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EXPLOSIVE

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This invention relates generally to explosives and particularly to the manufacture of spherical grains of smokeless powder and a method of controlling the gravimetric density of such grains.

In the manufacture of propellent powder grains in accordance with the processes described in the copending application of Fredrich Olsen, Gordon C. Tibbitts, and Edward B. W. Kerone, now Patent No. 2,027,114, and Serial No. 57,662, filed January 6, 1936; in which a lacquer is made from a smokeless powder base and solvent, the lacquer being distributed in a non-solvent medium in the form of globules, and the globules hardened to form spherically shaped grains, there is often a tendency for the non-solvent medium in which the globules are suspended to diffuse into the globules of lacquer or even become emulsified therein. Consequently assuming the non-solvent medium to be water, it will be understood that as the solvent is evaporated from the globules of lacquer some water may remain therein. This results in the final grains having interiors of porous structure while the exterior is a continuous substantially impervious surface.

The degree of porosity existing in such propellent powder grains is to some extent determined by the solvent employed in making the lacquer. For instance, with a given series of solvents, such for instance as the acetates (e. g., ethyl, isopropyl, butyl), the degree of the porosity of the powder grains will generally parallel the boiling temperature of the solvent, that is to say, the emulsification of water within the lacquer is easier with the solvents of lower boiling point in a given series, and hence when grains of high density are desired, solvents of the higher boiling points may be employed.

The object of the present invention, generally stated, is to provide a propellent powder grain of the type referred to wherein the porosity or gravimetric density of the grain may be controlled.

Another object of the invention is to provide a process of controlling the porosity of powder grains made according to the processes referred to.

A more specific object of the invention is to provide a process of making powder grains wherein globules of smokeless powder base lacquer are suspended in a nonsolvent medium and hardened, and wherein the action of nonsolvent medium within the globules is controlled.

Other objects will become apparent to those skilled in the art when the following description is read:

In accordance with the present invention, generally stated, while the globules of smokeless powder base lacquer are dispersed in a non-solvent medium and the globules thus hardened to form grains in the manner described in said copending application of Olsen, Tibbitts and Kerone, and while some non-solvent may be contained within the globules of such lacquer, the physical properties of the non-solvent medium within and without the globules are differentiated so as to cause relative migration of the non-solvent within the globules and the non-solvent without the globules. For instance, in case the lacquer has been first formed in the presence of the non-solvent vehicle, such as water, in the manner described under the heading "Distributed solvent process" in said application of Olsen, Tibbitts and Kerone, Serial No. 598,332, some water is emulsified within each globule so that the globules, instead of being composed entirely of smokeless powder base lacquer, are indeed an emulsion of water in smokeless powder base lacquer. Now when the globules of smokeless powder base lacquer containing the non-solvent such as water are hardened by heating while suspended in a non-solvent vehicle having the same physical properties as that emulsified within the globules (i. e., water), it will be understood that the globule may be completely hardened to form a grain before the water in the interior thereof is removed. Now in accordance with the present invention the non-solvent vehicle employed as the continuous phase of the system in which the globules are hardened has different physical properties than the non-solvent medium within the globules. In the case of water the change of physical properties may be brought about by the addition of a solute. The result may even be obtained by emulsifying distilled water within the globules while the continuous or external phase is composed of undistilled or untreated water.

A great variety of substances may be employed as the solute to be added to the water. For instance, sodium sulphate, barium nitrate, and potassium nitrate, are particularly suitable, but any substance which actually dissolves in the non-solvent medium, such as water, and changes its physical properties, for instance, sugar, may be suitable.

Likewise, it will be understood that entirely different non-solvent media may be employed, that is to say, the non-solvent within the globules may be a different one from that which constitutes the continuous or external phase of the system.

