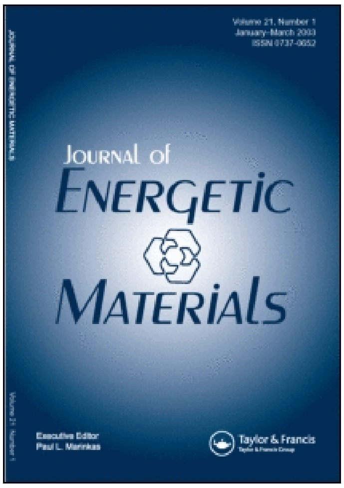

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PROBLEMS CONNECTED WITH POSSIBILITY OF THE SOLVENTLESS PRODUCTION OF MODIFIED BALL-POWDER COMPOSITIONS

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ABSTRACT

The present paper deals with verifying the potential preparation of ball powders using a solventless procedure in laboratory. The solution is based on such a principle that the powder mass consisting of nitrocellulose, a non-volatile meltable explosive gelatinizing agent (such as TNT), and a non-volatile meltable nonexplosive gelatinizing agent (such as triacetine), which is an energy-deficient composition, is enriched in its energy content by adding an energy donor (such as PETN), other additives, such as wax E (improving the rheological properties)

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and Centralite, being introduced in minor amounts.

The powder preparation is run in a reactor under stirring with a temperature control in the presence of protective colloid. The process run under the given conditions yields a plastic powder mass forming discrete ball grains which are hardened by cooling the charge as a whole. Various factors of importance are discussed with respect to the granulometric composition and shape quality of the product obtained.

The ball powder gained was subjected to informative ballistic tests with cartridges of the 7.62 mm calibre using the central initiation and those of .22 calibre rimfire cartridges. With some types of the .22 calibre cartridges really good ballistic parameters were achieved though the performance of the new powder was inferior to that of conventional powders.

INTRODUCTION

At present the production technology of ball powders is used widely for producing an assortment of small-grain smokeless powders. A number of modifications of the process concerned exist, which differ in details of some production stages, e.g., in the art of introducing the liquid nitrate ester into the powder mass[1,2,3,4], the art of controlling powder-grain porosity[5], the art of treating the grain surface[6], and the like. The common principle remains to comprise, in any case, processing a nitrocellulose solution (containing, eventually, other components or additives) to convert the originally continuous phase to a form of discrete spherical grains. This process is run in a two-phase system comprising a nitrocellulose solution and water with a protective colloid and, eventually, also an emulgator added, the volatile solvent being removed by a distillation run in a subsequent stage. This technology is only in a special case of preparing the large powder grain (diam. above 0,8 mm) modified by using a cutting process[1], which secures a mechanical preshaping of the grain. All the variances of the process for preparing ball powder use ehtylacetate (occasionally with other solvent - components added) as the volatile solvent, which is the most appropriate solvent usable for the respective purpose. Evaporating the solvent is then one of the critical stages of the production cycle due to its duration, and, in addition to it, ethylacetate as a volatile inflammable solvent presents a steady fire risk in the production area.

The present paper examines the possibility to exclude the use of volatile inflammable solvent from the production process by replacing it with a nonvolatile gelatinating system which remains in the product as a permanent component thereof.

Principle of Solving the Problem

The role of volatile solvent in the process is substituted by using a two-component gelatinizing system consisting of an explosive meltable component and a nonexplosive liquid component. At an appropriate processing temperature the system turns liquid and able of gelatinizing or solving nitrocellulose. This gives rise to the formation of a homogeneous powder mass, which is of such a consistency, that it can be processed to give the ball powder grain provided an appropriate protective colloid is present in the aqueous medium under stirring. This grain can be hardened by merely cooling the system, which results in the solidification of meltable gelatinizing agent, and the resulting product turns solid as a whole.

