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PROCESS FOR THE MANUFACTURE OF SMOKELESS POWDER

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This invention relates to an improved process of making smokeless powder and to the product so made.

In the prior art it has been customary to manufacture smokeless powder for use in shot shells, rim fire rifle cartridges, pistol cartridges and center fire rifle cartridges, by colloiding nitrocellulose with solvents such as, for example, a mixture of ethyl alcohol and acetone, ethyl ether and alcohol with or without the addition of nitroglycerine. Various modifying agents have also been added such as stabilizers, oxidizing salts, deterrents, etc., in order to impart various desired properties to the powders. After the powder is sufficiently colloided it is pressed through a die into strands and these strands then fed through rolls against revolving knives, which cut the strands into small flakes, as many at 325 flakes being cut from a 1 inch strand of powder.

It has also been the practice of the prior art to colloid nitrocellulose with nitroglycerine or other explosive plasticizers with or without the use of additional solvents and then passing the resulting colloid between rolls to produce sheets of the desired thickness. These sheets were then cut by various means to produce flakes of the desired shape and size.

Powders prepared in this manner, however, have had the disadvantage of not charging very uniformly in automatic measuring machines, because they had flowing properties which did not insure that substantially the same weight of powder would fill the measuring cup of the machine each time. An explanation for this may lie in the fact that when a knife cuts off a flake from the strand or a grain from a sheet, it leaves a feather edge turned up at an angle to the face of the flake which then catches against other flakes and thus prevents them from sliding over each other in a satisfactory manner.

In order to obtain powders giving uniform loading qualities and the ballistic advantages obtained thereby, it was proposed that the colloided powder be passed between the rolls or subjected to other suitable means to produce sheets of desired thickness, but instead of cutting these sheets to produce the individual powder grains, grinding the sheets in a suitable apparatus to produce flat, irregularly shaped grains having, however, a uniform predetermined web thickness. For this process to be effective, however, compositions are necessary which will produce a brittle sheet after rolling or after any other treatment in which a sheet is formed, so that the sheet can be reduced to the proper grain size in any convenient manner without necessitating cutting to the desired size with knives, thus eliminating the disadvantages of the rolled and cut type of powder.

While this process has operated satisfactorily with single base powders using solvents easily eliminated during a rolling treatment and using powders containing only sufficient plasticizer, explosive or non-explosive to produce a brittle sheet after the rolling treatment, it has not been satisfactory for producing powders having a high plasticizer content. Heretofore, the total non-volatile plasticizer content has been limited on the low side by a minimum that could be used, still obtaining a plasticized sheet on the rolls. There is considerable variation in this lower limit depending upon the type nitrocellulose used, the plasticizer or plasticizers used, and the temperature of the rolls, but when, for example, nitroglycerine constitutes the major portion of the plasticizer, this lower limit is about 25% by weight of the mixture. In a similar manner the upper limit is about 35% by weight of the mixture, quantities in excess of this not producing a sufficiently brittle sheet which can be ground or broken into suitable sized grains.

Now in accordance with my invention, a process is provided whereby smokeless powder with a non-volatile solvent plasticizer content greater than about 35% by weight of the mixture, and having a predetermined web thickness, can be reduced to the proper grain size by grinding, or breaking by other means, without necessitating cutting to the desired size by means of knives or similar cutting machines.

More particularly, I have found that powders with a non-volatile solvent plasticizer content greater than 35% by weight of the mixture after being formed into sheets of predetermined uniform thickness by suitable means, such as by rolling, or otherwise may be broken into the proper grain size by cooling the sheeted powder to subnormal temperatures and grinding the sheets at these low temperatures in suitable equipment. To maintain the sub-normal temperature and at the same time avoid fire or detonation through impact or friction, cold water, a brine solution or other non-explosive liquids may be used during the grinding.

With nitroglycerine as the non-volatile solvent plasticizer I have found that when the content of nitroglycerine in the powder is about 35% by weight thereof, grinding under water at a temperature of about 60° F. will produce powder grains of the desired fineness. With a nitro-

glycerine content of 60%, on the other hand, it is necessary to cool the sheet to a temperature of —50° F. before it is brittle enough to be ground to the desired grain size.

In proceeding in accordance with my invention, I may take water-wet, alcohol-wet, or dry nitrocellulose or ground scrap cannon powder, substituted wholly or in part for the new nitrocellulose, and colloid said nitrocellulose with an explosive solvent plasticizer such as, for example, nitroglycerine, ethyleneglycol dinitrate, propyleneglycol dinitrate, diethyleneglycol dinitrate, glycerol dinitrate monochlorohydrin, etc., or mixtures thereof. I may also colloid the nitrocellulose with non-explosive solvent plasticizers among which are, for example, diethyl phthalate, diamyl phthalate, diethyldiphenylurea, etc., or I may colloid the nitrocellulose with what I term a low potential explosive plasticizer such as, for example, dinitrotoluene and trinitrotoluene. Alternatively, I may colloid the nitrocellulose with explosive plasticizers of the type before mentioned in admixture either with non-explosive plasticizers or with low potential explosive plasticizers or with combinations thereof.

