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Thermal Stability and Detonation Characteristics of Pressed and Elastic Explosives on the Basis of Selected Cyclic Nitramines^{*)}

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Abstract: Bicyclo-HMX (cis-1,3,4,6-tetranitro-octahydroimidazo-[4,5-d]imidazole or BCHMX) was studied as a plastic explosive bonded with the C4 matrix and with Viton A. Also a series of nitramines namely RDX (1,3,5-trinitro-1,3,5-triazinane), HMX (1,3,5,7-tetranitro-1,3,5,7-tetrazocane) and HNIW (ε-2,4,6,8,10,12-hexanitro-2,4,6,8,10,12-hexaazaisowurtzitane, ε-CL-20) were studied for comparison with the same types of binders. The detonation velocity, D, of all prepared mixtures was measured. Their thermal stability was determined using

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non-isothermal differential thermal analysis (DTA). While the C4 matrix lowers the thermal stability of the resulting explosives, Viton A enhances this stability. Approximate relationships between the peaks of exothermic decomposition and the D values were found. The detonation parameters were also calculated by means of Kamlet & Jacobs method, CHEETAH and improved EXPLO5 code for all the mixtures. From the measured D values and the calculated detonation parameters, it is obvious that the detonation parameters of BCHMX-mixtures are very close to HMX-explosives and better than those of RDX-mixtures. It was found that the C4 matrix reduces the difference between the values of energy of detonation in the studied C4 mixtures. As expected, the pressed HNIW-Viton A mixture has the highest detonation parameters of all of the prepared mixtures.

Keywords: detonation, C4 matrix, BCHMX, HMX, HNIW, RDX, stability, Viton

Introduction

The development in the field of energetic materials is based on preparation of new materials with high performance, high thermal stability and low sensitivity. One of the important factors that affect the performance of energetic materials is the crystal density. This density is affected by molecular structure and conformation; a linear structure has lower density than a cyclic one, and similarly the polycyclic and the cage structures have higher density than monocyclic molecules [1]. At the Institute of Energetic Materials (IEM), we have succeeded in preparation of a new bicyclic-nitramine which is designated as bicyclo-HMX or BCHMX (*cis*-1,3,4,6-tetranitrooctahydroimidazo-[4,5-d]imidazole) [2]. It has theoretical maximum density (TMD) of 1.86 g cm^{-3} , calculated detonation velocity of 9050 ms^{-1} , detonation pressure of 37 GPa and explosion heat of 6.518 MJ kg^{-1} [3].

The practical applications of the BCHMX have not been described until now. Therefore, this study deals with the formation of two kinds of plastic explosives based on this compound. One kind was made on the basis of C4 matrix which consists of polyisobutylene as a main part of this plastic binder [4]. The second kind is bonded by Viton A which is a copolymer of vinylidene fluoride with hexafluoropropylene [5]. For comparison a series of plastic explosives based on the C4 matrix and another series based on Viton A, containing one of three selected nitramines – RDX (1,3,5-trinitro-1,3,5-triazinane), β -HMX (β -1,3,5,7-tetranitro-1,3,5,7-tetrazocane), and ϵ -HNIW (ϵ -2,4,6,8,10,12-hexanitro-2,4,6,8,10,12-hexaazaisowurtzitane, ϵ -CL-20) were prepared and studied in this paper. The experimentally measured detonation velocities and calculated detonation

parameters of all the prepared explosives were a groundwork for elicitation of the corresponding conclusions; the mentioned calculations were done by a modified Kamlet & Jacobs method [6-8], CHEETAH [6, 9] and EXPLO5 [6, 10] codes. Likewise the results of differential thermal analysis (DTA) of all the explosives used in this study are included as subsidiary information.

