United States Patent Office 3,745,078
Patented July 10, 1973

3,745,078

DISPERSION OF FINE AMMONIUM PERCHLORATE, ALUMINUM OR FERRIC OXIDE PARTICLES IN PROPELLANTS

Bernard J. Alley and Herbert A. Carter, Huntsville, Ala., assignors to the United States of America as represented by the Secretary of the Army

No Drawing. Filed Oct. 19, 1967, Ser. No. 677,846
Int. Cl. C06b 11/00
U.S. Cl. 149—76 8 Claims

Mechanical methods of dispersion other than those employing conventional mixers have also been developed to disperse fine ammonium perchlorate particles in propellants. One such method involves the mechanical incorporation of fine ammonium perchlorate particles, such as by the use of a roll mill, in a carboxyl-terminated polybutadiene polymer to form a paste. The paste is subsequently added to the propellant mix. However, this method has several disadvantages. Only a portion of the total propellant oxidizer can be added in the paste form because of the limitation of the amount of ammonium perchlorate which can be held in the paste. The remainder must be added in the dry nondispersed state. Furthermore, a solvent must be added to the paste during dispersion to facilitate processing. Additionally, complete dispersion is usually not achieved by this mechanical process, and a portion of the ammonium perchlorate is ground. This affects the repeatability of the finished propellant properties.

Logically, chemical dispersion methods have been investigated as a means of improving the dispersion of the ammonium perchlorate, aluminum or ferric oxide particles in the propellant.

Accordingly, it is the principal object of this invention to provide an improved method for dispersing fine ammonium perchlorate, aluminum or ferric oxide particles in a propellant.

It is a particular object of this invention to provide such a method which involves chemical dispersion.

SUMMARY OF THE INVENTION

Fine ammonium perchlorate (or aluminum or ferric oxide) particles can be completely dispersed in propellants by first preparing a slurry of fine ammonium perchlorate particles (and other propellant solid or liquid ingredients, if desired) with a suitable surfactant and an organic liquid. This is normally done by conventional mixing. The slurry is then allowed to stand, until the surfactant reacts with the ammonium perchlorate surface and establishes equilibrium at the ammonium perchlorate-liquid interface. The surfactant reaction produces complete dispersion of the ammonium perchlorate, ferric oxide or aluminum particles in the slurry by effectively neutralizing the charges and reducing the cohesive forces between the particles. Preferred organic liquids are normal-hexane, normal-heptane, 1,1,2-trichloro-1,2,2-trifluoroethane and 1,1,2,2-tetrachloro - 1,2 - difluoroethane. The type and amount of surfactant are critical. Sodium sulfonate of mineral oil has been found satisfactory as a surfactant. Preferably 0.1 to 0.5 weight percent (on a total solids basis) should be used. Examples of other suitable surfactants are lecithin and Asolectin.

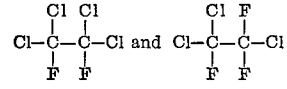
After the ammonium perchlorate (and other propellant solid ingredients, if desired) is dispersed, the propellant binder is mixed in, and the organic liquid is then removed. If desired, the binder can be added after the organic liquid is removed.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Fine ammonium perchlorate (or aluminum or ferric oxide) particles can be completely dispersed in propellants by means of a chemical method of dispersion. The method involves preparing a slurry of fine ammonium perchlorate particles (together with other propellant solid or liquid ingredients, if desired) with a suitable surfactant and an organic liquid. This is done by conventional mixing. These other propellant solids include (usually) fine aluminum powder and any solid catalyst such as ferric oxide. After the solids are dispersed by allowing equilibrium to be established at the ammonium perchlorate-liquid interface or other solid-liquid interface, the propellant binder is mixed in, and the organic liquid is then removed. If desired, the binder can be added after removal of the organic liquid. The curing agent is generally added and mixed in after the removal of the organic liquid, but can instead be added before removal of the organic liquid.

It must be emphasized that the slurry must be allowed to stand until equilibrium is established at the solid(s)-liquid interface, before the propellant binder and other propellant ingredients are mixed in. This allows the surfactant to react with the ammonium perchlorate or other solid surface. The surfactant reaction produces complete dispersion of the ammonium perchlorate in the slurry by effectively neutralizing the charges and reducing the cohesive forces between the particles. The surfactant reduces the interfacial surface tension. Furthermore, the use of a surfactant and an organic liquid enables the surfactant to more readily establish equilibrium at the binder-solid interface as the organic liquid is removed.

