



Cent. Eur. J. Energ. Mater. 2021, 18(4): 545-569; DOI 10.22211/cejem/145427

Article is available in PDF-format, in colour, at:

<https://ipo.lukasiewicz.gov.pl/wydawnictwa/cejem-woluminy/vol-18-nr-4/>



Article is available under the Creative Commons Attribution-Noncommercial-NoDerivs 3.0 license CC BY-NC-ND 3.0.

Research paper

A New Testing Method for the Mechanical Properties of Elastic Explosive Compositions

Dorota Powal^{1,*}, Marcin Nita¹, Andrzej Orzechowski¹,
Andrzej Maranda²

¹ *Military Institute of Armament Technology, 7 Prymasa Stefana Wyszyńskiego Street, 05-220 Zielonka, Poland*

² *Łukasiewicz Research Network – Institute of Industrial Organic Chemistry, 6 Annopol Street, 03-236 Warsaw, Poland*

* *E-mail: powalad@witu.mil.pl*

Abstract: In recent years, Polymer Bonded Explosives (PBX) have been used in a wide variety of military applications. Many different kinds of polymers are used for such explosive formulations. This component determines both the preparation method and the properties of a PBX. In this paper, results are presented of studies on the preparation and testing of explosive compositions based on pentaerythritol tetranitrate (PETN, penthrite) and silicone rubber. These studies were undertaken to obtain elastic formulations of PBXs. Therefore, the possibility of applying a silicone rubber as a component of the composition was checked. In the first stage of this study, several compositions were prepared in order to choose the optimal mixture ratio with respect to cohesion of the explosive. For this purpose, a new method using the Brookfield Texture Analyser was developed. Subsequently, compatibility tests using thermal analysis methods were carried out. The best of composition was subjected to tests for determining its physicochemical and explosive characteristics.

Keywords: PBX, silicone rubber, elastic explosives, compression testing

1 Introduction

Currently, explosives containing polymers are widely used, both in military and civil applications. The variety of polymers commercially available makes it possible to form explosives with different properties for many applications. This encourages further research on their modifications, even though Polymer Bonded Explosive (PBX) explosives were developed by James and Smith almost fifty years ago [1]. Experiments concerning selected parameters of PBXs are performed and the results obtained indicate the areas for their applications [2-6].

A PBX can be in the form of high-density pressed charge, as well as cast, plastic, or elastic charges, where the crystalline explosive content is lower. The development of each of the above-mentioned formulations requires a series of tests to determine the properties of the explosive composition itself, as well as the explosive charge made with it. This is an essential element in the design each new product.

The present paper describes the results of research that was carried out in the development of an elastic explosive for special purposes. It was assumed that the final product should be elastic to such an extent that would not exceed its elastic limit during manual operation in its use. Regarding the functional parameters of the explosive, it was assumed that the critical diameter should be in the range of 4-8 mm and the value of the detonation velocity should exceed 6000 m/s. In addition to the development of PBX compositions with set parameters, the essence of the research was to develop a method that would allow optimization of the technological process of producing explosive compositions with elastic properties.

Based on the available sources, a PBX composition containing silicone resin and PETN was selected for investigation. The literature provides information on explosive compositions containing various types of silicone resins [7-9]. The authors of these studies mainly present the explosive properties and sensitivity to external stimuli [10-12], or the basic physicochemical properties of these types of compositions [13-15]. This type of information is important and determines the basis for the development of new explosive compositions, however they often do not contain any data regarding the processes for the preparation of such compositions. The present work focused on determining the rheological properties of the developed composition to determine the optimal mixture ratio of the materials for the desired explosive parameters.

As part of the experimental work, compositions containing a mixture of PETN and silicone resin (EFx) were prepared. The main goal of the research was to select the optimal ratio of the PETN/EFx mixture. For this purpose, a new method of assessing the mechanical properties was used, using an

AMETEK Brookfield CT3 texture analyser. This is a device widely used in the food, cosmetics and pharmaceutical industries to assess the texture properties of solids and liquids [16]. It was decided to check the possibility of using the analyser to evaluate the mechanical properties of the prepared elastic explosive composition (PBX-Si). In the literature on the subject, there is no clear definition of texture. It should also be emphasized that this is not about the texture as understood as a feature of the surface of an object. This term can be defined as a set of physical properties of a solid or liquid resulting from its structure. This property of matter, which is difficult to define, has a specific set of features and is closely related to sensory tests. Texture analysis is based on only mass, length, and time measurements. These measurements provide the possibility to quantify and compare features such as brittleness, solidity, elasticity or stickiness, which are usually determined only subjectively by means of the senses. Based on the measurements, parameters such as hardness, cohesiveness, springiness or adhesiveness can be estimated.

