

UNITED STATES PATENT OFFICE

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PROCESS OF MAKING DOUBLE BASE POWDERS

No Drawing.

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This invention relates to propellant powders and more particularly to those of the double base type, which may contain nitro-cellulose with nitro-glycerin, ethylene glycol dinitrate, diethylene glycol dinitrate, or any nitrate which is a solvent for nitro-cellulose, and will not be decomposed by the acid used in nitrating the nitro-cellulose.

In the manufacture of nitro-cellulose-nitro-glycerin powders as heretofore practiced, the cellulose, after being nitrated in the usual manner, is separated from the acid and purified to remove all traces of the acid. The nitrated cellulose has then mixed therewith nitro-glycerin and the combination is treated to secure colloidizing. In accordance with one process, nitro-cellulose in the form of a fine pulp and made, for instance, by the nitration of cotton linters, is purified by repeated boiling and washing, first, with an alkali and then with pure water; the nitro-cellulose being in the form of a fine pulp in a large quantity of water, is then mixed with nitro-glycerin by introducing the latter in the form of a spray, in order to form an emulsion in the water; the mixture being agitated so as to cause the nitro-glycerin to combine with the finely divided nitro-cellulose. The particles of nitro-cellulose and the nitro-glycerin combined therewith are then separated from the water in a centrifugal machine, dried and subjected to the action of heated rolls, in order to secure the desired colloidizing. While that process is practical, where the cellulose is in the form of cotton linters, the fibres of which are sufficiently large that they can be separated from the nitrating acid by centrifuging without losing very much of the nitro-cellulose; when the cellulose is made from straw or similar material, the fibres are so reduced in size and character that the loss encountered by separation, as by centrifuging, is increased to such a point where it effects economical considerations. It has, therefore, been proposed, where straw cellulose is used, first, to make the cellulose up into paper and then to cut the same into pieces of sufficient size so they can be readily separated from the nitrating acid; the grains

of nitro-cellulose are then purified of all acid by repeated boiling and washing.

One of the objects of this invention, therefore, is to provide a process of making double base powders whereby the objectionable features of the prior art processes are obviated, and more particularly whereby the losses due to the employment of the finely divided nitro-cellulose are avoided, more particularly in the separation of the same from the liquid.

Another object of this invention is to obviate the necessity, more particularly where straw cellulose is employed, of, first, making up the cellulose into sheets and cutting the same into grains.

Another object is to provide a process of making double base powders whereby the preliminary separation of the nitrated cellulose from the acid is obviated.

When diethylene glycol is nitrated, it is extremely difficult to separate the resulting nitrate from the spent acid; for when strong nitrating acids are used, the diethylene glycol dinitrate forms in a layer on top of the acid and decomposes rapidly if allowed to stand in contact with the spent acid; on the other hand, with weak nitrating acid, the nitrate forms as an emulsion in the spent acid and can only be separated by reducing the temperature, as for instance, pouring the mixture into ice and water.

Another object, therefore, of this invention is to provide a means of more easily separating such nitrates from the acid used in their nitration.

Further objects will appear from the detail description, in which will be described a practical embodiment of this invention; it is to be understood, however, that this invention is susceptible of various embodiments other than that described.

Generally stated, in accordance with one embodiment of this invention, the double base component such as nitro-glycerin or similar nitrate, or a suitable explosive solvent or gelatinizer for nitro-cellulose is added to the nitrated cellulose after its nitration and in the presence of the nitrating agent; the combined nitro-cellulose and nitro-glycerin are then separated from the nitrating agent and

purified and treated in any suitable manner to secure grains in which nitro-cellulose is colloided with the nitro-glycerin. With nitrates such as diethylene glycol dinitrate, however, which form emulsions with the nitrating acid as heretofore noted, it is preferable to reverse the procedure and nitrate the cellulose in the spent acid containing the emulsion of diethylene glycol dinitrate; for in this way a means is provided for collecting the diethylene glycol dinitrate and separating it from the acid. There is a further advantage in proceeding in this way as it makes possible the combination of the two nitrogen products at a percentage of nitrogen in the nitro-cellulose at which it is most soluble.