The specific organic liquid employed in the tests referred to hereinafter is normal-heptane. Equivalent organic liquids other than normal-heptane can also be employed. Examples are: normal-hexane,



Other organic liquids which can also be employed are ethyl acetate, tetrahydrofuran, benzene, toluene, hexane, acetone and ethyl alcohol. Although still other organic liquids can also be employed, the liquid boiling point and vapor pressure should be low enough so that it can be readily removed during subsequent propellant processing, but not so low that it will readily evaporate during slurry preparation and standing. The ammonium perchlorate must be insoluble in the organic liquid and the surfactant must be soluble in the organic liquid.

The type of surfactant used is critical. A surfactant which will work is Twitchell Base –8240, a trademark of Emery Industries, Cincinnati, Ohio. This surfactant is sodium sulfonate of mineral oil. The amount of surfactant used is also important, because it affects the final propellant properties. The amount of Twitchell Base –8240 used is preferably from about 0.1 to about 0.5 percent by weight (on a total solids basis). Amounts of surfactant less than about 0.1% may not be completely effective while amounts in excess of 0.5% may have detrimental effects on final propellant properties. If the organic liquid is used without the surfactant, the desired ammonium perchlorate dispersion is not achieved.

Generally speaking, the method of this invention is concerned with dispersing fine ammonium perchlorate, aluminum or ferric oxide particles having less than 50μ weight-median diameter. The particles to be dispersed must be in the organic liquid-surfactant slurry.

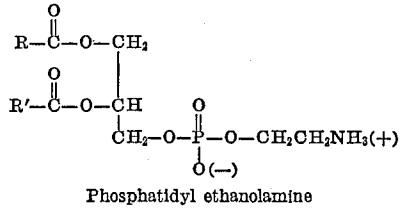
The foregoing procedure has been tested and has resulted in complete dispersion of the fine ammonium perchlorate particles in the normal-heptane slurry (as established by using an accepted packing fraction measurement procedure). Using a nominal 4μ weight-median diameter ammonium perchlorate particle size (which size is normally very difficult to disperse), the dispersion anal-

ysis for a normal-heptane slurry without the surfactant showed a measured packing fraction (ϵ) of 0.34. The same analysis for a normal-heptane slurry with 0.2% of Twitchell Base –8240 showed ϵ to be 0.77. The much larger ϵ for the slurry with the surfactant added shows that the 4μ ammonium perchlorate was completely dispersed.

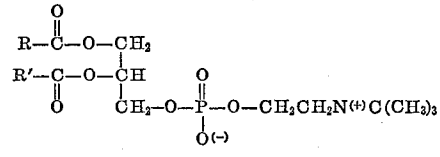
Certain other equivalent proprietary surfactants (both of Emery Industries and other companies) can also be employed. In general, these surfactants are sodium sulfonates of mineral oil. By mineral oil is meant that portion of petroleum which distills at 626–734° F., is purified and redistilled (boiling at 680° F.). Mineral oil is a mixture of long-chain aliphatic hydrocarbons.

Surfactants other than sodium sulfonates of mineral oil can also be employed to achieve the solids dispersion. In particular, the following surfactants have also been demonstrated to be effective:

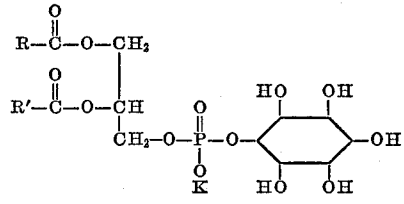
(1) Asolectin (marketed by Associated Concentrates, Inc., Long Island, N.Y.). This material from soybeans is a mixture consisting essentially of approximately equal parts (by weight) of phosphatidyl cholines (lecithin), ethanolamines, and inositols. This is a qualitative estimate. Numerous unknown liquids are also present as well as antioxidant pigments. The structures of the main ingredients are:



Phosphatidyl ethanolamine



Phosphatidyl choline (lecithin)



Phosphatidyl inositol

(2) Lecithin (marketed by Central Soya Co., 1825 N. Laramie Avenue, Chicago, Ill.). This is a fluid natural product from soybean oil. The structure is given above.

