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Investigation of the influence of additives on the detonation parameters of nitromethane 2. Aluminum powder
Badanie wpływu dodatków na parametry detonacyjne nitrometanu 2. Pył aluminiowy

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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 aluminum powder content of different grain sizes were used. The dimensions of the aluminum 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 pyłów aluminiowych o różnym rozdrobnieniu. Wykazano, że wymiary ziaren pyłu aluminiowego 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, aluminum powder, detonation velocity, critical diameter*
Słowa kluczowe: *nitrometan, pył aluminiowy, prędkość detonacji, średnica krytyczna*

1. Introduction

Alongside nitroglycerine and nitroglycol, nitromethane is one of the few liquid explosives in use. It is used as a component of propellants [1] which are considered 'green' [2]. The physicochemical properties and detonation and thermochemical parameters of nitromethane have been presented, among others, in monographs by Urbański [3] and Liu [4], as well as others [5-8]. The detonation parameters of nitromethane have been modified by various additives. Research has been conducted on mixtures of nitromethane with liquid compounds [9-11], ammonium nitrate [12-18], silicon dioxide [19], glass microspheres [20-26], aluminium-magnesium powder [27], and aluminium powder [28-37].

Aluminium powders began to be used as modifiers of these parameters at the end of the 19th century. Urbański [38] quotes the first patents and publications: by Dressler (1897), Goldschmidt (1898), Escales (1899) and Roth (1900), as well as works published by Kasta between 1902 and 1908 in the journal *Jahresber Mil. Vers. Amt.* concerning aluminised explosives. They were found in the composition of explosives used in military technology and in the mining industry. In military explosives, it is used as an additive to increase energy parameters, and in mining explosives, it increases detonation parameters as well as detonation capability. These properties have led to research into its impact on the parameters of both high explosive materials and propellants, including those containing nitromethane.

Kato and Brochet studied the detonation velocity [28] and detonation temperature [29] of nitromethane thickened with 3% polymethyl methacrylate and its mixtures with 99% pure aluminium powder with a grain size of 8-15 µm. The detonation velocity (D) was measured in cylinders of the following diameters (d): 13, 18, 25 and 31 mm. Based on the $D = f(1/d)$ dependency, they determined the graphical detonation velocity in an unlimited charge diameter and for aluminium powder content of 0, 5, 10 and 15%, obtaining detonation velocities of 6227, 6220, 6163 and 6104 m/s, respectively. Based on photographs taken with a high-speed camera, Kato and Brochet claim that the delay in aluminium combustion is several 10^2 of nanoseconds. In the case of pyrometric measurements of mixture temperatures, they obtained a maximum temperature at 30% metal content and, on this basis, suggested that aluminium reacts with the water produced by the decomposition of nitromethane [29].

The phenomenon of combustion, including aluminium nanopowder types, in nitromethane decomposition products was investigated in [30]. Baudin *et al.* [30] tested mixtures of concentrated nitromethane with four types of aluminium powder: micrometric (grain size 5 µm) and three nanopowders with grains smaller than 100 nm. The detonation velocity and the propulsion velocity of a copper tube wall were determined as a function of time for two types of composite mixtures: NM gel/Al 20/80 and 40/60 and, for comparison, a pressed mixture of TNT/Al 80/20. The addition of micrometric powder caused a smaller decrease in detonation velocity than nanopowders, for which significant differences were found, this resulting, among other things, from different charge densities. Also, in the case of TNT/Al composite mixture, a lower detonation velocity was found than for pure TNT. The detonation velocity results obtained for both NM/Al and TNT/Al Baudinet et al. put down to the narrow chemical reaction zone of the detonation wave, characteristic of high-energy individual explosives, which include TNT and nitromethane. The speed-time graphs of the driven copper tube wall for mixtures containing 20% aluminium powder were identical.

The results of cylinder tests were also reported by Milne *et al.* [31]. The tests were performed on NM/Al mixtures containing 20, 30, 40, 50 and 60% aluminium with an average grain size of 10.5 µm. The increase in aluminium powder content caused the speed of the driven copper tube wall to decrease. Comparing these experimental results with the modelling results, Milne *et al.* [31] concluded that the ignition and burning times of aluminium powder grains are longer than the detonation time, presumably referring to the chemical reaction time of the detonation wave.

