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SMOKELESS POWDER AND PROCESS OF MANUFACTURING SAME

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This invention relates to an improved process for the manufacture of smokeless propellent powders. While the invention is adapted to the manufacture of nitrocellulose propellent powders generally, it is applicable particularly to the manufacture of such powders for small arms, such for example as pistols, revolvers, rifles and shotguns.

Prior to the present invention propellent powders have been made by a rather wide variety of processes. For example, smokeless powders of the so-called dense type have usually been made by colloiding dehydrated nitrocellulose, in whole or in part, with a mixture of ethyl ether and ethyl alcohol, or with other volatile solvents, such, for example, as acetone, and either with or without the incorporation of non-volatile solvents or plasticizers, and with or without the incorporation of nitroglycerine. After mixing, it has been the practice to press the powder into blocks, these blocks being subsequently pressed through dies so as to form the powder into rods, tubes, ribbons, strips, and the like, or the mass has been passed through or between rolls to form sheets, which are then cut into smaller lengths or grains of various sizes and shapes. These powder grains were then subjected to a solvent recovery treatment so as to reclaim the volatile solvents, such as ether and alcohol, and the like, and subsequently subjected to a drying treatment to reduce the residual volatile solvent to a low point. The dried powder grains were then passed through a screen or sieve to remove imperfect grains and then finally blended and packed. It has also been the practice in some cases to surface treat the powder grains thus formed with deterrent materials or to glaze them with graphite so as to make them burn more progressively.

It is well known that smokeless powders produced by the foregoing processes have a more or less definite web or burning thickness obtained by mechanical means. This web thickness in the case of tubular grains with one or more perforations is the average thickness of the walls of the powder after drying; that is, after shrinkage. In the case of ribbons or films of powder, the web is the average thickness of the same when dried. In the case of powder grains cut very thin, the web may then be the thickness of the cut after shrinkage.

On the other hand, powders of the so-called bulk type are usually granulated in stills or pans into more or less spherical grains of varying sizes which do not possess a definite web or burning thickness. This type of powder is usually more or less porous or spongy, which condition permits a more rapid rate of burning for the same grain size than is obtained with the more completely colloided grains of the dense type.

For example, in the well known Schultze process of manufacture, the ingredients comprising wet nitrocellulose, inorganic nitrates, nitrohydrocarbons, starch, paraffin oil, coloring material, etc. are incorporated under edge-runners similar in type to those used for the manufacture of black powder. After incorporation, the material is placed in large inclined pans which rotate through a tank of hot water or in a steam bath. The heat from the bath causes the water to evaporate slowly from the composition, and as this takes place the powder grains of varying sizes and more or less spherical in shape are formed as the material rolls across the inclined pan. These powder grains are sieved to a more or less definite grain size, dried and hardened by spraying the surface with a mixture of acetone and ethyl alcohol or some other suitable volatile solvent mixture having a colloiding action on the nitrocellulose. This treatment causes the outer surface of the gains to be colloided to a greater extent than the inner parts of the grains. The finished powder, after drying, is sieved again between two sieves to give a more or less definite grain size.

In still another process for the manufacture of bulk smokeless powders, the nitrocellulose together with deterrents or plasticizers are agitated in water or in an aqueous solution of barium or potassium nitrate. During agitation there is added a water insoluble solvent mixture to the nitrocellulose. This solvent may consist of amyl acetate, butyl acetate, or other suitable solvents diluted with benzol or other suitable diluent. The solvent mixture being insoluble in water, is separated into globules by the agitation, and these globules colloid a certain amount of nitrocellulose causing it to form into soft grains or pellets. By applying heat to the mass, which is usually contained in a still, the excess volatile solvent is driven off leaving the powder in the form of relatively hard grains which are more or less spherical in form, and which are more or less colloided depending upon the character, quantity, strength, etc. of the solvent and methods used. The powder grains thus formed are dried and sieved, after which they may or may not be further treated by any of the known methods familiar to manufacturers of smokeless powders.

An object of our invention is the manufacture of an improved propellent powder having a more

or less definite web thickness of predetermined dimensions. A further object of our invention is an improved process of producing propellent powders.

According to one embodiment of our invention we prepare a nitrocellulose-water mixture and partially colloid the nitrocelullose in the presence of water and with the aid of volatile solvents for nitrocellulose which are substantially insoluble in water. The wet mass is then subjected to the action of pressure, by means of rolls or otherwise, in order to further colloid and form the mass into sheets or flakes of predetermined thickness and consistency. The volatile solvents are then substantially all removed from the so treated material, and the sheets or flakes are subsequently reduced to the proper grain sizes suitable for loading purposes.

