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PROGRESSIVE-BURNING SMOKELESS POWDER AND METHOD OF MANUFACTURE

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This invention relates to an improved method of surface coating smokeless powder with a deterrent material, and more particularly to an improved method of surface coating smokeless powder with a synthetic lacquer resin and to a smokeless powder coated with such deterrent material.

As is well known, in order to render possible close control of the burning rate of smokeless powder and produce a high velocity of the projectile without high breech pressures, it has been customary to coat the grains of the smokeless powder with a deterrent material, explosive or non-explosive, which will retard the initial burning rate of the powder grains. Such coating material penetrates the powder grains more or less, so that as the powder burns, the successively exposed surfaces contain gradually less and less of the deterrent material, thus causing the combustion of the grains to proceed with increasing speed.

In order to effect the coating of smokeless powder grains with a deterrent material, numerous processes have been devised, e. g., using dinitrotoluene as the deterrent material, powder grains coated with dinitrotoluene are subjected to heat, with or without the presence of moisture or water. By another method, the powder grains are coated by treatment with a solution of dinitrotoluene in benzene, the benzene evaporated, and penetration of the dinitrotoluene into the grain of powder obtained by immersing the coated powder grains in a hot water bath for several hours or several days. By still another method, using as the deterrent material a mixture of dimethyldiphenyl urea and dinitrotoluene, or dimethyldiphenyl urea alone, the powder grains and deterrent material are covered with water, and after mixing, heat is applied for varying periods.

By still another method the powder grains are surface treated by an alkyl phthalate, either in solution or in aqueous emulsion.

By the use of deterrents which are solvents for the nitrocellulose of the powder grain, great difficulty is experienced in the sticking together or the clustering of the powder grains. This is completely avoided by the use of my invention described below.

Treatment of smokeless powder grains to render their combustion progressive by applying thereto a deterrent by methods heretofore known, while producing the desired results to a degree, has been open to a number of objections. The deterrents heretofore used have been more or less good solvents for the nitrocellulose of the smoke-

less powder, and consequently, even if applied on the surface of the powder grain, have penetrated to a depth into the powder grain depending upon the conditions of treatment, e. g., a longer time of heating of the powder grains with the deterrent or solution of the deterrent causing a deeper penetration of the deterrent into the powder grains than would otherwise be the case.

Powder grains treated with a deterrent which is a solvent for the nitrocellulose of the powder grains tend to penetrate more and more deeply into the powder grains during storage of such progressive-burning smokeless powder, particularly during storage of said powder during hot periods of the year or in hot climates, and consequently the concentration of the deterrent on the surface of the powder grain becomes less and less with the age of the powder grain, and the deterrent action on the surface of the powder grain consequently becomes less and less on storage. The result thereof is that powder grains deterred by a deterrent which is a solvent of the nitrocellulose in the powder grain, do not remain ballistically stable, i. e., the ballistics of the powder change with the age of the powder due to the disappearance of the deterrent from the surface of the powder grains.

Now in accordance with our invention we may utilize as a deterrent for smokeless powder grains, a non-volatile, non-explosive organic substance which is compatible with the nitrocellulose of the powder grains, but which is a non-solvent therefor. More particularly, we may use as a deterrent for smokeless powder grains a synthetic lacquer resin which is characterized by the general properties just shown. As a consequence of being compatible with the nitrocellulose, without being a solvent therefor, the deterrents which we may use adhere strongly to the surface of the powder grain without rendering the grains sticky or penetrating deeper into the grain or storage, and thus altering the characteristics of the powder on storage. We have been unable to determine definitely whether or not the deterrents which we may use penetrate into the surface of the powder grains at all. We believe that they penetrate mechanically to a slight extent. If, however, such penetration takes place, it either does not continue on storage, or if it does, it progresses so slowly as to have practically no effect on the ballistics of the powder.

We may apply our improved deterrents to the smokeless powder grain in solution in a solvent for the deterrent which is not a solvent for the

smokeless powder, in an aqueous emulsion or otherwise as may be desirable and convenient.

As examples of deterrents which we may utilize to coat the grains of smokeless powder in accordance with this invention, we may use the alkyl esters of the fatty acids, as, alkyl esters of ricinoleic acid, alkyl esters of polymerized ricinoleic acid, etc.; rosin esters, as, monohydric alcohol esters of abietic acid, polyhydric alcohol esters of pimelic acid, polyhydric alcohol esters of pimelic acid, etc.; synthetic lacquer resins, as, the modified or unmodified polyhydric alcohol-polybasic acid resins (Alkyds, Rezyls, etc.), phenol-formaldehyde type resins, etc.; rosins, as, wood rosin, gum rosin, etc.; hydrogenated rosins, as, hydrogenated wood rosin, hydrogenated gum rosin, etc.; and the like. Thus, we may use butyl ricinoleate, butyl acetyl ricinoleate, butyl ester of polymerized ricinoleic acid, methyl abietate, ethyl abietate, hydrogenated methyl abietate, hydrogenated ethyl abietate, ester gum, hydrogenated ester gum, modified or unmodified glycerolphthalate resin, oil-modified phenol-formaldehyde type resins, glycerol sebacate, glycol sebacate, glycerol-glycol sebacate, modified or unmodified ethylene glycol terpinene maleic anhydride resins, modified or unmodified diethylene glycol terpinene maleic anhydride resins, modified or unmodified triethylene glycol terpinene maleic anhydride resins, modified or unmodified glycerol terpinene maleic anhydride resins, etc.

