UNITED STATES PATENT OFFICE.

ALBERT LECOEUR, OF ROUEN, AND PAUL RUDOLF, OF ST. AUBIN-JOUXTE-BOULLENG, FRANCE, ASSIGNORS TO LE CRINOID SOCIÉTÉ ANONYME, OF ROUEN, FRANCE.

MANUFACTURE OF THREADS FROM CUPRO-AMMONIACAL SOLUTIONS OF CELLULOSE.

980,294. Specification of Letters Patent. Patented Jan. 3, 1911.

No Drawing. Application filed June 25, 1910. Serial No. 568,839.

To all whom it may concern:

Be it known that we, ALBERT LECOEUR, of 98 Avenue du Mont Riboudet, Rouen, France, manufacturer, and PAUL RUDOLF, of Rue Raspail, St. Aubin-jouxte-Boulleng, France, chemist, have invented certain new and useful Improvements in the Manufacture of Threads From Cupro-Ammoniacal Solutions of Cellulose, of which the following is a specification.

This invention relates to improvements in the manufacture of threads from cupro-ammoniacal solutions of cellulose, and more particularly to improvements in or relating to alkaline precipitating baths employed in the manufacture of threads from cupro-ammoniacal solutions of cellulose, whereby the solidity of the thread may be improved, while the efficiency of the precipitating bath is increased and prolonged and the recovery of the copper from the bath is facilitated.

As the cupro-ammoniacal solution of cellulose is pressed through the spinning capsules into the alkaline precipitating bath and formed into threads, the bath gradually acquires a bluish color from the presence of copper salts. In a short time the bath becomes so colored that it is almost impossible to distinguish the blue threads in the same, and it becomes so difficult for the workmen to observe any broken threads and re-attach the broken ends to the bobbin that it is found necessary to recharge the bath with fresh uncolored liquor long before the bath becomes exhausted. To meet this difficulty it has been proposed to add to the alkaline precipitating bath glucose, saccharose, or analogous substances (viz:—such alcohols or alcohols with ketone or aldehyde groups as are adapted to form loosely combined compounds with bases), which at an elevated temperature reduce the blue copper salts in solution to metallic copper or red oxid of copper, which forms a reddish brown deposit against which the blue threads can be readily distinguished.

We have discovered that the reduction of the blue copper salts in solution is effected more economically and at a lower temperature by the addition of a dilute solution of formic aldehyde, as its reducing action is so energetic that a very small addition of formic aldehyde to the precipitating bath suffices for the complete reduction at comparatively low temperature of the copper salts which pass into solution in the alkaline bath, while an excess of formic aldehyde has a particularly favorable influence on the quality of the thread, rendering it more solid and resistant to strain than the thread obtained from alkaline glucose baths. We have found, for instance, that in a concentrated alkaline bath such as a bath of caustic soda and carbonate of soda described in British Patent No. 21,191 of 1908, used at a temperature of 40° C. or thereabout, the presence of even 1.5 per cent. of the commercial 40 per cent. formaldehyde solution in the precipitating bath suffices for the immediate reduction of the copper salts which pass into solution during formation of the thread, and causes the copper to deposit as metal or lower oxid at the bottom of the bath, from which it can be easily recovered and again used in the manufacture. With the addition of 5 per cent. formic aldehyded solution to the bath (*i. e.*, 2 per cent. formic aldehyde) at a temperature of 30° to 40° C., the coagulation of the thread is sensibly improved, and after washing and drying, the thread is more resistant to strain and less susceptible to moisture than thread which has been spun in a bath to which no formic aldehyde has been added.

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

1. Process for the manufacture of threads from cupro-ammoniacal solutions of cellulose, comprising precipitation of the threads in an alkaline bath containing formic aldehyde.

2. Process for the manufacture of threads from cupro-ammoniacal solutions of cellulose, comprising precipitation of the threads in a concentrated alkaline bath, as a bath of caustic soda and carbonate of soda, containing about 2 per cent. formic aldehyde, substantially as specified.

In witness whereof, we have hereunto signed our names in the presence of two subscribing witnesses.

ALBERT LECOEUR.
PAUL RUDOLF.

Witnesses:
L. PAPILLON,
MAURICE D'ANJOU.