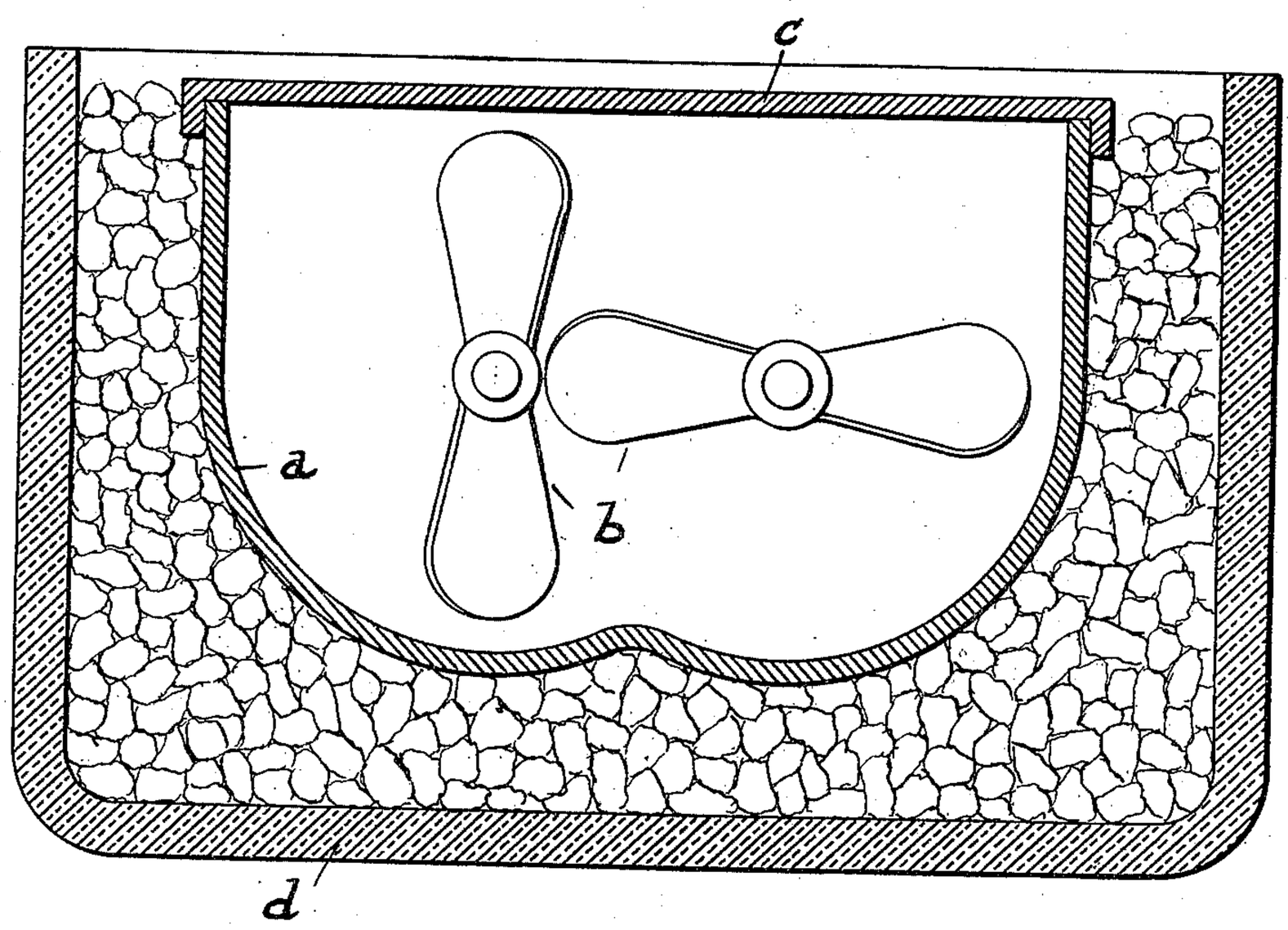
No. 652,455. Patented June 26, 1900.

J. B. BERNADOU.

PROCESS OF MAKING SMOKELESS POWDER.

(Application filed Nov. 17, 1898.)

(No Model.)



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UNITED STATES PATENT OFFICE.

