Influence of Different Polymeric Matrices on the Properties of Pentaerythritol Tetranitrate

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ABSTRACT

Six different polymeric matrices were fabricated to reduce the sensitivity of PETN (Pentaerythritol tetranitrate). The polymeric matrices used were individually based on Acrylonitrile butadiene rubber (NBR) softened by plasticizer, styrene-butadiene rubber (SBR) softened by oil, polymethyl methacrylate (PMMA) plasticised by dioctyl adipate (DOA), polydimethylsiloxane (PDMS), polyurethane matrix, and Fluorel binder. A computerised plastograph mixer was utilised for producing three polymer-bonded explosives (PETN-NBR, PETN-SBR, and PETN-PDMS) based on the non-aqueous method. A cast-cured method was used to prepare PBX based on polyurethane (PETN-HTPB), while the slurry technique was used to prepare beads of PETN coated by either fluorel binder (PETN-FL) or based on PMMA forming (PETN-PMMA). The heat of combustion and sensitivities were investigated. The velocity of detonation was measured, while the characteristics of the detonation wave were deduced theoretically by the EXPLO 5 (thermodynamic code). The ballistic mortar experiment was performed to determine the explosive strength. By comparing the results, it was found that PDMS has the highest influence on decreasing the impact sensitivity of PETN, while the cast cured PETN-HTPB has the lowest friction sensitivity. On the other side, PETN-FL has the highest detonation parameters with high impact sensitivity. Several relationships were verified and the matching between the measured results with the calculated ones was confirmed.

Keywords: Polymer composites; Rubbers; PETN; Sensitivity; Detonation

1. INTRODUCTION

Polymers have been extensively employed in the explosives field to tolerate their mechanical properties accompanied by their physical characteristics and also to expand their performance[1-3]. In 1952, 1,3,5-trinitro-1,3,5-triazinane (RDX) has been mixed with polystyrene and dioctyl phthalate fabricating the first known plastic bonded explosive (PBX)[4]. Afterward, many researches have been published to investigate the influence of polymeric matrices of the explosive powder[5]. Polymers showed a significant ability to mitigate the shocks and thus decreasing the accidental explosions as in the case of insensitive munitions (IMs)[6]. Also, PBXs showed high flexibility during various processes such as safe pressing into various three-dimensional shapes and casting at room temperatures[7].

One of the most serious challenges that affect the usage of the known modern explosives is their high sensitivity to different stimuli[8,9] which makes them unsafe to be used in their pure state. Therefore, many efforts have been attained to investigate the ability of polymers to increase the safety manipulations of these modern explosives[10-12]. Interestingly, polymers showed a significant influence on the sensitivity of explosives without an effective decrease in their performance[13,14]. Polyurethanes (PUs) are one of the most frequently used synthetic polymer materials in civilian applications[15]. It is commonly synthesised through the reaction between hydroxyl-terminated polybutadiene (HTPB) with an isocyanate agent[16]. Polyurethane plays an important role in the rocket propellant industry as well as binding the explosive crystals[17,18]. PU not only binds all the solid ingredients in a homogeneous polymeric matrix but also considered as the main source from which the whole propellant system acquires the necessary mechanical properties[18-20]. Similarly, Polydimethylsiloxane (PDMS) is a well-known viscoelastic silicon rubber with a wide range of applications including contact lenses, lubricants, and antifoaming agents in the food industry[21]. PDMS was found to possess interesting mechanical properties that enabled it to be used as a modern binder, instead of the conventional polyurethane-based ones, for either explosive or propellant formulations[22-24]. Also, Acrylonitrile butadiene rubber (NBR) is a commercial synthetic rubber widely used in the production of hoses, gloves, sealants, and footwear[25]. However, NBR has been used as a binder in the military field to improve the mechanical properties of explosives producing different types of sheet explosives and flexible blasting cartridges[26,27]. Moreover, the thermoplastic styrene-butadiene synthetic rubber (SBR) was effectively used in the field of energetic materials as adhesives in the improvised explosive devices and as phlegmatizers to decrease the initiation energy of hexanitrohexaazaisowurtzitane (CL-20)[28], while the

transparent thermoplastic polymethyl methacrylate (PMMA), which is commercially known as acrylic glass, has found its direction in the field of energetic systems as propellants holder and as confinements for the reinforced materials and emulsion explosives[29-31].