Experimental

Preparation of Plastic Explosives based on C4 matrix

The plastic explosives with C4 matrix were prepared at Explosia Pardubice (Research Institute of Industrial Chemistry) [6]. The used RDX is a product of Dyno Nobel (mixture of Classes 2 and 5 according to the standard [11]), β -HMX was imported from Russia and its particle size was close to Class 3 according to the standard [12]), technical-grade ϵ -HNIW was a product of Explosia pilot plant, and BCHMX was prepared by a two step continuous laboratory synthesis at the IEM [3]. The C4 matrix is a mixture of polyisobutylene (PIB) 25%, dioctyl sebacate (DOS) 59% and oily material (HM46) 16%. The preparation of the binder was produced by mixing small pieces of polyisobutylene with the sebacate and the oil under specific conditions. The explosive 91% wt. was mixed with the binder matrix 9% wt. at 70 °C for 70 min under vacuum using computerized mixer Plastograph BRABENDER. The prepared samples were extruded with a 40 mm screw extruder to obtain long charges of plastic explosive with 16 mm diameter.

Preparation of plastic bonded explosives based on Viton A

All the samples based on Viton A 200 as a binder were prepared by modified water-solvent slurry method [13]. Viton A 200, producer DuPont Performance Elastomers, is an elastomer with a fluorine content of 66% and a density of 1.78- 1.82 g cm⁻³. This fluorine content implies a 60/40 weight ratio of vinylidene fluoride to hexafluoropropene monomers or approximately 77.83 mole % vinylidene fluoride [14]. The nitramine is slurred in water (aqueous phase) and Viton A is dissolved in an organic solvent with low boiling point, immiscible with water, which is added during the process. This process is carried out under vigorous stirring (500-600 rpm). The solvent is removed by distillation under continuous stirring, and the polymer precipitates on the surface of the crystals of the explosive at the end of the solvent elimination. All the prepared samples have 91% wt. of explosive and 9% wt. of Viton A.

Elemental analysis

Fisons - EA-1108 CHNS-O elemental analyzer was used for the determination of C, H and N in the prepared samples [6]. The results of the elemental analysis were recalculated to match the N content with the individual explosive and reported in Table 1 as a hypothetical formula. Then the calculated formula was used as if it were a single explosive and it was submitted to CHEETAH [6, 9], EXPLO5 [6, 10] and Kamlet & Jacobs [6-8] methods for calculations.

Thermal stability

A DTA 550 Ex apparatus was used for thermal analysis of explosives [15]. The measurements were carried out at atmospheric pressure, with the tested sample in a direct contact with the air. The tested sample (0.05 g) was placed in a test tube made of Simax glass, 5 mm in diameter and 50 mm long. The reference standard was 0.05 g aluminum oxide. A linear heating rate of $5\text{ }^{\circ}\text{C min}^{-1}$ was used. The results presented in Figure 1 show the exothermic effect of the thermal decomposition and the endothermic peaks of the samples based on C4 matrix and the pure explosives, while the results for the samples based on Viton A are presented in Figure 2, in addition to results for the pure explosives as a reference. Peak temperatures of exothermic thermal decomposition at a linear heating rate of $5\text{ }^{\circ}\text{C min}^{-1}$ for all samples are presented in Table 1.

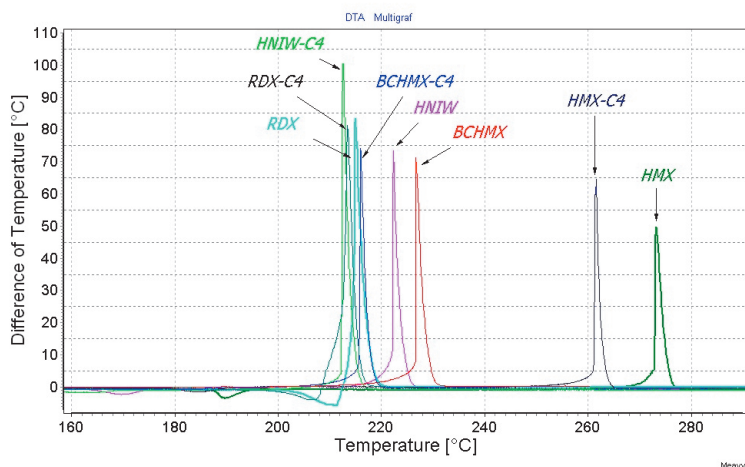


Figure 1. The typical DTA curves of the pure and by C4 matrix bonded nitramines (at heating rate $5\text{ }^{\circ}\text{C min}^{-1}$).

