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Investigation of the influence of additives on the detonation parameters of nitromethane. 1. Ammonium nitrate(V)
Badanie wpływu dodatków na parametry detonacyjne nitrometanu. 1. Azotan(V) amonu

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Abstract: *The paper presents results of an investigation into the detonation velocity and critical diameter of nitromethane mixtures thickened with polymethyl methacrylate, where ammonium nitrate(V) (NH_4NO_3, AN) contents of different grain sizes were used. The dimensions of the AN grains were shown to be decisive in their effect on the measured detonation parameters. The experimental results are appended with the results of numerical estimates of the detonation parameters of the materials tested. A physical and chemical interpretation of the experimental results is also provided.*
Streszczenie: *W pracy przedstawiono wyniki badań prędkości detonacji i średnicy krytycznej mieszanin nitrometanu zagęszczonego polimetakrylanem metylu z różnymi zawartościami azotanu(V) amonu (NH_4NO_3, AN) o różnym jego rozdrobnieniu. Wykazano, że wymiary ziaren AN decydują o wartościach mierzonych parametrów detonacyjnych. Wyniki eksperymentalne uzupełniono rezultatami szacowań numerycznych parametrów detonacyjnych testowanych materiałów. Zaprezentowano również interpretację fizykochemiczną wyników badań eksperymentalnych.*

Keywords: *nitromethane, ammonium nitrate(V), detonation velocity, critical diameter*
Słowa kluczowe: *nitrometan, azotan(V) amonu, prędkość detonacji, średnica krytyczna*

1. Introduction

Liquid explosives are chemical compounds or mixtures of chemical compounds, usually in the form of a gel. According to Liu [1], the most important LE groups are: nitrates(V), nitroalkanes, nitroalcohols, azides, and aromatic nitrogen compounds. One of the representatives of liquid nitroalkanes is nitromethane (NM), which has found applications in liquid rocket fuels. The detonation capability of nitromethane was first identified by Bellinger *et al.* [2], who observed its high-energy transformation during impact of an NM sample, placed in an airtight thick walled steel vessel with, by a 12.5 mm calibre projectile. In contrast,

Cass [3] demonstrated that nitromethane explodes upon impact by a 2 kg weight, free-falling from a height of 195 cm. The final, and tragic, proof of the fact that nitromethane is an explosive was provided by two rail disasters in the United States in 1958, at Niagara Junction and near Mount Pulaski. Both involved explosion of rail tankers carrying nitromethane [4]. Thermodynamic parameters of nitromethane were first determined by Holcome and Dorsey [5], while Medard [6] and Urbanski and Pawlec [4] determined its detonation parameters. Mader [6], using the Dautriche method, measured the detonation velocity (D) in a 30/35 mm diameter glass tube and a 30/35 mm diameter aluminium tube, obtaining a value of 6600 m/s. Much lower detonation velocities were obtained by Urbanski and Pawlec – for example, 6360 m/s in 22/27 mm steel pipe. According to the latest data available and reported in [1], the temperature-dependent detonation velocity of NM is 6380 m/s (4°C) and 6320 (25°C). The authors of [4] determined the capacity of NM to do work using a lead block method, which, depending on the mass of the tetryl charge used as a booster (after subtraction of its contribution), was 420 cm^3 (1 g) or 495 cm^3 (5 g).

The detonation parameters of NM have been modified by various additives. Mixtures of NM with AN [7-12], silicon dioxide [13], carbon black plus dialuminium trioxide, talc and tungsten [14], glass microspheres [15-21], aluminium dust [14, 22-25] and aluminium-magnesium dust [26] have been researched.