It is to be understood where, in the claims, I refer to a non-volatile solvent plasticizer, I include thereby a substance, having a solvent action on the nitrocellulose, which may be either an explosive plasticizer, a non-explosive plasticizer, a low potential explosive plasticizer and admixtures of an explosive plasticizer with either or both of the other types.

I may admix the nitrocellulose with the non-volatile plasticizers above mentioned in suitable equipment such as, for example, a mixer of a type well known to those skilled in the art, in order to produce a uniform mixture of nitrocellulose and said plasticizer and at the same time effect a partial colloiding. Complete colloiding of the nitrocellulose with a plasticizer in an ordinary mixer is difficult to obtain unless a volatile solvent or an excessive amount of heat is used. I have found that after the preliminary admixing and partial colloiding obtained in the mixer, it is preferable to colloid the mass completely by means of rolls, for example, through the combined action of heat, pressure and mixing and after colloiding is complete, to form the colloided mass into sheets of predetermined thickness by means of rolls, properly adjusted to give the thickness desired, or by any other means whereby a sheet or ribbon of predetermined thickness may be obtained.

Instead of using rolls to obtain the desired thickness, I may press the colloided mass through a "ribbon" die and in this manner produce a flat ribbon or strand having the thickness desired for any particular purpose.

When colloiding nitrocellulose with a liquid high explosive plasticizer as, for example, nitroglycerine, nitroglycol or the like, I may make a stable emulsion of the said compounds in dispersed phase in water, which when admixed with water-wet nitrocellulose will produce a partial colloid which is almost non-inflammable and non-explosive until after the water is largely removed, which greatly reduces the hazard of manufacture of smokeless powder of this type.

Prior to or at the time of colloiding of the nitrocellulose, in the manner before indicated, various other ingredients may be added, among which are, for example, stabilizers, inorganic salts and deterrents.

The time for rolling the partially colloided mass is not a critical one and will depend on various factors such as composition of the powder, size and temperature of the rolls, etc. During the time of rolling the insufficiently colloided powder sheet may be removed and put back in the rolls three or four times to give faster and more uniform colloiding. When heated rolls are used, the temperature thereof will depend on the composition of the powder, type of plasticizer being used, etc., but in general a temperature of the rolls obtained by circulating water therethrough of about 120° F. to about 180° F., has been found satisfactory. After the sheet has been sufficiently colloided, it may be given two or three single passes through rolls with the proper adjustment in order to obtain the predetermined desired thickness sheet. In this manner a sheet is obtained which has two smooth parallel surfaces a predetermined distance apart.

After the powder composition has been rolled to the desired predetermined thickness, which is controlled by the degree of rolling, and the final setting of the rolls, the sheet is broken into the proper grain size by grinding the material at subnormal temperatures in the presence of water, a brine solution or the like, in a machine such as an attrition mill or a Jordan engine, or other similar apparatus. After sieving to the desired size, the powder may be dried, glazed with graphite, or it may be coated with a suitable deterrent such as, for example, dinitrotoluene, diethyldiphenylurea and the like, by any of the well-known methods commonly used and then glazed with graphite. Where a brine solution is used in grinding, the powder will be washed free of brine before it is dried and, thereafter, it may be treated as shown before. A propellant powder is thus obtained having a predetermined burning thickness which together with the composition and subsequent coating treatments may be regulated to give ballistic requirements well understood by those skilled in the art.

Having now indicated the procedure, in accordance with this invention, in general terms, I will now proceed to a more detailed description thereof by reference to the following examples.

Example I

A double base powder containing 35% nitroglycerine, 20% of nitrocellulose having a nitrogen content of 13.2%, 43.25% ground pyro powder, 0.75% diethyldiphenylurea and 1% of potassium sulphate was colloided and rolled into sheets having a thickness of 0.005 inch. The sheets so made were then ground under water at a temperature of about 58–60° F. to a fineness such that the powder will pass a 26 mesh screen and be retained on a 60 mesh screen. This powder was then dried, glazed with graphite, and was ready to be loaded into cartridges and fired.

For the purpose of making a progressive-burning smokeless powder, for example, I may coat 20 parts of the above powder before it has been glazed with graphite with 0.4 part of hydrogenated methyl abietate and 0.2 part of ethyl phthalate, dissolved in 2.5 parts of ethyl alcohol. The powder and coating solution is tumbled together in a sweetie barrel at room temperature for ½ hour, after which the powder is dried for 40 hours at 55° C., glazed with graphite and rescreened.