Pentaerythritol tetranitrate (PETN) is one of the most usable explosives that exhibit a relatively high power index of 167%. This enabled PETN to be a promising candidate in a diversity of applications[32]. However, decreasing its sensitivity is still a motivation for many types of research to extend its applications accompanied by high safety levels[33-35]. Recent studies by Elbeih[36], *et al.* have investigated the explosive properties of several plastic bonded explosives (PBXs) formulations based on cis-1,3,4,6-tetranitrooctahydroimidazo-[4,5-d]imidazole (BCHMX) in comparison with PETN. Interesting relationships have been achieved between the sensitivity and performance of PETN and several nitramines by Zeman[13,37], *et al.* PETN has shown obvious changes in its characteristics as a result of the interfacial interactions between its crystals and several materials which clarify its importance to perform PBXs[38-40]. Nevertheless, there is still scarcity in studying the optimum polymeric matrix to be used with PETN. Therefore, in this study, six diverse polymeric matrices have been fabricated. Firstly, the non-aqueous method was used to synthesize PBX samples based on polydimethylsiloxane (PDMS), Acrylonitrile butadiene rubber (NBR) softened by plasticizer, and Styrene-butadiene rubber (SBR) softened by oil. Moreover, a cast cured PBX based on hydroxyl-terminated polybutadiene (HTPB) was synthesised. Also, fluorel binder and poly-methyl methacrylate (PMMA), plasticised by dioctyl adipate (DOA), were used to coat PETN crystals using the slurry method. The heat of combustion, the velocity of the detonation, and the sensitivity of the studied formulations towards impact and friction stimuli were experimentally measured. Also, the explosive strength was investigated using a ballistic mortar. Detonation characteristics were calculated theoretically by employing the thermochemical computer software EXPLO 5.

2. EXPERIMENTAL WORKS
2.1 Materials
PETN, Acrylonitrile butadiene rubber (NBR), and Styrene-butadiene rubber (SBR) were obtained from Heliopolis Company for Chemical Industries, Egypt. Polydimethylsiloxane (PDMS) with a selective viscosity was obtained from a

siloprene trademark, Germany. Polymethyl Methacrylate (PMMA, Akrylon EGG 025; has a tensile strength of 70 MPa and 1.19 g/cm^3 density) was acquired from Quinn Plastics, Slovakia. Hydroxyl-terminated polybutadiene prepolymer (HTPB R-45M, a hydroxyl value: 0.84 m$_{eq}$/gram, obtained from ARCO Co.), and hexamethylene diisocyanate curing agent (HMDI, NCO equivalence value: 11.83 m$_{eq}$/gram, were obtained from Shandong YuchengYiao Technology Co. Ltd., China. Tris-1-(2-methylaziridinyl) phosphine oxide bonding agent (MAPO, acquired from Hangzhou Yuhao Chemical Technology Co. Ltd., China) together with dioctyl adipate plasticizer (DOA, attained from Island Pyrochemical Industries, China) were utilised without any further modifications. Fluoroelastomer Dyneon FT 2481 acquired from 3M Company, (fluorine content: 68.6%, density: 1.86 g cm^{-3}, and Mooney viscosity: 75) was utilised as a binder.

2.2 The Particle Size Distribution of PETN
The particle sizes of PETN samples were determined using particle size distribution analyzer Partica LA-950 (produced by HORIBA). This analyzer utilizes the laser scattering technique which is based on recording the intensity and the light scattered angle. The particle sizes are calculated by the Mie-scattering theory (Laser diffraction). Typical particle size curves are presented in Fig. 1.