In the foregoing process we preferably remove the excess water present in the mixture before the second step of colloiding and gelatinizing by means of pressure applied to the mixture. While we preferably pass the material through a set of rolls to accomplish this second colloiding and gelatinizing effect, various ways of accomplishing this may be used such as kneading or churning the material. In any case considerable pressure should be applied. One of the advantages accomplished in passing the mixture through rolls is that the rolls may be set to give a predetermined web thickness to the grains or flakes. The reduction of the material to proper grain size may be accomplished in various ways but we preferably pass the material in the presence of water through a cutting machine such as an attrition mill or a Jordan engine, or other similar apparatus.

A further and more specific embodiment of our invention comprises preparing the nitrocellulose-water mixture by agitating the materials together to form a slurry. Other ingredients, as desired, such as solvents, stabilizers, inorganic salts, deterrents, and/or plasticizers, may be added to the slurry during agitation. The excess water is then removed from this slurry by passing the mass over a screen or otherwise. The slurry may be conveyed to a second screen for removing further amounts of water or this may be done by means of a screw conveyor which further presses out excess water. The material is then subjected to the desired pressure to further colloid and gelatinize the mass. This may be done by passing the mass through rolls set to form the mass into sheets or flakes of predetermined thickness. The material may then be subjected to regulated heat treatment to remove the volatile solvent, and may be cut to the desired grain or flake size. Before the solvent is removed it may be desirable in some instances to subject the material to further agitation after leaving the rolls so as to break up the sheets and facilitate the removal of the solvents when heat is applied. A further and more specific embodiment of our invention is given in the following illustration:

We make a slurry of water, or of a water solution of inorganic nitrates and/or carbonates, such for example as potassium and/or barium, or the like, and nitrocellulose of a quality suitable for propellent powders, together with, if desired, any stabilizing material and also any deterrent or plasticizing material such as charcoal, starch, paraffine oil, dinitrotoluol, dimethyldiphenylurea, diethyldiphenylurea, dibutylphthalate, and like compounds suitable for this purpose. To this slurry we add, during agitation, a quantity of volatile solvent or solvent mixtures for the nitrocellulose. The volatile solvent which we prefer to use is one substantially insoluble in water, such as amyl, butyl or ethyl acetate or mixtures of these with other water insoluble solvents. These solvents may or may not be diluted by the addition of water insoluble diluents such as benzol, toluol, petroleum spirits, and the like. The paraffine oil, dinitrotoluol, dimethyldiphenylurea, etc., referred to in the foregoing may, if desirable, be dissolved in a solvent rather than mixed with the nitrocellulose and then added to the slurry with the solvent mixture. After the addition of the solvent mixture the mass is agitated until it shows a tendency to granulate due to the colloiding action of the solvent mixture on the nitrocellulose. At this point excess water is drained off and the partially colloided mass of nitrocellulose composition, together with occluded water is then passed through one or more sets of rolls. Before doing so however, we may also pass the material through a mechanical straining machine such as a rubber straining apparatus which assists in the removal of the excess water and at the same time aids in further colloiding the material. The pressure exerted by the rolls tends to further colloid or gelatinize the nitrocellulose. The extent of this rolling treatment can be modified to some degree according to the character of the finished powder desired. In the final passage of the material through the rolls, the rolls are set to give a more or less definite and predetermined thickness to the leaf-like masses or sheets of colloided material. No one thickness can be specified as this must be regulated according to the characteristics of the guns or ammunition for which the powder is being made. However, for powder for a specific purpose, a certain thickness is predetermined and this is controlled by the degree of rolling and final setting of the rolls. The material at this point is relatively soft and rich in solvent. For this reason the soft sheets or leaf-like masses of powder, together with a quantity of water, are transferred to a still and the excess solvent is boiled off and recovered. This operation is continued until the residual volatile solvent left in the powder mass is less than approximately 1%. This operation also has the effect of hardening the colloided composition. The resulting material is cut to a suitable size by various means or by any suitable machine such as the Jordon engine or an attrition mill, and is sieved to the desired grain size, after which it may be dried, glazed with graphite, or the surface may be impregnated with dinitrotoluol, dimethyldiphenylurea or other suitable impregnating material by any of the well known methods used for this purpose. In this manner we obtain a propellent powder possessing a rather definite web or burning thickness which together with the composition and subsequent surface impregnating treatment may be regulated to give certain desired ballistics which will be readily understood by those skilled in the art.

Powder made according to our process of manufacture is suitable for use in small arms such as revolvers, pistols, rifles, and shotguns and for various purposes to which small arms powders are applicable. The powder may also be made in larger granulations for use in military ordnance, such for example as howitzers, field guns, etc.

