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3,702,271

AZEOTROPIC REMOVAL OF WATER FROM ORDNANCE

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America as represented by the Secretary of the Navy
No Drawing. Filed Oct. 23, 1970, Ser. No. 83,660
Int. Cl. C06f 11/00

U.S. Cl. 149—38

6 Claims

ABSTRACT OF THE DISCLOSURE

A process for removing water from explosives and propellants wherein the explosive or propellants are mixed with a liquid hydrocarbon, which exhibits an azeotrope with water, and the resultant azeotrope is volatilized from the remaining composition.

BACKGROUND OF THE INVENTION

This invention relates generally to a process for the purification of ordnance materials and more particularly to a process for the removal of water from explosive or propellant ingredients or compositions.

Prior art techniques for the separation of trace quantities of water from explosives and propellant are essentially ineffective. These prior art processes have entailed, for example, time consuming convection air drying or the costly vacuum drying of the ordnance ingredient prior to mixing, with subsequent vacuum mixing and casting of the material.

Since the removal of the water is dependent upon the water's vapor pressure alone, with respect to the final pressure and surface area of the fluid explosive or propellant, only extremely low rates of removal are obtained at the practical vacuums of 1 to 5 mm. of Hg absolute.

Thus, since even the presence of only very small amounts of water in an explosive or propellant ingredient, i.e., 0.1 to 0.2 percent, can react adversely with the other ingredients present in the formulation, such as energetic fuels and oxidizers, and high energy cross-linking agents, a need exists for a more efficient water separation technique.

SUMMARY OF THE INVENTION

Accordingly, one object of the present invention is to provide a process for the separation of water from explosive or propellant ingredients and compositions.

Another object of the present invention is to provide a process for the separation of water from explosive or propellant ingredients and compositions which is simple, fast and economical.

A further object of this invention is to provide a process for providing an essentially water free explosive or propellant.

These and other objects are accomplished by a process wherein the water to be removed is separated from the ordnance material via the volatilization of a low-boiling azeotrope mixture of said water and a liquid hydrocarbon.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The process of the instant invention is applicable to the removal of water from water-wet or merely moisture-wet ordnance materials. It is intended that ordnance material be defined as a single explosive or propellant ingredient, as well as mixtures of various explosive and propellant ingredients or complete explosive or propellant formulations. For example, the process is applicable to the removal of water from single base, double base, composite, and composite-modified double base propellant in-

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gredients and final formulations. Furthermore, it is intended that ordnance materials carrying more than about 2 percent by weight water be considered water-wet while materials carrying less water be considered moisture-wet.

Accordingly, the process of the instant invention is performed by initially slurring the water-wet or moisture wet solid explosive or propellant ingredient or ingredients (including binders and cross-linking agents, if employed) in an organic liquid hydrocarbon which is capable of forming an azeotrope when mixed with water. In practice, any of the dry ingredients such as the cross-linking agents, etc. may be added at a later stage in the process to avoid prolonged contact with the remainder of the propellant formulation.

Organic liquid hydrocarbons which are within the scope of this invention must exhibit an azeotrope with water and, therefore, are pure materials (as contrasted to a mixture of hydrocarbons or oils, etc.) which exhibit a definite boiling point. Furthermore, the liquid hydrocarbons of this invention are preferably volatile, i.e. exhibit a relatively low-boiling point. Specifically, liquid hydrocarbons which are encompassed herein include, for example, hexane, heptane, toluene, benzene and the like. Hexane and heptane are preferred. For the purpose of more conveniently separating the azeotrope components, it is preferred that the liquid hydrocarbon be one that is immiscible with water.

If liquid ingredients of low vapor pressure and high shock sensitivity, such as nitroglycerin, are being employed these may be then added to the initial slurry as an emulsion in the same volatile hydrocarbon or one that forms a ternary azeotrope with the first hydrocarbon and water.

The quantity of liquid hydrocarbon utilized is dependent upon the amount of water quantitatively determined to be present in the various ingredients of the explosive or propellant employed. A sufficient quantity of the hydrocarbon to remove all the water, via the formation of an azeotrope mixture therewith, is used. Thus, to insure the most efficient removal of water, the use of an excess amount of volatile liquid hydrocarbon is preferred.

Additional mixing of the explosive or propellant ingredients is then performed while all the ingredients are dispersed or suspended in the hydrocarbon or hydrocarbons, until a homogenous slurry is attained. It is unnecessary to provide heat or vacuum to the system during these initial mixing and slurring steps of the process although to do so is within the scope of this invention.

In turn, upon stopping the agitation, the mixed explosive or propellant materials settle and allow for the excess hydrocarbon to be decanted. Excess hydrocarbon is defined as that hydrocarbon which has not been mixed with the water in the explosive or propellant material.

At this point, the slurried explosive or propellant materials are further agitated, under vacuum, generally from about 10-40 mm. Hg absolute, and sufficient heat is applied to volatilize off the particular azeotropic mixture of hydrocarbon and water. Generally, when the characteristic odor of the solvent is gone, essentially all of the azeotrope mixture has been removed. This normally takes from about one-half hour to about an hour, depending upon various factors, such as the strength of the vacuum, the type of mixer used, the efficiency of the decantation of the excess solvent, etc.