The mean particle size of PETN (grade I) was found to be 16 µm, while the diameter of cumulative (90%) was 20 µm. The small particle sizes are crucially required to fill the spaces between the large particles to increase the mass ratio of PETN in the polymeric matrix. PETN (grade II) has a mean particle size of 201 µm and the diameter of cumulative (90%) is 426 µm. The importance of the large particle size of PETN is to keep on the mechanical properties of the plastic explosives and to keep the homogeneity between explosive crystals and the polymeric matrix. PETN used for the preparation of all the plastic explosives types were mixture of PETN (grade I) and PETN grade (II) with a mass ratio of 1:2.

2.3 Preparation of Polymer Bonded PETN
2.3.1 Non-aqueous Method
The PBX samples based on the non-aqueous technique were prepared by mixing PETN crystals with a binder matrix in a mass ratio of (15% PETN: 85% Binder). A computerising mixer plastograph, shown in Fig. 2(a), was utilised for the production of three polymer-bonded explosives (PBXs) based
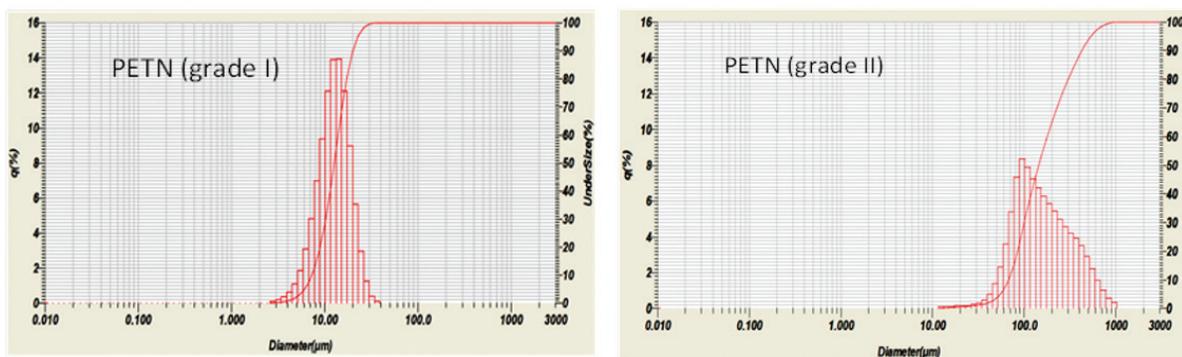

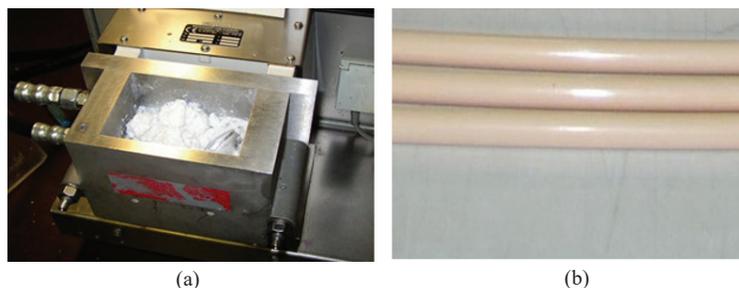**Figure 1. PETN grades Particle size distribution.**

Figure 2. **(a) Mixing of PETN with thermoplastic matrix in plastograph and (b) photo of the extruded sample.**

on the non-aqueous method (PETN-SBR, PETN-PDMS, and PETN-NBR). PETN-PDMS plastic explosive was successfully obtained by mixing of PETN explosive with PDMS polymeric matrix for 60 min using the plastograph. The temperature was kept constant at room temperature during the mixing process. However, PETN-SBR and PETN-NBR were produced by mixing PETN crystals with either NBR plasticised by dibutyl phthalate or with SBR plasticised by oil at 70°C for 60 min. The samples were extruded in the form of cylinders as shown in Fig. 2(b)

2.3.2 Slurry Method

Herein, the PMMA binder system (ratio of 1 part PMMA to 3 parts DOA) was dissolved in methyl ethyl ketone (MEK) and then added to the PETN which was slurried in water. In this technique, 91 wt.% of PETN crystals were mixed with 9 wt.% of binder in order to synthesize PETN-PMMA. This desensitisation process occurred with vigorous stirring at 500–600 rpm. The stirring continues until the solvent elimination is achieved. At this stage, full coverage of the surface of the explosive crystals with a polymeric layer was observed. The same procedure was applied to prepare PETN-FL using Fluorel binder instead of PMMA binder system. The preparation setup as well as the obtained beads of PBXs and the pressed sample are shown in Fig. 3.