Francis patented two types of explosives based on nitromethane and containing AN in the form of a gel [7, 8] or slurry [9]. For the gelatinised explosive [7], the continuous phase was formed by concentrated NM. The proposed thickeners were cellulose, ethyl cellulose, cellulose acetate, and polyoxyethylene. The AN/NM mass ratio was in the range of 45:55 to 65:35. The detonation velocities of these explosives ranged from 3600 to 5400 m/s. In another patent by Francis [8], the gelatinised explosive contained a mixture of nitropropane (NP) and NM at a concentration of 40:60 to 55:45. In the final mixture, the ratio of AN/NP-NM ratio was higher than that of the NM, ranging from 75:25 to 85:15. The detonation velocity of AN/NM-PM mixtures was lower than those of AN/NM, within 3600 to 4500 m/s, which was affected by the lack of detonation capability of the nitropropane. It is only the dinitrogen derivatives of propane which have explosive properties. A guar thickened slurry explosive [9] contained water as an additional solvent, urea as a crystallization inhibitor, and AN and was sensitised with microballoons made of plastic. Its detonation velocity in the 20-30% NM content range increased from 4640 to 5770 m/s.

Walker and Ballard [10] proposed the use of gelled mixtures containing 10-30% NM and ammonium, sodium or lithium nitrates(V) for mine blasting applications, but did not specify any detonation parameters. The lack of data on detonation parameters is evident in another patent by Ballard and McMillan [11], according to which a gelled NM system contained 2.5-5.0% of glass microspheres (GMS) and plastic microspheres, with 10-20% of a desensitising component, barium sulphate(VI).

A small amount of data on the detonation parameters of gelled NM-AM systems can be found in a patent by Edwards [12]. An explosive with a composition [expressed in mass ratios] comprising:

– NM: 15;
– aqueous AN solution (50%): 20.0,
– granular AN: 60.1,
– GMS: 2.0, and
– a gelling agent: 2.9,

had a detonation velocity of 4230 m/s and would be initiated using a #6 detonator.

Engelke [13] conducted a very detailed study of the detonation velocity of commercial-grade, 94.13% pure nitromethane thickened with 1% guar gum and a 92.75/6/1.25 NM/silicon dioxide/guar mixture, as a function of charge diameter (d). The experiments were conducted in tubes of different diameters, made from different materials. Based on the relationship $D = f(d)$, Engelke established the detonation velocities in charges of unlimited diameter (D_∞), which were 6262 m/s for NM and 6167 m/s for the 92.75/6/1.25 NM/silicon dioxide-guar gum mixture. Engelke also carried out critical diameter (d_{cr}) measurements. In a Pyrex glass tube, the NM had a d_{cr} of 16.2 mm, while the silicon dioxide containing mixture had a d_{cr} of 9.6 mm.

The results of d_{cr} measurements of mixtures of NM with carbon black, tungsten, dialuminium trioxide talc and aluminium dust over a wide range containing 5% to 70%, are summarised in [14]. The d_{cr} was measured

in glass tubes with a wall thickness of 0.6-1.3 mm. The addition of the above substances initially resulted in a decrease in critical diameters, followed by an increase. Minimum critical diameters were found at tested substance levels of approximately 30%. Kurbangalina [14] obtained the lowest critical diameters (around 2 mm) for mixtures containing dialuminium trioxide with a grain size of 7 μm and the highest critical diameters for carbon black (around 18 mm).

GMS are the most commonly used sensitisers for cartridge emulsion explosives [29, 30]. Therefore, they have also been used in the composition of explosive mixtures containing NM, and their effect on the detonation parameters of such explosives has been reported in a number of papers. In these, detonation parameters were determined for NM-GMS with a varying GMS content and diameter, and experiments were conducted on various diameters of the charges made of different materials. Presles *et al.* [15] studied detonation velocities in PVC pipes of various diameters, of NM-GMS mixtures containing glass microspheres with a bulk density of 0.132 g/cm^3 and a grain size of 5-170 μm. The GMS content was 0-30%. Presles *et al.* determined the detonation velocity and pressure at an unlimited diameter, from the relationship $D = f(1/d)$. At GMS contents of 0% and 30%, the density decreased from 1.14 to 0.39 g/cm^3,while the detonation velocity fell from 4540 to 2322 m/s.

