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DUSTLESS PROPELLENT POWDER CONTAINING COATED SPHERICAL NITROCELLULOSE

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This invention relates to propellent explosives and particularly to improved propellent powders and methods of making them.

Single or double base grains for use in rocket motors, missiles, and gas generating devices of various types, are quite large. For example, they can be a foot or more in diameter and several feet in length. Such grains have been conventionally prepared by the methods employed in the manufacture of extruded powder. However, the curing and solvent removal of these relatively large grains have presented a formidable problem. These conventional manufacturing difficulties are readily overcome when the large propellent or gas generating grains are prepared in accordance with the teachings of U.S. Patent 2,916,775, issued to John J. O'Neill, Jr. In the manufacture of large propellent grains by this process, finely divided particles of a nitrocellulose base propellent are mixed with a plastisol solvent consisting of relatively non-volatile nitrocellulose plasticizers to form a flowable paste. The paste is then formed into the shape of a rocket powder grain and maintained in the assumed shape until it becomes consolidated to form the desired rocket powder grain.

Molding or casting processes of this type are particularly well adapted for the manufacture of large diameter propellent powder grains. However, the handling of the exceedingly small propellent powder particles used in making up the large grains poses several inherent difficulties. The nitrocellulose propellent particles have an average size of about 0.003 inch and because of this exceedingly small size, are quite dusty. This presents an intense safety problem due to the high concentration of explosive dust in the atmosphere, and also necessitates frequent cleaning of the operating area. In addition, the finely divided nitrocellulose particles are quite susceptible to the accumulation of static charges. Thus, extreme care must be exercised to avoid the generation of a spark in this raw material. Also, the finely divided nitrocellulose particles are not readily wetted by the plastisol solvent. Thus, mixing is necessarily prolonged because it must be continued until all particles are uniformly wetted with the plastisol solvent.

It is, therefore, an object of this invention to provide propellent powders overcoming the disadvantages of the prior art. It is also an object of this invention to provide propellent powder grains exhibiting little or no dusting while retaining their free-flowing characteristics. A further object of this invention is to provide an improved astatic propellent powder charge. In addition, it is an object of this invention to provide propellent powder grains having improved wettability by a casting solvent. A more specific object of this invention is to provide an improved method for the manufacture of cast propellent powder grains for use in rocket motors and the like.

In accordance with this invention, these and other objects are accomplished, generally speaking, by coating the grains of a propellent nitrocellulose-base powder charge with a substantially non-volatile liquid which is compatible with nitrocellulose and which has no appreciable plasticizing action on nitrocelluiose. More specifically, this invention contemplates surface coating nitrocellulose propellent powder grains with a substantially non-volatile liquid ester formed by the reaction of

phthalic acid or the dibasic saturated aliphatic acids having between 4 and 8 carbon atoms in their alkyl chain with saturated aliphatic alcohols having between 6 and 12 carbon atoms in their alkyl chain. In addition to phthalic acid, the acids that can be employed in accordance with the present invention are adipic acid, pimelic acid, suberic acid, azelaic acid, and sebacic acid. Dioctyl phthalate is the preferred ester employed in accordance with the present invention, but the objects of the present invention can also be realized by using one or more of the di-esters of the acids and alcohols referred to above. Since the di-esters employed have no appreciable solvent power for nitrocellulose, they do not penetrate the surface of the grains. They are present only as a molecular external coating.

In order to minimize the dusting and static characteristics of the nitrocellulose propellent powder grains while maintaining their free-flowing characteristics, the amount of the di-ester present on the surface of the powder grains must be maintained between about 0.02% and about 0.5% of the weght of the powder. Amounts of the di-ester less than about 0.02% have no appreciable effect. On the other hand, when more than approximately 0.5% of the di-ester is used as a coating agent, it detracts from the free-flowing qualities of the powder and merely acts as a diluent for the propellent charge.