Using the energetically deficit gelatinizing system requires to supply the powder mass with a source of energy, which is done by adding a crystalline high explosive being a chemical individuum, playing no substantial active part in the gelatination process. This principle applied results in the formation of a multicomponent composition representable by a scheme: nitrocellulose - non volatile binary gelatinizing agent system - crystalline high explosive.

Trinitrotoluene was used in the role of the meltable explosive gelatinizing agent, and PETN acted as the energy donor. Triacetine (TAC) and dibutyl phthalate (DBP) were considered, alternatively, as a nonexplosive liquid gelatinizing agents.

There have been tested also several sorts of nitrocellulose take from the usual assortment produced, presuming, however, that under the conditions given a sufficient degree of gelatinability will be shown by a sort characterized by a comparatively low nitrogen content and, appropriately low viscosity.

Some of the products prepared were subjected to preliminary ballistic tests.

Experimental Section

Laboratory-scale preparation of the ball powder was done under the following conditions:

1) By means of a dispergator fitted with a knife-type stator so as rotor (of the Turrax system) a suspension of solid powder-mass components was prepared, which

gave a PETN grain size 0.05 mm, provided that PETN was used in the respective experiment.

2) The suspension thus obtained was subjected to heating under stirring for 50 min., increasing the temperature to reach 95 °C at the end, the mixture being held at 95 °C for further 25 min. During this period of constant temperature the final grain shaping took place, and the mixture was cooled down at a water bath afterwards.

3) The laboratory-scale process used a special stirring speed regime, the stirring speed being changed in a range of 350-1200 rpm.

4) At a definite process stage an aqueous protective - colloid (arabic gum) solution and, simultaneously, a liquid gelatinizing agent were dosed to the mixture under stirring.

5) The powder grain having been washed and filtered off, matured on being left laying overnight was processed by graphitizing, drying, and screening operations in subsequent stages, as usual in processing

the fine-grain powders.

The process factors mentioned in points (3) and (4) present the ways of controlling the shape quality and size distribution of the resulting ball grain-powder. A principal condition of the formation of discrete powder balls consists in a suitable gelatinization degree of powder mass resulting in an appropriate viscosity at a processing temperature. With respect to this a decisive criterion for running the process comprised ability of the powder mass to create, under the given conditions, the ball grains of appropriate size and shape qualities at a satisfactory yield.

With respect to the circumstances outlined in the leading part of experiments a basic powder mass was prepared which showed the composition as follows: Nitrocellulose 28 % - TNT 34 % - TAC 36 % - Centralite I 1 % - wax E 1 %.

Wax E (ethylene glycol ester derived of higher fatty acids, m.p. 70 - 74 °C) showed in connection with

TNT a positive effect on the resulting-grain sphericity, which agrees well to the use of wax E as an additive improving the rheological properties of castable explosives based on TNT as necessary for their filling to cartridges.

During these experiments it has been verified that TAC is as a liquid gelatinizing agent for the purpose of interest superior to DBP. From the six nitrocellulose types the most appropriate properties were exhibited by industrial nitrocellulose of the type A-500 showing a low solubility in esters and a high solubility in ethyl alcohol, a nitrogen content of 10.73 % and a viscosity of 0.75 mPa s (as measured in a 2 % acetone solution by a Hoeppler viscosimeter). Using DBP so as five other nitrocellulose types with a nitrogen content in a range of 11.55 - 13.22 % and a viscosity of 5.80 - 19.50 mPas gave the product in a low yield (below 50 %) or showing an inappropriate grain shape.

The main series of experiments was directed to preparing a powder using the basic mass dotted with varying PETN amounts. This examination was performed on the basis of two experimental arrangments.

On the one side by means of the leading factors used in the laboratory-scale process two series of experiments (E and F) were done, which were directed to obtaining the samples of constant PETN content, the process being in case of the series (E the mass containing 50 % PETN) orientated to give a product characterized by a maximum content of 0.1 - 0.2 mm fraction, whereas in case of the series F (the mass containing 45 % PETN) a maximum yield of the 0.6 - 0.7 mm fraction was desired. These ranges represent essentially the achievable dimension limits of the product-showing appropriate shape.

