(No Model.)

H. MAXIM.

METHOD OF MAKING NITRO-CELLULOSE.

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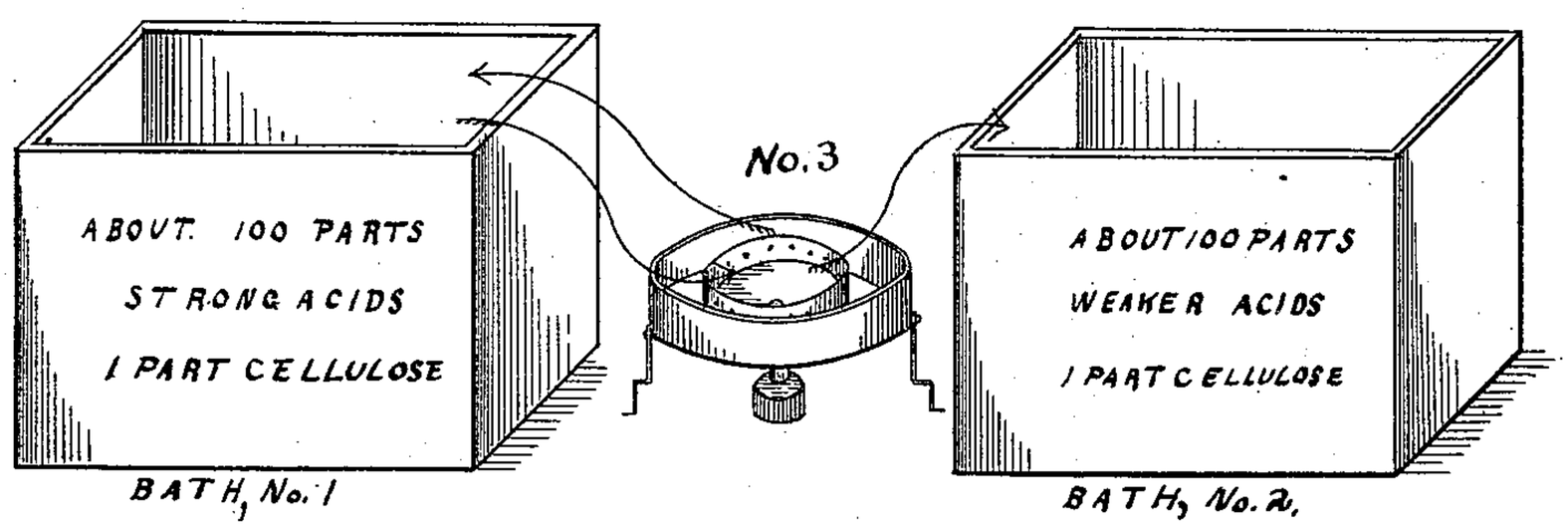

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UNITED STATES PATENT OFFICE.

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METHOD OF MAKING NITRO-CELLULOSE.

SPECIFICATION forming part of Letters Patent No. 465,280, dated December 15, 1891.

Application filed May 12, 1891. Serial No. 392,514. (No specimens.)

To all whom it may concern:

Be it known that I, HUDSON MAXIM, at present residing at the city of New York, in the county of New York and State of New York, have invented certain new and useful Improvements in Methods of Making Nitro-Cellulose, of which the following is a specification, reference being had therein to the accompanying drawing.

My invention relates to the manufacture of nitro substitution compounds of cellulose, such as pyroxyline or gun-cotton, and is chiefly designed to provide in a more simple and efficient manner than heretofore for the production of trinitro-cellulose.

According to the methods of manufacture heretofore employed the cellulose has been first immersed in a bath containing a large excess of a mixture of highly-concentrated nitric and sulphuric acids. After remaining in this bath for about five or six minutes the partially-converted cellulose is taken out and put into earthen pots, each holding, say, about one and one-fourth pounds of cellulose and containing about fourteen pounds of the mixed acids from the immersion-bath, or about eleven and one-fifth pounds of acids for each pound of cellulose. The cellulose is allowed to remain in the earthen pots for, say, twenty-four hours, to digest or finish the process of nitration. As the reaction going on generates heat, and as the quantity of acids is small in most factories, not being enough to fully cover the cellulose, the pots are placed in a vat of cold water, and the water is made to circulate about them to prevent fuming off of the contents. After about twenty-four hours the converted cellulose is placed in a centrifugal machine to remove the excess of acids, and the remainder of the acids is removed by copious washings and boilings with water. The quantity of the acids consumed per pound of cellulose is therefore about eleven and one-fifth pounds of the mixed acids, which are usually mixed in the proportions of one part of nitric acid to three parts of sulphuric acid, this quantity being supplied to the immersion-bath for each pound of cellulose dipped in it, for the purpose of replacing the acids carried in the cellulose and removed therewith to the above-mentioned digest-pots.