The influence of grain size on the detonation parameters of NM/Al was also determined. Kato *et al.* [33, 34] presented the results of studies on NM/Al mixtures and, for comparison, NM/Cu mixtures containing metallic additives of different grain sizes. They showed that the detonation velocity limits of NM/Al decreases from 5800 to 4700 m/s with increases in grain size from 8 to 350 µm. In the case of NM/Cu, with grain sizes of 98 and 560 mm, the detonation velocity limits were similar – approximately 3500 m/s. A different dependence of the limit of detonation velocity on the amount of aluminium powder in NM/Al mixtures was presented by Kato *et al.* [33, 34]. Nitromethane charges containing grains with dimensions of 39, 110 and 350 µm had detonation velocity limits of 5230, 5350 and 3940 m/s, respectively, *i.e.* the highest detonation velocity was obtained for aluminium powders with the average grain size tested in the experiments. Kato et al. also states that the critical diameters of NM/Al and MN/Cu increase with increases in the grain size of the metallic additive, although no numerical data were provided.

2. Experiments

2.1. Materials and preparation of explosive specimens

The tests used nitromethane produced by Spółdzielnia Pracy Chemików in Łódź, flaked aluminium powder (Al_{fl}) with a surface area of 4000 cm^2/g and granulated aluminium powder (Al_{gr}) with a grain size of less than 0.15 mm, as well as polymethyl methacrylate (PMMA). Mixtures for detonation parameter testing were prepared by mixing nitromethane with crushed polymethyl methacrylate (2%) and stirring until dissolved. Next, aluminium powder was added to the gelatinous solution and the whole mixture was stirred until visually homogeneous.

2.2. Detonation velocity measurement

Detonation velocity was determined using a contact sensor method. The explosive material was placed in a vinidur (uPVC) tube with an internal diameter of 38 mm and an external diameter of 40 mm and a length of 230 mm, in which four sensors were placed. The detonator used was a plane-wave generator made of hexolite 50/50. The measurement results are given in Tables 1 and 2 and Figure 1.

Table 1. Detonation velocities of mixtures of nitromethane with flaked aluminum powder

Component content [%]			Density [g/cm^3]	Detonation velocity [m/s]
Nitromethane	Flaked aluminum powder	Polymethyl methacrylate		
98.0	0	2.0	1.14	6300
88.2	10	1.8	1.17	6180
78.4	20	1.6	1.19	5970
68,6	30	1.4	1.21	5410
58,8	40	1.2	1.23	4240
49.0	50	1.0	1.27	2600

Table 2. Detonation velocities of mixtures of nitromethane with granulated aluminum powder

Component content [%]			Density [g/cm^3]	Detonation velocity [m/s]
Nitromethane	Granulated aluminum dust	Polymethyl methacrylate		
98.0	0	2.0	1.14	6300
88.2	10	1.8	1.20	6050
78.4	20	1.6	1.25	5930
68.6	30	1.4	1.35	5840
5.,8	40	1.2	1.42	5750
49.0	50	1.0	1.50	5430
39.2	60	0.8	1.58	5150
29.4	70	0.6	1.65	4700

