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NITROCELLULOSE PARTICLES

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This invention relates generally to explosives and more particularly to a method for making globular particles of nitrocellulose of small granulation.

A process for making globular grains of smokeless powder base is disclosed in U.S. Patent 2,027,114 granted to Olsen, Tibbitts and Kenone, January 7, 1936. The process disclosed in this patent is admirably suited for making globular powder grains of the size ordinarily used as propellants in small arms cartridges, i.e., within the granulation of about 0.010 inch to about 0.025 inch, but it is unsuitable for making products predominating in powder grains having a diameter of about 0.012 inch or less. Particles of such small size are particularly advantageous for use in molding large propellant grains and other articles from nitrocellulose.

It is, therefore, an object of this invention to provide a method for making particles of nitrocellulose in relatively small particle size. Another object of the invention is to provide a method for making globular particles of nitrocellulose having a granulation of about 0.012 inch or less. Still another object of the invention is to provide a process for making globular nitrocellulose particles from fibrous nitrocellulose without completely destroying the fibrous structure of the nitrocellulose, the resulting globules being spheres of about 0.012 inch diameter or less. Another object is to provide a method for making substantially globular particles of nitrocellulose particularly well suited for use in casting large propellant grains.

In accordance with this invention, the foregoing objects as well as others are achieved, generally speaking, by suspending ungelatinized nitrocellulose fibers in a non-solvent medium, dispersing a protective colloid in the resulting slurry, adding to the slurry at a predetermined rate a solvent for the nitrocellulose which is substantially immiscible with the non-solvent medium in sufficient quantity to soften the fibers of nitrocellulose but to only partially gelatinize them, agitating the slurry to break down the softened nitrocellulose fibers and causing them to assume a spherical form, and thereafter removing the solvent from the globular nitrocellulose particles.

It is essential that the fibers of nitrocellulose be treated with a protective colloid before sufficient solvent is added to the slurry to soften the fibers, in order to avoid agglomeration of the softened fibers. Fibrous nitrocellulose must be used instead of gelatinized nitrocellulose in order to obtain nitrocellulose particles having the characteristics contemplated by this invention.

The size distribution of the fibers of conventional nitrocellulose varies from minute particles of about 0.0005 inch diameter to about 0.020 inch long and 0.002 inch in cross-sectional diameter. A majority of the fibers have dimensions of approximately 0.001 inch by 0.010 inch. In practicing this invention, a suitable protective colloid is added to a slurry of such fibers suspended in water or other suitable non-solvent medium and the slurry is agitated until a protective coating of the colloid is placed on

each fiber. Solvent is then added slowly to the slurry as agitation is continued. The solvent may be ethyl acetate or any other solvent for nitrocellulose which is substantially immiscible with the non-solvent medium. Preferably, the solvent has a boiling point below that of the non-solvent. Examples of suitable solvents in addition to ethyl acetate include methyl ethyl ketone, ethyl formate, butyl acetate and the like. A suitable mixture of solvent such as 80 percent methyl ethyl ketone and 20 percent toluene may also be used. The addition of solvent brings about softening of the minute particles which gradually assume a globular shape without complete gelatinization of the nitrocellulose as the slurry is agitated. The large fibers disintegrate under the influence of agitation and the resulting smaller particles then assume a substantially spherical shape.

The addition of solvent must be controlled in such a manner that the particles of nitrocellulose are only softened and are rounded up without complete dissolution of the nitrocellulose, or, in other words, without the formation of a lacquer of the type formed by the process disclosed in U.S. Patent 2,027,114. The particles having a fibrous structure made by the process of this invention impart greater strength to products molded therefrom than is imparted by completely gelatinized particles of nitrocellulose made by the process disclosed in U.S. Patent 2,027,114. The solvent may be added to the slurry at any rate up to the point the slurry contains sufficient solvent to begin softening the nitrocellulose. With a water-ethyl acetate system, this occurs when about 1.6 parts solvent per part of nitrocellulose are substantially uniformly distributed in from about 10 to about 17 parts water per part of nitrocellulose. The solvent concentration is then increased gradually by adding solvent to the slurry at a rate of not more than about 0.25 part solvent per one hundred parts of nitrocellulose per second. Under most conditions, a final concentration in a water-ethyl acetate system of not more than about two hundred parts solvent per one hundred parts of nitrocellulose will be required to produce only partially gelatinized globular nitrocellulose particles of the type contemplated by this invention, but it has been found that as much as about three hundred parts solvent to one hundred parts nitrocellulose may be utilized without the nitrocellulose becoming completely dissolved and coalescing to form a continuous lacquer phase of the type described in the aforementioned patent. This ratio may vary somewhat with other systems but in any system it is advantageous to utilize only that amount of solvent required to soften and not completely dissolve the nitrocellulsoe and less than that amount of solvent which will permit coalescence. After the globules have assumed a globular shape, the solvent is removed from the slurry by any suitable method such as by distillation or by purging the slurry with air or other suitable gas.

Any suitable protective colloid may be utilized. For example, gum arabic or an animal glue such as Swift's Colloid No. 1 may be used to advantage.

In order better to described and further clarify the invention, the following is a detail description of a preferred embodiment thereof:

About 100 parts by weight fibrous nitrated cotton linters having a nitrogen content of about 12.6 percent and a viscosity of about 8 seconds as determined by the Hercules Falling Ball Method are slurried in about 1500 parts water. About 50 parts by weight gum arabic are added to the slurry and the slurry is agitated until the fibers of nitrocellulose are completely coated with gum arabic. About one part by weight dinitro diphenylamine is added.