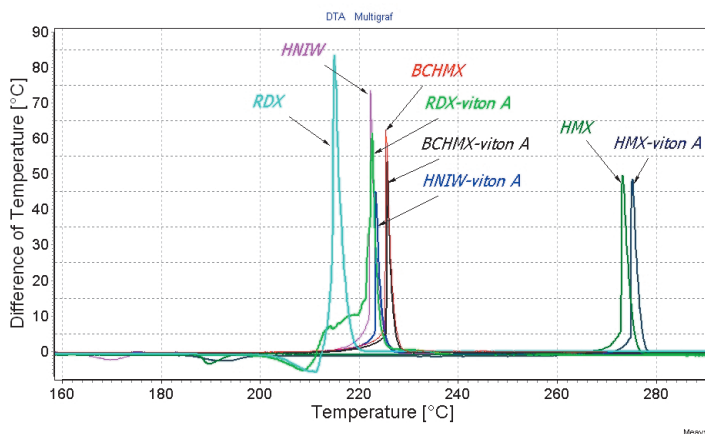


Figure 2. The typical DTA curves of the pure and by Viton A bonded nitramines (at heating rate $5\text{ }^{\circ}\text{C min}^{-1}$).

Heat of combustion

Automatic Combustion Calorimeter MS10A was used for measuring the heat of combustion of the samples. The samples were prepared and placed in bomb containing 5 ml of water then filled with an excess of oxygen [16]; the data obtained from the measurements are reported in Table 1 as heat of combustion. These data were used for calculation of the heat of formation of the samples (it is obtained by the summation of the heat of combustion of the sample and the heat of formation of the products from the combustion process which are CO_2 (g) and H_2O (L)), and these heats were used for the theoretical calculations.

Pressing of the samples

The plastic bonded explosives based on Viton A were pressed at the Explosia Co. Pardubice using hydraulic pressing machine (PYE 100). All the samples were pressed at a pressure of 486 MPa under vacuum for 10 seconds. Cylinders with diameter 16 mm and height 20 mm were made. For each composition, 18 cylinders were prepared and glued together as three charges made of 6 cylinders.

Detonation velocity measurements

The detonation velocity of all prepared mixtures was measured by the ionization copper probe method [17] where the data were recorded on an oscilloscope (Tektronix TDS 3012). The plastic samples were prepared in the form of cylinder 16 mm in diameter and 200 mm long. The copper probes were

inserted in the charge. The first probe was inserted at distance of 50 mm from the booster and the second probe was inserted at 100 mm distance from the first probe. In the case of the pressed charges, the first probe was inserted between the first and the second prepared cylinder while the second probe was inserted between the fifth and the last cylinder where the distance between the two probes was 80 mm. Both probes were connected to the oscilloscope via coaxial cable. Charges were set off using a booster charge (Semtex 1A with mass of 3 g and diameter of 16 mm) initiated with an electric detonator. For each sample, three measurements were performed and the mean value (max. ± 60) for each is reported in Table 1.

Calculation of the detonation characteristics

The theoretical detonation characteristics (detonation velocity, D , detonation pressure, P , detonation energy, $E_{det.}$, heat of detonation, $Q_{det.}$ and heat of explosion, Q_{max}) for all prepared samples as well as pure nitramines were calculated by the use of the CHEETAH code [9], EXPLO5 code [10, 19] and Kamlet & Jacobs method [7, 8]. In the case of the CHEETAH calculations, the BKWS set of parameters for the BKW EOS, i.e., $\alpha = 0.5$, $\beta = 0.298$, $\kappa = 10.50$, $\Theta = 6620$ [19] was used, while in the EXPLO5 code, the BKWN set of parameters were applied, this parameters are: $\alpha = 0.5$, $\beta = 0.176$, $\kappa = 14.71$, $\Theta = 6620$ [18]; the original EXPLO5 code (version 3) was modified by Sućeska also for calculation of the fluorine containing explosives and its first utilization is in this paper as a version 4. The heat of explosion in the Kamlet & Jacobs method [7, 8] is the heat released in a constant volume explosion and it is determined by subtracting the heats of formation of the explosive (reactants) from the sum of heats of formation for the detonation gasses (products). The detonation heats calculated by EXPLO5 are obtained in similar way, but the composition of products is taken from the Chapman-Jouguet (CJ) state. The detonation energy calculated by CHEETAH code is a sum of mechanical and thermal energies. The mechanical energy represents the amount of energy available to do mechanical work and it is equal to the expansion work of detonation products and determined for the volume of detonation products corresponding to pressure $p = 1$ atm, while the thermal energy represents the amount of energy locked up as heat of detonation products at $p = 1$ atm [4], for calculations, the composition of detonation products was frozen at a temperature of 1800 K on the isentrope beginning at the CJ point. The calculated detonation parameters of all prepared samples and the pure explosives are reported in Table 2.