JOHN B. BERNADOU, OF THE UNITED STATES NAVY.

PROCESS OF MAKING SMOKELESS POWDER.

SPECIFICATION forming part of Letters Patent No. 652,455, dated June 26, 1900.

Application filed November 17, 1898. Serial No. 696,725. (No specimens.)

To all whom it may concern:

Be it known that I, JOHN B. BERNADOU, lieutenant, United States Navy, at present stationed in the city of Washington, District of Columbia, have invented certain new and useful Improvements in Smokeless Powder, of which the following is a specification.

My invention relates to an improved process for making colloid powders; and the object of my invention is to furnish a new and simple colloid which may be used itself after drying as a powder or which may be used as an explosive cementing agent or binder in connection with other materials used in the manufacture of smokeless powders.

At present it is the general practice in the manufacture of colloid smokeless powders to mix the nitro-cellulose with a colloiding agent, as acetone or ether alcohol, to place these materials in a mechanical mixer, in which they are thoroughly mixed, and to then form the resultant pasty mass into the desired shapes, as rods, strips, or grains, which are then exposed to the air or to a moderate heat until the excess of solvent is expelled. The drying must be conducted with care and uniformity. If the powder be dried too quickly, as by exposure to too great a heat, the surface of the grains will be formed into a pellicle, while part of the interior content of the solvent will vaporize, causing the grain to swell and split. If the powder be not dried uniformly, the damper grains will ignite more slowly than the dryer ones, and hence irregularity in ignition and in ballistic performance will result. If the powder be left in an undried state, the traces of water, alcohol, and ether remaining in it operate to render it, in comparison with other lots of the same powder properly dried, slow in ignition.

Experience has shown that the complete and uniform drying of the colloid must be effected if the resultant mass is to prove a effective powder. The more effectually that the residual solvent, that remaining after the pasty colloid has been formed into the final shape, can be driven off the more efficient will be the powder, the greater its ballistic power, and the less the chance of hang-fires.

The solvent commonly employed in the manufacture of smokeless powders is ether alcohol, which varies from a mixture of about two parts, by weight, of ether to one of alcohol, to a mixture of about one part, by weight, of ether to two of alcohol. Thus in specifications issued by the United States Navy Department a ratio of sixty-four parts, by weight, of ethyl ether to thirty-six parts, by weight, of ethyl alcohol is called for. On the other hand, the form of soluble nitro-cellulose employed for this powder, known as "pyro-cellulose," is soluble in a mixture of two parts, by weight, of ethyl alcohol and one part, by weight, of ethyl ether, and for any mixture between these limits colloidization can be readily effected. In the processes hitherto in use further reduction in the relative amount of alcohol employed with the ether than that stated, thirty-six parts of alcohol to sixty-four of ether, by weight, tends to make the nitro-cellulose insoluble.

The colloiding agent is generally mixed with the nitro-cellulose while the latter is in a dried state, or else part of the alcohol is employed for its dehydration and the balance of the solvent, containing the ether, is added upon the introduction of the dampened mass into the mixer. These operations are now conducted at ordinary atmospheric temperatures. The addition of large quantities of alcohol, which is permitted to contain as much as 7.7 per cent of water, by weight, necessarily introduces a considerable amount of water into the powder, and this water is very difficult to get rid of.

I have discovered that if ether-alcohol-soluble nitro-cellulose be immersed in ethyl ether and be then exposed to a temperature of about that of freezing water or lower the nitro-cellulose will go into solution or form a jelly with the ether, resulting in the formation of a colloid. Once in solution or jelly the nitro-cellulose will not again revert to its original cellular form, but constitutes a colloid which may be employed when dried as a powder or as a cementing agent or binder in the manufacture of powders containing other ingredients. This property of dissolving in ether upon application of cold is common to all forms of soluble nitro-celluloses. By "soluble nitro-cellulose" I mean those nitro-celluloses that will go into solution at ordinary atmos-

pheric temperatures in a mixture of two parts, by weight, (about,) of ethyl ether and one part, by weight, (about,) of ethyl alcohol. The property above referred to is possessed by soluble nitro-hydro-cellulose prepared by nitrating hydro-cellulose, while the form of soluble nitro-cellulose known as "pyro-cellulose," prepared by the hot process of nitration, also possesses the same property.