Example II

A double base powder containing 43% nitroglycerine, 53.5% of nitrocellulose having a nitrogen content of 13.2%, 0.5% diphenylamine and

3% diethylphthalate was colloided and rolled into sheets having a thickness of 0.010 inch. The powder sheets so made were then ground under water, cooled to a temperature of about 43° F. Screen tests on this powder showed the following results:

		Per cent
	Held on 24 mesh	9.6
Through 24	Held on 32 mesh	2.9
Through 32	Held on 40 mesh	4.3
Through 40	Held on 60 mesh	43.3
Through 60	Held on -- mesh	39.4
		100.0

The powder grains above prepared were then dried, glazed with graphite, and were ready to be loaded into cartridges and fired.

A progressive-burning powder can be made from this in the same manner as indicated in Example I above.

Example III

A double base powder containing 50% nitroglycerine, 48% nitrocellulose, having a nitrogen content of 12.6%, and 2% diethyl-diphenylurea was colloided and rolled into sheets having a thickness of 0.010 inch. The sheet so made was then ground in a brine solution at a temperature of 12° F. The screen tests on this powder showed the following results:

		Per cent
	Held on 24 mesh	11.9
Through 24	Held on 32 mesh	2.2
Through 32	Held on 40 mesh	4.2
Through 40	Held on 60 mesh	40.8
Through 60	Held on -- mesh	40.9
		100.0

Powder grains so prepared may then be washed free of brine solution, dried, glazed with graphite, and loaded into cartridges and fired. A progressive-burning powder can be made therefrom in the manner before indicated.

I have found that the smokeless powder produced as shown in the examples above burns very cleanly due to its uniform thickness. In fact when the small amount of residue is wiped out of the gun barrel after firing shells loaded with powder made according to my process, no unburned powder will normally be found, the residue being small parts of charred paper from the paper shot shell. Furthermore, the smokeless powder produced in accordance with my invention loads more uniformly on automatic loading machines than prior art powders of the same composition prepared by a process in which the grains are cut from a sheet or from a strand. I attribute the better flowing qualities of my powder to the fact that the grinding leaves a broken edge and the two parallel surfaces formed during the rolling of the powder have no projections or "feather edges" to catch other grains and prevent them from sliding.

It will be understood that where I refer to sub-normal temperatures, I mean thereby temperatures less than those normally found in buildings where smokeless operations are carried out. There will necessarily be a wide sub-normal temperature range where my process may be carried out, as is indicated by the examples above, depending upon the non-volatile solvent plasticizer content of the smokeless powder.

It will also be understood that the details and examples given hereinbefore are illustrative only, and in no way limiting on my invention as broadly described hereinbefore and on the appended claims.

What I claim and desire to protect by Letters Patent is:

1. The process of making smokeless powder containing more than about 35% by weight of a non-volatile solvent plasticizer which comprises colloiding nitrocellulose with said plasticizer present in an amount which is more than about 35% by weight of the powder, subjecting the colloided mass to pressure by mechanical means to produce a colloided smokeless powder sheet having two smooth parallel surfaces a predetermined distance apart cooling said sheet to brittleness and reducing said sheet to grains by grinding.

2. The process of making smokeless powder containing more than 35% of non-volatile solvent plasticizer which comprises colloiding nitrocellulose with said plasticizer present in an amount which is more than about 35% by weight of the powder by means of rolls, further rolling the colloided mass to produce a colloided smokeless powder sheet having two smooth parallel surfaces a predetermined distance apart cooling said sheet to brittleness and reducing said sheet to grains by grinding.

3. The process of making smokeless powder containing more than 35% of non-volatile solvent plasticizer which comprises colloiding nitrocellulose with said plasticizer present in an amount which is more than about 35% by weight of the powder in the presence of substances selected from the group consisting of an inorganic salt, a deterrent and a stabilizer and combination of these, by means of rolls, further rolling the colloided mass to produce a colloided smokeless powder sheet having two smooth parallel surfaces, a predetermined distance apart cooling said sheet to brittleness and reducing said sheet to grains by grinding.

4. The process of making smokeless powder containing more than 35% of non-volatile explosive solvent plasticizer which comprises colloiding nitrocellulose with an aqueous emulsion of said plasticizer present in an amount which is more than about 35% by weight of the powder by means of rolls, further rolling the colloided mass to produce a colloided smokeless powder sheet having two smooth parallel surfaces, a predetermined distance apart cooling said sheet to brittleness and reducing said sheet to grains by grinding.

5. The process of claim 1 in which nitroglycerine present in an amount which is more than about 35% by weight of the powder is used as the non-volatile solvent plasticizer.

6. The process of claim 1 in which diethyleneglycol dinitrate present in an amount which is more than about 35% by weight of the powder is used as the non-volatile solvent plasticizer.

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