Results and Discussion

The DTA measurements of plastic explosives based on C4 matrix show that the temperature difference ΔT (peak height) at the decomposition of these mixtures is higher than ΔT of the corresponding explosive in its pure state (compare each other of Figures 1 and 2). At the same time the peak temperatures for all the prepared C4 mixtures are lower than those of pure explosives as shown in Figure 1; this is most likely due to the presence of the polar plasticizer (DOS) in binders which acts as a solvent for all the nitramines used and leads to the decrease of the mixture's decomposition temperature as reported in [6, 21]. In the case of the explosives bonded by Viton A, it is clear that the peak temperatures for all prepared mixtures are slightly higher than those of pure explosives within range from 1-5 C as shown in Figure 2; this Figure proves that the presence of Viton A in the mixture enhances the thermal stability of the nitramines applied in these mixtures. It is obvious that mixtures bonded by Viton A have higher thermal stability than the mixtures based on C4 matrix. The above mentioned observation are reminiscent of the older finding [24] that a film of inert liquid (here it might be Viton A) inhibits the thermal decomposition of RDX, while an additive of solvent type (here DOS) accelerates this decomposition.

An approximate comparison of experimental detonation velocities and DTA peaks of exothermic decomposition of the studied explosives (see Figure 3) is interesting (these peak temperatures are in a linear correlation with autoignition temperatures of explosives [25]). In the sense of this comparison the studied group is divided into three sub-groups according to the thermal reactivity of the given explosives (Figure 3): the most reactive mixtures are HNIW and crystalline RDX, less reactive are plastic explosives on the basis of BCHMX and RDX and the most stable are HMX explosives. Figure 3 shows that the increase of thermal stability corresponds to the increase of detonation velocity of explosives in each of these groups. This finding conforms to influence of binder on both the thermal and shock reactivities of the applied nitramines (here disadvantage of the C4 mixture application is evident from the point of view of obtained D values for other binders). The mentioned relationship is not so much pronounced and deserves to be investigated further.

Table 1. The data obtained from the experimental

Code designation of explosive	Formula	Mol. weight (g mol ⁻¹)	Heat of combustion (J g ⁻¹)	Heat of formation (kJ mol ⁻¹)	Density (g cm ⁻³)*	Detonation velocity experiment (m s ⁻¹)	DTA peak at 5 °C min ⁻¹ (°C)
RDX cryst.	C ₃ H ₆ N ₆ O ₆	222.14	9438	47.5	1.76*	8750*	214.8
RDX - C4	C _{4,66} H _{8,04} N ₆ O _{5,99}	243.7	12356±74	22.0	1.61	8055±31	212.9
RDX-Viton A	C _{3,63} H _{6,46} F _{0,77} N ₆ O _{5,95}	244.00	9726	-90.3	1.76	8285	222.6
β-HMX cryst	C ₄ H ₈ N ₈ O ₈	296.18	9485	77	1.90*	9100*	273.3
HMX - C4	C _{6,12} H _{11,40} N ₈ O _{8,14}	327.1	12382±21	2.6	1.67	8318±35	261.5
HMX-Viton A	C _{4,82} H _{8,51} F _{1,02} N ₈ O _{7,93}	324.80	9808	-75.4	1.84	8602	275.2
BCHMX -3% Viton B**	C _{4,23} H _{6,14} N ₈ O ₈ F _{0,33}	303.34	9147±29	173.5	1.79	8650	225.4
BCHMX-C4	C _{6,18} H _{10,24} N ₈ O _{8,08}	326.01	12163±39	55.3	1.66	8266±54	215.9
BCHMX-Viton A	C _{4,85} H _{6,67} F _{1,02} N ₈ O _{7,99}	324.27	9436	48.6	1.81	8474	225.9
ε-HNIW	C ₆ H ₆ N ₁₂ O ₁₂	438.23	8311	397.8	1.96***	9440***	222.4
HNIW-C4	C _{9,17} H _{12,09} N ₁₂ O _{12,19}	485.40	11452±13	201.1	1.77	8594±22	212.3
HNIW-Viton A	C _{7,22} H _{6,83} F _{1,51} N ₁₂ O _{11,93}	481.28	8661	127.7	1.94	9023	223.5