The resulting powder according to our invention, while having substantially all of the advantages of both the bulk and dense type of powders, the process has very definite economic advantages, a principal one being in reduced cost of manufacture of powder having a definite web

thickness. For example, the manufacture of pressed nitrocellulose and nitroglycerine powders involves the dehydration of the nitrocellulose, colloiding of the dehydrated material, pressing the colloid after mixing through dies and conveying the strings to a cutting machine to be cut into grains. All of these steps require excessive handling of the material. The costs of dehydrating, pressing, cutting and handling the powder, together with high solvent losses, are relatively large. These costs are materially reduced by pressing a wet colloid through rolls, which may be enclosed to reduce solvent losses, using a water insoluble solvent mixture which may be recovered by boiling the leaf-like masses in water and finally cutting up the hardened leaf-like masses. By our process these operations can be carried out in the manner indicated, thus eliminating cost of handling of powder.

While various embodiments of our new process exist and may be practiced within the scope of our invention, it is to be understood that we do not intend to be limited in the foregoing description and illustrations except as indicated in the following patent claims.

We claim:

1. The process of making propellent powders which comprises preparing a nitrocellulose-water mixture, partially colloiding the nitrocellulose with a substantially water-insoluble volatile solvent, removing the excess water, further colloiding and gelatinizing the nitrocellulose by mechanical means, and removing substantially all the solvent therefrom.

2. The process of making propellent powder which comprises preparing a nitrocellulose-water mixture, incorporating therewith an inorganic salt, partially colloiding the nitrocellulose with a substantially water-insoluble volatile solvent, removing the excess water, further colloiding and gelatinizing the nitrocellulose by mechanical means, removing substantially all the solvent therefrom, and reducing the material to predetermined grain or flake size.

3. The process of making propellent powder which comprises preparing a nitrocellulose-water mixture, partially colloiding the nitrocellulose with a substantially water-insoluble volatile solvent, in the presence of a deterrent or plasticizer, removing the excess water, further colloiding and gelatinizing the nitrocellulose by means of pressure, removing the solvent therefrom and reducing the material to predetermined grain or flake size.

4. The process of making propellent powder which comprises preparing a nitrocellulose-water mixture, partially colloiding the nitrocellulose with a substantially water-insoluble volatile solvent, removing the excess water, further colloiding and gelatinizing the nitrocellulose by mechanical means, forming the mass into sheets or flakes of predetermined thickness, removing substantially all the solvent therefrom, and reducing the material to predetermined grain or flake size.

5. The process of making propellent powder which comprises preparing a nitrocellulose-water mixture, partially colloiding the nitrocellulose with a substantially water-insoluble volatile solvent, removing the excess water, further colloiding and gelatinizing the nitrocellulose by mechanical means, forming the mass into sheets or flakes of predetermined thickness, agitating the material to break up the sheets or flakes of material, removing substantially all the solvent therefrom, and reducing the material to predetermined grain or flake size.

6. The process of making propellent powder which comprises preparing a nitrocellulose-water mixture, partially colloiding the nitrocellulose with a substantially water-insoluble volatile solvent, in the presence of an inorganic salt and a deterrent or plasticizer, removing the excess water, and further colloiding and gelatinizing the nitrocellulose by forming the material into sheets or flakes, agitating the material to break up the sheets or flakes of material, heating the material to remove substantially all the volatile solvent therefrom, and reducing the material to predetermined grain size.

7. The process of making propellent powder which comprises preparing a nitrocellulose-water mixture, partially colloiding the nitrocellulose with a substantially water-insoluble volatile solvent, in the presence of a stabilizer, removing the excess water, and further colloiding and gelatinizing the nitrocellulose by forming the material into sheets or flakes, agitating the material to break up the sheets or flakes of material, heating the material to remove substantially all the solvent therefrom, and reducing the material to predetermined grain size.

8. The process of making propellent powder which comprises preparing a nitrocellulose-water mixture, partially colloiding the nitrocellulose with a substantially water-insoluble volatile solvent, removing the excess water and further colloiding and gelatinizing the nitrocellulose by passing the mass through rolls whereby the material is formed into sheets or flakes, heating the material to remove substantially all the volatile solvent therefrom, and reducing the material to predetermined grain size.

9. The process of claim 8 in which an inorganic salt is incorporated with the partially colloided nitrocellulose before the excess water in removed.

10. The process of claim 8 in which a deterrent or plasticizer is incorporated with the partially colloided nitrocellulose before the excess water is removed.

11. The process of claim 8 in which an inorganic salt and a deterrent or plasticizer is incorporated with the partially colloided nitrocellulose before the excess water is removed.

12. The process of claim 8 in which the sheets or flakes are broken up before the volatile solvents are removed.

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