As a practical embodiment of this invention, cellulose in a finely divided state is nitrated with acid, such as sulfuric and nitric acid, in the usual manner. Before the separation of the nitrated cellulose and the acid, however, the nitro-glycerin is added, preferably in the form of a spray. The mixture of nitro-cellulose and nitro-glycerin is agitated either mechanically or by air. This causes the nitro-glycerin to combine with the nitro-cellulose particles, and these particles agglomerate to form a spongy mass, which readily separates from the acid. This spongy mass can now be readily separated from the acid by either drawing off the latter or by centrifuging. The agglomerated mass can now be purified by boiling and washing, preferably with some alkali at the start, followed by pure water. The purified nitro-cellulose-nitroglycerin can now be rolled into sheets or pressed into strings, with or without the application of heat, and cut into grains in the usual manner.

The cellulose which can be employed in this process may be any of the cellulose materials employed for that purpose. This invention is, however, particularly applicable to the employment of cellulose made from straw, such as wheat straw, pulped and preliminarily purified in accordance with any usual process well known to those skilled in the art. The cellulose may be nitrated to any desired degree of nitration, in accordance with the desired characteristics of the powder, and the relative quantity of nitro-glycerin or other explosive solvent or gelatinizer for nitro-cellulose may be such as to produce the desired combination. While nitro-glycerin is particularly useful in this process, other suitable nitrates and explosive solvents or gelatinizers for nitro-cellulose may be employed; it will, therefore be understood that the term "nitro-glycerin" as well as the term "nitro-cellulose" is used descriptively and functionally and not limitatively. It has been found that a double base propellant powder made in accordance with the process described is free from traces of acid and stable; it will be understood, of course, that suitable stabiliz-

ing agents may be employed when desired or required.

It will be understood that certain features and sub-combinations are of utility and may be employed without reference to other features and sub-combinations; that is contemplated by and is within the scope of the appended claims. It is furthermore to be understood that various changes may be made in details and operations within the scope of the appended claims without departing from the spirit of this invention; it is, therefore, to be understood that this invention is not to be limited to the specific details and operations described.

Having thus described the invention, what is claimed is:

1. A process of making double base powders in which cellulose is nitrated, characterized by the feature that nitro-glycerin is added immediately after such nitration.

2. A process of making double base powders in which cellulose is nitrated, characterized by the feature that nitro-glycerin is added to the nitrated cellulose in the presence of the nitrating agent.

3. A process of making double base powders in which cellulose is nitrated with acid, characterized by the feature that nitro-glycerin is added to nitrated cellulose in the acid.

4. A process of making double base powders in which finely divided cellulose is subjected to a nitrating agent, characterized by the feature that nitro-glycerin is mixed with the nitrated cellulose in the presence of the nitrating agent.

5. A process of making double base powders in which finely divided cellulose is nitrated with acid, characterized by the feature that nitro-glycerin is added to the nitrated cellulose in the acid.

6. In the art of making double base powders, the process comprising, treating cellulose with a nitrating agent, adding nitro-glycerin thereto and separating the nitrating agent.

7. In the art of making double base powders, the process comprising, treating cellulose with nitrating acid, adding nitro-glycerin thereto, and separating the combined nitro-cellulose and nitro-glycerin from the acid.

8. In the art of making double base powders, the process comprising, treating cellulose with nitrating acid, adding nitro-glycerin thereto, separating the combined nitro-cellulose and nitro-glycerin from the acid and washing the combined nitro-cellulose and nitro-glycerin.

9. In the art of making double base powders, the process comprising, treating finely divided cellulose with nitrating acid, mixing nitro-glycerin therewith and separating the colloided nitro-cellulose from the acid.

10. In the art of making double base powders, the process comprising, treating finely

divided cellulose with nitrating acid, mixing nitro-glycerin therewith, separating the colloided nitro-cellulose from the acid and washing the colloided nitro-cellulose.

5 11. In the art of making double base powders, the process comprising, treating cellulose with a nitrating agent, adding nitro-glycerin thereto, agitating the mixture and separating the nitrating agent.

10 12. In the art of making double base powders, the process comprising, treating cellulose with nitrating acid, adding nitro-glycerin thereto, agitating the mixture and separating the combined nitro-cellulose and nitro-glycerin from the acid.

15 In testimony whereof, I affix my signature this 1st day of April, 1930.

WILLIAM E. WAGNER.

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