Nitrocellulose powders can be coated with the di-esters of the present invention in any convenient manner. It is only necessary that the grains making up the charge are substantially uniformly coated. Thus, the propellent powder charge can be mixed with the liquid di-ester in a sweetie barrel or other conventional tumbling apparatus to effect the coating. This tumbling operation can be performed in the presence or absence of water. If desired, the powder and/or the coating agent can be mixed with water prior to the coating operation. It is preferred, however, to treat an aqueous slurry or suspension of the powder grains to be coated with an aqueous emulsion or suspension of the liquid di-ester in a continuous manner. This is most readily accomplished by dewatering an aqueous slurry of the powder grains in a continuous centrifuge and then adding an aqueous emulsion of the coating material to the centrifuge. The emulsified di-ester readily replaces the water wetting the powder, and the excess emulsion is conveniently removed from the powder charge by continued centrifugation. Any water remaining on the powder grains can then be removed by conventional drying methods.

The di-ester coatings of the present invention can be applied to nitrocellulose powder of any size or shape. However, they are more advantageously utilized with spherical propellent powder grains having an average diameter of about 0.003 inch and used as the base material in the manufacture of giant rocket grains and the like. Although the present invention is concerned primarily with the treatment of such casting powder, the qualities of standard small arms ammunition propellent are also materially improved when coated with the di-ester of the present invention. The present coating materials can be used to replace or augment the conventional graphite glaze on commercial and military propellents. Thus, the present invention is applicable to any type of nitrocellulose based powder regardless of its size or shape.

When the propellent powder grains coated in accordance with this invention are used as a base material in the manufacture of rocket grains, they are mixed with a plastisol solvent which has relatively poor solvent power for the propellent at ordinary room temperature, but which is very rapid acting and has a relatively great solvent power at elevated temperatures. The plastisol solvent can be either a deterrent or an energizing modi-

fier or a mixture of energizers and deterrents. Suitable energizers include nitroglycerin, triethylene glycol dinitrate, butane triol trinitrate, diglycol dinitrate, ethylene glycol dinitrate, and the like. Suitable deterrents that may be used include dibutyl phthalate, dimethyl sebacate, dibutyl succinate, dibutyl adipate, dipropyl adipate, triacetine, ethyl diphenol phosphate, tributyl phosphate, and the like. The di-esters of the present invention are readily compatible with plastisol solvents of this type and greatly reduce the time required for obtaining a uniform plastisol mixture. Also, because the di-esters have no appreciable solvent power toward the nitrocellulose propellent, the "pot life" of the plastisol mixture is not lessened.

In order that the invention may be further clarified, following is an example illustrating a preferred embodiment thereof. In this embodiment and throughout the specification, all proportions are expressed by weight unless otherwise indicated.

About 1000 parts of an aqueous powder slurry obtained in accordance with the process of U.S. 2,888,713, granted to Eugene A. Andrew, was fed to a continuous centrifuge. The powder was substantially spherical in form and had an average diameter of approximately 0.003 inch. The powder slurry was about 95% water and 5% powder. The water content of the powder was reduced to approximately 8% by centrifuging. At this point, about 7 parts of an aqueous emulsion of dioctyl phthalate was gradually added to the centrifuge over about 5 minutes. This aqueous emulsion consisted of about 1.15% dioctyl phthalate and about 98.85% water. This emulsion was run into the centrifuge and through the wet powder charge until the original water content of the powder was replaced with the aqueous dioctyl phthalate emulsion. The powder was then removed from the centrifuge and the water driven off of the powder by drying in a tray dryer for about 10 hours at a temperature of approximately 65° C. At the end of the drying period, the powder was substantially free of water and contained a uniform surface coating of dioctyl phthalate. The dioctyl phthalate on the surface of the grain constituted about 0.1% of the weight of the powder. This powder charge was completely dust-free and substantially astatic. Also, the coating had no deleterious effects on the free-flowing characteristics of the powder charge. This material was particularly well adapted for the manufacture of rocket grains by the plastisol process because of its improved wettability by the casting solvents.

