

UNITED STATES PATENT OFFICE.

ALBERT LECŒUR, OF ROUEN, FRANCE.

MANUFACTURE OF THREADS, FILAMENTS, OR BANDS OF CELLULOSE.

967,397.

Specification of Letters Patent. Patented Aug. 16, 1910.

No Drawing.

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To all whom it may concern:

Be it known that I, ALBERT LECŒUR, of 98 Avenue du Montriboudet, Rouen, Seine Inferieure, France, manufacturer, have invented certain new and useful Improvements in the Manufacture of Threads, Filaments, or Bands of Cellulose, of which the following is a specification.

This invention relates to the manufacture of artificial silk or other threads or filaments or bands of cellulose from cupro-ammoniacal solutions of cellulose.

In the manufacture of artificial silk from solutions of cellulose in cupro-ammoniacal liquors, the baths usually employed contain mineral acids (hydrochloric, sulfuric or nitric) or organic acids (formic, acetic) as precipitating agents, that is to say, when the cellulose solution is pressed through capillary orifices into these acids, the cellulose is regenerated and the ammoniacal and metallic constituents of the solvent converted into salts. I have however found that the cellulose threads thus precipitated have a milky, opaque aspect and are not strong. The employment of solutions of caustic alkalies for like purposes has also been described, and on substitution of caustic soda for acid in the baths, the threads of sodic cupro-cellulose obtained are supple and transparent and are stronger. But the caustic lyes, although applicable to the manufacture of coarser threads, such as artificial horsehair, are not suitable for the manufacture of artificial silk. For the latter manufacture the employment of soda or potash with subsequent acidification is unsuccessful because when caustic alkalies are used as precipitating or coagulating agents, capillary orifices cannot be used of sufficiently small diameter to produce a fine silk or even a silk of medium fineness (titre). I have discovered that the best results can be obtained with a solution of bisulfate of the alkalies. These acid salts possess a very great hydrolytic power particularly suitable to the coagulation of the thread and conversion of the ammonia and copper into dialyzable salts.

In order to carry out the invention I may prepare a solution of bisulfate of soda or potash by dissolving the requisite quantity of commercial bisulfate in water, or by adding the hydroxids, carbonates or other salts of sodium or potassium to a solution of sulfuric acid in water, the solution being

made of sufficient strength that the bath thus prepared possesses the power at a suitable temperature of instantly coagulating solutions of cellulose. Thus for the manufacture of artificial silk from the solutions of colloidal cuproammonium cellulose manufactured in accordance with British Letters Patent No. 16442 of 1906, a bath containing a saturated solution of bisulfate of sodium equal in degree of acidity to 30 per cent. sulfuric acid, gives at a temperature slightly above normal most satisfactory results. On passing from the first bisulfate bath the cellulose thread filament or band is preferably caught up on a revolving bobbin which is immersed in a second and more dilute solution of bisulfate. The threads or the like thus formed are supple, elastic and exceptionally strong and when submitted to the usual treatments of washing, bleaching and drying furnish an artificial silk of excellent quality.

In the manufacture of coarser threads, such as artificial horsehair, bisulfate of sodium or potassium may also be used in the first bath, but it is preferable to employ a more dilute solution, of about 2 or 3 per cent., and a second bath of caustic soda or potash, instead of a second bath of bisulfate.

In addition to the other advantages of solutions of bisulfate of sodium or potassium as precipitating baths, they possess an important economic advantage as they can be obtained at a very cheap price as by-products of other manufactures.

What I claim and desire to secure by Letters Patent is:—

1. A process for the manufacture of cellulose threads, filaments or bands, consisting in forcing a solution of cellulose in a cupro-ammoniacal liquor through orifices into a solution of bisulfate of an alkali.

2. A process for the manufacture of cellulose threads, filaments or bands, consisting in forcing a solution of cellulose in a cupro-ammoniacal liquor through orifices into a solution of bisulfate of soda.

3. A process for the manufacture of cellulose threads, filaments or bands, consisting in forcing a solution of cellulose in a cupro-ammoniacal liquor through orifices into a saturated solution of bisulfate of an alkali.

4. A process for the manufacture of cellulose threads, filaments or bands, consisting in forcing a solution of cellulose in a cupro-ammoniacal liquor through orifices into a

saturated solution of alkali bisulfate equal in acidity to about 30 per cent. sulfuric acid.

5. A process for the manufacture of cellulose threads, filaments or bands, consisting in forcing a solution of cellulose in a cupro-ammoniacal liquor through orifices into a saturated solution of bisulfate of alkali and subsequently passing the precipitated cellulose material into a more dilute solution of caustic soda.

6. A process for the manufacture of cellulose threads, filaments or bands, consisting

in forcing a solution of cellulose in a cupro-ammoniacal liquor through orifices into a solution of bisulfate of alkali, and passing the precipitated cellulose material into a second solution of alkali.

In witness whereof, I have hereunto signed my name in the presence of two subscribing witnesses.

ALBERT LECŒUR.

Witnesses:

OSCAR MALMROS,
PAUL RUDOLF.