For the purposes of the research, original methodology was developed empirically. The essence of the method was to determine the so-called texture profile (Texture Profile Analysis, TPA).

2 Experimental Procedures

2.1 Formulations

The first stage in the investigation involved the preparation of a mixture of crystalline PETN and a two-component silicone resin under the trade name Ecoflex. The ingredients were mixed in three different mass ratios of PETN/EFx 90/10, 80/20 and 70/30, respectively. The crystalline PETN, synthesized in MIAT laboratory, contained two particle size fractions, *viz.* 200-450 and 50-100 μm . Furthermore, two types of silicone resins were tested in the mixtures, *i.e.* Ecoflex-50 and Ecoflex-30. The resins differed significantly in three parameters as follows: hardness on the scale (Shore A), tensile strength and viscosity at 20 °C (Table 1).

Table 1. Properties of Ecoflex-50 and Ecoflex-30

Silicone resin	Hardness (Shore A)	Tensile strength [N/mm ²]	Viscosity [mPas]
Ecoflex-50	50	2.17	8000
Ecoflex-30	30	1.38	3000

The silicone manufacturer (Smooth-On, Inc) does not specify the exact structure of the polysiloxane. It is an addition silicone cross-linked with platinum compounds at room temperature. All mixtures were prepared manually by mixing all of the ingredients together. The cross-linking of the silicone rubber was at room-temperature from 3 to 16 h, for Ecoflex-50 and Ecoflex-30, respectively.

2.2 Methodology for measuring the mechanical properties of PBX-Si

For the strength tests, samples were prepared for six measurement series. Three series, one differing in the mass ratio of PETN/EFx with the Ecoflex-50 silicone, and another three series with the Ecoflex-30 silicone. In each series, five single samples were subjected to compression, of which the two extreme values were rejected and the arithmetic mean was calculated from the remaining three. A single sample was a cylinder with a diameter of 10 mm and a height of 10 mm. The samples were formed using a specially prepared multi-cavity mould. Figure 1 shows a view of typical samples made of the PBX-Si composition (also on a measuring table) and the elements of the mould used.

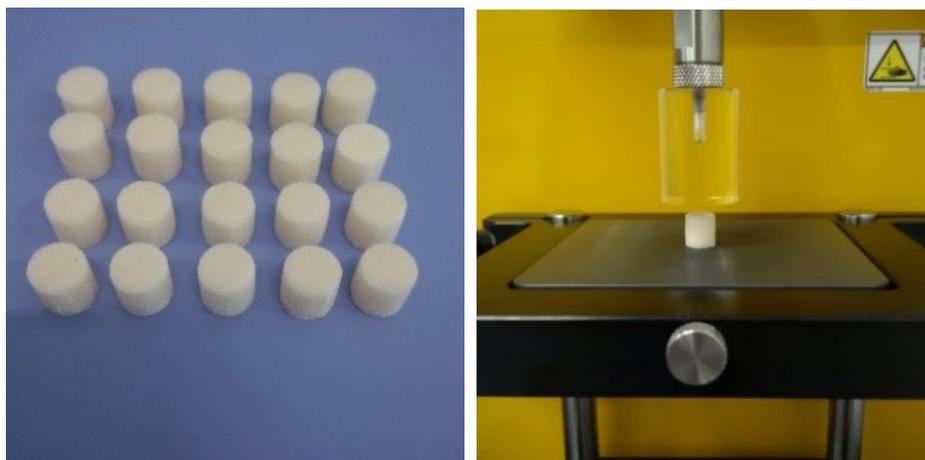


Figure 1. A view of the samples for TPA analysis and the mould used

The determination of the texture profile for the samples was carried out in two compression cycles of the same sample. The target point of a single measurement for all series was 20 and 40% deformation (change of the initial sample height by 20 and 40%, respectively). The following assumptions were made for establishing a target point based on the sensory test. A sample under service conditions will not deform more than 20%. Whereas a 40% deformation should be sufficient

to determine significant differences in the structure of individual compositions differing in chemical constitution. The parameters determining the course of the analysis are presented below. Table 2 contains a detailed list of the samples used in these measurements.

- target: 20% or 40% of deformation,
- trigger load: 0.1 N,
- compression speed: 0.50 mm/s,
- return speed: 0.50 mm/s,
- probe – cylinder, made of PMMA – diameter: 25.4 mm,
- temperature – 21 °C.