2.3.3 Cast Cured Method

For the synthesis of the PETN-HTPB cast formulation, HTPB prepolymer, MAPO bonding agent, and DOA plasticizer were mixed for 20 min at a constant temperature of 40 °C in a 4L upright mixer, acquired from Sigma Company.

Afterward, the mixture was introduced to a 20 min vacuum mixing to get rid of any trapped air molecules. Once air bubbles removal was achieved, PETN was gradually added in three equal portions over 30 min. Then, HMDI as a curing agent was added to the mixture at 55 °C, and stirring continues for another 30 min. The obtained mixture was then cast at 60 °C and left for seven days in a vacuum medium until curing occurs. Finally, the binder system based on the HTPB binder was successfully obtained with an NCO/OH ratio of 1.3. The cast cured samples contain 85 wt% PETN and 15 wt% polymeric matrix as shown in Fig. 4.

2.4 Measurement of Combustion Heat

Herein, a constant-volume bomb calorimeter, model MS10A, was used to measure the combustion heat of the entire explosive formulations. This experiment takes place in a bomb containing an excess of oxygen to ensure the complete combustion of the elements[41]. Table 1 summarizes the results of the combustion heat.
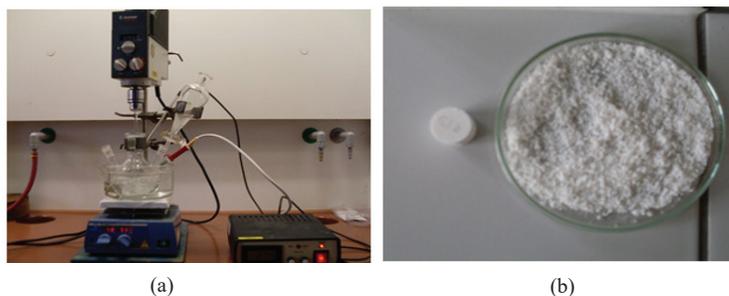

Figure 3. **(a) The setup for preparation of PBX by slurry technique, (b) PBX beads prepared.**

Figure 4. **(a) The melt cast sample production, (b) The cast cured sample.**

Table 1. Results of the explosive properties of the tested explosive formulations

Formulation	Combustion heat (J g⁻¹)	Impact energy (J)	Friction force (N)	Loading density (g cm⁻³)	VOD (*measured*) (m s⁻¹)	Strength of explosive % TNT
PETN	8182	2.9	44	1.70	8400	-
PETN-NBR	11942	14.9	213	1.53	7522	134.2
PETN-PDMS	12284	22.3	187	1.54	7563	136.8
PETN-PMMA	11384	12.7	167	1.56	7618	137.1
PETN-SBR	12943	16.5	226	1.52	7490	133.1
PETN-HTPB	13227	12.4	276	1.50	7432	132.4
PETN-FL	8464	6.3	238	1.68	7958	138.7

2.5 Measurement of the Sensitivity to Different Stimuli

The BAM impact sensitivity instrument was used to predict the impact energy needed to initiate the investigated explosive formulations. In this experiment, different drop weights were used to hit the explosive samples from variable distances. The friction sensitivity of the investigated explosives samples was experimentally measured by employing the BAM Friction-Test Instrumentation[41]. In this experiment, 0.01 g of explosive sample is placed on a porcelain panel. A loading arm is used to apply diverse loads between the porcelain panel and the pistil. This load affects directly the explosive sample. This may lead to the observation of sound, smoke, or igniting smell. The probit analysis method was used to determine the sensitivity of the samples prepared[42]. Table 1 gives the initiation information for the investigated explosive formulations at which 50% successful initiations were achieved.