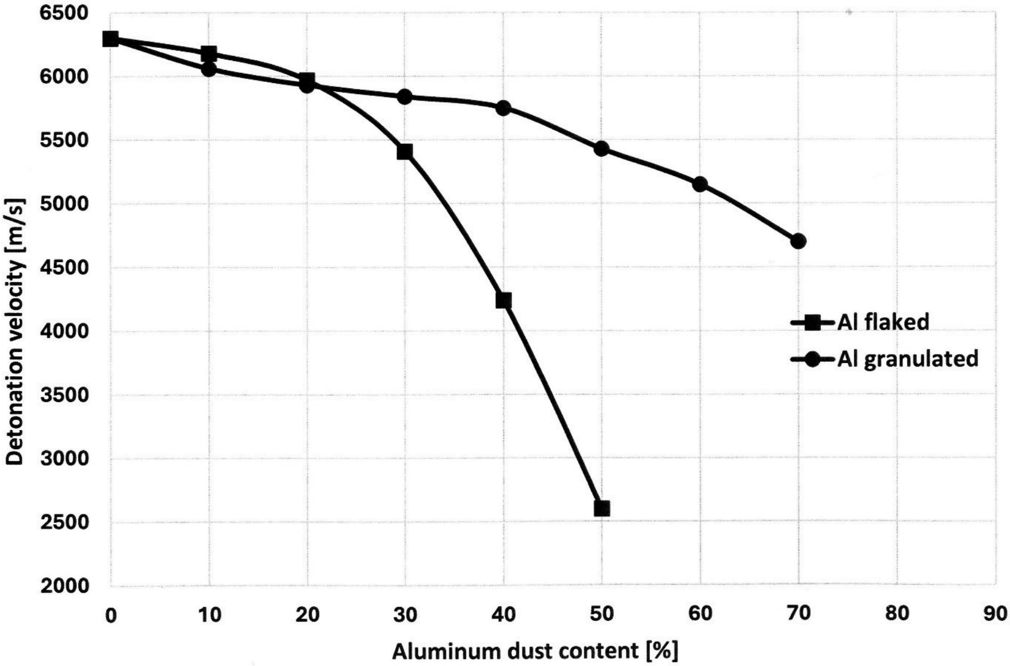

Figure 1. Detonation velocities of NM/Al v aluminum powder content

2.3. Critical diameter measurement

The critical diameter (d_{cr}) was measured initially using the conical charge method and, to refine the experimental results, using the telescopic charge method. The charges were placed on a steel plate marked with lines indicating the diameters. The results of the critical diameter measurements are given in Tables 3 and 4 and Figure 2.

Table 3. Critical detonation diameters of mixtures of nitromethane with flaked aluminum powder

Component content [%]			Density [g/cm^3]	Critical diameter [mm]
Nitromethame	Flaked aluminum powder	Polymethyl methacrylate		
98.0	0	2.0	1,14	16
88.2	10	1.8	1,17	10
78.4	20	1.6	1,19	7
68,6	30	1.4	1,21	8
58,8	40	1.2	1,23	12
49.0	50	1.0	1,27	18

Table 4. Critical detonation diameters of nitromethane mixtures with granulated aluminum powder

Component content [%]			Density [g/cm³]	Critical diameter [m/s]
Nitromehtane	Granulated aluminum powder	Polymethyl methacrylate		
98.0	0	2.0	1.14	16
88.2	10	1.8	1.20	18
78.4	20	1.6	1.25	12
68,6	30	1.4	1.35	8
58,8	40	1.2	1.42	6
49.0	50	1.0	1.50	8
39.2	60	0.8	1.58	12
29.4	70	0.6	1.65	18