The other type of experiments arranged was represented by the series G directed to obtaining a product characterized by the maximum content of 0.1 - 0.4 mm fraction, individual samples of this series being dotted with PETN added in an amount of 0 - 70 %.

The grain size or the fraction limits are meant as the fractions above the sieves as used in doing the sieve analysis.

Laboratory Results and their Discussion

In Table 1 there is given the chemical composition as derived from the basic-mass composition for the samples prepared in individual series.

In verifying the individual variances of control factors of the process run in laboratory the Series E samples gave a yield of 37 - 70 % for the fraction 0.1 - 0.2, where as the series F samples gave a 0.6 - 0.7 fraction in a yield of 35 - 90 %. The yield of 0.1 - 0.4 mm fraction obtained in the series G samples processed under unchanged laboratory - preparation conditions but with a varying PETN content was in a range of 70 - 91 %, the maximum yields of the fraction given being achieved for the samples G2 and G3 containing 30 resp. 40 % PETN. In this way the results obtained suggest that crystalline PETN (of a maximum crystal size of 0.05 mm) present in the powder mass supports the process tending to give small spherical grains.

The effect of PETN content on the shape quality powder grain is evidenced by the photomicrographs (Figs. 1 through 4) representing selected samples of the series G. The photomicrographs were taken to obtain evidence on possibility of checking the spherical shape of grain contours by means of microphotographs taken in transmitted light, as no appropriate equipment was available for taking the photomicrographs in reflected light. The microphotographs were taken for a semibounded <0.2, 0.4)mm size interval, the appropriate fraction having been separated from the samples.

The images obtained show clearly the effect of increases in the amount of PETN added on shape quality and homogeneity of the spherical grains obtained. Both the parameters suffered a deterioration due to high-dotation effects. In Fig. 4 there are evident protruding PETN crystals which are not covered with the basic powder mass due to dotation of the powder with high (70 %) amounts of PETN added.

In Table 2 there are given the density and heat of explosion values measured for the selected fractions of the grain powder samples prepared by the laboratory-scale process in individual experimental series. In case of E2 and F1 samples the residual fractions of low product yields are concerned.

The powder-grain density was determined pycnometrically in water and the heat of explosion was measured calorimetrically operating at a constant volume.

Ballistic tests

Selected samples of the ball powder containing 40 - 60 % PETN prepared in the framework of three experiment series were subjected to ballistic test runs using standard cartridges of 7.62 and .22 calibres.

Table 2 brings standard data for the cartridges used in the tests and the basic prescribed data thereof, namely $\bar{v}_x$ - the mean projectile velocity obtained by performing a set of shots, measured at a prescribed x meters base.

$\bar{p}_{max}$ - the mean value of measured maximum pressure calculated from the set of shots, the number of shots in a set being (of Table 3). The usual weighed amount of a standard powder for the cartridge given is designated as w_o. The tests were run using a powder sample kept at a constant temperature of +20 ± 2 °C.

For the 7.62 mm cartridges the samples used showed to be in appropriate.

With a Model 59 cartridge (tested with a sample E2) filled with 50 % weight amount of powder a set of 3 shots gained approximately 70 % projectile velocity v_{25} and 140 % pressure p_{max} compared to prescribed values.

The cartridges Model 43 (tested with the samples F1 and F2) there filled with 60 wt. % amounts showed a velocity v_{25} ranging 78 - 85 % and a pressure p_{max} ranging 105 - 180 % of the respective permitted values, two shots being fired for both the samples.

The shooting results obtained with the calibre .22 cartridges are given in Table 4.

The results obtained show clearly that in the .22 SS cartridges the powder F1 sample (containing 45 % PETN) behaves really effectively, reaching about 120 % of the prescribed projectile velocity at a sufficient pressure reserve. The powder of the same composition but of a comparatively large grain size (sample F2) showed, however, quite an insufficient performance accompanied with ignition difficulties when used in the same-type cartridge.