2.6 Measurement of the Detonation Velocity

KONTINITRO EXPLOMET-FO-2000 Device was used to measure the detonating velocity (VoD) of the samples prepared. Herein, the initiation process was performed with the aid of the standard detonator no. 8. The samples tested were in the form of cylinders with a diameter of 21 mm and a 200 mm length. Measurements were achieved through the existence of three optical sensors placed at different distances from the initiation spot. The average value among the three sensors could be counted as the detonating velocity of that sample as recorded in Table 1.

2.7 Measurement of Explosive Strength

The explosive strength of PBXs was obtained by the ballistic mortar experiment relatively to 2,4,6-trinitrotoluene TNT[41]. A weighted sample of 10 g was inserted in the mortar. A safety fuse was attached to the back of the projectile as an igniting tool to start the initiation of the detonator. Under the action of the explosion, the pendulum is moving and the angle is recorded. The result was compared with the calibration curve of TNT. The explosive strength was reported relatively to TNT (as a percentage of TNT). Three samples were tested for each composition and the mean values were taken as the representative value for each sample as represented in Table 1.

2.8 Determination of the Detonation Characteristics

The EXPLO 5 thermochemical software was utilised to calculate the detonation characteristics (detonation velocity, pressure, and the heat of detonation) for each explosive formulation[43]. These detonation characteristics were obtained by applying the Becker-Kistiakowsky-Wilson equation of state (BKW EOS). The obtained detonating characteristics results are reported in Table 2.

3. RESULTS AND DISCUSSION

The safety during the explosives handling depends on the sensitivity of explosives to different stimuli. Friction and impact sensitivities of the formulations prepared are presented in Fig. 5. These results showed a major decrease in the sensitivity of the fabricated PBXs in comparison to the pure PETN. It confirms the dramatic influence of the binder systems on the sensitivity of PETN. Interestingly, PETN-PDMS was found to possess the lowest sensitivity to impact, while PETN-HTPB has the lowest friction sensitivity of the different compositions. To optimize these results, Fig. 5 has been divided into four parts; the first part includes PETN as a sensitive explosive and PETN-PMMA which has friction sensitivity lower than 175 N and also impact sensitivity lower than 14 J.

The second group (includes PETN-HTPB and PETN-FL) is composed of the PBX formulations which have relatively high impact sensitivity and low friction sensitivity. In this group, PBX formulations based on fluorinated binders are
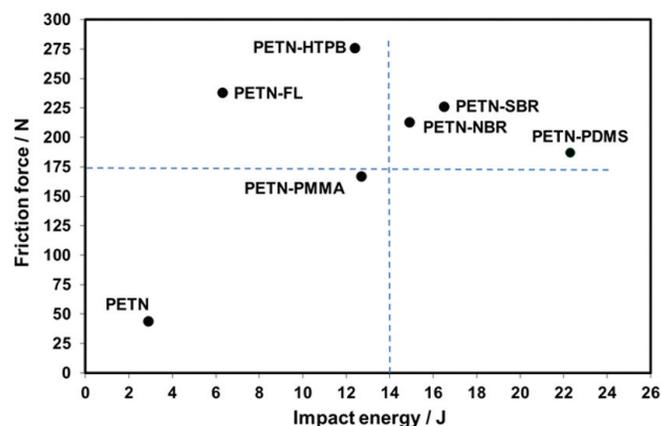

Figure 5. Results of the friction force in comparison with the impact energy of the different formulations.