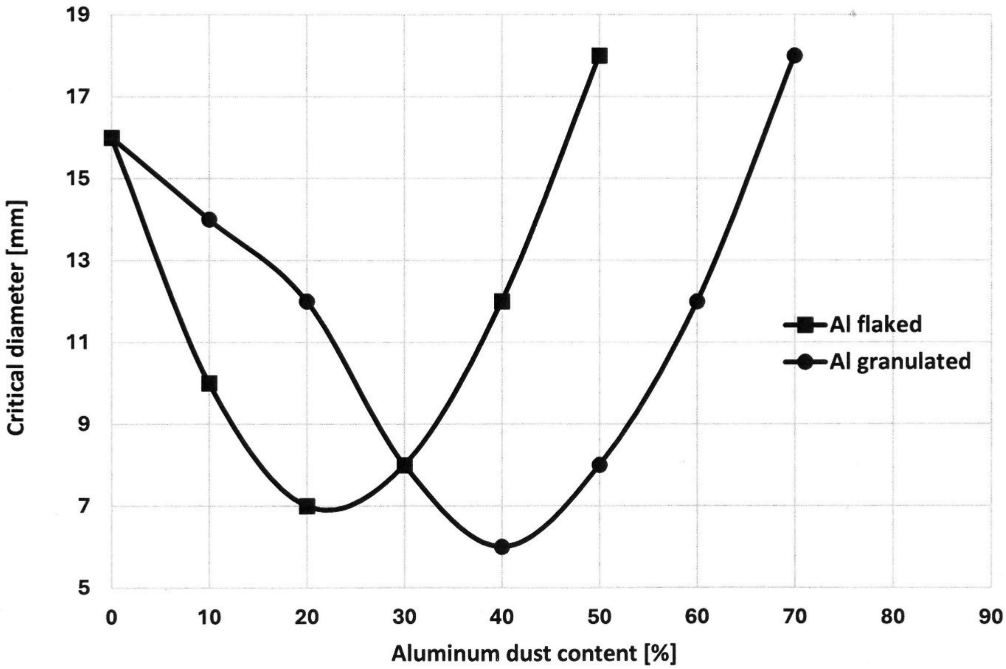

Figure 2. Critical diameters of NM/Al depending on aluminum powder content

2. Numerical estimates

The calculations were performed using EXPLO5 v8.01.01 [50] with the BKW standard parameter library (α = 0.499; β = 0.403; κ = 10.864 and θ = 5441). Cases were analysed in which the aluminium used was chemically active in the detonation wave at 60, 30 and 0%. The actual densities of the mixtures used in the experiments were used for the calculations. The results of the calculations are presented in Tables 5-10 and compared with the experimental data in Figures 3 and 4.

Table 5. Calculated values of detonation velocity of mixtures of nitromethane with flaked aluminum powder - 60% of chemical activity

Component content [%]			Density [g/cm^3]	Detonation velocity [m/s]
Nitromethane	Flaked aluminum dust	Polymethyl methacrylate		
98.0	0	2.0	1.14	6971
88.2	10	1.8	1.17	6686
78.4	20	1.6	1.19	6309
68.6	30	1.4	1.21	5898
58.8	40	1.2	1.23	5136
49.0	50	1.0	1.27	4139

Table 6. Calculated values of detonation velocity of mixtures of nitromethane with granulated aluminum powder - 60% of chemical activity

Component content [%]			Density [g/cm^3]	Detonation velocity [m/s]
Nitromethane	Granulated aluminum dust	Polymethyl methacrylate		
98.0	0	2.0	1.14	6971
88.2	10	1.8	1.20	6789
78.4	20	1.6	1.25	6499
68.6	30	1.4	1.35	6268
58.8	40	1.2	1.42	5765
49.0	50	1.0	1.50	4937
39.2	60	0.8	1.58	4545
29.4	70	0.6	1.65	3972

Table 7. Calculated values of detonation velocity of mixtures of nitromethane with flaked aluminum powder - 30% of chemical activity

Component content [%]			Density [g/cm^3]	Detonation velocity [m/s]
Nitromethane	Flaked aluminum dust	Polymethyl methacrylate		
98.0	0	2.0	1.14	6971
88.2	10	1.8	1.17	6664
78.4	20	1.6	1.19	6262
68.6	30	1.4	1.21	5833
58.8	40	1.2	1.23	5335
49.0	50	1.0	1.27	4770

Table 8. Calculated values of detonation velocity of mixtures of nitromethane with granulated aluminum powder - 30% of chemical activity

Component content [%]			Density [g/cm^3]	Detonation velocity [m/s]
Nitromethane	Granulated aluminum dust	Polymethyl methacrylate		
98.0	0	2.0	1.14	6971
88.2	10	1.8	1.20	6772
78.4	20	1.6	1.25	6468
68.6	30	1.4	1.35	6299
58.8	40	1.2	1.42	5955
49.0	50	1.0	1.50	5543
39.2	60	0.8	1.58	4908
29.4	70	0.6	1.65	3972

Table 9. Calculated values of detonation velocity of mixtures of nitromethane with flaked aluminum powder - 0% of chemical activity

Component content [%]			Density [g/cm^3]	Detonation velocity [m/s]
Nitromethane	Flaked aluminum dust	Polymethyl methacrylate		
98.0	0	2.0	1.14	6971
88.2	10	1.8	1.17	6602
78.4	20	1.6	1.19	6156
68.6	30	1.4	1.21	5665
58.8	40	1.2	1.23	5117
49.0	50	1.0	1.27	4571

Table 10. Calculated values of detonation velocity of mixtures of nitromethane with granulated aluminum powder - 0% of chemical activity

Component content [%]			Density [g/cm^3]	Detonation velocity [m/s]
Nitromethane	Granulated aluminium dust	Polymethyl methacrylate		
98.0	0	2.0	1.14	6971
88.2	10	1.8	1.20	6717
78.4	20	1.6	1.25	6383
68.6	30	1.4	1.35	6213
58.8	40	1.2	1.42	5867
49.0	50	1.0	1.50	5498
39.2	60	0.8	1.58	5048
29.4	70	0.6	1.65	4430