Table 2. List of sample compositions used in the measurements

Batch number	Target point of TPA		Mass ratio [g/g]	
			PETN/EFx-30	PETN/EFx-30
B1	20% Deformation	40% Deformation	70/30	70/30
B2			80/20	80/20
B3			90/10	90/10
			PETN/EFx-50	PETN/EFx-50
B4	20% Deformation	40% Deformation	70/30	70/30
B5			80/20	80/20
B6			90/10	90/10

In the first compression cycle, the measuring probe was positioned on the sample surface (pressure 0.1 N) at a place corresponding to its initial height. In the second measuring cycle, the probe position was determined based on the first cycle. The main goal was to determine the load-distance characteristics, based on which the following parameters were determined: hardness (maximum load), cohesiveness, springiness and others. Figures 2 and 3 show the method for the determination of the indicated parameters.

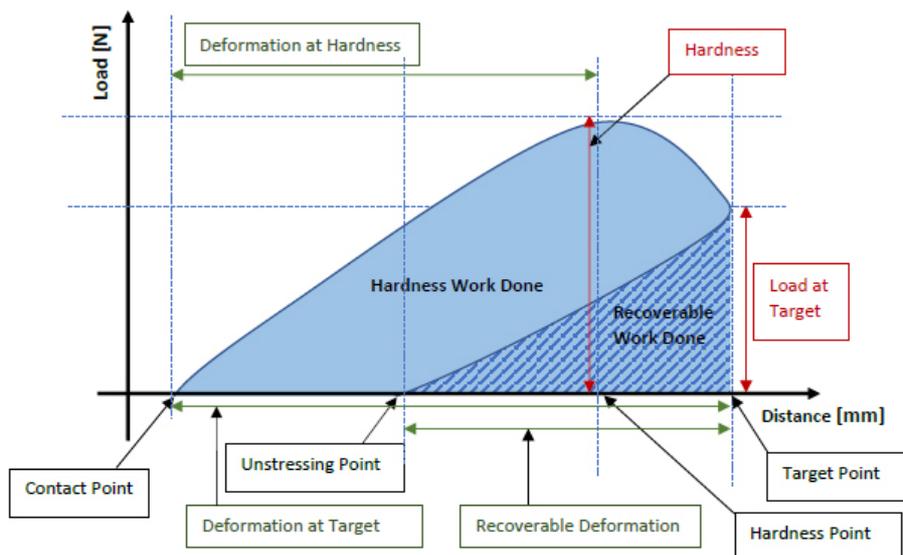


Figure 2. Parameters that can be determined based on the load-distance characteristics – 1st cycle of compression

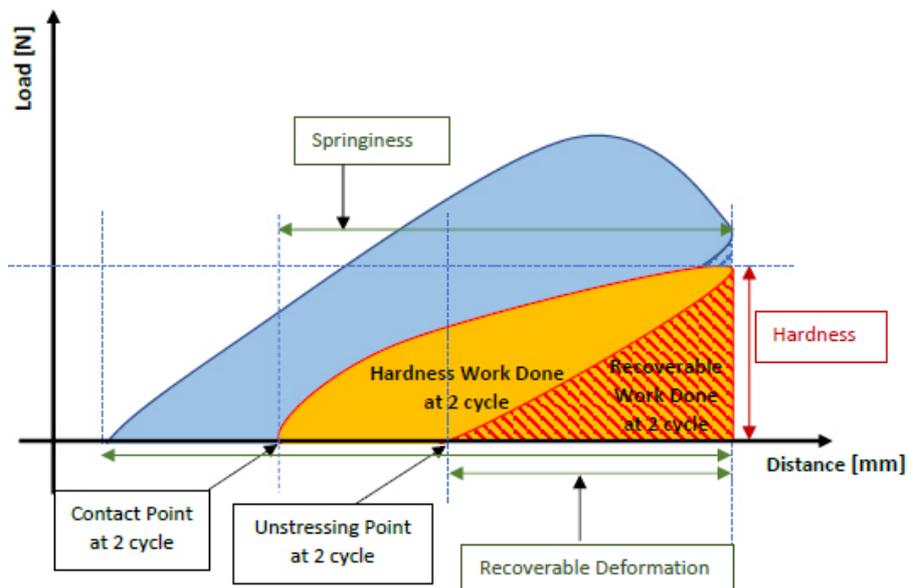


Figure 3. Parameters that can be determined based on the load-distance characteristics 2nd cycle of compression (TPA analysis)

2.3 Compatibility and thermal stability

The assessment of the compatibility of explosives is an important factor in the development of new explosive compositions. This parameter is significant, primarily for safety reasons. There are many publications in the literature concerning the compatibility and chemical stability testing of explosives and their mixtures [17-19]. These literature data clearly show that the proper assessment of stability and compatibility is not an easy process. The properties of the test substance, sample preparation, and selection of the test conditions are the factors that determine the final result [20]. In order to make a proper assessment, it is advisable to make the measurements by more than one test method. Publication [21] discusses in detail the terms of chemical and physical stability, mutual compatibility of the explosive components in mixtures, as well as references to the commonly used test methods.