Table 2. Calculated detonation properties by EXPLO 5 code

Explosive formulation	Density (g cm^{-3})	EXPLO5			
		Detonation velocity (m s^{-1})	D_{calc}-D_{exp} D_{exp}/100 (%)	Detonation pressure (GPa)	Detonation heat (J·g^{-1})
PETN	1.70	8350	-0.6	28.62	6258
PETN-NBR	1.53	7354	-2.2	20.93	5725
PETN-PDMS	1.54	7391	-2.3	21.04	5654
PETN-PMMA	1.56	7433	-2.4	21.45	5690
PETN-SBR	1.52	7326	-2.2	20.03	5581
PETN-HTPB	1.50	7217	-2.9	18.8	5472
PETN-FL	1.68	7736	-2.8	24.71	5869

composed of beads coated by a high-density polymer. This polymer acts as an inhibitor and consequently decreases the sensitivity of PBX against the sliding action. Besides, the cast cured PETN-HTPB has the lowest friction sensitivity due to the influence of the thermoset binder system which successfully isolates the crystals from the friction force. The third group contains plastic explosives prepared by a non-aqueous process, where these samples include oil or plasticizers which optimize their sensitivities to the different stimuli.

A well-known foundation that proved the dependency of the detonation velocity on the loading density of the explosive could be deduced from Fig. 6. It is obvious that all the PBXs prepared have a perfect linear relationship. The pure PETN has high detonation velocity and is outside this relation due to the absence of any inert material. PETN-FL sample has the highest detonation velocity of all the PBXs studied due to the high density of the fluorinated binder (fluorel), which affects the loading density of its PBX formulation. PETN-FL has a 9 wt.% of Fluorel which decreases the detonation velocity of PETN, while its density is still close to that of the pure PETN explosive. At the same time, it is obvious that PETN-PMMA has a high detonation velocity but with an undesirable high sensitivity, as discussed in Figure 5. This probably affects the safety of its applications in the energetic materials field. Interestingly, PETN-PDMS was shown as an interesting sample which has the highest detonation velocity in comparison to the rest of the studied samples. On the other hand, PETN-HTPB formulation has the lowest detonation velocity amongst all the compositions.
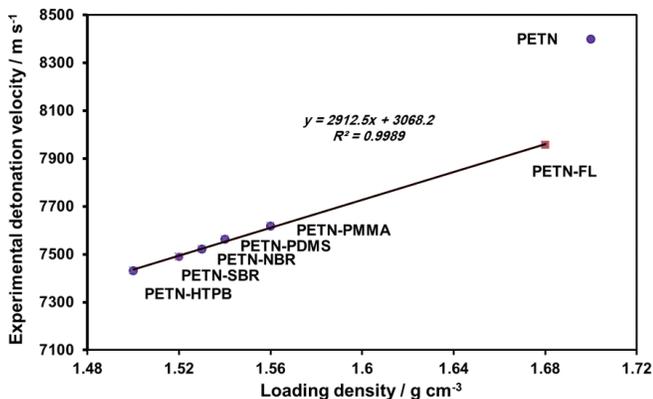

Figure 6. Relationship between the density and detonation velocities of PETN based PBXs.

As discussed in several publications[5], an interesting inverse relationship between the heat of combustion, measured in this study, and the heat of detonation, calculated by EXPLO 5, is presented in Fig. 7. The results proved that the polymeric matrices increase the combustion heat of the pure PETN explosive, while the heat of detonation decreases as a result of decreasing the weight percentage of the explosive in the PBX samples. Also, a linear relationship was observed between all the studied samples except the PETN-FL. This might be due to the high percentage of the explosive filler in PETN-FL (91 wt.% of PETN), while the other samples have only (85 wt.% of PETN). Also, PETN-FL is a binder without any oil
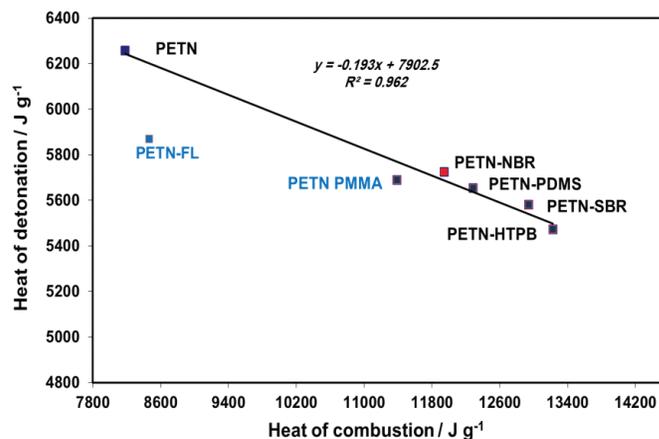