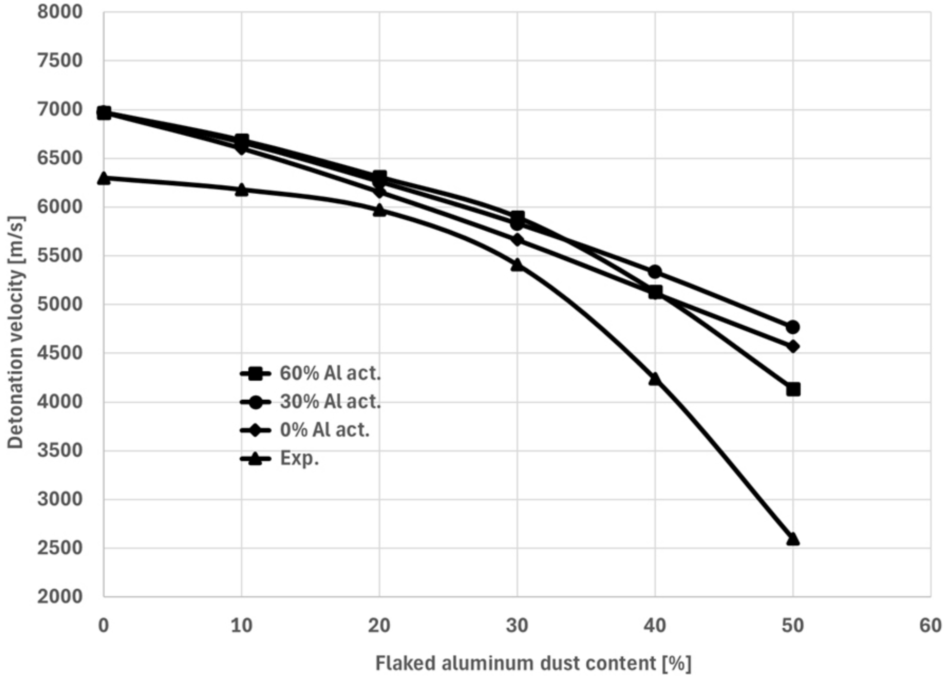

Figure 3. Dependence of detonation velocity on the ratio and activity of flaked aluminium powder

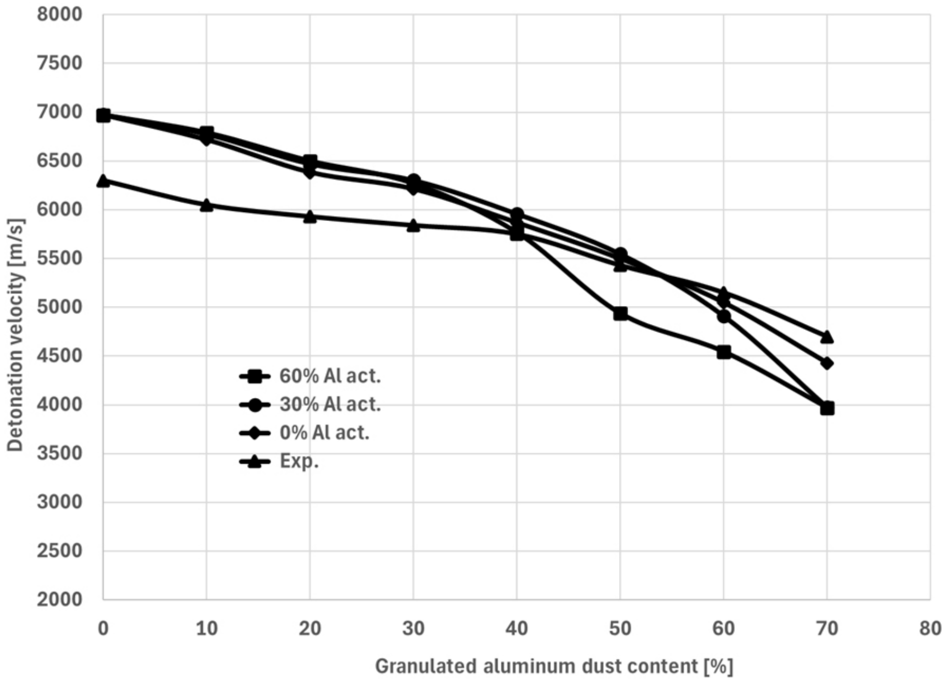

Figure 4. Dependence of detonation velocity on the ratio and activity of granulated aluminum powder