Grois *et al.* [16] measured the detonation velocity and pressure of a NM-GMS mixture with the addition of 47 μm diameter GMS at 0-3% content range. The addition of 3% GMS provided a decrease in detonation velocity from 6360 to 5062 m/s, determined for an unlimited charge diameter, and a decrease in detonation pressure from 12.6 to 9.8 GPa. Lee *et al.* [17] studied NM-GMS systems containing 15% microspheres with diameters varying between 0.065-2.40 mm. The minimum d_{cr} value obtained was 20 mm. The decrease in detonation velocity and pressure with increasing GMS content was reported in the work of Gois *et al.* [18], who studied NM-GMS containing 0-8% GMS.

The decrease in NM-GMS detonation velocity was also confirmed in [19], where 70 μm GMS were used. Increasing the GMS content from 0.5% to 13% resulted in a decrease in detonation velocity from 6300 to 3700 m/s, and a decrease in d_{cr} from 16 mm (at 0.5% GMS) to 3.5 mm (8.0% GMS). Mochalova *et al.* presented a hypothesis that the addition of GMS in the content range of interest did not cause a qualitative change in the structure of the detonation wave's chemical reaction zone, which was confirmed in [20] for extreme GMS content, 16-30%.

The varying values of the detonation parameters presented in the referenced literature are caused by various factors, such as nitromethane purity, the thickening agent content, the type of explosive charge casing materials, and the experimentally used diameters of GMS. Bouton *et al.* [21] confirmed that the detonation parameters of NM-GMS depends not only on the GMS content, but also the GMS size by testing NM-GMS explosives with two selected sphere sizes, 47 and 102 μm. The results of the detonation velocity tests using both steel and PMMA pipes, revealed that the tested parameter was higher for the compositions containing smaller GMS in the all of pipe diameters used in the experiments.

Aluminium dust has become a popular ingredient in all types of explosives, including those based on NM, since it was first used in 1897 by Deissler [22, 23] in explosive mixtures. Kato and Brochet investigated the detonation velocity [24] and detonation temperature [25] of NM with aluminium dust of 99% purity and a grain size in micrometres. They determined the detonation velocity of NM of unlimited diameter to be 6277 m/s. As the content of aluminium dust grew between 5-10% with a grain size of 8-15 μm, the detonation velocity decreased. This is explained by the delay in the combustion of aluminium by several tens of 10^2 ns.

Milne *et al.* [26] carried out a cylindrical test was on an NM-Al explosive containing 20-60% spherical aluminium dust with an average grain size of 10.5 μm. Comparing the cylindrical test results with the modelling results, they found that the combustion time of the aluminium dust grains tested was longer than the detonation propagation time obtained in the experiments.

An NM-Al explosive containing nanometre-grain aluminium was investigated. Sabourin *et al.* [27] studied NM-Al systems containing aluminium dust of 38 nm grain size. As the content of aluminium dust increased, so did combustion velocity, with the minimum d_{cr} being 12 mm. According to the authors of [28, 29], despite

its nanometric fineness, aluminium dust is not involved in the reactions occurring in the NM detonation zone. The detonation parameters and characteristics of the blast waves generated by the detonation of mixtures of NM and aluminium-magnesium alloy (Al_3Mg_4, PAM) with an average grain size of 63 μm, was investigated in [30]. NM was thickened with 4% PMMA and a PAM content of 15, 30, 45 and 60%. The detonation velocity in 37/40 mm PVC pipes and the intensity of the blast waves for 200 g NM-Al charges placed in 46/50 mm PVC pipes, were determined. The cylindrical test used 25/30 mm copper pipes. The addition of 0-60% aluminium-magnesium dust reduced the detonation velocity from 6211 to 5613 m/s. The ballistic capacity in the range of 15-30% PAM is similar to NM without metallic dust. The authors found, based on experimental data and numerical estimates, that both aluminium and magnesium burn during the expansion of the copper tube, and that the presence of magnesium has a positive effect on the combustion velocity of aluminium in the detonation products of NM. Both the maximum positive pressure of the air blast wave and its impulse, increase with PAM content.