*) Taken from monograph [23] (the values from measurements under confined conditions);

**) Detonation velocity of pure BCHMX was not measured because we have not possibility to realize this measurement under confined conditions, therefore, we used unconfined condition with the pressed charge of phlegmatized nitramine by 3% of Viton B [3];

***) Taken from Ref. [22];

*) The TMD values of pure nitramines (in g cm⁻³) are: 2.04 for ε-HNIW [23], 1.96 for β-HMX [23], 1.86 for BCHMX [2, 3] and 1.82 for RDX [23].

Table 2. Results for the calculated detonation characteristics (detonation velocity, D , detonation pressure, P , detonation energy, E_{det} , heat of detonation, Q_{det} , and heat of explosion, Q_{max})

Explosive	CHEETAH calculation				EXPLO5 calculation				Kamlet & Jacobs calculation			
	D (m s ⁻¹)	$\frac{D_{calc}-D_{exp}}{D_{exp}/100}$ (%)	P (GPa)	Energy of detonation E_{det} (J g ⁻¹)	D (m s ⁻¹)	$\frac{D_{calc}-D_{exp}}{D_{exp}/100}$ (%)	P (GPa)	Heat of detonation Q_{det} (J g ⁻¹)	D (m s ⁻¹)	$\frac{D_{calc}-D_{exp}}{D_{exp}/100}$ (%)	P (GPa)	Heat of explosion Q_{max} (J g ⁻¹)
RDX cryst.	8771	0.2	31.52	5886	8718	-0.4	32.12	6102	8621	-1.5	32.53	6127
RDX - C4	7956	-1.2	24.42	5278	7856	-2.5	23.36	5512	7675	-4.7	24.37	5671
RDX-VitonA	8329	0.5	28.9	5518	8300	0.2	29.03	5614	8024	-3.1	28.18	5505
β-HMX cryst.	9382	3.1	37.76	5925	9225	1.4	38.00	6075	9105	+0.05	37.97	6145
HMX-C4	8271	-0.6	27.02	5376	8130	-2.3	25.86	5564	7912	-4.9	26.51	5693
HMX-VitonA	8678	0.9	32.31	5692	8627	0.3	32.44	5756	8331	-3.2	31.19	5640
BCHMX- 3%Viton B*	8686	0.4	32.28	6039	8705	0.6	33.02	6296	8641	-0.1	33.02	6386
BCHMX-C4	8145	-1.5	26.23	5385	8056	-2.5	25.49	5602	7868	-4.8	26.13	5763
BCHMX-VitonA	8472	0	31.00	5851	8504	0.4	31.83	6006	8299	-2.1	30.66	5938
ε-HNIW	9372	-0.7	40.16	6557	9338	-1.1	40.82	6462	9365	-0.8	40.88	6609
HNIW-C4	8442	-1.8	29.77	5547	8425	-2	29.64	5744	8246	-4	29.86	5922
HNIW-VitonA	8770	-2.8	34.97	5892	8850	-1.9	36.33	6022	8667	-4.3	34.81	5939

*) Data for pure BCHMX are not presented in this Table because they are missed also in Table 1.