3. Analysis of test results

The results of detonation velocity tests showed that the addition of aluminum powder to nitromethane, regardless of its grain size, causes a decrease in the measured parameter. This was confirmed by the results of experiments carried out on other individual explosives classified by Price [40] as belonging to the first group of explosives, presented in [41-49]. The tests were carried out for: TNT [41, 42, 44, 46, 47], hexogen [41, 42, 49], octogen [42, 43, 47, 49] and bis(2,2,2-trinitro)nitroamine [43, 45]. Only in [46] have the results of detonation velocity measurements been presented, indicating that the addition of aluminum powder to TNT causes an increase in the determined parameter. The addition of aluminum powder to a PBX explosive containing octogen and polyurethane binders also causes a decrease in detonation velocity [48]. On the other hand, a different nature of the $D = f(\%\mathrm{Al})$ dependency was found in [49], where, among other things, the detonation and thermochemical parameters of mixtures of wax- and graphite-phlegmatised hexogen (20%) with aluminum powder and ammonium chlorate(VII) (NH_4ClO_4) were measured and theoretically estimated. The addition of aluminum powder at a level of 30-55%, with a simultaneous reduction in NH_4ClO_4 content to 45-20%, resulted in an increase in detonation velocity from 6020 to 6400 m/s. The nature of this $D = f(\mathrm{Al})$ dependency may result from the fact that, at such low hexogen content, the priority chemical reaction in the detonation wave zone is the reaction between aluminium and oxygen generated by the decomposition of NH_4ClO_4. As demonstrated by Moyseyenko [51], aluminium powder, in certain concentrations, increases the detonation velocity and work capacity and decreases the critical diameter of NH_4ClO_4.

Baudin and Burgues [52], following the work of Finger *et al.* [53] and the model of detonation of gas mixtures containing aluminium powder developed by Veyssiere [54, 55], propose four stages of explosive/Al detonation:

- initiation of the most explosive component under the influence of a shock wave,
- exothermic decomposition of the above-mentioned component and, for example, the binder contained in PBX,
- heating of aluminum powder grains by detonation products to the ignition temperature, which occurs earlier under the influence of the shock wave but is less intense than in explosives because the metallic additive has lower compressibility [56],
- oxidation of aluminum in the rarefaction wave, preceded by the breaking of the oxide layer, caused by the higher thermal expansion of aluminum than its oxide [57].

The results of the research presented in almost all of the previously cited publications, as well as those carried out as part of this work, show that the addition of aluminum powder to high-energy individual explosives causes a decrease in the detonation velocity. A number of behaviours during detonation of the explosive/Al mixture, which contribute to a decrease in the detonation velocity, are proposed. [58].

- Aluminum powder behaves as an inert additive both in physical and chemical terms. There is no thermodynamic equilibrium between it and the decomposition products of the explosive material, and aluminium, which does not react with them, acts as ballast, lowering the detonation heat. According to Dremin *et al.* [59], such an effect on the detonation parameters of the explosive/Al mixture is typical for high charge densities containing metallic additive grains with dimensions of 80-270 μm [59].
- Aluminum powder is chemically neutral and absorbs the heat generated by the detonation of the explosive material. When oxidised in a wave of rarefaction, it increases the ability to perform work [60].
- A disproportionation reaction occurs, resulting in the formation of lower aluminum oxides (Al_2O, AlO) in gaseous form, and their presence in detonation products reduces the overall reaction balance [59, 61, 62]. This mechanism was adopted for the entire range of explosive-Al load densities in [61, 62], and for low densities in [59].
- A reaction occurs in which aluminum(III) oxide generated during the oxidation evaporates. The evaporation process is a strongly endothermic phenomenon, which leads to a decrease in the parameters of the detonation wave. This hypothesis is acceptable only if aluminum(III) oxide is present in gaseous

form under detonation conditions and is rejected in [59].

– Aluminum reacts to a small extent in the chemical reaction zone of the detonation wave, which results from the low oxidation rate of metal grains compared to the decomposition rate of the high explosive material [63]. The aluminum atoms on the surface of the grains undergo oxidation, while the remaining atoms are only heated. The energy balance of these transformations is negative and results in a reduction in the amount of energy transferred to the detonation wave front.

