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[54] **METHOD FOR PRODUCTION OF SPHERICAL PARTICLES OF A SOLID MATERIAL**

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[51] **Int. Cl.** **C06b 21/02**

[58] **Field of Search** ..... 264/3 C, 3 D, 3 E, 3 R; 149/46, 61, 76, 105, 109

[56] **References Cited**

UNITED STATES PATENTS

1,612,167	12/1926	Beardsley et al.	264/3 D
2,691,800	10/1954	Seavey	264/3 E
2,852,360	9/1958	By	264/3 E X
3,459,607	8/1969	Paustian et al.	264/3 R X

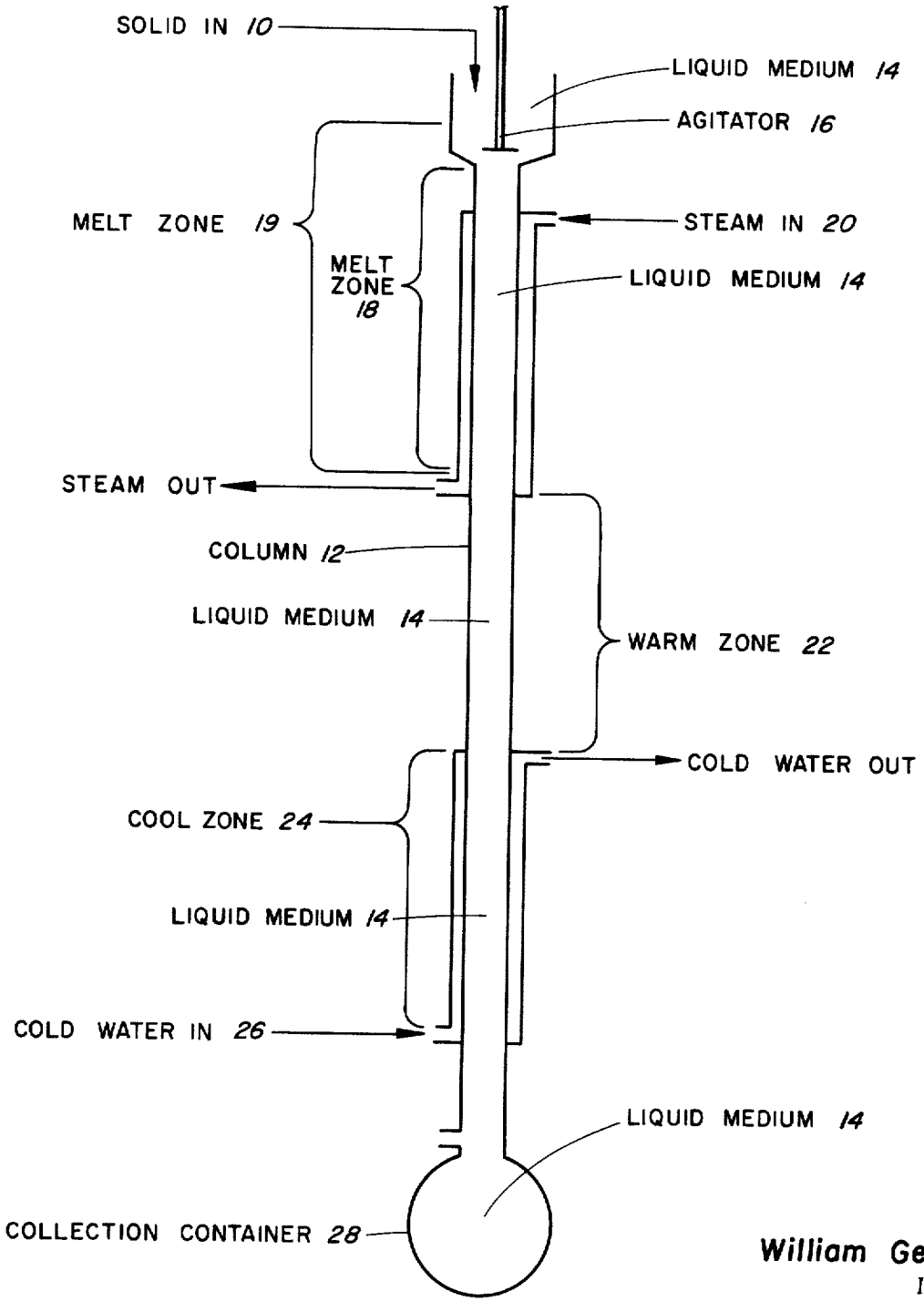
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[57] **ABSTRACT**

A process for making spherical solid particles, especially useful for solid explosives and propellant ingredients, wherein the solid material, such as hydroxylammonium perchlorate, is subjected to a temperature above its melting in a liquid medium, such as tetrachloroethylene, and thereby being transformed into its molten state is passed through a series of successively lower temperatures of the same liquid medium to form the rounded particles.

9 Claims, 1 Drawing Figure



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METHOD FOR PRODUCTION OF SPHERICAL PARTICLES OF A SOLID MATERIAL

BACKGROUND OF THE INVENTION

This invention relates generally to a process for making spherical particles of solid materials and more particularly to a process for making spherical particles of a solid crystalline material especially useful as a propellant ingredient.

