonium spinning cell having a spinneret 2 mounted therein through which cuprammonium cellulose solution is extruded. Coagulating fluid, such as pure water, is introduced into a glass cylindrical casing 3 surrounding the spinneret through conduit 4 and the flow of water is such that it will fill the casing and overflow into funnel 5, contacting the filaments 6 as they are drawn through the funnel.

A second funnel-like member 7 is positioned in the bottom wall 8 of the casing and is provided with a fluid inlet 9 through which salt solution is introduced into the space between the lower portion of the upper funnel 5 and the lower funnel 7. The upper funnel 5 is retained in spaced relation to the lower funnel 7 by means of a perforated ring or gasket 10 having openings 11 therein, so that the water introduced into the casing 3 flows downwardly through the lower funnel and in contact with the thread after it leaves the upper funnel. The salt solution introduced into the lower funnel stratifies in the coagulating fluid and, therefore, the spinning bath includes an upper stratum of substantially pure water and a lower stratum of a solution comprising water and a salt as will be hereinafter described in detail.

We have found by experimentation that the cuprammonium cellulose spinning process may be improved by using a stratified spinning bath in the spinning funnels, this bath being produced by continuously injecting into the lower portion of the spinning funnel a dilute solution of two or more chemically different salts, such as magnesium and ammonium compounds; provided the concentration of this solution is carefully controlled to avoid visible precipitations of copper and magnesium compounds in the funnel. For this purpose we may use a combination of any ammonium salt with any magnesium salt provided this combination does not result in precipitations by chemical interaction. Thus, for example, ammonium phosphate and ammonium arsenate cannot be used in accordance with the present invention. We have found that a solution containing magnesium sulfate and ammonium carbonate is very suitable for being injected into the lower portion of the spinning funnel. When this mixture is used, a portion of the magnesium sulfate is converted into colloidal magnesium hydroxide by the ammonium contained in the wash water, this wash water comprising the upper stratum of the bath contained in the funnel. The concentration of these salts and their ratios must be so selected that the magne-

sium hydroxide formed in the spinning funnel fails to cause a visible turbidity of the spinning water. By injecting such a solution into the lower portion of the spinning funnel in comparatively small amounts, such as for example, 80 cc. per minute, it is possible to reduce the amount of spinning water (conventionally about 750 cc. per minute) to approximately 400 cc. per minute without deleteriously affecting the spinning process. In addition, besides reducing the quantity of wash water, it is also possible to reduce the temperature thereof. Thus, the temperature of the wash water may be reduced from about 35° C. to about 26° C. without precipitation of copper salts, etc., in the lower part of the spinning funnel. These precipitations, as is well known to those skilled in the art, are very annoying in the continuous spinning of cuprammonium cellulose and hamper the visual control thereof.

We have found that any soluble magnesium and ammonium salt may be used in this process with the exception of ammonium phosphate and ammonium arsenate. Magnesium chloride, ammonium carbonate, ammonium sulfate and ammonium acetate are examples of compounds which may be used in the present process, although we wish to emphasize that these examples are merely given to illustrate our invention and that we do not wish to be limited to the use of these compounds and no others.

We have found by experimentation that it is necessary to inject these dilute salt mixtures into the lower portion of the spinning funnel with the formation of colloidal magnesium hydroxide, and we wish to emphasize that solutions of low concentration give the desired results. About 2.4 g. of magnesium sulfate per liter of water is generally sufficient while the concentration of ammonium carbonate, etc., may be lower than that of the magnesium sulfate.

We have also found that it is possible to partially produce the dilute salt mixture in the lower portion of the spinning funnel itself. This may be accomplished, for example, by preparing outside of the spinning funnel a specific solution of a magnesium salt, particularly of magnesium sulfate, and an acid or an acid salt. It is not necessary to feed the magnesium salt and the acid or acid salt to the spinning funnel in the form of finished mixture, since it suffices that the aforementioned salts and/or acids be present in the lower portion of the spinning funnel in such a concentration that the desired formation of magnesium hydroxide in the spinning funnel takes place to such an extent that no visible precipitation thereof occurs therein.

Example I.—A cuprammonium cellulose solution is spun through a spinning nozzle containing 90 holes of about 0.8 mm. diameter into a conventional funnel provided with a lower inlet for the magnesium salt mixture. Soft water of about 26° C. is introduced in conventional manner into an upper inlet of the funnel at the rate of about 400 cc. per minute. A salt solution of about 26° C. containing about 2.4 g. of magnesium sulfate and about 1.25 g. of ammonium carbonate per liter of water is injected into the lower inlet of the funnel at the rate of about 80 cc. per minute. The thread issuing at the lower portion of the spinning funnel is led in conventional manner over thread guides whereby the major portion of the spinning water separates therefrom. The thread is then continuously decoppered, wound at a speed of about 30 meters per minute and thereafter finished. The

artificial silk produced in this manner has a dry strength of about 200 g. per 100 denier and a wet strength of about 140 g. per 100 denier.