With the .22 LR cartridge the sample F1 gave sufficient projectile velocities when the weighed-in amount was increased a little, the permitted pressure being exceeded, however, by 63 %. The sample F2 exhibited here, similarly as was the case with the preceding cartridge type, an insufficient performance even at the maximum cartridge filling.

The samples of small grain diameter and of different PETN content (G3 through G5) exhibited when used in .22 LR cartridges a sufficient performance but at pressures reaching about 150 - 180 % of the permitted value. These results show also that increases, in PETN content in a range of 40 - 60 % manifest themselves in the rising pressure encountered in the weapon, the projectile pressure remaining almost unchanged.

The sample E1 (containing 50 % PETN) showed satisfactory in cartridges characterized by increased demands on performance such as .22 LR-US but only when the weighed-in amount was increased to reach nearly 150 % of the usual powder amount.

Conclusions

Laboratory-scale experiments were used to verify the potential viability of preparing the ball powder using a solventless process based on a nontraditional principle with respect to both the absence of volatile solvent in the process so as the powder-mass composition giving possibility of being processed in a two-phase system containing water under stirring, with a protective colloid added.

The basic powder mass showed good fillability with crystalline PETN (the powder could contain up to 70 % PETN), but a negative effect of high PETN content on the shape quality of spherical grain was observed at the same time.

Possibility of reaching a high yield (about 70 - 90 %) of the dimensional grain fractions of interest has been demonstrated as indicated by changes in the process conditions, especially the regimes of stirring and feeding the protective-colloid (arabic gum) solution and also the liquid gelatinizing agent (TAC) into the stirred system.

The process does not require the evaporation of volatile solvent, so that the time necessary for producing the ball grains of the type given is significantly shorter compared to the time necessary for producing the hitherto usual ball powder.

The informative ballistic tests applied to the final grain product have shown that the powder does not behave typically compared to usual types of fine-grain powder. The powder subjected to tests showed reduced ignitability in the .22 calibre rimfire cartridges, in the 7.62 mm cartridges it, however, showed a tendency to rapid burning with excessively sharp pressure rise and a reduced performance.

Character of the powder corresponds evidently better to being used in the form of fine-grain product (in a range of 0.1 - 0.4 mm) and this in comparatively small-calibre and low-performance cartridges. In cartridges of the .22 Short Sport and .22 Long Rifle-US types quite satisfactory parameters were obtained, though the weighed-in amounts indicate that the powder tested was when used in the latter cartridge less efficient than the usual powder charge.

TABLE 1. Chemical compositions of the samples prepared

in individual	PETN /%/	nc A-500 /%/	TNT /%/	TAC /%/	C-I /%/	wax E /%/
Series E:	50,0	13,7	16,7	17,6	1,0	1,0
Series F:	45,0	15,1	18,4	19,5	1,0	1,0
Series G:						
Samples:						
G1	0,0	28,0	34,0	36,0	1,0	1,0
G2	30,0	19,4	23,6	25,0	1,0	1,0
G3	40,0	16,6	20,1	21,3	1,0	1,0
G4	50,0	13,7	16,7	17,6	1,0	1,0
G5	60,0	10,9	13,2	13,9	1,0	1,0
G6	70,0	8,0	9,7	10,3	1,0	1,0

TABLE 2. Experimental density and heat of explosion values obtained for the selected fractions in individual process series.

Sample	PETN /%/	Fraction /mm/	Density /kg.m^{-3}/	Heat of Explosion /kJ.kg^{-1}/	Notes
E1	50	0,1-0,2	1410	3964	B
E2	50	0,3-0,7	1450	4198	B
F1	45	0,3-0,5	1380	3864	B
F2	45	0,6-0,7	1400	-	B
G1	0	0,1-0,4	1325	1344	-
G2	30	0,1-0,4	1440	2976	B
G3	40	0,1-0,4	1460	3587	B
G4	50	0,1-0,4	1475	3889	B
G5	60	0,1-0,4	1480	4521	-
G6	70	0,1-0,4	1527	5019	-

Notes: B - the sample was used for the ballistic tests - see the subsequent Section.