As can be seen from the data cited above, a number of substances have been added to NM to modify its detonation parameters. Of these, two types of additives can be singled out: thermodynamic parameter enhancers (aluminium dust and aluminium-magnesium dust) and detonation enhancers, which include GMS and all the substances used in [14]. One of the NM parameter modifiers was AN, which, since the patents of Norrbin and Ohlsson [31, 32], has become a basic ingredient in all types of mining explosives developed ever since, as well as military explosives. The patent literature cited above lacks detailed test results for NM/AN systems, and only single results can be found, from which it is not possible to determine what effect the addition of AN has on the basic detonation parameters of nitromethane, i.e. D and d_{cr}.

2. Experiments

2.1. Preparation of explosive specimens

NM produced by the Spółdzielnia Pracy Chemików in Łodz (Poland), the crystalline AN with a grain size of less than 0.3 mm and the granular AN with an average granule size of less than 1.5 mm (produced by Zakłady Chemiczne in Puławy, Poland), and polymethyl methacrylate (PMMA) produced by Zakłady Chemiczne in Oświęcim (Poland), trade name “Metapleks”, were used in this investigation.

PMMA was ground prior to the preparation of explosive compositions. The mixtures for detonation parameter testing were prepared as follows. A known mass of NM was placed in a beaker, to which PMMA (2%) was added while stirring until dissolved. The appropriate mass of AN was added to the resulting gelatinous solution and the mixture stirred until a homogenised mixture was visually apparent.

2.2. Detonation velocity measurement

Detonation velocity was measured using a contact sensor method. The explosive was placed in a hard PVC tube with an inner diameter of 38 mm, an outer diameter of 40 mm and a length of 230 mm. There were four sensors in the tube, spaced 40 mm apart. The distance between the booster and the first sensor was 120 mm. The booster used was a plane-wave generator made of hexolite (50/50 TNT/hexogen). The results obtained are given in Tables 1 and 2 and Figure 1.

Table 1. Detonation velocity of NM and crystalline AN mixtures

Component content [%]			Density [g/cm^3]	*D* [m/s]
NM	Crystalline AN	PMMA		
98.0	–	2.0	1.14	6300
88.2	10	1.8	1.18	6000
78.4	20	1.6	1.22	5980
68.6	30	1.4	1.25	5900
58.8	40	1.2	1.29	5790
49.0	50	1.0	1.32	5660
39.2	60	0.8	1.35	5420
29.4	70	0.6	1.37	5190
19.6	80	0.4	1.40	5030

Table 2. Detonation velocity of NM and granular AN mixtures

Component content [%]			Density [g/cm^3]	*D* [m/s]
NM	Granular AN	PMMA		
98.0	0	2.0	1.14	6300
88.2	10	1.8	1.20	6200
78.4	20	1.6	1.22	6060
68.6	30	1.4	1.24	4310
58.8	40	1.2	1.26	2600
49.0	50	1.0	1.30	no detonation