Figure 7. Inverse relationship between the combustion heat and the detonation heat of PETN based PBXs.

or plasticizers, so its combustion is low when compared with the other polymeric matrices. PETN-HTPB has the highest combustion heat in comparison to the other compositions. This could be attributed to the high content of carbon in the HTPB structure, thus its efficiency to act as a fuel in the composite solid rocket propellants increases. Furthermore, PETN-NBR has a high heat of detonation when compared with the other samples (except PETN-FL). This result is compatible with the application of nitrile rubber in the military plastic explosive (Semtex 10) which has several applications[36].

Concerning the detonation physics, the detonation pressure could be demonstrated by the square of the detonation velocity multiplied by its density. In this study, the compatibility between the experimental measurements and the calculated results obtained by EXPLO5 were checked as presented in Fig. 8. A clear linear relationship with high accuracy was realised between the experimental ρD^2 and the calculated detonation pressure. This figure confirms the accuracy of the EXPLO5 software in the determination of the detonation characteristics, where the maximum difference between the experimental and the calculated detonation velocity was less than -3 %. Also, it is obvious that the detonation pressure of PETN-FL is the highest of all the studied PBXs, while PETN-NBR and PETN-PDMS have nearly the same detonation pressure.
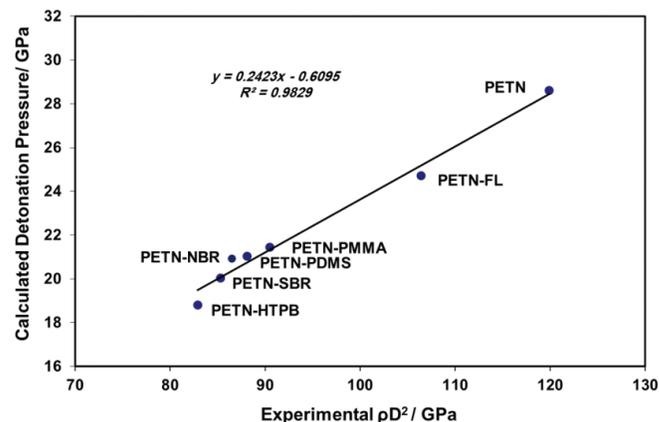

Figure 8. Relationship between the experimental ρD^2 and the pressure of detonation obtained by software.

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The values of the relative explosive strength of PETN-based PBXs are presented in Table 2, while the dependence of these values on the detonation pressure (represented by the experimental value ρD^2) is clarified in Fig. 9. An approximately linear relationship was observed between the relative explosive strength and the experimental ρD^2 of all the studied PBXs except PETN-FL. The high density of fluorel binder and the high percentage of PETN in this composition caused an obvious increase in the detonation velocity with a slight increase in its explosive strength. PETN-PMMA has high explosive strength but is still highly sensitive as mentioned previously. PETN-PDMS has high explosive strength when compared with PETN-NBR formulation. This relationship confirms the idea that the explosive strength depends on the detonation pressure of the explosive and simultaneously depends on the detonation velocity. Besides, it was discussed by Elbeih[44], *et al.* that the explosive strength represents the power of the explosive and it depends on both the volume of gases and the heat of detonation. In this paper, the effect of the detonation pressure is quite clear on the explosive strength of the explosives, while the relationship is approximately linear due to the absence of an important parameter (volume of gases) which might improve the linearity of this relationship. It means that the explosive strength depends on the detonation pressure but also the heat of detonation and the volume of gases are important parameters and should be considered.
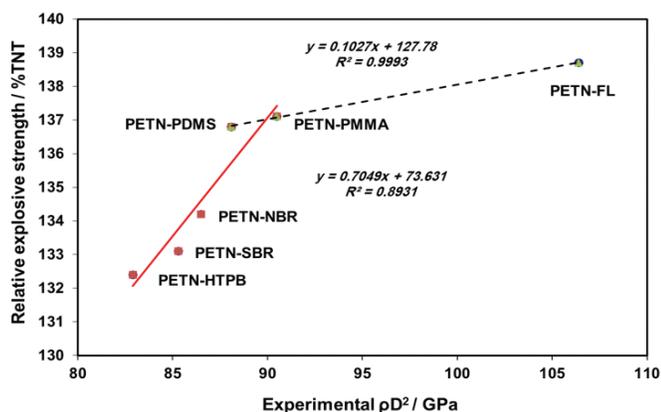