The contents of the vessel are then heated to about

65° F. to 68° F. and about 160 parts by weight ethyl acetate are added to the slurry. Agitation is continued until the solvent is substantially uniformly dispersed in the slurry. A total of about 140 parts ethyl acetate is then added at a rate of about 0.25 part by weight per second while agitation is continued for about 30 minutes or until the particles have a substantially globular shape. About 90 parts sodium sulfate dissolved in about 300 parts water are added and agitation is continued until the solution is substantially uniformly mixed with the slurry. The temperature of the slurry is raised to the boiling point and held at this temperature until substantially all of the solvent has been removed therefrom and the softened globules of nitrocellulose have become hardened while retaining their globular shape. About 87 percent of the globular particles of nitrocellulose thus obtained have a diameter of about 0.012 inch or less. About 77 percent of the particles have a diameter of 0.010 inch or less. About 46 percent of the particles have a diameter of 0.006 inch or less. About 37 percent have a diameter of about 0.005 inch or less and about 28 percent are about 0.004 inch or less in diameter. The globular shaped particles are only partially gelatinized.

The foregoing procedure may be duplicated with the exception that after about 160 parts solvent have been added to the slurry of about 100 parts nitrocellulose and about 1500 parts water, additional solvent is added at a rate of about 35 parts solvent per second until a total of about 300 parts solvent have been added. Only about 52 percent of the resulting particles have a diameter of 0.012 inch or less, about 37 percent are less than 0.010 inch in diameter, about 16 percent are less than 0.006 inch in diameter, about 13 percent are less than 0.005 inch in diameter, and about 11 percent are less than 0.004 inch.

When the foregoing procedure is followed with the exception that after the original 160 parts solvent have been added the rate of solvent addition is about 15 parts per second until a total of about 300 parts are added, the product contains about 50 percent globular particles having a diameter of about 0.012 inch or less, about 43 percent globular particles having a diameter of 0.010 inch or less, about 32 percent globular particles having a diameter of about 0.006 inch or less, about 28 percent globular particles having a diameter of about 0.005 inch or less and about 26 percent particles of 0.004 inch or less diameter.

About 90 percent of the particles obtained in a typical batch of powder made in accordance with the process of U.S. Patent 2,027,114 in which the smokeless powder base is completely dissolved to form a lacquer and this lacquer is then comminuted into globules has a diameter of from about 0.0248 to 0.0092 inch.

No definite theory as to the reason why the product of this invention is predominately 0.012 inch or less in size has been forthcoming up to this time. However, it is believed that because of the structure of the nitrocellulose each fiber breaks up into particles of the desired size as the fiber is softened by the solvent and because of the presence of a film of a protective colloid on the surface of the fibers the resulting small particles do not coalesce. It appears from the results of some of the experimental work that as the fiber is softened by the solvent a break occurs at each node. In any event, the product obtained by the process of this invention consists predominately of only partially gelatinized nitrocellulose particles having a maximum dimension of not more than about 0.012 inch. Although the examples have been described with water as a non-solvent medium it is to be understood that any other suitable non-solvent for nitrocellulose can be utilized provided it is substantially immiscible with the solvent for the nitrocellulose.

It is contemplated that the particles may be treated with a non-explosive plasticizer such as dibutyl phthalate, dinitrotoluene, diphenyl phthalate, diamyl phthalate, ethylcentralite or any other deterrent, if desired. Likewise, the particles may be coated with an energizing plasticizer such as a liquid nitric ester of a polyhydric alcohol including nitroglycerine, nitroglycol, or the like, as desired. Moreover, various modifying agents such as red lead oxide, Pb_3O_4, litharge, lead stannate, or other material which modifies the burning rate of powder grains may be placed on the surface of the particles of nitrocellulose by any suitable method, if desired.

Although the invention has been described in considerable detail in the foregoing, it is to be understood that the purpose of such detail is only for clarification of the invention and that many modifications can be made by those skilled in the art without departing from the spirit and scope of the invention except as it is limited by the appended claims.

I claim:

1. A method for making substantially globular particles of nitrocellulose having a diameter not substantially greater than 0.012 inch which comprises forming a slurry by suspending fibers of nitrocellulose in an aqueous medium in the ratio of about 1 part by weight nitrocellulose per 10 to 17 parts by weight aqueous medium, coating the fibers with a protective colloid, adding about 1.6 parts by weight ethyl acetate for each part by weight nitrocellulose, thereafter adding ethyl acetate to the slurry at a rate not substantially greater than about 0.25 part by weight per 100 parts by weight nitrocellulose per second while maintaining the ethyl acetate to nitrocellulose ratio below about 3:1, agitating the slurry until the fibers have assumed a globular form, and removing the solvent from the nitrocellulose.

2. A method for making substantially globular particles of nitrocellulose having a diameter not substantially greater than 0.012 inch which comprises forming a slurry by suspending about 100 parts by weight fibrous nitrocellulose in about 1500 parts by weight of an aqueous medium, dispersing throughout the resulting slurry about 50 parts by weight gum arabic, adding about 160 parts by weight ethyl acetate, and subsequently adding an additional 140 parts by weight ethyl acetate at a rate of about 0.25 part per second, maintaining the suspension by agitation for about 30 minutes, and removing the solvent.

References Cited in the file of this patent

UNITED STATES PATENTS

2,027,114	Olsen et al.	Jan. 7, 1936
2,206,916	Olsen et al.	July 9, 1940