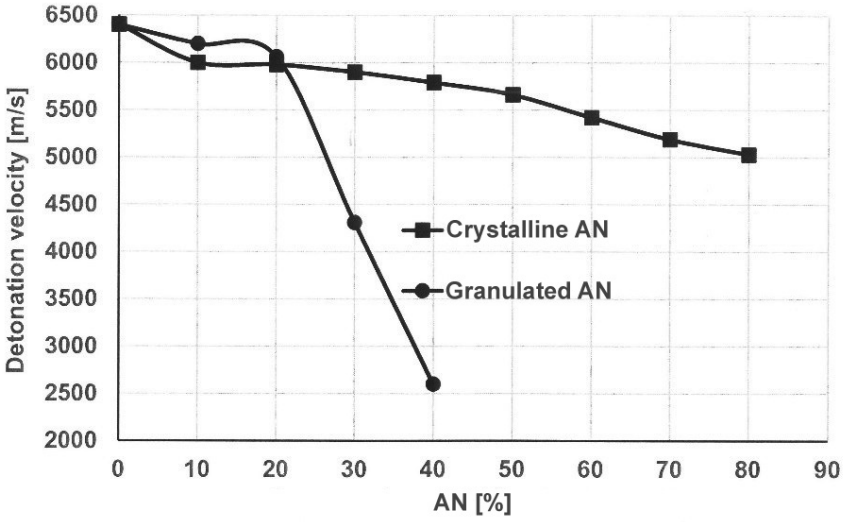

Figure 1. Detonation velocities of NM-AN vs. AN content

2.3. d_{cr} measurement

The d_{cr} was measured initially using the conical charge method and, to refine the experimental results, using the telescopic charge method. In both cases, the charges were placed on a steel plate which featured engraved lines denoting diameter values. The results of the d_{cr} measurements are listed in Tables 3 and 4 and Figure 2.

Table 3. d_{cr} of detonation for NM and crystalline AN mixtures

Component content [%]			Density [g/cm^3]	d_{cr} [m/s]
NM	Crystalline AN	PMMA		
88.2	10	1.8	1.18	25
78.4	20	1.6	1.22	23
68.6	30	1.4	1.25	20
58.8	40	1.2	1.29	18
49.0	50	1.0	1.32	17
39.2	60	0.8	1.35	18
29.4	70	0.6	1.37	20
19.6	80	0.4	1.40	22

Table 4. d_{cr} of detonation for NM and granular AN mixtures

Component content [%]			Density [g/cm^3]	d_{cr} [m/s]
NM	Granular AN	PMMA		
88.2	10	1.8	1.14	28
78.4	20	1.6	1.20	30
68.6	30	1.4	1.22	32
58.8	40	1.2	1.34	34

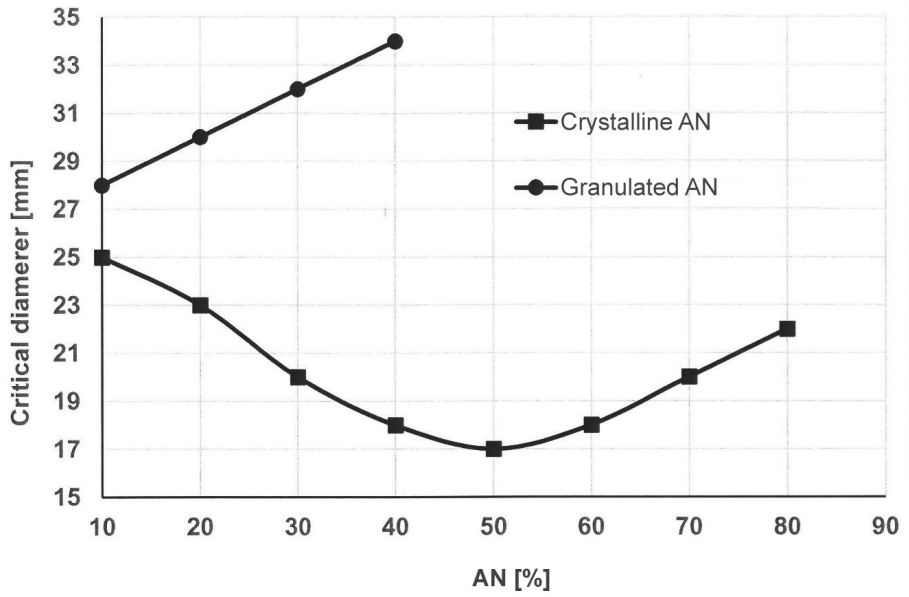

Figure 2. Critical diameters of NM-AN vs. AN content

3. Detonation parameter estimation

To estimate the theoretical values of the detonation parameters, TIGER software was used along with the Becker-Kistiakowsky-Wilson (BKW) equation of state, due to the widespread use of the latter in describing the properties of mixtures under high temperature and pressure conditions. The BKW equation of state is:

