

# UNITED STATES PATENT OFFICE.

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## PROCESS OF NITRATING CELLULOSE.

SPECIFICATION forming part of Letters Patent No. 526,752, dated October 2, 1894.

Application filed November 10, 1893. Serial No. 490,520. (No specimens.)

*To all whom it may concern:*

Be it known that I, ROBERT C. SCHÜPPHAUS, a citizen of the United States of America, residing at the city of Brooklyn, Kings county, and State of New York, have invented certain new and useful Improvements in Processes of Manufacturing Gun-Cotton, of which the following is a specification.

10 This invention relates generally to the manufacture of gun cotton and more particularly to the nitration step of the process in such manufacture.

It is well recognized in the process of nitrating cellulose that success is based somewhat on the uniformity of composition, which when practiced in the usual way is hard to attain. As the acids bath is weakened after each successive treatment of the cellulose, different methods have been proposed by which to restore the acids bath to the proper strength. It is also well recognized that it is absolutely essential in order to obtain a uniform product that the proportions of the composition of the acids shall be at all times uniform.

It is obvious that no uniform product can be obtained of the highest grade of nitration by using the acids bath over again upon simply replenishing such bath with a fresh concentrated mixture, for the reason that in the conversion of cellulose into nitro-cellulose, a certain quantity of water is formed. Hence the acids mixture grows richer in sulfuric acid and water and poorer in nitric acid; these relative changes being the smaller the larger the proportion of acids mixture employed. If we suppose that one hundred pounds of acids to each pound of cellulose are used, and that the mixture consists of three parts of the strongest sulfuric acid and one part of the strongest nitric acid, say of ninety-eight per cent. strength, then, after the nitration, on the assumption that no water has been introduced into the acids mixture other than that formed in the reaction, that no nitric acid has evaporated, and that all the cellulose has been converted into true gun cotton, the percent-

age of composition of the remaining acids bath would be: 75.63 sulfuric acid of ninety-eight per cent. strength, twenty-four nitric acid of ninety-eight per cent. strength, and .37 water.

If the nitro-cellulose is withdrawn from the bath and the bath be replenished with a quantity of the original mixture, three parts sulfuric acid and one part nitric acid, the acids solution must necessarily grow less and less fit to produce gun-cotton, until at last a point is reached when it is essential to start with an entirely fresh acids bath; the weakened bath in some instances being used in the preparation of collodion cotton and in others is subjected to distillation.

By rejecting a larger quantity of spent acid than that unavoidably lost in the washing process and replacing it by an equal amount of the original strong mixture, a bath may be produced yielding a high grade gun-cotton; but such product will always contain more of the lower pyroxyline and be variable in composition. Besides, a large stock of weakened acids accumulates that must be disposed of in some way.

The present invention is designed to overcome these difficulties and to provide a simple and economical method of replenishing or bringing the weakened acids bath to its proper strength and proportions; and it consists essentially in adding nitric acid and sulfuric anhydrid to the weakened acids bath.

In practicing the improved method, after withdrawing the nitro-cellulose from the acids bath, the adhering acids are removed either by means of a centrifugal machine, a press, or by a suction filter, and the acids thus recovered are returned to the remainder of the bath. The bath is then analyzed and the percentage of sulfuric and nitric acids of ninety-eight per cent. strength is ascertained. The surplus of water in this bath is then combined with sufficient sulfuric anhydrid, i. e., four times its weight to form ninety-eight per cent. strength sulfuric acid, and enough ninety-eight per cent. strength nitric acid is also added to re-establish the pre-existing pro-

portions of three parts sulfuric acid to one part nitric acid. By this means the original bath in its wasted state is restored to its proper strength and proportions by the simple addition of sulfuric anhydrid and nitric acid.

In the case above cited it would take to restore the weakened acids bath 1.42 pounds sulfuric anhydrid, and 1.83 pounds nitric acid of ninety-eight per cent. strength changing the one hundred pounds of waste acids into one hundred and three and thirty-one one hundredths pounds of acids of the original strength and proportions.

It may be stated that if this gain in weight of 3.31 pounds should not be sufficient to restore the unavoidable loss of acids through the washing process, a solution of sulfuric anhydrid in oil of vitriol may be employed to build up the strength of the waste acids;

and a correspondingly large amount of nitric acid should also be added in such case.

What is claimed is—

1. In the art of nitrating cellulose, the herein described method of restoring the weakened acids bath, which consists in adding thereto sulfuric anhydrid and nitric acid.

2. In the art of nitrating cellulose the herein described method of restoring the weakened acids bath, which consists in adding thereto a solution of sulfuric anhydrid and oil of vitriol and nitric acid.

In witness whereof I have hereunto set my hand in the presence of two witnesses.

ROBERT C. SCHÜPPHAUS.

Witnesses:

GEO. H. GRAHAM,  
E. L. TODD.