It is sometimes desirable for certain materials and for special applications of other materials to be in the form of spherical, pellet-like particles. For example, to minimize the harmful effects in shipping, handling and storing certain poisonous materials, it is desirable to have them in the form of relatively dense spherical particles rather than in fine particle, dust or lump form.

In the propellant industry, high solids loading, that is the capability to utilize as much solid propellant ingredient as possible, such as oxidizer, fuel, etc., in as little space as possible to reduce rocket weight, is also highly desirable. Thus, bulk density is an important consideration in determining the feasibility and performance of a material to be added to or used as a propellant ingredient.

Often because of its crystal size and shape, it is difficult to provide a material, which possesses all the other desirable characteristics for a propellant ingredient, with a high enough bulk density to make it useful as a propellant ingredient. Bulk density can be increased, however, by homogeneously mixing different average sizes of spherical particles. In addition, spherical particles avoid the stress problems associated with irregular particles and also increase the fluidity of propellant formulations.

In the past, the processes for manufacturing solid spherical particles of materials, although satisfactory for the most part, have been unsatisfactory particularly for propellant ingredients due to the tendency of these processes to alter more than just the physical appearances of these solids and also their incapability to cope with the hazards associated with the customary explosive nature of propellant ingredients.

SUMMARY OF THE INVENTION

Accordingly, it is one object of this invention to provide a process for making spherical particles of a solid material.

Another object of the present invention is to provide a process for making spherical particles of a solid wherein the solid is not altered in any way except to change its physical configuration.

Still another object of this invention is to provide a process for making spherical particles of a solid especially suited as an explosive or propellant ingredient.

These and other objects are achieved by a process wherein the solid particles are made molten in a liquid medium and then permitted to pass through a series of progressively lower temperatures of the same liquid medium and recovering the rounded particles.

BRIEF DESCRIPTION OF THE DRAWING

The sole figure is a simplified diagrammatic representation of the type of apparatus which may be employed in carrying out the process of the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

In general, the present invention is applicable to the production of spherical particles of any solid material having a defined melting point or melting range provided the solid does not undergo decomposition at a temperature below its melting point or range. More specifically, the process of the present invention is particularly suited for the production of spherical particles of crystalline materials employed as explosives, explosive ingredients, propellants or propellant ingredients, which also have a defined melting point or melting range and also with the proviso that the substance does not undergo decomposition at a temperature below its melting point or range.

Among solid substances within the scope of the present invention are, for example, urea, dinitrobenzene, hydroxylammonium perchlorate, trinitrotoluene (TNT), ammonium nitrate, potassium perchlorate, etc.

The process of the present invention may be better understood by reference to the drawing wherein the desired solid substance is added to a receiving section **10** of a column **12** which are both filled with a liquid medium **14.** The choice of fluid medium **14** utilized Is dependent upon the solid substance employed. The liquid medium, however, must be defined by the following characteristics: (1) the material must be a liquid at the temperature at which the solid substance to be rounded melts, (2) the material should not dissolve the solid substance to any significant extent, (3) the liquid medium must not be reactive with the solid, (4) the medium and solid must be compatible, and (5) the liquid density must be less than the density of the solid. Some liquid media which are particularly well suited for the purposes of this invention include, for example, tetrachloroethylene, trichloroethylene, carbon tetrachloride, nitropropane, toluene and the like. Even water may be utilized if it meets the hereinbefore described criteria set forth for the liquid media.

The receiving section **10** of the column **12** is preferably fitted with an agitator **16** to prevent any undue coalescing of the solid particles and too further provide for an even distribution of the solid as it falls through the column **12.** Moreover, at times it may be desirable to decrease the original particle size and therefore to employ a high speed agitator **16.**

A significant distinguishing characteristic of the process is that the solid substance is melted in the liquid medium **14** by permitting the solid particles to fall freely and directly from the receiving preferably **10** to a melt zone **18** (filled with the liquid medium **14**) which has been heated to a temperature, preferfably not more than 10° to 15°, and even more preferably not more than about 4° to 5° above their melting point. The heat may be provided by any energy source, such as the steam jacket arrangement **20,** capable of supplying sufficient heat to the liquid medium to allow the solid particles to melt.

In the prior art processes, the solid particles are traditionally melted in mass and in a "dry" state prior to being passed through a screen plate and then into a cooling fluid. This procedure has two major disadvantages. Firstly, the solids are kept in a dry molten state for long periods of time thereby encouraging decomposition, which is especially undesirable for explosive materials, and secondly, the resulting rounded particles do

not retain their original size (which is sometimes desirable) due to their loss of individuality as a result of the screening plate.

These disadvantages are avoided by the instant process by eliminating any screening device and permitting the solid material to become molten only in the liquid medium **14** for only a short time.

Accordingly, in one embodiment of this invention the solid particles fall through the melt zone **18** and become molten while in another embodiment of this invention, if a smaller than original particle size is desired, the solid particles are made molten in melt zone **19** (the initial portion of which initiates in receiving section **10** and the remaining portion of which coincides with melt zone **18**) and a high speed agitating action is applied wherein the larger molten droplets are made into smaller droplets.