TABLE 3. Properties of the cartridges used for the ballistic tests of the ball powder prepared.

Cartridge	Dimensions /mm, inch/	Notes	w_o /g/	$\overline{v}_{12,5}$ /m.s^{-1}/	$\overline{v}_{25}$ /m.s^{-1}/	$\overline{p}_{max}$ /MPa/
Model 59	7,62x54	1)	3,0	-	828±5	279,5
Model 43	7,62x39	1)	1,55	-	717±5	265,0
Model .22 LR	.22	2)	0,090–0,115	350±7,5	-	105,0
.22 LR–US	.22	3)	0,090	368±7,5	-	117,7
.22 SS	.22	4)	0,060	245±7,5	-	78,5

Notes: 1) Czech army cartridge
2) .22 Long Rifle /rimfire cartridge/
3) .22 Long Rifle according to the american conditions, i.e. for an increased power
4) .22 Short Sport /rimfire cartridge/

TABLE 4. The shooting results obtained with the calibre .22 rimfire cartridges filled with the samples given as stated.

Cartridge	Sample	Moisture /%/	n	w /g/	$\bar{v}_{12,5}$ /m.s^{-1}/	$\bar{p}_{max}$ /MPa/	Note
.22 SS	F1	0,16	3	0,090	293,0	75,7	-
	F2	0,18	1	0,060	120,0	-	-
	F2	0,18	1	0,070	-	-	was not ignited
.22 LR	F1	0,16	5	0,120	352,4	171,0	-
	F2	0,18	1	0,120	294,1	92,4	max
	G3	0,08	5	0,120	344,0	154,4	-
	G4	0,05	5	0,098	347,8	166,5	-
	G5	0,09	5	0,080	345,2	185,0	-
.22 LR-US	E1	0,27	5	0,138	368,3	108,5	-

Footnotes: w - real amount of the tested powder used
max - the maximum powder amount to charge the cartridge was used
Moisture content was determined by the K.Fischer method

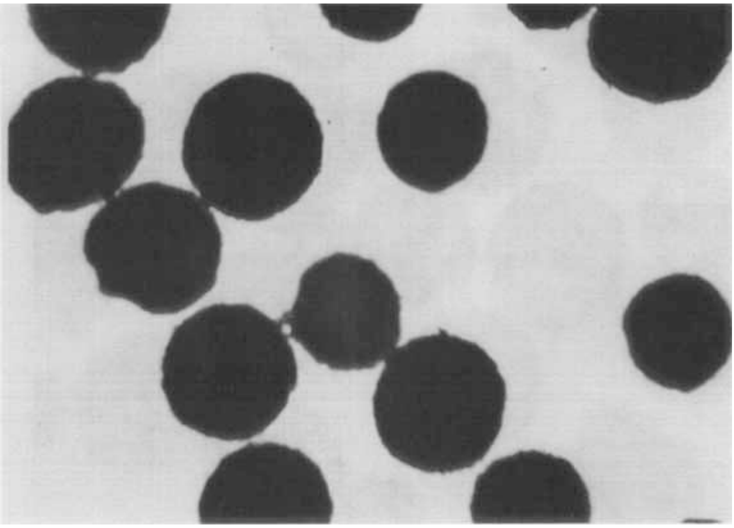

FIGURE 1.

Sample G1 /without PETN added/, fraction ⟨0.2;0.4) mm.
Magnified 50x.