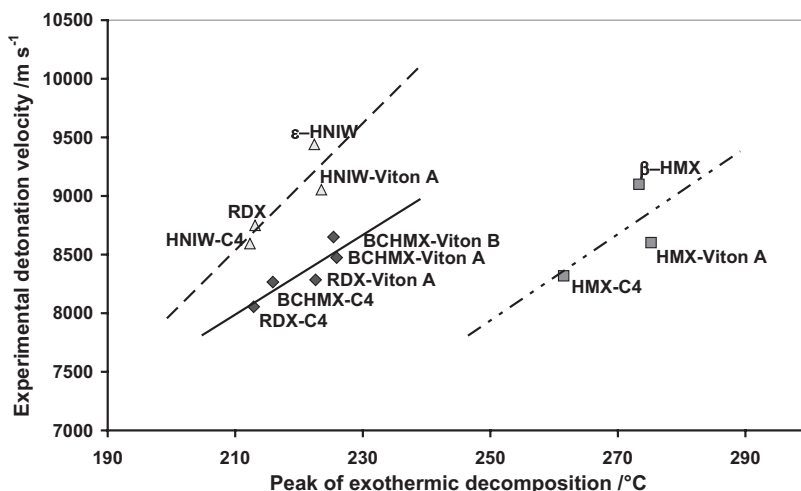


Figure 3. Approximate relationship between the experimental detonation velocities and DTA-peak temperatures (at 5 °C min⁻¹).

According to the Kamlet & Jacobs method, the D values for all the samples are different from the experimental values within range of 5% difference. Further, CHEETAH calculations for all the samples (pure explosives, pressed and elastic samples) showed difference than the experimental values in the range between +3 to -2.8 %. Results of the EXPLO 5 code showed D values close to the experimental results with difference in the range between +1.4 to -2.5% (see also Table 2).

In case of plastic explosives based on C4 matrix, it was observed that the measured detonation velocity of plastic BCHMX-C4 was 200 m s⁻¹ higher than the one of plastic RDX-C4 and 60 m s⁻¹ lower than in the case of plastic HMX-C4. While comparing the results of the detonation parameters, according to the three methods of calculations, it was observed that BCHMX-C4 has slightly lower detonation velocity and detonation pressure than HMX-C4 and slightly higher heat of explosion than HMX-C4. As expected the highest detonation parameters of all the prepared plastic samples based on C4 matrix was observed for HNIW-C4.

Regarding to the three methods of calculation (see Table 2), it was observed that the detonation velocity and the detonation pressure of samples based on BCHMX are lower than that based on HMX, while the heat of detonation and energy of detonation of BCHMX-Viton A are higher than HMX-Viton A and close to HNIW-Viton A; this fact is due to the rigidity of BCHMX molecule (crowded molecule [2, 3]) and relatively high heat of formation of the BCHMX-Viton A explosive.

When comparing the results of the samples containing the C4 matrix with those based on Viton A, it was observed that the difference in the detonation velocities between BCHMX and HMX in the C4 matrix is 60 m s^{-1} but in case of Viton A, it is 130 m s^{-1} ; this is mostly due to the higher density in the case of the Viton A mixtures.

A mutual comparison of experimental results (here a term $\rho \cdot D^2$) and calculated detonation pressures by Kamlet & Jacobs method, CHEETAH and EXPLO5 codes is shown in Figure 4 which confirms the conformity of the experimental and the calculated results in this paper.

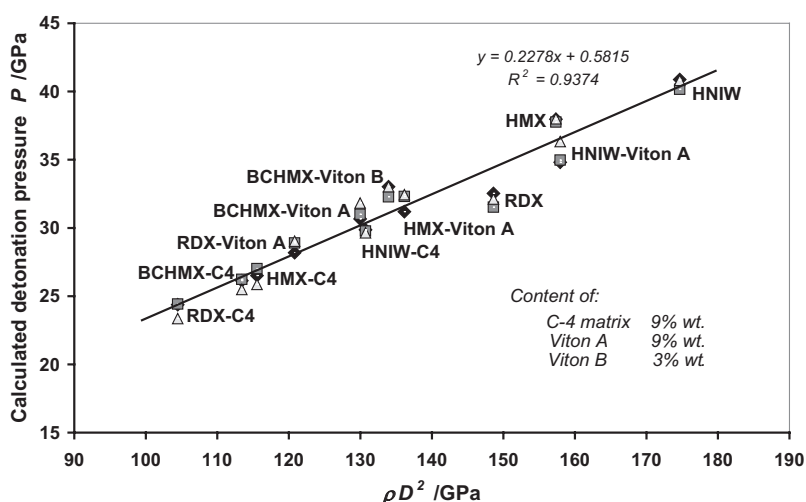


Figure 4. Mutual comparison of the calculated values of detonation pressures and square of experimental detonation velocity, D^2 , multiply by loading density, ρ : points \blacklozenge correspond to the Kamlet & Jacobs method, points \blacksquare to the CHEETAH code and points \triangle to code EXPLO5.