In the present study, the DTA/TG analysis technique was used to determine the chemical compatibility of the components of the PETN/Ecoflex composition. The study was conducted according to two research procedures. The first was carried out according to the methodology for Test 3 (Procedure A) in STANAG 4147 [22]. The DTA/TG analyses were performed for crystalline PETN, EFX resin and the PETN/EFX mixture using an Hitachi STA 7200 analyser. Hermetically sealed aluminium vessels were used. Samples weighing about 1.5 mg were heated at 2 °C/min. The carrier gas was ultra-high purity nitrogen, which was fed at a rate of 50 mL/min. The analysis conditions for the individual components and the mixture were the same. In order to determine the compatibility, the weight loss of the PETN/EFX composition was assessed in relation to the sum of the weight losses exhibited by the individual components.

The second evaluation method was the procedure included in the standard PN-V-04011-21: 1998 [23]. According to this procedure, it is assumed that the tested explosive formulation is stable if the course of its decomposition curve coincides with the standard sample decomposition curve. Pure PETN with the same particle size distribution as in the tested PETN/Ecoflex mixture was used as a standard sample. DTA/TG tests were carried out in the temperature range covering the full decomposition of the sample, *i.e.* from 30 to 300 °C. Measurements were performed with the sample heating rate of 5 °C/min. in a nitrogen atmosphere at gas flow 100 mL/min. The tested samples, weighing about 5 mg, were placed in open aluminium vessels.

2.4 Explosive characteristics

2.4.1 Detonation velocity

The detonation velocity was measured using the ionization probes technique with the explosive confined in a PCV pipe. The tested charges were placed in the pipe having an internal diameter of 28.5 mm and walls 2.5 mm thick. The pipe was 285 mm long and had 4 holes drilled at 49.7 mm apart, perpendicular to the lateral surface and in line parallel to the axis of the charge. Ionization probes were placed into the holes in the pipe. For initiation of detonation of the charge, an RDX booster and electric blasting cap were used. The time of shock wave propagation was recorded on a nanosecond counter. The detonation velocity was calculated based on the propagation time measured between the probes.

2.4.2 Critical Diameter

The critical diameter of the PETN/EFx composition was measured using a procedure with a right circular cone [24]. The charge was prepared by filling the plastic cone, of 29 mm diameter at its base and 165 mm long, with the explosive. The mass of PETN/EFx composition of 80/20 wt.% ratio was 42 g. The charge was placed on a steel witness plate and initiated by an RDX booster and electric blasting cap. The critical diameter of the explosive was taken as the diameter of the cone at which the point of detonation failure is visible on the witness plate.

2.4.3 Detonation pressure

The detonation pressure of PETN/EFx was estimated based on the results of a Plate-Dent Test [25]. This test was carried out for a pressed charge of TNT and a pressed charge of PETN/EFx of 80/20 wt.% ratio. The characteristics of the charges are listed in Table 3. The trial involved detonating a cylindrical charge of each explosive on a thick steel plate and measuring the depth of the dent produced in the plate.

Table 3. Characteristics of the charges prepared for the Plate-Dent Test

Parameter	TNT charge	PETN/EFx charge
Mass [g]	39.9	39.9
Diameter [mm]	37.3	37.3
Height [mm]	23.2	25.7
Density [g/cm ³]	1.58	1.42

2.4.4 Sensitivity to Mechanical Stimuli

The sensitivity to impact of the tested composition was measured according to the European Standard PN-EN 13631-4: 2002 [26] using a drop hammer. According to the standard, the sensitivity to impact is the lowest impact energy that causes at least one reaction (crack, flame) in six trials. The measurements of sensitivity to friction were made according to European Standard PN-EN-13631-3:2004 [27] using a Joulus-Petri apparatus. According to this standard, the sensitivity to friction is the minimum pressure (in Newtons) of stamp, which causes a minimum of one positive result (explosive reaction) in six trials.

