

UNITED STATES PATENT OFFICE.

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PROCESS FOR OBTAINING THREADS OF ARTIFICIAL SILK, FILMS, AND OTHER OBJECTS.

1,214,931.

Specification of Letters Patent.

Patented Feb. 6, 1917.

No Drawing.

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To all whom it may concern:

Be it known that we, LEON LEDUC, engineer, a subject of the French Republic, and resident of Couture St. Germain, Belgium, and HENRI JACQUEMIN, engineer, a subject of the King of the Belgians, and resident of Maransart, Belgium, have invented a certain new and useful Process for Obtaining Threads of Artificial Silk, Films, and other Objects, of which the following is a specification.

Cross and Bevan have shown in the year 1893 that sodium chlorid precipitates the xanthogenate of alkali cellulose from aqueous solution or viscose, but without decomposing it and have based on this fact a process for the purification of raw viscose. Although this property has been known for such a long time, no inventor has been able to utilize sodium chlorid even with the addition of an acid either strong or weak so as to precipitate viscose as a substance suitable for the manufacture of threads of artificial silk, of photographic films or other similar object of regenerated cellulose.

Patents relating to precipitation baths are however numerous: the chief of them claim the ammoniacal salts, sulfuric acid with the addition of a salt, or acid salts such as the bi-sulfates. The others relate to the addition to these baths of accessory substances such as glucose, the sulfites, etc., for different purposes.

The object of the present invention is a precipitation bath composed of a salt other than the ammoniacal salts and of a strong mineral acid other than sulfuric acid, by which a relatively slow precipitation is insured and a slow regeneration of the cellulose.

We have found a mixture of 9 liters of a solution of sodium chlorid (NaCl) saturated at ordinary temperature and one liter of concentrated commercial hydrochloric acid, which mixture consequently contains 3 to 4% free HCl, to be particularly suitable.

The precipitation bath should only be

slightly heated. The relatively low temperature prevents the violent liberation of gas (H_2S , CO_2 , and the like) and thus we obtain a very smooth, lustrous and solid fiber.

We have also found that it is not indispensable to subject the raw viscose to a preliminary purifying process; it should however have a suitable composition, although this may vary within wide limits.

We may mention as typical, viscose containing 60 to 70 kilos of NaOH to 100 kilos of cellulose and 10 to 15 kilos of cellulose in 100 kilos of viscose. When the operation is only carried on in a bath composed as mentioned above of sodium chlorid and hydrochloric acid which amply suffices for the coagulation process, the accessory products of the viscose decomposed by the coagulation bath are partly deposited on the thread giving it a brick-red tint which further increases in intensity if the thread is left in the open air for some time.

In order consequently to avoid these impurities which have no other disadvantage than to render the twisting a little less easy, we have found it advantageous to pass the thread through a second bath of the same composition as the first.

We are assured by proceeding in this manner that in spite of the gradual weakening of the first bath, the thread will always be suitably coagulated before arriving at the bobbin, at the reel or other apparatus on which it is wound.

This second bath is not weakened, and is only slightly soiled; it can therefore finally serve as the first bath. The latter may also moreover be regenerated by the addition of hydrochloric acid after filtration or decantation of the impurities.

What we claim and desire to secure by Letters Patent of the United States is:—

1. A process of forming filaments comprising subjecting viscose to a precipitation bath composed of a solution of sodium chlorid with the addition of hydrochloric acid, substantially as described.

2. A process of forming filaments comprising subjecting viscose to a precipitation bath composed of a solution of sodium chlorid and hydrochloric acid in the proportion of 5 5 liters of a concentrated solution of sodium chlorid to 1 liter of concentrated commercial hydrochloric acid containing about 4 per cent. hydrochloric acid.

In witness whereof we have hereunto set our hands in the présence of two witnesses. 10

LEON LEDUC.
HENRI JACQUEMIN.

Witnesses:

CHAS. ROY NASMITH,
CHARLES A. JOHNSON.