Relationship between the experimental D values and loading density, ρ , of the studied explosives is presented in Figure 5. It is obvious that, in the sense of this Figure, the mixtures with Viton A are not compatible with the trend for pure nitramines and their mixtures bonded by C4 matrix and with data for BCHMX, bonded by 3% wt. of Viton B. Due to the higher density of the mixtures containing Viton A, as compared with the C4 mixtures the detonation parameters of all the firstly mentioned plastic explosives are higher than the C4 explosives. At the same time, the prepared HNIW-Viton A showed the highest detonation parameters and the highest measured detonation velocity (9023 m s^{-1}). The D value of HNIW-C4

is higher than that of RDX-Viton A at nearly the same loading density by more than 300 m s^{-1} . The density of HNIW-C4 is lower than of HMX-Viton A but they have nearly the same detonation velocity. These facts show that compositions based on HNIW have the highest detonation characteristics compared with the corresponding compositions which contain the other used nitramines.

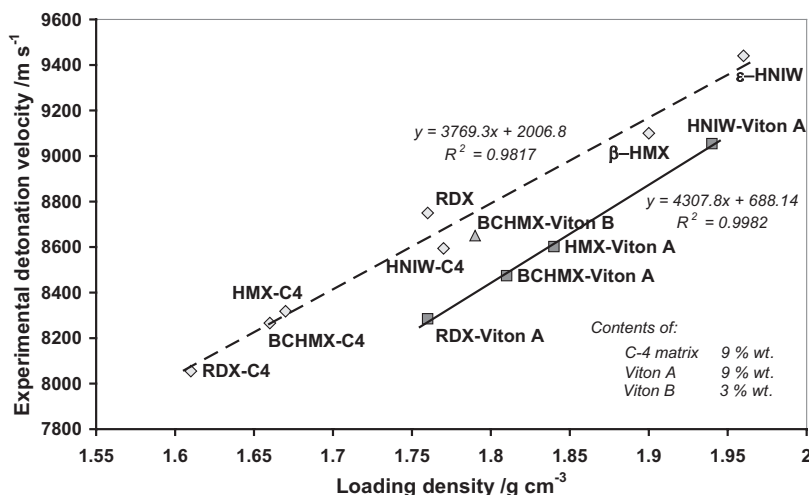


Figure 5. Relationship between the experimental detonation velocity and loading density.

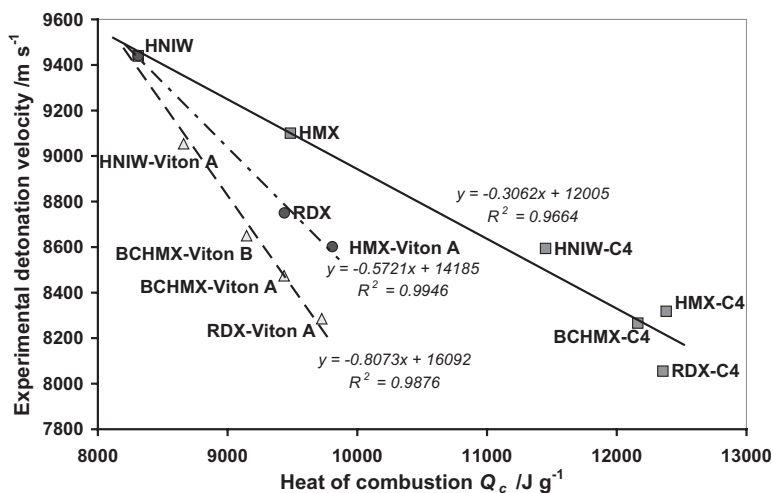


Figure 6. Relationship between the experimental detonation velocities and heats of combustion of the studied explosives.