As an example of the carrying out of the process involving our invention and producing our improved product, 908 parts by weight of a double base smokeless shotgun powder, containing 20% nitroglycerin, were placed in a cold sweetie barrel, and to this was added slowly 27 parts by weight of butylacetyl ricinoleate dissolved in about 160 parts by weight of cold ethyl alcohol, the mixture tumbled in a closed sweetie barrel at room temperature for about ½ hour, after which the powder was removed to a dry house and dried for one day at 55° C., then graphited, screened and exposed to an atmosphere of 50-60% relative humidity to bring the moisture content to equilibrium.

In a further example of carrying out our process and the making of the product according to our invention, 908 parts by weight of a double base shotgun powder containing 20% nitroglycerin was treated in a manner identical with the above example with 27 parts by weight of hydrogenated methyl abietate.

In a further example of our process and product, 908 parts by weight of a 10% nitroglycerin shotgun powder were coated with 5% of hydrogenated methyl abietate in the manner described above.

In the coating operation we may use only sufficient volatile solvent containing the deterrent to wet the powder grains, or we may use a large excess of solvent which can be removed by evaporation or otherwise. We may treat smokeless powder with the deterrent at temperatures ranging between 0° and 100° C., and we may treat the smokeless powder grains with our improved deterrent, alone or in solution, for varying lengths of time, depending upon the composition of the powder treated, and the size of the powder grains. As solvent for the deterrent, we may use any convenient solvent which has no appreciable solvent action upon the nitrocellulose of the powder grains, for example, ethyl alcohol, propyl alcohol, isopropyl alcohol, petroleum hydrocarbons,

as benzene, petroleum ether, and ether such as, diethyl ether; and we may coat either single base powder grains or double base powder grains.

As an example of the heat stability of smokeless powder prepared in accordance with this invention as compared with smokeless powder prepared in accordance with the old art, using as deterrent a solvent of nitrocellulose, the following table is given:

Powder charge grains	Deterrent used	Velocity foot/sec.	Pressure, lb./in. ²	Condition of charge
38.5	Di-ethylphthalate...	1015	11,000	Fresh.
38.5	do	1022	14,600	Stored 10 days at 55° C.
36.5	Butyl ester of polymerized ricinoleic acid.	979	10,300	Fresh.
36.5	do	978	11,100	Stored 10 days at 55° C.
36.5	Butylacetyl ricinoleate.	989	11,100	Fresh.
36.5	do	989	11,400	Stored 10 days at 55° C.
37.0	Butyl ricinoleate.	989	12,100	Fresh.
37.0	do	980	11,700	Stored 10 days at 55° C.
36.5	Hydrogenated methyl abietate.	1011	11,100	Fresh.
36.5	do	1016	11,200	Stored 20 days at 50° C.

It will be noted that with the old art powders (coated with diethyl phthalate), there was an increase in pressure after hot storage of 3500 lbs. per sq. in., whereas with the smokeless powder prepared in accordance with the present invention, there is either a decrease in pressures for the same charge and the same velocity, or at most an increase in pressure of only 800 lbs. per sq. in.

It will be understood that where we have referred to a compound as being a non-solvent for nitrocellulose, we mean a compound which, by reason of its relatively low content of chemical groups which are known to promote solvent action, its high molecular weight, its high viscosity at temperatures to which the compound may be subjected, or its structural arrangement, does not dissolve nitrocellulose of the degree of nitration used in a particular case, at a measurable rate of solution.

It will 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 in the appended claims.

This application is a division of our application Serial No. 117,890, filed December 28, 1936, relating to progressive-burning smokeless powder and method of manufacture.

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

1. In the method for the preparation of progressive-burning smokeless powder, the step which comprises surface coating smokeless powder grains with a synthetic lacquer resin.

2. In the method for the preparation of progressive-burning smokeless powder, the step which comprises surface coating smokeless powder grains with a polybasic acid polyhydric alcohol resin.

3. In the method for the preparation of progressive-burning smokeless powder, the step which comprises surface coating smokeless powder grains with a glycerol phthalate resin.

4. In the method for the preparation of progressive-burning smokeless powder, the step which comprises surface coating smokeless powder grains with a hydrogenated rosin ester.

5. In the method for the preparation of progressive-burning smokeless powder, the step which comprises surface coating smokeless powder grains with a hydrogenated monohydric alcohol ester of rosin.
6. In the method for the preparation of progressive-burning smokeless powder, the step which comprises surface coating smokeless powder grains with hydrogenated methyl abietate.
7. In the method for the preparation of progressive-burning smokeless powder, the step which comprises surface coating smokeless powder grains with a hydrogenated rosin.
8. As a new product smokeless powder grains surface coated with a synthetic lacquer resin.
9. As a new product smokeless powder grains surface coated with a polybasic acid polyhydric alcohol resin.
10. As a new product smokeless powder grains surface coated with a glycerol phthalate resin.
11. As a new product smokeless powder grains surface coated with a hydrogenated rosin ester.
12. As a new product smokeless powder grains surface coated with a hydrogenated monohydric alcohol ester of rosin.
13. As a new product smokeless powder grains surface coated with hydrogenated methyl abietate.
14. As a new product smokeless powder grains surface coated with a hydrogenated rosin.
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