

## UNITED STATES PATENT OFFICE

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PROCESS FOR THE MANUFACTURE OF  
NITRO-CELLULOSES

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This invention relates to a process for the manufacture of nitro-celluloses from chemical wood pulp.

In the nitration of cellulose use has hitherto been made of nitrating acids, consisting of sulphuric acid, nitric acid and water, in varying proportions.

It has now been ascertained that substantial advantages are obtained by using a nitrating acid containing in an amount corresponding substantially to about one fifth of the weight of the wood pulp chlorine, such as can be obtained for example by adding small quantities of hydrochloric acid to a normal nitrating acid mixture.

In this manner the advantage is obtained in the first place that the product obtained is of a lighter color, which is particularly important in the nitration of less pure raw materials. Moreover the process of the present invention enables the viscosity of the solutions made from the resulting nitrocelluloses to be varied within certain limits, in a simple manner.

It is known that wood pulps with the usual resin content, of for example 0.8–1%, when nitrated with the aid of the hitherto normal nitrating acids consisting of nitric acid, sulphuric acid and water, furnish products having a more or less pronounced tinge of yellow, and that even when these products are made into other products, such as for example lacquers, plastic masses (celluloid) and the like, such products are not completely white. Some grades of cotton that are not a pure white also behave in similar fashion. However, when white cotton is used as the originating material colorless products can be obtained. It is true that lighter products have been obtained by removing the resin from the raw cellulose prior to nitration, but apart from the fact that the effect thus achieved was often still incomplete, the additional cost of the resin extraction renders the process commercially impracticable.

In carrying out the invention, tests made have shown that the viscosity of the solutions made from the resulting nitrocellulose falls as the proportion of chlorine contained in the nitrating acid rises, so that it is possible to regulate the viscosity of the solutions to be made from the nitrocellulose in simple manner within certain limits. In particular it is easily possible in accordance with the invention to make collodion cottons direct, these being directly utilizable as lacquer cottons owing to the low viscosity of their solutions, whereas a collodion cotton furnishing highly viscous solutions is obtained without the addition of the hydrochloric acid, and the vis-

cosity can only be reduced to the required extent by subsequent treatment in an autoclave.

The process and the particular advantages arising therefrom will hereinafter be illustrated with the aid of the following examples:—

*Example I*

5 kgs. of a bleached pine wood cellulose with a resin content of 0.8% were nitrated for 30 minutes at a temperature of 30° C. with 95 kgs. of an acid mixture consisting of 60% H<sub>2</sub>SO<sub>4</sub>, 25% HNO<sub>3</sub> and 15% H<sub>2</sub>O. After the usual washing and ripening, there was obtained a collodion cotton containing 11% of nitrogen having a distinct yellow tinge, and a viscosity of 203 taking acetone as unity (measured in a 2% acetone solution).

When the treatment was carried out in the same manner but with the addition of 3 litres of hydrochloric acid (specific gravity 1.19) to the above-specified quantity of nitrating acid, there was obtained a collodion cotton containing 10.9% of nitrogen and having a relative viscosity of 47 taking acetone as unity. This collodion cotton was of a pure white color without the slightest tinge of yellow, and was completely equal to a cotton made, for example, from de-resinized cellulose as regards color. It could thus also be made into products of a clear, white color.

*Example II*

5 kgs. of cotton linters were nitrated for 30 minutes at 30° C. with 95 kgs. of a nitrating acid composed of 60% H<sub>2</sub>SO<sub>4</sub>, 25% HNO<sub>3</sub> and 15% H<sub>2</sub>O. After washing and ripening which was carried out in the usual manner, a collodion cotton was obtained which contained 12.1% nitrogen and had a relative viscosity (in a 2% acetone solution) of 294, taking acetone as unity.

By operating in the same manner, but adding 3 litres of hydrochloric acid (specific gravity 1.19) to the above-specified quantity of nitrating acid, there was obtained a collodion cotton which contained 10.9% of nitrogen and which had a relative viscosity of 90, taking acetone as unity.

I claim:—

1. A process for manufacturing nitro-celluloses, which process comprises nitrating cellulose raw materials for about 30 minutes at about 30° C. with a nitrating acid containing approximately 57.8% sulfuric acid, 24.3% nitric acid, and 3.7% hydrochloric acid.

2. A process for manufacturing nitro-celluloses, which process comprises nitrating cellulose raw materials for about 30 minutes at about 30° C.

with a nitrating acid containing approximately 57.8% sulfuric acid, 24.3% nitric acid, 3.7% hydrochloric acid, and 14.2% water.

3. A process for manufacturing nitro-celluloses, which process comprises nitrating cellulose raw materials for about 30 minutes at about 30° C.,

with about 20 times its weight quantity of a nitrating acid containing approximately 57.8% sulfuric acid, 24.3% nitric acid and 3.7% hydrochloric acid.

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