In the initial range of aluminum powder content (<20%), the decrease in detonation velocity is very similar for both types of metallic additives tested. Then it is more intense in the case of flaked aluminum powder (Fig. 1). Under the test conditions, NM/Al mixtures detonated at a granular aluminum powder content of 70% and a flaked aluminum powder content of 50%, with minimum values of 4700 and 2600 m/s, respectively (Fig. 1).

The diverse nature of the obtained $D = f(\%\text{Al})$ dependencies may result from two reasons: the larger specific surface area of flaked aluminum powder and the lower density, at the same Al content, of NM/Al mixtures based on it. The larger surface area results in a faster heat exchange between the nitromethane decomposition products and the aluminum powder. The result of lower NM/Al density is lower nitromethane concentration per unit volume, which means that less heat is released, affecting the detonation velocity.

The dependency of the experimentally determined detonation velocity on changes in the content of flaked aluminum dust (Fig. 3) coincides with the theoretical dependence in the range below 40% aluminum content. The complexity and heterogeneity of the mixture determine the occurrence of disturbances in the detonation process, which is reflected in the clearly lower experimental values compared to the theoretical ones.

Similarly, the dependency of the experimentally determined detonation velocity on changes in the content of granulated aluminum dust (Fig. 4) coincides with the theoretical dependence in the range below 40% aluminum content. When Al content exceeds 50%, the experimentally determined detonation velocity is higher than the theoretical value. The use of a higher proportion of granulated aluminum probably causes partial sedimentation of the solid fuel, which may improve the detonation parameters of the nitromethane-rich layer of the charge. In both cases, the nature of the experimental graph, in the range of Al 0–30%, is very similar to the theoretical output determined for 60% Al.

The $d_{cr} = f(\%\text{Al})$ dependencies are very similar for NM/Al containing flaked and granulated aluminium powder (Tables 3 and 4, Fig. 2). With increasing aluminum powder content, the critical diameter first decreases and then increases after reaching a minimum. A similar dependency of $d_{cr} = f(\%\text{Al})$ for NM/Al was presented by Kurbangalina [64]. This type of dependency is typical for aluminised explosives containing compounds with poorly defined explosive properties – ammonium nitrates and guanidines [65]. In the case of NM/Al flaked aluminum powder, the smallest critical diameter is 7 mm (20%) and for granulated powder it is 6 mm (40%). Quantitative differences also occur at the highest critical diameter (19 mm). NM/Al have them at Al_{fl} 50% and Al_{gr} 70%. This may be due to the fact that, due to its increased surface area, Al_{fl} contains a larger amount of aluminum(III) oxide, which is an inert substance from the point of view of potential chemical reactions which may occur during the NM/Al detonation process.

In the case of individual explosives, a reduction in critical diameter as a result of the addition of aluminum powder was obtained for cast TNT. Brousseau *et al.* [66] investigated the detonation velocities of TNTs containing 10, 20 and 30% of aluminum powder with nano- and micrometric particle sizes. For example, for a powder content of 30%, they determined detonation velocities in charges with a diameter of 25.4 mm in all cases, which is lower than the critical diameter of cast TNT – 27.4 mm [67]. The results of research conducted by Kurbangalina [64] and Brousseau *et al.* [66] show that if the charge matrix is a homogeneous explosive material, the aluminum powder contained therein, forming a dispersed phase, is the source of hot spots which determine the initiation of the detonation process.

4. Conclusions

- ♦ The detonation of condensed explosives is a highly complex phenomenon, as it involves both chemical reactions and physical processes occurring under extreme temperatures and pressures. The situation becomes even more complicated when aluminium powder is present in the explosive mixture, as its physical and chemical properties differ significantly from those of individual explosives. Therefore, despite the fact that research on explosive/Al mixtures has been conducted for over 120 years, no theory has been developed to explain the behaviour of aluminum powder in the chemical reaction zone of a detonation wave of individual high-energy explosives.
- ♦ Considering the detonation velocity measurements presented in this paper it can be assumed that due to the narrow width of the chemical reaction zone of nitromethane, aluminum does not participate in reactions with the detonation products of explosives. However, to a certain extent, its content facilitates the NM/Al initiation process.

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