Figure 9. Relationship between the experimental ρD^2 and the relative explosive strength.

Licht[45] has stated the general finding that increasing of the explosive performance is normally followed by increasing of its sensitivity. In this study, a comparison between the relative explosive strength of the studied PBXs and their sensitivity to friction impulse is presented in Fig. 10. The increase in the explosive strength of the studied PBXs was found to be accompanied by an increase in their friction sensitivity; PETN-HTPB has the lowest friction sensitivity with low explosive strength and PETN-PMMA has the highest sensitivity and explosive strength. As mentioned previously, PETN-FL does not correlate with the rest of the samples due to the high density of the inert Fluorel binder which decreases the friction sensitivity of PETN while its impact sensitivity is still high. In Fig. 10, despite the absence of a linear relationship, the direct proportional connection between the performance and the sensitivity is observed.
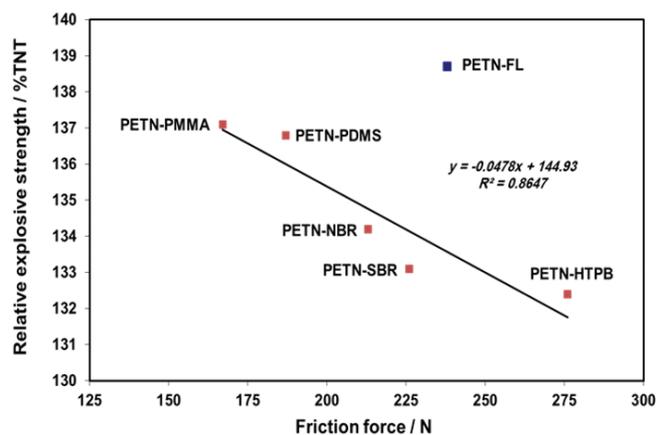

Figure 10. Relationship between friction sensitivity and relative explosive strength for the studied formulations.

4. CONCLUSIONS

PETN-PDMS was found to possess the lowest impact sensitivity, while PETN-HTPB has the lowest friction sensitivity among all the PBX compositions prepared. The Fluorel high-density polymer acts as an inhibitor and thus decreases the sliding influence (decreases the friction sensitivity of PETN-FL) and plays an important role to improve the detonation velocity. The presence of a polyurethane matrix in PETN-HTPB is responsible for the highest heat of combustion and the low detonation heat value. On the other hand, the high percentage of the energetic filler in PETN-FL formulation causes a relatively low heat of combustion and high detonation parameters. The relationship between the detonation pressure (demonstrated by the experimental ρD^2) of the prepared compositions and the relative explosive strength was confirmed. The types of polymer used have a significant effect on the PBXs performance, where the pressed formulations were found to have higher strength in comparison to the PBX formulations based on either the non-aqueous process or cast-cured type. However, PETN-PDMS formulation was found to have a relatively lower sensitivity, higher detonation velocity, and higher explosive strength. PDMS seems to be the optimum polymer for obtaining PBXs based on PETN. This study results confirm the matching between the experimentally measured results and the theoretically calculated ones.

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