The object of my invention is to produce pyroxyline of various degrees of nitration, such as dinitro-cellulose and trinitro-cellulose, but more especially the highest nitrated compound—that is to say, trinitro-cellulose or gun-cotton proper—with the consumption or expenditure of a less quantity of acids than has been heretofore required, and consequently at a correspondingly lower cost per pound for the gun-cotton produced, and a saving of labor.

The figure is a diagram illustrating apparatus with which my invention may be carried out.

No. 1 indicates one bath; No. 2 indicates another bath; No. 3 indicates a centrifugal or other drying machine.

In carrying my said invention into practice I prefer to employ two immersion-baths of the mixed acids, each bath being of different strength and the proportions of the nitric and sulphuric acids being slightly different in each bath. Each bath should contain from fifty to one hundred times as much of the mixed acids as of the cellulose to be immersed therein. The cellulose is dipped in the first bath in much the same manner as has been practiced heretofore—namely, for about five or six minutes, during which time the cellulose has become thoroughly saturated with the acids, and about from one-half to two-thirds of the conversion of the cellulose into the gun-cotton or trinitro-cellulose has taken place. The cellulose is then removed from the first bath and all but from seven and one-half to nine pounds of the acids to each pound of cellulose, or thereabout, is removed or squeezed out of it, and these acids thus removed are allowed to run back into the first bath. The preferred method is to pass the cellulose through a centrifugal machine between baths No. 1 and 2 to remove the excess of acids. The partially-converted cellulose thus removed from the first bath is then placed in the second and last bath, which I term the "digest-bath," where it is allowed to remain from sixteen to forty-eight hours to become thoroughly converted into trinitro-cellulose.

The strength of acids employed for the first bath are the strongest obtainable in commerce—viz., nitric acid of 1.52 specific gravity, one part, and sulphuric acid of 1.84 spe-

cific gravity, three parts. After the immersion of the cellulose in the first bath the acids become slightly diluted with water evolved by the action of the acids upon the cellulose. The acids in the first bath, which at first should be about one part nitric acid to three parts sulphuric acid, become slightly altered in their relative proportions, part of the nitric acid being taken up in the conversion of the cellulose into the nitro-cellulose. I therefore add to this bath, after the removal of the cellulose therefrom, a quantity of the strongest mixed acids in the above proportions, or thereabout—viz., one part nitric acid to three parts sulphuric acid—to restore or partially restore the somewhat weakened acids.

A great advantage of my said invention is that in the last or digest bath a large excess of acids is employed. Therefore the cellulose material is fully immersed and flooded with the acids, so as to preclude any possibility of fuming off from the heat evolved from the reaction, which at this stage of the process is yet necessary to complete the nitration.

By the potting-out method hereinbefore described, which has heretofore been practiced, it has been found necessary to employ, or to allow to remain in the cellulose material, from ten to twelve pounds of acid (usually about eleven and one-third pounds of acid) for each pound of cellulose, in order that the cellulose material shall be sufficiently saturated with the acids to complete or finish the process of nitration, whereas by my process herein described, in consequence of employing a large excess of acids in a large digest-bath, in which all the cellulose material of a day's batch may be immersed, I am enabled to complete the conversion of the nitro-cellulose taken from bath 1 into trinitro-cellulose in the digest-bath with a less expenditure of the acids—that is, with a less quantity of acids added to the first bath and a less quantity removed from the said bath in the cellulose material. As the cellulose material is about one-half to two-thirds converted in the first bath, and as only one-half to one-third more conversion is possible in the second bath, there is no extra expenditure or consumption of the acids in consequence of employing a large excess thereof. After the cellulose has become fully converted into trinitro-cellulose in the digest-bath it is removed and the excess of acids squeezed out or extracted with a centrifugal machine in the usual way, after which it is washed, boiled, treated, and dried in the usual way. The acids extracted from the nitro-cellulose are allowed to return to bath No. 2; but after all of the gun-cotton has been taken out of bath No. 2 a quantity of the weakened acids is removed from bath No. 2 sufficient to make room for those which come over and into it with the cellulose material from the first bath in a day's batch.