The melt zones **18** or **19**, in which the particles begin to become spherical, is extended only enough to allow for the melting of all the particles falling through. In other words, the melt zones **18** and **19** should be as small as possible so that the particles do not remain molten for any unnecessary amount of time.

In both embodiments of the instant invention the spheres of molten solid are allowed to continue to fall through a warm zone **22** of the liquid medium **14** where they are slowly cooled and further solidified before falling to the next lower cool zone **24.** The warm zone **22** is merely a transition zone of melt temperature liquid medium and below melt temperature liquid medium. Thus, no outside source of energy is required to set the temperature of the liquid medium **14** therein. The temperature within the cool zone **24** may be provided by anny suitable means capable of cooling the liquid medium **14** to any temperature below the melting point of the solid, such as the cold water jacket arrangement **26.**

After passing through the cool zone **24,** the solid spherical particles fall into a collection container **28** and thereafter are removed from the column **12.** The desired spherical particles are removed from the liquid medium **14** and recovered by any conventional means such as decantation, centrifugation, filtration, or distillation, etc.

The general nature of the invention having been set forth, the following examples are presented as specific illustrations thereof. It will be understood that the invention is not limited to these examples but is susceptible to different modifications that will be readily recognized by one of ordinary skill in the art.

EXAMPLE 1

Hydroxylammonium perchlorate (HAP) particles (m.p. 95° C) of approximately 500–1000 micron size were fed at the rate of about one gram per minute through a ½ inch diameter column filled with tetrachloroethylene (b.p. 121° C) and arranged similar to the one shown in the sole figure of the drawing. The HAP solid was melted in the liquid medium and the molten droplets were then subjected to a high speed agitation provided by a "Vertis 45" mixer. The resulting molten droplets were then permitted to fall through the remaining portion of the melt zone and subsequently through the warm and cool zones of the column. By controlling the temperature in the melt zone and the rate of agitation, solid spherical particles of HAP ranging in size from about 100 to about 600 microns were recovered. The spherical particles were removed from the tetrachloroethylene by decantation of the liquid and subsequent washing with carbon tetrachloride in which they were stored. The yield was virtually 100 percent, with any loss being associated with the melted material adhering to the walls of the column or agitator.

EXAMPLE 2

Hydroxylammonium perchlorate (HAP) particles (m.p. 95° C) of approximately 200 micron diameter and 700 microns in length were fed through a column which was set up similar to the one shown in the sole figure of the drawing. The solid particles were only mildly agitated in the tetrachloroethylene liquid medium (B.P. 121° C) before being allowed to fall through the melt zone and subsequent warm and cool zones of liquid medium in the column. Upon complete solidification, rounded particles of approximately 350 microns in diameter were recovered by decantation of the liquid medium and washings with carbon tetrachloride in which the particles were stored.

Obviously, numerous modifications and variations of the present invention are possible in the light of the above teachings. For examole, to prevent any significant loss of solid, it may be advantageous to provide for agitation throughout the various temperature zones of the process. Such agitation will help to prevent the solid from sticking to the walls of the column or coalescing because of contact of molten droplets. 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 as new and desired to be secured by Letters Patent of the United States is:

1. A process for making spherical particles of a solid explosive or propellant material which comprises subjecting said solid explosive or propellant material to a temperature above its melting point in a liquid medium, passing the molten solid thereby formed through a progression of successively lower temperature zones of the same liquid mediums and collecting the solidified spherical particles.

2. The process of claim **1** wherein the solid material is subjected to a temperature of not more than about 4 to 5° centigrade above its melting point and wherein at least the last of said successfully lower temperature zones of liquid medium has a temperature below the melting point of said solid material.

3. The process of claim **2** wherein said liquid medium is defined by the characteristics of: (*a*) remaining a liquid at the temperature at which said solid material melts, (*b*) non-dissolving said solid material to any large extent, (*c*) unreactive with said solid material, (*d*) compatible with said solid material and, (*e*) having a density of less than the density of said solid material.

4. The process of claim **3** wherein the solid material is added to the liquid medium and agitated therein prior to being subjected to a temperature above its melting point.

5. The process of claim **3** wherein the solid material is added to the liquid medium, subjected to a temperature above its melting point in said liquid medium and simultaneously subjected to high speed agitation.

6. The process of claim **4** wherein the solid material is selected from the group consisting of hydroxylammonium perchlorate, urea, dinitrobenzene, trimitrotoluene, ammonium nitrate and potassium nitrate.

7. The process of claim 4 wherein the solid material is hydroxylammonium perchlorate and the liquid medium is tetrachloroethylene.

8. The process of claim 5 wherein the solid material is selected from the group consisting of hydroxylammonium perchlorate, urea, dinitrobenzene, trinitrotoluene, ammonium nitrate and potassium nitrate.

9. The process of claim 5 wherein the solid material is hydroxylammonium perchlorate and the liquid medium is tetrachloroethylene.

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