$$\frac{pV}{nRT} = 1 + \chi e^{\beta\chi} \tag{1}$$

where: $\chi = \frac{k}{V(T+\theta)^{\alpha}}$, $k = \kappa \sum_{i=1}^{N_g} n_i k_i$, N_g – quantity of gaseous detonation products; n_i – molar fraction of component i; k_i – co-volume; p – pressure; V – specific volume; n – number of moles; R – gas constant; T – temperature.

There are four constants, α, β, κ and θ, in the equation of state, which were adjusted using experimental data. The most commonly used values in this type of thermochemical calculation software is to program BKW_C [33], with: $\alpha = 0.50$, $\beta = 0.403$, $\kappa = 10.85$ and $\theta = 5441$. Using these, the pressure (P_{CJ}), velocity and temperature (T) of detonation were calculated, assuming full involvement of the mixture components and full thermodynamic equilibrium. Table 5 lists the results of calculating the fundamental detonation parameters for 100% and 50% active AN, while Figure 3 compares the detonation velocities estimated at different AN levels with the experimental data obtained for crystalline AN.

Table 5. Results of the parameter estimates for NM-AN systems

AN type	Component content [%]			Density [g/cm^3]	100% active AN			50% active AN		
	NM	AN	PMMA		D [m/s]	P_{CJ} [GPa]	T [K]	D [m/s]	P_{CJ} [GPa]	T [K]
Crystalline	98.0	0	2.0	1.14	6094	11.86	3424	6094	11.86	3424
	88.2	10	1.8	1.18	6241	12.42	3372	6009	11.59	3238
	78.4	20	1.6	1.22	6405	13.06	3321	5931	11.32	3054
	68.6	30	1.4	1.25	6555	13.64	3279	5830	10.93	2875
	58.8	40	1.2	1.29	6745	14.54	3238	5762	10.71	2697
	49.0	50	1.0	1.32	6829	14.37	3193	5666	10.36	2524
	39.2	60	0.8	1.35	6820	14.67	3238	5571	10.01	2353
	29.4	70	0.6	1.37	6708	14.31	3106	5411	8.92	2172
	19.6	80	0.4	1.40	6488	13.27	2653	5213	8.28	2054
Granular	98.0	0	2.0	1.14	6094	11.86	3424	6094	11.86	3424
	88.2	10	1.8	1.20	6294	12.69	3362	6060	11.85	3229
	78.4	20	1.6	1.22	6405	13.06	3321	5931	11.32	3054
	68.6	30	1.4	1.24	6523	13.47	3282	5802	10.80	2878
	58.8	40	1.2	1.26	6645	13.94	3246	5674	10.28	2704