Example II.—The identical solution is spun with the identical arrangement as set forth in Example I at a spinning speed of 62 meters per minute. 400 cc. of water are injected per minute into the upper part of the funnel, this water having a temperature of about 30° C. A dilute solution of magnesium sulfate and ammonium carbonate is injected at a speed of 80 cc. per minute into the lower portion of the funnel. This solution contains 2.5 g. of magnesium sulfate and 1.25 g. of ammonium carbonate per liter.

Example III.—Cuprammonium cellulose is spun following Example I, at a speed of 31 meters per minute with the exception that the injected solution contains 2.5 g. of magnesium sulfate and 1.87 g. of ammonium acetate per liter.

Example IV.—Cuprammonium cellulose is spun in accordance with the process set forth in Example I, at a speed of 31 meters per minute with the exception that the injected solution contains 2.5 g. of magnesium sulfate and 3.7 g. of potassium bisulfate per liter of water.

Example V.—Cuprammonium cellulose is spun, following Example I, at a speed of 31 meters per minute and a temperature of about 28° C., with the exception that the injected liquid contains 2.5 g. of magnesium sulfate and 1.8 g. of ammonium sulfate per liter of water.

Example VI.—Cuprammonium cellulose is spun, following Example I, at a speed of about 31 meters per minute, with the exception that a liquid is injected containing 2.5 g. of magnesium sulfate and 1.33 g. of sulfuric acid per liter of water.

While the aforementioned examples set forth the spinning of threads of 120 denier at a speed of about from 30 to 62 meters per minute, a bath consumption of 400 to 480 cc. per minute, and an injection of salt solution containing about 0.04 to 0.05% of magnesium sulfate, calculated on the total consumption of spinning water it must be emphasized that titers, other spinning water quantities and differently formed spinning funnels, etc., require a change of concentration of the injected salt solutions. These concentrations and amounts of injected salt solutions must be predetermined by experiments.

Having thus described our invention as required by the Patent Statutes what we claim is:

1. In the process of producing artificial silk by the stretch-spinning process, the step which comprises continuously extruding at a speed of about 30 meters per minute a cuprammonium cellulose solution through a spinneret having approximately 90 openings of about 0.8 mm. diameter each, into the top of a funnel provided with an upper and a lower inlet, said upper inlet being furnished with about 400 cc. of water per minute of about 26° C., and said lower inlet being furnished with about 80 cc. per minute of water of about 26° C. containing about 0.24% of magnesium sulfate and about 0.125% of ammonium carbonate by weight.

2. In the process of producing artificial silk by the stretch-spinning process, the step which comprises continuously extruding at a speed of about 62 meters per minute a cuprammonium cellulose solution through a spinneret having approximately 90 openings of about 0.8 mm. diameter each, into the top of a funnel provided with

an upper and a lower inlet, said upper inlet being furnished with about 400 cc. of water per minute of about 30° C., and said lower inlet being furnished with about 80 cc. per minute of water of about 30° C. containing about 0.25% of magnesium sulfate and about 0.18% of ammonium sulfate by weight.

3. In the process of producing artificial silk by continuously extruding with stretching a cuprammonium cellulose solution into a funnel, the step which comprises extruding a cuprammonium cellulose solution through a spinneret into the top of a funnel provided with an upper and a lower inlet, continuously supplying water to said extruded solution through said upper inlet and continuously supplying a mixture comprising an aqueous solution of an ammonium salt and an aqueous solution of a magnesium salt to said extruded solution through said lower inlet whereby the formation of a visible precipitate of copper salts is prevented, the am-

monium salt used being chemically non-reactive with said magnesium salt to prevent formation of a visible precipitate.

4. In the process of producing artificial silk by continuously extruding with stretching a cuprammonium cellulose solution through a spinneret into the top of a funnel provided with an upper and a lower inlet, continuously supplying water to said extruded solution through said upper inlet and continuously supplying a mixture comprising an aqueous solution of an ammonium salt and an aqueous solution of an inorganic magnesium salt to said extruded solution through said lower inlet whereby the formation of a visible precipitate of copper salts is prevented, the ammonium salt used being chemically non-reactive with said magnesium salt to prevent formation of a visible precipitate.

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