Nitrocellulose is the most commonly used propellent powder base. The nitrocellulose may be used alone or in combination with deterrents such as dinitrotoluene, dialkyl and diaryl phthalates, etc. Energizers such as nitroglycerin, aluminum, trinitrotoluene, cyclotrimethylenetrinitramine, ammonium perchlorate, and the like can also be incorporated with the nitrocellulose or mixed with the surface coated powder. Whenever desirable, the nitrocellulose can be replaced in whole or in part by other gelatinizable polynitro materials such as nitrostarch, polyvinyl nitrate, and similar materials.

While one complete embodiment has been disclosed in detail and modifications therein suggested, it is to be understood that the example given is for the purpose of illustrating the invention, and not by way of limitation. It is recognized that those skilled in the art will make appropriate adjustment of the several variables in the process in order to adapt it to the peculiarities of any given operation, and consequently it is to be distinctly understood that the invention is not limited to the examples given, save as indicated in the appended claims.

Having thus described the invention, what is claimed and desired to be secured by Letters Patent is:

1. A dustless propellent powder charge consisting essentially of non-porous solidified globules of nitrocellulose base powder grains having an average diameter of less than about 0.003 inch and substantially uniformly surface coated with from about 0.02% to 0.5% of dioctyl phthalate.

2. A dustless propellent powder charge consisting essentially of spherical nitrocellulose propellent powder grains having an average diameter less than about 0.003 inch and coated substantially uniformly with between about 0.02% and 0.5% of a liquid ester formed by the esterification of a dibasic acid selected from the group consisting of phthalic acid and the dibasic saturated aliphatic acids having between 4 and 8 carbon atoms in their alkyl chains with a saturated aliphatic alcohol containing between 6 and 12 carbon atoms in the alkyl chain.

3. In the manufacture of rocket propellent grains the steps comprising introducing a water slurry of finely divided nitrocellulose powder grains to a centrifuge, centrifuging the slurry to remove the water therefrom, adding an aqueous emulsion of dioctyl phthalate to the powder remaining in the centrifuge, the amount of dioctyl phthalate being between about 0.02 and 0.5% based on the weight of the powder grains, mixing the emulsion with the powder, centrifuging the resultant mixture to remove liquid therefrom, drying the powder grains, mixing the dried powder grains with a casting solvent to form a plastisol mixture, and forming the plastisol mixture into grains.

4. In the manufacture of rocket propellent grains the steps comprising introducing a water slurry of finely divided nitrocellulose powder grains to a centrifuge, centrifuging the slurry to remove the water therefrom, adding an aqueous emulsion of dioctyl phthalate to the powder remaining in the centrifuge, the amount of dioctyl phthalate being between about 0.02 and 0.5% based on the weight of the powder grains, mixing the emulsion with the powder, centrifuging the resultant mixture to remove liquid therefrom, and drying the powder grains.

5. A method of preparing fine diameter spherical particles of nitrocellulose propellent powder for casting into a rocket grain comprising mixing the powder grains with from about 0.02% to about 0.5% of a liquid alkyl diester formed by the esterification of a dibasic acid selected from the group consisting of phthalic acid and the dibasic saturated aliphatic acids having between 4 and 8 carbon atoms in their alkyl chains with a saturated aliphatic alcohol containing between 6 and 12 carbon atoms in the alkyl chain, whereby a substantially uniform coating of said ester is formed on the surface of said grains.

6. A dustless astatic propellent powder charge consisting essentially of nitrocellulose base propellent powder grains provided with a substantially uniform surface coating of from about 0.02 to 0.5% by weight of a liquid ester formed by the esterification of a dibasic acid selected from the group consisting of phthalic acid and the dibasic saturated aliphatic acids having between 4 and 8 carbon atoms in their alkyl chains with a saturated aliphatic alcohol containing between 6 and 12 carbon atoms in the alkyl chain, the interior of the grains being substantially devoid of the ester.

References Cited in the file of this patent

UNITED STATES PATENTS

2,235,298	Olsen	Mar. 18, 1941
2,916,775	O'Neill	Dec. 15, 1959
2,917,379	Ryker	Dec. 15, 1959