The method of this invention eliminates most of the disadvantages of the prior art mechanical methods of dispersion. The advantages of this invention are:

(1) The procedure is safer than mechanical methods of dispersion, because little work is required and the slurry is less sensitive than the dry ammonium perchlorate.

(2) All of the ammonium perchlorate in the final propellant formulation can be added using the chemical dispersion method of this invention.

(3) The particle size of the ammonium perchlorate is not altered.

(4) Better dispersion of the ammonium perchlorate is achieved, which results in better propellant mechanical properties and easier propellant processing.

(5) Higher solid loadings for high density impulse propellants can be obtained by optimum combinations of fine ammonium perchlorate sizes with larger ammonium perchlorate sizes.

(6) High burning rates can be obtained by high solid-loading of fine ammonium perchlorate sizes.

The method of this invention is beneficial for any propellant which contains ammonium perchlorate, aluminum or ferric oxide that is difficult to disperse, but is especially applicable to high burning rate propellants that contain high percentages of fine ammonium perchlorate and aluminum and/or ferric oxide.

Various other modifications and variations of this invention will readily suggest themselves to those skilled in the art in the light of the above teachings, which modifications and variations are within the spirit and scope of this invention.

We claim:

1. A method for dispersing fine ammonium perchlorate, aluminum or ferric oxide particles of less than 50μ weight median diameter comprising the steps of mixing a slurry of said fine ammonium perchlorate, aluminum or ferric oxide particles, an organic liquid and a surfactant, said surfactant being of about 0.1 to about 0.5 percent by weight on a total solids basis, which lowers the interfacial surface tension between said fine ammonium perchlorate, aluminum or ferric oxide particles and said organic liquid, and allowing said slurry to stand until said surfactant reacts with said ammonium perchlorate, aluminum or ferric oxide particles to effectively disperse said ammonium perchlorate, aluminum or ferric oxide particles in said slurry, said surfactant being selected from the group consisting of sodium sulfonate of mineral oil, lecithin, and a mixture consisting essentially of approximately equal parts by weight of phosphatidyl cholines, ethanolamines and inositols, and said organic liquid being selected from the group consisting of normal-heptane, normal-hexane, 1,1,2-trichloro - 1,2,2 - trifluoroethane, 1,1,2,2-tetrachloro-1,2-difluoroethane, ethyl acetate, tetrahydrofuran, benzene, toluene, hexane, acetone, and ethyl alcohol.

2. The method of claim 1 wherein said particles are solid propellant ingredients, wherein a propellant binder and other propellant ingredients that are liquid are added to said slurry after dispersion, and wherein said organic liquid is thereafter removed.

3. The method of claim 2 wherein said organic liquid is normal-heptane, normal-hexane, 1,1,2-trichloro-1,2,2-trifluoroethane or 1,1,2,2-tetrachloro-1,2-difluoroethane.

4. The method of claim 1 wherein said organic liquid is normal-heptane.

5. The method of claim 4 wherein said surfactant is said lecithin in an amount of about 0.2 percent by weight on a total solids basis.

6. The method of claim 4 wherein said surfactant is sodium sulfonate of mineral oil.

7. The method of claim 1 wherein said particles are solid propellant ingredients, wherein said organic liquid is removed from said particles and thereafter a propellant binder and other propellant ingredients that are liquid are added to said dispersed particles.

8. The method of claim 1 wherein said organic liquid is tetrahydrofuran.

References Cited

UNITED STATES PATENTS

2,938,780	5/1960	Proell et al.	149—19
2,942,964	6/1960	Burgwald et al.	149—19
2,946,671	7/1960	Marti	149—19
2,946,672	7/1960	Marti	149—19
2,967,098	1/1961	Weil	149—76 X
2,970,046	1/1961	Cutforth	149—19
2,974,026	3/1961	Batchelder	149—19
2,991,167	7/1961	Burton	149—19
3,027,283	3/1962	Bice	149—76 X
3,109,761	11/1963	Cobb et al.	149—76 X

STEPHEN J. LECHERT, JR., Primary Examiner

U.S. Cl. X.R.

149—40, 42, 44, 87, 109, 113, 114; 44—51; 252—351, 363.5