

# UNITED STATES PATENT OFFICE.

ERNST BERL AND MAX ISLER, OF TUBIZE, NEAR BRUSSELS, BELGIUM.

## PROCESS FOR SPINNING SOLUTIONS OF NITROCELLULOSE.

1,188,718.

Specification of Letters Patent. Patented June 27, 1916.

No Drawing.

Application filed June 6, 1914. Serial No. 843,556.

*To all whom it may concern:*

Be it known that we, ERNEST BERL, a subject of the Emperor of Austria-Hungary, and MAX ISLER, a citizen of Switzerland, residing at Tubize near Brussels, in the Kingdom of Belgium, have invented certain new and useful Improvements in Processes for Spinning Solutions of Nitrocellulose, of which the following is a specification.

The processes which have hitherto been used for spinning solutions of nitrocellulose for the production of artificial silk, lustrous threads, artificial horse-hair, artificial straw, films, bands or the like from a collodion of nitrocellulose by means of what is termed the "wet-spinning process" have very important inconveniences. In the first place, it is necessary to employ dry nitrocellulose to secure its solution in the required solvents. It has been found, for instance, that if it is intended to spin in water a collodion which has been prepared with wet nitrocellulose as commonly used in the dry spinning process, the products which are finally obtained are opaque, unelastic and possess only a very low industrial value. The drying of the nitrocellulose, however, which has been considered hitherto as necessary, requires a very great stability of the nitrocellulose in order to prevent the accidents due to the spontaneous decomposition of the nitrocellulose. Notwithstanding the said precautions, however, the danger of working with dry nitrocellulose remains very great. On the other hand, as it is well known, it is advisable, in the wet-spinning process, to substitute certain mixtures of solvents, which are relatively heavy, as for example a mixture comprising methyl alcohol, ethyl alcohol and a little proportion of ether to the usual and cheaper mixtures of ether and alcohol originally used.

Now it has been found that the above stated inconveniences of the wet spinning process can be completely avoided if use is made of ordinary wet nitrocellulose such as that dried in a centrifugal machine and if said nitrocellulose, containing from 20 to 30% of water, is dissolved in a mixture of ether and alcohol, the usual proportions of the mixture of 60 parts of ether and 40 parts of alcohol being replaced by a mixture in which alcohol prevails for example a mixture containing from 40 to 50 parts of ether and 60 to 50 parts of alcohol. Fur-

ther it has been found that very transparent and elastic threads, having an absolutely regular and uniform section can be obtained if, instead of the usual precipitating liquid of pure water, a precipitating liquid is employed composed of diluted alcohol conveniently heated, said liquid comprising preferably from 25 to 50 volumes of alcohol for 100 volumes of water.

It has been further found that the expulsion of the ether from the products, after coagulation, can be effected in particularly good conditions if the precipitating or coagulating liquid is maintained at a temperature corresponding approximately to the boiling temperature of the ether, consequently at about 35.5° C. If said conditions are observed it is possible to obtain transparent threads (the transparency of which may be compared with the transparency of glass) having a circular and regular section, said threads being produced at a spinning velocity which is not less than the velocity which is obtained in the dry-spinning process and which attains a rate of 40 to 50 m. per minute. It will be evident that by conducting the thread during a sufficiently long time through the coagulating liquid, the main portion of the ether will be recovered; further approximately the total quantity of alcohol can be recovered if the products are subsequently treated with water.

We are aware that the use of concentrated alcohol is already known for the coagulation of threads obtained from solutions of nitrocellulose, said alcohol having been used in order to compensate the solvent capacity of the precipitating solution for the nitrocellulose. In this known process, however, in order that the precipitating solution be able to act rapidly, it is necessary to use alcohol from 85 to 90%. If it was proposed to use alcoholic solutions of such a concentration in the new process as above described, in which the spinning velocity stands above 40 m. per minute, the threads would be adhesive and the products obtained, specially artificial silk, would be absolutely useless in practice. Said inconveniences are avoided when it is worked according to the above described process by using an alcoholic solution containing only from 25 to 50% of alcohol and having a temperature less than the temperature of ebullition of the ether. A high velocity of spinning can thus be used

and the artificial silk which is produced, is particularly soft and nonadhesive. A further advantage of the new process consists in that the recuperation of the solvent can  
5 be very easily carried out. The possibility of using wet nitrocellulose also constitutes a great advantage in view of the suppression of danger in the manufacture.

Having thus described our invention, what  
10 we claim is:

1. A process for the spinning of solutions of nitrocellulose for the manufacture of artificial silk, lustrous threads, artificial horse-  
hair and straw, films, bands and the like,  
15 said process consisting in preparing a collodion with a nitrocellulose containing from 20 to 30% of water, then spinning said collodion and then coagulating said collodion, with alcoholic solutions containing from 25  
20 to 50% vol. of alcohol, said coagulation be-

ing effected at a temperature less than the temperature of ebullition of the ether.

2. A process for the manufacture of artificial silk, lustrous threads, artificial horse-  
hair and straw, films, bands, and the like, 25  
said process consisting in dissolving a nitrocellulose containing from 20 to 30% of water in a mixture of ether and alcohol in which alcohol prevails, then spinning said solution and finally passing the product in an alco- 30  
holic solution containing from 25 to 50% vol. of alcohol the temperature of said solution being maintained at about 35.5° C.

In testimony whereof we have affixed our signatures in presence of two witnesses.

ERNEST BERL.  
MAX ISLER.

Witnesses:

JACQUES BEDE,  
CHAS. ROY NASMITH.