While in the foregoing general description and in the specific embodiment to be described hereinafter, reference is particularly made to the addition of solutes to the continuous or external phase of the system in which the globules are hardened, it is to be understood that this is the procedure which is followed when dense grains, that is grains of low porosity are desired. It is to be understood, however, that the solute may be added to the non-solvent vehicle which becomes emulsified within the globules and omitted from the continuous external phase in order to cause a diffusion of the water from the external phase into the globules during hardening, thus to produce a grain of high porosity. Of course, the solute need not be entirely omitted from the water of the continuous phase, it being understood that control of the degree of porosity is made possible by the addition of varying amounts of solute to the non-solvent vehicle within or without the globule.

When ethyl acetate is used as the solvent, as described under the heading "Distributed solvent process" in said Olsen, Tibbitts and Kerone patent and the non-solvent medium within and without the globules has the same physical properties, powder having a gravimetric density between 0.250 and 0.350 may be expected. By the addition of a solute, for example 1% of sodium sulphate (weight of nitrocellulose), to the water constituting the continuous phase of the system the gravimetric density of the powder grains is increased to about 0.800, other conditions remaining constant.

When isopropyl acetate is used as the solvent in said "Distributed solvent process" and the non-solvent vehicle within and without the grains has the same physical properties, nitrocellulose grains having a gravimetric density of about 0.500 may be expected. By the use of solutes in accordance with the present invention the gravimetric density may be controlled within wide limits. For example, other conditions remaining constant, the addition of 0.1% of sodium sulphate (by weight of nitrocellulose) is effective to increase the gravimetric density of the powder to 0.675; 0.2% of sodium sulphate increases the gravimetric density to 0.720; 0.4% of sodium sulphates increases the gravimetric density to 0.830; 0.6% increases the gravimetric density to 0.850. These gravimetric densities may be further increased by surface treating with nitroglycerine, for instance, the powder having a grivimetric density of 0.850 will readily take up enough nitroglycerine to reach a gravimetric density of 0.950. Likewise by the use of larger amounts of solute, gravimetric densities as high as 1.0 may be obtained. Comparable results are obtained by the use of other solutes in amounts depending upon their effect upon the physical properties of the water. For instance, 25% less barium nitrate than sodium sulphate is required to give comparable results.

Whether the results achieved by the use of solutes are attributable to an osmotic action, a differential of vapor pressure, or other phenomenon or a combination of them is not determined and hence no theory of action is proffered herein. While the scientific reasons for the action of the solutes as above described are largely uncertain, it is definitely established that when the physical properties of the non-solvent medium constituting the external phase of the system in which the globules of lacquer are hardened, are different from the physical properties of the non-solvent contained within the globules, definite control of the action of the non-solvent medium within the globules, and hence control of the porosity of the resulting grains is made possible. The amount of a particular solute to use in order to produce powder grains of a given porosity or gravimetric density is dependent upon many factors which may be encountered in commercial operation and hence simple experimentation will be necessary in each instance in order to determine the amount of solute necessary to accomplish the desired result. When once obtained, however, for specific operating conditions powder grains of uniform porosity may be expected, other things remaining the same.

While in the foregoing description and the specific illustrative embodiments which have been given, nitrocellulose has been taken as the smokeless powder base, it is to be understood that the present invention is not limited to the treatment of nitrocellulose powder but is applicable to the treatment of other smokeless powder bases, such as nitrocellulose-nitroglycerine, nitrocellulose and deterrents, nitrocellulose-nitroglycerine with deterrents, etc.

From the foregoing description it will be apparent that the present invention accomplishes its objects and provides its propellent powder grain of controlled porosity whereby, although the exterior surface of the grain may be impervious, the internal voids or porosity is so controlled as to achieve the desired gravimetric density. Moreover, in accordance with the present invention powder grains having higher gravimetric densities than have heretofore been attained may be produced.

Since it is apparent that there are various features of the present invention which may be utilized and applied independently of other features and without reference to the specific examples hereinbefore given, it is to be distinctly understood that the invention is not limited to the specific embodiments hereinbefore set forth. Various modifications of the procedure will present themselves to those skilled in the art and, while the present invention is particularly applicable to the manufacture of spherically shaped grains in accordance with the processes of said Olsen, Tibbitts and Kerone applications, it is not intended to limit this invention thereto. It is to be distinctly understood, thereofore, that such modifications and the use of such individual features and sub-combinations of features as may present themselves to those skilled in the art without departing from the spirit of this invention are, although not specifically described herein, contemplated by and within the scope of the appended claims.