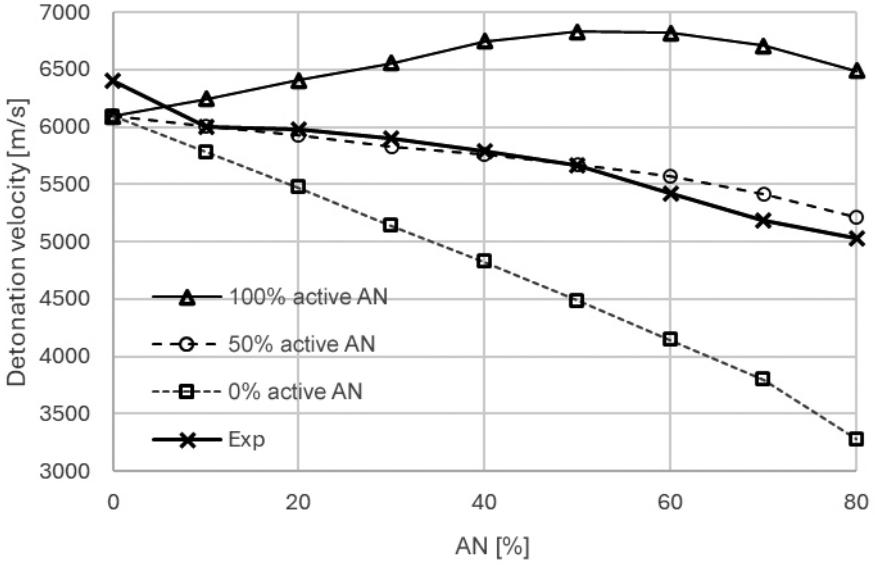

Figure 3. Dependence of MN-AN detonation velocity from the AN content and assumed AN conversion

4. Analysis of test results

For both added crystalline and granular AN, the detonation velocity decreased with increases in the content in the explosive of the oxidant mixture. However, the relationship $D = f$ (%AN) was quantitatively different for the AN types tested. Under the conditions of this research i.e. using a 38/40 mm hard PVC tube, those mixtures with crystalline AN detonated with at least 80% AN (Table 1, Figure 1) and at least 40% granular AN (Table 2, Figure 2). The nature of the decrease in D with increasing AN content also varied. For granular AN in the range up to 20%, the decrease was low, followed by a higher rate of decrease (Figure 2). In contrast, for crystalline AN, it was almost monotonic (Figure 1). Numerical estimates of the detonation parameters of NM-AN systems revealed that the parameter values were determined by the assumed degree of AN conversion (Table 5, Figure 3). Very similar D values from numerical estimates, assuming 50% AN activity, and experimental data for the addition of crystalline AN, were obtained.

For the d_{cr} tests, quite different patterns were obtained for the relationship $d_{cr} = f$(%AN) depending on the AN grain size. The addition of crystalline AN at a 10-50% content range, reduced the d_{cr} to a minimum value of 17 mm, which then would grow as the AN content increased. The NM-AN mixtures had the ability to detonate even at 80% crystalline AN content. On the other hand, for granular AN, in its examined levels of 10-40%, only a monotonic increase of detonation capacity was found with increasing oxidant levels; once the oxidant increased to 50%, the detonation process would not ensue. The effect of solid additives to liquid explosives on changing the mode of detonation propagation from homogeneous to heterogeneous is discussed by Campbell *et al.* [34]. According to Włodarczyk [35], there are six causes which can determine the formation of activation hot spots which are decisive in the initiation of the detonation process. Analysing the structure of NM/AN mixtures, it can be concluded that, in their case, the determining factor for the formation of activation hot spots could be the effect of blast waves near the additives featuring a high wave impedance. Mullay [36] proposed the sensitisation of emulsion explosives with grains of high hardness: ferrosilicon, ferrophosphorus, ferromanganese, and dialuminium trioxide measuring less than 0.3 mm of grain. Kurbangalina [14], on the other hand, demonstrated that to sensitise NM, the sensitiser need not be of high hardness – she used talc and carbon black – albeit with a small grain size. The small grain size ensures a high density of activation hot

spots. Therefore, NM-ANs are sensitised by crystalline AN, while granular AN is only an inert additive. Also confirmed by numerical estimates, the activity of crystalline AN contributed to a lowering of the d_{cr}.

5. Conclusions

The results from the experiments and the numerical estimations allow some general conclusions to be proposed.

- The detonation parameters of NM and AN mixtures depend on the oxidant content and the fineness of its grain (size).
- The addition of crystalline AN results in a less severe decrease in D compared to granular AN and, over a wide content range, a decrease in d_{cr}. The nature of the trend of the relationships $D = f(\%AN_{kr})$ and $d_{cr} = f(\%AN_{kr})$ are indicative of a partial reaction of AN in the reaction zone of the detonation wave, which was confirmed by numerical estimation results.
- The presence of granular AN in the NM-AN mixture highly reduces, over the entire range of the content tested (10-40%), the D and its explosive capability.

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