Ordinary forms of soluble nitro-celluloses prepared under the old process, which have been unduly heated or treated with alkali, causing partial disintegration or modification of their substance, display the phenomenon imperfectly, yet will colloid in ether sufficiently under the influence of cold to illustrate the principle.

The phenomenon of the action of the ether upon the nitro-cellulose may be illustrated in a very perfect manner by placing the soluble nitro-cellulose—for example, a gram of dry soluble nitro-hydro-cellulose, defined above—in a test-tube, adding an excess of ether, tightly corking it, and immersing it in a freezing mixture of salt and ice. The soluble nitro-cellulose, which at first remained undissolved like so much paper-pulp at the bottom of the clear ether, goes into solution under influence of the cold, producing a syrupy but mobile liquid. If the tube be now removed from the cooling mixture and warmed by immersion in lukewarm water or even by being held tightly in the palm of the hand, the contents will congeal under influence of the heat into a dense jelly which will not obey the law of liquid flow. Reimmersion of the tube into the cooling mixture will again render its contents liquid, and the alternate rendering of its contents liquid and solid may be effected by alternate reëxposures to sources of cold and heat, respectively.

The heating on the above experiment is resorted to to illustrate the principle of the process—viz., that soluble nitro-cellulose is more soluble in the cool than in the warm liquid. If the tube be now uncorked and the excess of solvent be evaporated off, a colloid will be formed which may be used as a smokeless powder or as a cementing agent for binding together other ingredients of smokeless powders.

The ease with which the soluble nitro-cellulose can be colloided in the presence of cold is materially increased by mechanical agitation—that is, by kneading or incorporating the ether and soluble nitro-cellulose together in a closed vessel, whereby the colloid can be developed with the expenditure of a minimum amount of solvent. The process of forming the colloid powder, therefore, consists in subjecting the ether and soluble nitro-cellulose to mechanical agitation or kneading in some approved form of mechanical mixer, such as the Werner and Pfleiderer, in the presence of a sufficient degree of cold and in subsequently removing the mass after colloidization has ensued, forming into shapes or grains, and drying.

In the accompanying drawing I have illustrated the Werner and Pfleiderer apparatus, *a* being the vessel in which the mixing takes place; *b*, blades which are revolved in any suitable manner; *c*, a cover, and *d* a jacket adapted to contain a cooling mixture.

The cooling-jacket may be simply a cool atmospheric envelop in which the vessel *a* is placed.

Some nitro-celluloses are more refractory than others—that is, require more cold to produce colloidization. The addition of a small quantity of alcohol in such cases, say up to five per cent., will facilitate colloidization at temperatures below but near the freezing-point.

In a division of this application, Serial No. 739,654, filed December 8, 1899, the colloid itself is claimed, the present application being confined to the process for making the colloid.

I claim—

1. The described method of forming a colloid consisting in subjecting soluble nitro-cellulose and a colloiding agent to a temperature equal to or below that of freezing water and in evaporating off the excess of solvent in the resultant product.

2. The described method of forming a colloid consisting in subjecting soluble nitro-cellulose and a colloiding agent, in a closed vessel, to a temperature equal to or below that of freezing water and in evaporating off the excess of solvent in the resulting product.

3. The described process of manufacture of a colloid powder, which consists in subjecting soluble nitro-cellulose and a colloiding agent, in a closed vessel, to a temperature equal to or below that of freezing water, mechanically agitating or kneading the cooled mixture, forming it into shapes or grains, and drying.

4. The described method of forming a colloid consisting in subjecting soluble nitro-cellulose and ether to a temperature equal to or below that of freezing water and in evaporating off the excess of solvent in the resultant product.

5. The described method of forming a colloid consisting in subjecting soluble nitro-cellulose and ether, in a closed vessel, to a temperature equal to or below that of freezing water and in evaporating off the excess of solvent in the resultant product.

6. The described process of manufacture of a colloid powder, which consists in subjecting soluble nitro-cellulose and ether, in a closed vessel, to a temperature equal to or below that of freezing water, mechanically agitating or kneading the cooled mixture, forming it into shapes or grains and drying.

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