Having thus described the invention, what is claimed is:

1. In the art of making explosives the process comprising, dispersing globules of smokeless powder base solution containing fluid non-solvent in a fluid non-solvent bath, the non-solvent within the globules having physical properties differentiating from those of the non-solvent of the bath so as to cause relative migration of the non-solvent within the globules and the non-solvent of the bath, and hardening the globules while the dispersion is maintained.

2. In the art of making explosives the process comprising, forming a lacquer from nitrocellulose and a solvent, agitating the lacquer with a fluid vehicle which is substantially insoluble with the solvent but which is emulsifiable within the

lacquer until some of the vehicle has emulsified within the lacquer, dispersing globules of the lacquer emulsion in the fluid vehicle, changing the physical properties of the fluid vehicle of the continuous phase so as to cause relative migration of the vehicle within the globules and the continuous phase, and hardening the globules while the dispersion is maintained.

3. In the art of making explosives the process comprising, forming a lacquer from nitrocellulose and a solvent, agitating the lacquer with a vehicle which is substantially insoluble with the solvent but which is emulsifiable within the lacquer until some of the vehicle has emulsified within the lacquer, dispersing globules of the lacquer emulsion in the vehicle, adding a solute of such a nature to the vehicle of the continuous phase so as to cause migration of the vehicle from the globules to the continuous phase, and hardening the globules while the dispersion is maintained.

4. In the art of making explosives the process comprising, forming a lacquer from a smokeless powder base and a solvent, agitating the lacquer with a fluid vehicle which is emulsifiable within the lacquer, dispersing the lacquer in the form of globules in a fluid vehicle having physical properties differentiating from those of the first vehicle so as to cause relative migration of the non-solvent within the globules and the non-solvent of the bath, and heating the mixture to harden the globules.

5. In the art of making explosives the process comprising, emulsifying a fluid non-solvent in a solution of a smokeless powder base, and removing the solvent from the solution in the presence of a fluid non-solvent having physical properties differentiating from those of the non-solvent which is emulsified within the solution so as to cause relative migration of the non-solvent within the globules and the non-solvent of the bath.

6. In the art of making explosives, the process comprising, emulsifying water containing a solute in a solution of a smokeless powder base, suspending globules of the emulsion in water the nature of the solute being such as to cause migration of water into the globules, and hardening the globules while the suspension is maintained.

7. In the art of making explosive the process comprising, forming a lacquer from a smokeless powder base and a solvent, agitating the lacquer with a fluid vehicle which is emulsifiable within the lacquer, dispersing the lacquer in the form of globules in a fluid vehicle having physical properties differentiating from those of the first vehicle so as to cause relative migration of the non-solvent within the globules and the non-solvent of the bath, heating the mixture to harden the globules, and surface treating the hardened globules with nitroglycerine.

8. A propellent powder grain composed of a solidified droplet of gelatinized smokeless powder base having a gravimetric density in excess of 0.500.

9. A propellent powder grain composed of a solidified droplet of gelatinized smokeless powder base having a porous interior and a substantially impervious surface, treated with nitroglycerine and having a gravimetric density of at least 0.950.

10. A propellent powder grain composed of a solidified droplet of gelatinized smokeless powder base having a porous interior and a substantially impervious surface, and having a gravimetric density of 0.675 to 0.950, the degree of porosity varying inversely as the gravimetric density.

11. In the art of making explosives, the process comprising, dispersing globules of smokeless powder base solution, containing occluded fluid, in a fluid external phase which differentiates from the occluded fluid to cause relative migration of the entrapped fluid and the fluid of the external phase through the globules, and hardening the globules.

12. In the art of making explosives, the process comprising, dispersing globules of smokeless powder base solution, containing occluded fluid, in a fluid external phase, one of the fluids containing sufficient of a solute of such a nature as to cause relative migration of the entrapped fluid and the fluid of the external phase through the globules, and hardening the globules.

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