With the above mentioned discussion about the relationships in Figure 5 also the dependence of the experimental D values on heats of combustion coheres as in Figure 6. It means that besides the influence of a binder chemical entity, also thermo-chemical factors of the given binder play a significant role during the detonation of studied mixtures; here a formation of hydrogen fluoride during detonation of the fluorine-containing explosives [8] is a most favorable influence on detonation compared with oxygen consumption by C4 matrix at enhanced formation of elemental carbon or CO in the detonation wave.

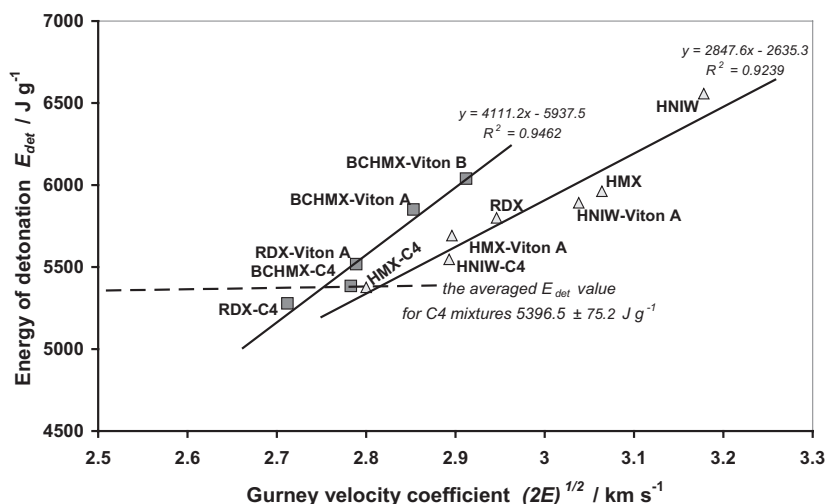


Figure 7. Relationship between the calculated energies of detonation (by CHEETAH code) and Gurney velocity coefficient, $\sqrt{2E}$, of the studied explosives.

Interesting is an elevation on the studied explosives from the point of view of fragmentation ability of the cylindrical or spherical metallic envelopes of the corresponding charges. The acceleration ability of a cylindrical metal envelope, driven by expanding products of detonation grazing along the internal surface of the envelope, can be described as the Gurney velocity coefficient, $\sqrt{2E}$ [26], which can be obtained for a given explosive from its D value by dividing of 2.97 [26]. Figure 7 presents a relationship between the calculated energies of detonation and the said coefficient. According to this Figure a group of the studied mixtures is divided into brisant explosives (mixtures on the basis of BCHMX and RDX) and the high brisant explosives, i. e. pure nitramines and mixtures with the HMX and HNIW contents. The C4 matrix seems to reduce the difference between the $E_{det.}$ values of the studied C4 mixtures.

Conclusion

Detonation parameters of BCHMX are slightly lower than those of HMX and better than RDX parameters in their applications as a filler of the plastic explosives bonded by C4 matrix and as pressed plastic bonded explosives with the Viton A binder. The thermal stability of the samples based on BCHMX is in the same range of the samples based on RDX. In comparison with Viton A the C4 matrix has a destabilizing influence on the studied nitramines; in this sense this polyfluorinated binder reminds an influence of inert liquid on the thermal reactivity of cyclic nitramines. According to thermal stability the studied group of explosives disintegrated into three subgroups; it was shown that in each from these subgroups a higher thermal stability of nitramine explosives should correspond to a higher detonation velocity. The C4 matrix as seems to reduce the difference between the values of energy of detonation in the studied C4 mixtures. Advantageous influence of Viton A on detonation of the studied nitramine explosives in comparison with C4 binder consists not only in a higher density of the resulted mixtures but also (significantly) in thermochemical aspects of polyfluorinated compounds decomposition in a detonation wave. The pressed HNIW-Viton A mixture has the highest detonation parameters of all of the prepared mixtures in this study, which verify the ranking of HNIW as the most powerful nitramine. The calculated detonation velocities by EXPLO5 code provided the very good results compared with the experimental detonation velocities also for polyfluorinated binders; the calculated detonation pressures by means of all three used methods, i.e. by the Kamlet & Jacobs method, CHEETAH and EXPLO5 codes, were in a good agreement.

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