Furthermore, any conventional azeotropic distillation technique may be employed to remove the azeotrope mixture from the ordnance materials and to subsequently separate the water and hydrocarbon.

After the removal of the azeotropic mixture the explosive or propellant formulation is essentially water-free and may then be cast, using a vacuum, to further insure the removal any remaining hydrocarbon or azeotrope.

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Thus, the process of the instant invention provides a technique for the removal of water contamination from ordnance compositions by the use of the formation of a low-boiling azeotrope mixture. The high efficiency of the process is evidenced by the quantitative determination of water remaining in the ordnance composition, subjected to the process, to be in the order of merely about 0.01 percent by weight.

Having generally described the invention the following examples are given for purposes of illustration. It will be understood that the invention is not limited to these examples, but is susceptible to different modifications that will be recognized by one of ordinary skill in the art.

EXAMPLE 1

196.1 grams of moisture-wet plastisol grade nitrocellulose were placed into a 60 LP ARC propellant mixing bowl along with 196.1 grams of fibrous nitrocellulose, 7.5 grams of potassium sulfate, 7.5 grams of lead carbonate and 50.0 grams of heptane and agitated for three minutes at 100° F. Thereafter, 358.5 grams of a pre-mixed plasticizer of metriol trinitrate and triethyleneglycol dinitrate were added to the mixer and agitated for ten minutes at 100° F. Vacuum conditions were applied to the mixer and the total composition was mixed further at 100° F. until the characteristic odor of the heptane was gone at which time essentially all of the azeotrope mixture of heptane and water was removed. The essentially water-free resultant product was subsequently cast for later use.

EXAMPLE 2

113.4 grams of moisture-wet plastisol grade nitrocellulose, 165.0 grams of aluminum, 180.2 grams of metriol trinitrate, 40.0 grams of triethyleneglycol dinitrate, 3.4 grams of ethyl centralite, 3.0 grams of resorcinol, 250 grams of ammonium perchlorate, and 30 grams of heptane were agitated in a 60 LP ARC propellant mixer bowl at 90° F. for ten minutes. An additional 245.0 grams of ammonium perchlorate were then added and the total slurried composition mixed, under vacuum, for thirty minutes at 100° F. at which time the removal of the heptane-water azeotrope mixture was complete. The essentially water-free propellant composition was cast and stored for later use.

EXAMPLE 3

327 grams of moisture-wet plastisol grade nitrocellulose and 40 grams of heptane were added to a 60 LP ARC propellant mixer at 120° F. under vacuum. 510 grams of metriol trinitrate was then added to this slurry mixture with continued agitation for about 10 minutes. Thereafter, an additional 163 grams of moisture-wet plastisol nitrocellulose was added to the mixer and agitated for another 10 minutes at 120° F. under vacuum, which resulted in the complete removal of the heptane-water azeotrope mixture. The resultant product was essentially water-free.

Obviously, numerous modifications and variations of the present invention are possible in the light of the above teachings. For example, nitrocellulose in physical forms other than the plastisol grade used in the hereinabove examples are also intended to be within the scope of the present invention. Moreover, propellant and explosive formulations containing materials such as cyclotetramethylenetetranitramine and/or cyclotrimethylenetetra-

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nitramine and the like also are obviously encompassed herein. It is therefore to be understood that within the scope of the appended claims the invention may be practiced otherwise than as specifically described herein.

What is claimed and desired to be secured by Letters Patent of the United States is:

1. A process for the removal of water from a water wet or moisture wet ordnance material wherein the ordnance material is any or all of the ingredients of an explosive or propellant composition, which consists of:

mixing said water wet or moisture wet ordnance material with a volatile water immiscible liquid hydrocarbon which is capable of forming an azeotropic mixture with said ordnance material so as to form an azeotropic mixture between said water and said liquid hydrocarbon, and wherein said mixing is performed in amounts such that an amount of said liquid hydrocarbon is used in excess of that needed to completely azeotrope said water; decanting any excess liquid; and volatilizing under vacuum to remove the azeotropic mixture so as to provide essentially liquid-free ordnance material containing no more than 0.01 percent water, by weight of ordnance material, and which is essentially free of said liquid hydrocarbon.

2. The process of claim 1 wherein said liquid hydrocarbon is selected from the group consisting of hexane, heptane, toluene and benzene.

3. The process of claim 1 wherein said water-wet or moisture-wet ordnance material is selected from the group consisting of a single base propellant, a double base propellant, a composite propellant and a composite-modified propellant.

4. The process of claim 1 wherein said water-wet or moisture-wet ordnance material is a mixture of nitrocellulose and metriol trinitrate.

5. The process of claim 1 wherein said water-wet or moisture-wet ordnance material is a mixture comprised of nitrocellulose, metriol trinitrate, aluminum, resorcinol, and ammonium perchlorate.

6. The process of claim 1 wherein said water-wet or moisture-wet ordnance material is a mixture comprised of nitrocellulose and a compound selected from the group consisting of cyclotetramethylenetetranitramine and cyclotrimethylenetetranitramine.

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U.S. Cl. X.R.

149—42, 44, 76, 92, 96, 97, 98, 94, 95, 100, 109