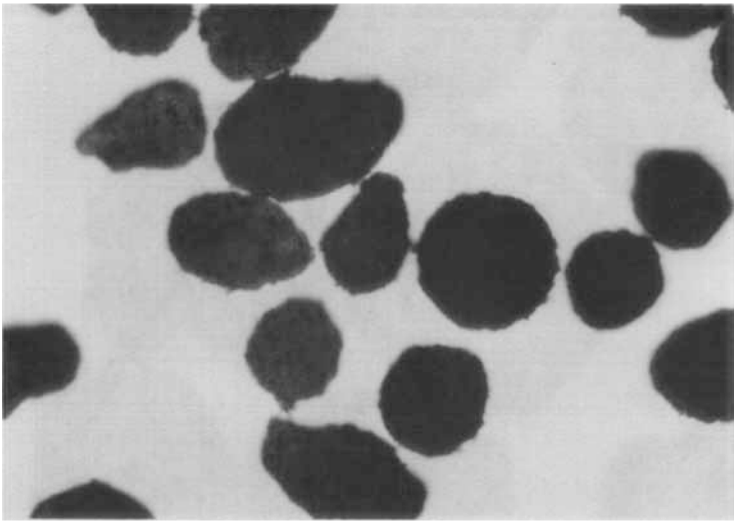

FIGURE 2.

Sample G4 /50 % PETN added/, fraction ⟨0.2;0.4) mm.
Magnified 50x.

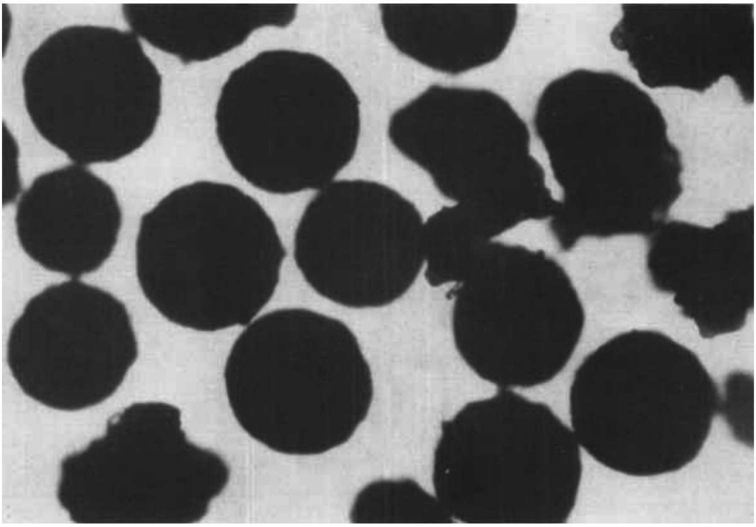

FIGURE 3.

Sample G6 /70 % PETN added/, fraction ⟨0.2;0.4) mm. Magnified 50x.

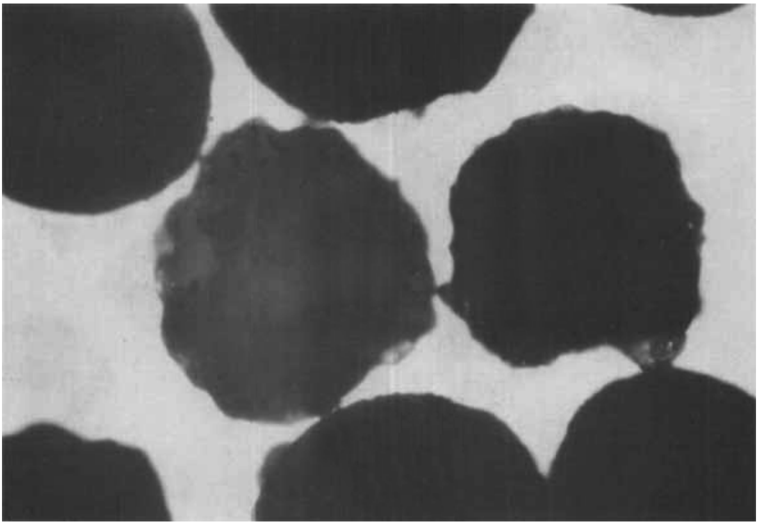

FIGURE 4.

Sample G6 /70 % PETN added/, fraction ⟨0.2;0.4) mm. Magnified 100x.

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