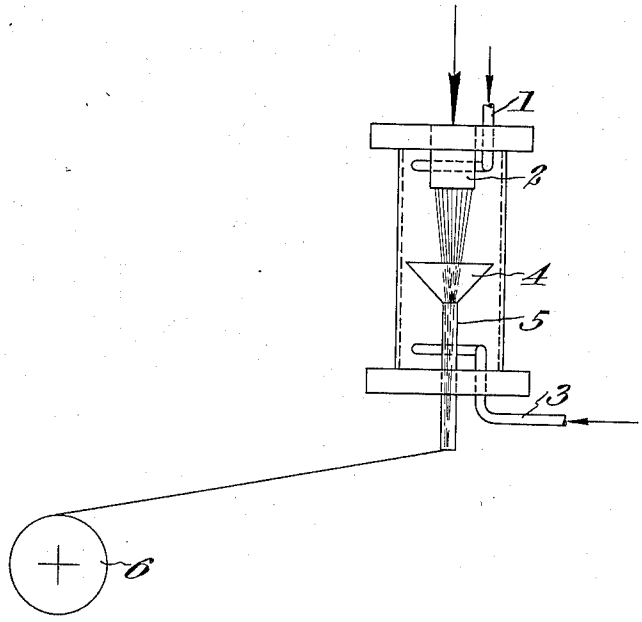
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E. HUBERT

ARTIFICIAL SILK AND PROCESS OF MAKING SAME

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ARTIFICIAL SILK AND PROCESS OF MAKING SAME.

Application filed December 23, 1922. Serial No. 608,747.

To all whom it may concern:

Be it known that I, EMIL HUBERT, a citizen of Germany, residing at Elberfeld, Roonstrasse 28, in the State of Prussia, Germany, have invented new and useful Improvements in Artificial Silks and Processes of Making Same, of which the following is a specification.

The United States Letter Patent No. 710,819 describes a process for the manufacture of artificial silk in which comparatively thick filaments formed from an ammoniacal copper oxide solution of cellulose are led through two superposed layers of liquid at differing temperatures, the filaments being drawn out mainly in the slowly acting precipitating layer which has a temperature of 40–50° C. and precipitated and hardened in the rapidly precipitating layer which has a temperature of 95–100° C.

The solutions of many valuable materials cannot be treated by this process even if the filaments so produced are further treated; this is especially the case for such solutions which on the one hand do not allow comparatively thick filaments to be drawn out to very fine filaments at the lower temperature employed, but which possess such property only at a high temperature, and which on the other hand at the requisite temperature of the precipitating baths are precipitated as milk white, tender filaments which are therefore worthless.

Heretofore it has been impossible to utilize the solutions of such materials in the manufacture of artificial silk where it was desired that individual filaments should have a fineness of 1–2 deniers, it has only been possible if at all to manufacture filaments of a stoutness of 20 deniers and more unless capillary nozzles 0.1 mm. and less in width were employed. The large range of cellulose esters and ethers belong to this class.

By this invention filaments having the fineness of cocoon silk can be made from the solutions of such materials with the employment of comparatively wide orifices by leading the comparatively thick filaments through two layers of liquid of which the first is hotter than the second.

It will be observed that this is the direct opposite to the known processes of drawing filaments in which the comparatively thick filament is led through two liquid layers of which the first is colder than the second and so in this process, to employ the phraseology of the Patent No. 710,819, on the contrary first employ a rapidly acting precipitating medium and then a slowly acting precipitating medium. This inversion alone renders it possible to manufacture the finest artificial silk from solutions of such substances. The column of the liquid layers of the type of liquid employed depends on the character of the substances used; the temperature of the first layer of liquid must, however, be such that filaments are not fractured by drawing them out to the required fineness, while its column should be so short that during the drawing out process the filaments are not largely precipitated and so enter the second layer in a semi-solid state where they gradually harden by prolonged contact with the colder liquid.

The drawing apparatus employed may be those now in use.

If due to a difference in specific gravity between the solution and the precipitating liquids, it is necessary to squirt the solution upwards, the lower layer or liquid must be hotter and have a higher specific gravity than the upper layer of liquid; when on the contrary the filaments are drawn in a downward direction out of nozzles, the upper layer is hotter and has the lower specific gravity. The first layer may be brought to and maintained at the required temperature by a heating worm, or fresh hot liquid may be added slowly to the bulk of the hot liquid. Preferably the second layer circulates continuously to impart to it a movement in the direction of the issue of the filaments. When necessary means may be provided for aiding the maintenance of the separation of the two layers of liquid and for preventing eddy currents at the surfaces of contact.

Example.—A cellulose acetate soluble in acetone is dissolved in dry pyridine bases to form a homogeneous 20–25% solution which after filtration is led to the nozzle of an apparatus such, for example, as that described in British Letters Patent No. 14655 of 1907. Apparatus suitable for carrying out the process is illustrated diagrammatically in the accompanying drawing. In the nozzles 2 there may be 50 holes of 0.6

mm. each. After the apparatus has been filled with cold water, hot water at 60° C. is delivered through the upper inlet 1 at the rate, for example, of 30–50 c. cms. per minute and contacts with the thick filaments issuing from the nozzles 2 in a downward direction such layer being 3–5 cm. long and possessing a temperature of 45–55° C. Water at 10–15° C. is delivered through the lower inlet 3. If the funnel 4 together with its leg 5 is 25–30 cm. long then the inlet of 300–500 c. cms. of water per minute suffices for a draw velocity of 40 metres per minute.

In the hot water zone the comparatively thick filaments passing into the funnel 4 are first stretched by gravity alone but subsequently they are drawn out by the pull of the water flowing in the leg 5 of the funnel and by the winding apparatus 6 to the fineness desired, and enter the cold water zone in a semi-solid and viscous condition; the filaments solidify in this zone and when drawn off by the winding apparatus 6 have a glossy appearance. The winding apparatus 6 may, if desired, for the purpose of removing any traces of pyridine from the filaments, rotate in the air or in a bath of water or dilute acid.

When dry, the filaments are in a condition to undergo any further treatment and each individual filament may have a fineness of 2–3 deniers. When stouter filaments are made, they may often, to commence with, appear to have an opal gloss, but this disappears on drying. Although in this example the difference in specific gravity between hot and cold water is small, yet after a few minutes there is a definite line of separation between the liquid layers. In order to enhance the drawing properties of a solution of cellulose acetate in pyridine, small quantities of suitable substances may be added; thus the addition of 10–30% acetylenetetrachloride $C_2H_2Cl_4$ relative to acetate renders the solution of higher specific gravity and more easily drawn, the individual filaments coming away from the nozzle more easily, thus allowing finer filaments to be obtained.

The process is not limited to the substances described and for example viscose and an ammoniacal copper oxide solution of cellulose as described in the Patent 710,819 may with advantage be employed.

I claim:—

The manufacture of artificial silk by a drawing process of the type employing two superposed liquids at different temperatures and of different specific gravities, in which comparatively thick filaments pass first into the hotter liquid where they are drawn and then into the colder liquid, substantially as described.

In testimony whereof I have hereunto set my hand.

EMIL HUBERT.