The acids which are carried along with the cellulose material from the first to the second bath, as hereinbefore described, will be of the strength and character of the acids of the first bath, or practically so, and will tend to restore the strength of the acids of the second bath. After the completion of the conversion of the cellulose material into gun-cotton in the second bath the acids therein will become altered in strength and in the proportions of the nitric to the sulphuric acid, from the acids of the first bath, in consequence of the conversion of the partially-converted cellulose brought from the first bath into trinitro-cellulose in the second bath, which is accomplished by the absorption of nitric acid by the cellulose and the production of water, which is taken up by the sulphuric acid.

By employing a large digest-bath, or a digest-bath with a large excess of acids, as I do, much saving of labor is effected, and the temperature of the digest-bath can be controlled with great accuracy and a more uniformly-nitrated product obtained, whereas by the "potting-out" method, hereinbefore mentioned, the cellulose material not being fully immersed or covered by the acids, and there being a large number of small pots, the temperature of these various pots cannot well be regulated with uniformity. Consequently the conversion takes place at a different temperature in some of them than others, and a lower nitrated product is frequently obtained in some of the pots, and the contents of some of the pots often become heated so much as to fume off.

I am aware that heretofore gun-cotton has been manufactured by the employment of two baths or more, one for the first immersion and one for a second immersion, and wherein the cellulose material has been allowed to remain to finish its conversion into gun-cotton; but in such case the strength of acids in the second bath has been equal to or greater than the strength of the acids of the first immersion-bath, the object having been to partially convert the cellulose in the first bath and to finish the nitration in a second and stronger bath, the only object of a second bath being to attain a higher and more thorough degree of nitration by immersion in a second bath of the strongest acids after an immersion in a preliminary bath, wherein and whereby the strength of acids has become somewhat weakened.

When a digest-bath as above described has been employed, a great loss of acids or a loss of acids of great strength has resulted, whereas by my process the acids which are removed from the second bath after the conversion therein of the cellulose material into trinitro-cellulose are of a minimum strength compatible with the manufacture of a trinitrate, the acids being renewed and restored therein again to working strength, as hereinbefore described, by the acids which are brought into it in the cellulose material from the first bath.

I have herein described my process as employing two baths only, one an immersion-

bath, the other a digest-bath; but of course two or three more first immersion-baths might be employed, together with one large digest-bath, to receive the partially-converted cellulose after it has been immersed in the said first baths, and all to attain the same result, hereinbefore described; also, of course two or more digest-baths might be employed to attain the same end, so long as the digest-bath or the digest-baths employed contain an acid mixture of the character hereinbefore specified for my second bath as compared with the herein-described first immersion-bath of my process, and so long as the acids in the second or digest bath or baths shall be in large excess sufficient to thoroughly immerse and cover the cellulose material to be placed therein.

While I have termed the acids mixture of the second bath a "weaker-acids bath," it must be understood that it is still a very strong mixture, probably about one per cent. weaker than the strongest-acids bath. By reason of the great excess of quantity and by the addition of the strong acids contained in each batch of nitro-cellulose added to the second bath the acids mixture contained in said bath remains of uniform character and the product has a uniformity not obtainable by the potting-out method.

In the production of dinitro-cellulose or pyroxyline of the lower grades of nitration by my process I employ suitable proportions of weaker acids in the baths above mentioned.

What I claim is—

1. The method herein described of producing nitro-cellulose or pyroxyline of a high grade, which consists in immersing the cellulose for a short time in a bath of strong-acids mixture, then conveying said cellulose with contained acids (amounting to, say, six or more times the weight of cellulose) to a second bath containing many times the weight of the cellulose of a weaker-acids mixture, and there completing the conversion by digesting for a considerable time, substantially as described.

2. The process of manufacturing pyroxyline of a high degree of nitration, which consists in immersing the material to be nitrated successively in two baths, each containing a large excess of mixed nitric and sulphuric acid, in the first and stronger bath of which the cellulose is placed until partially converted or converted into a lower nitro compound, and in the second and weaker of which baths the completion of the conversion into a higher nitro compound takes place, and the acids mixture in the second or last bath being maintained at a suitable working strength by the stronger acids of the first bath adhering to the cellulose material.

In testimony whereof I affix my signature in presence of two witnesses.

HUDSON MAXIM.

Witnesses:

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