3 Results

3.1 Assessment of the mechanical properties of PBX-Si compositions

The results obtained in the TPA tests for the PETN/EFx compositions can be presented in the form of various relationships, each of which provides quantifiable data describing the properties of the tested composition. The load-distance curves were the basis for determining all parameters describing the mechanical properties of the samples. Figures 4-6 show examples of load change characteristics of samples with different EFx-30 silicone resin contents for assuming the compression of 20% of their initial height. For all of the samples, this type of relationship was recorded in two compression cycles.

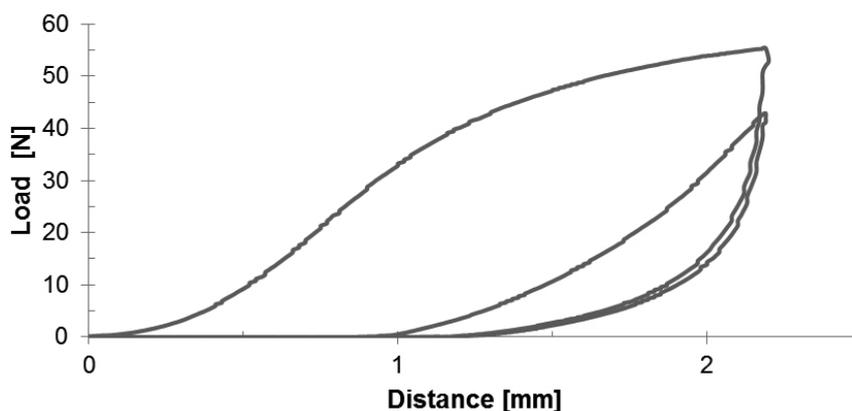


Figure 4. TPA analysis for PETN/EFx-30, 70/30 ratio at 20% of target deformation

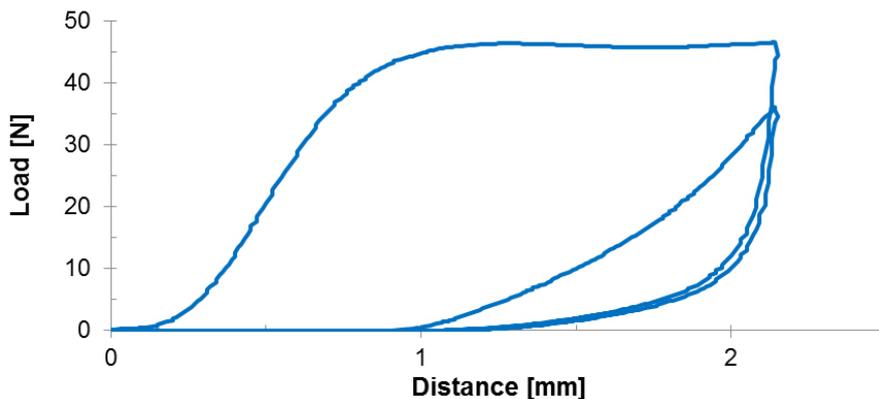


Figure 5. TPA analysis for PETN/EFx-30, 80/20 ratio at 20% of target deformation

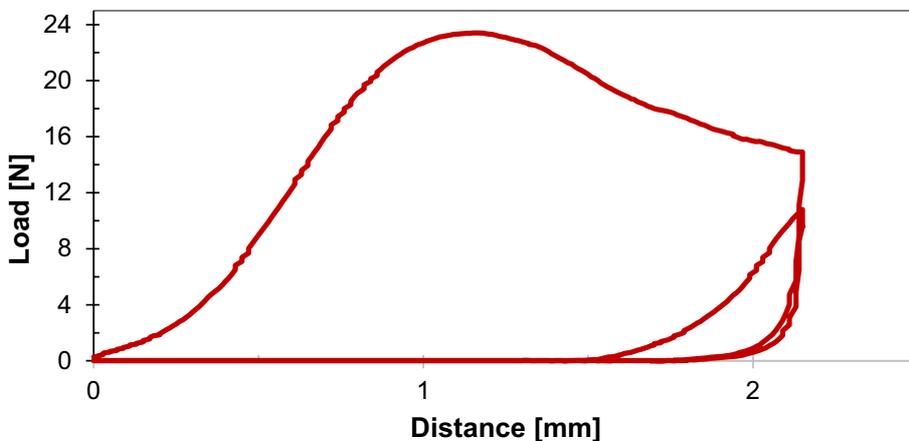


Figure 6. TPA analysis for PETN/EFx-30, 90/10 ratio at 20% of target deformation

General information about the mechanical properties of the tested samples is provided by the shape of the load-distance curves obtained. The same assumed change in sample height (20%) requires the application of a different load for each composition. In the samples containing 30% EFX, the load in the 1st cycle continuously increased until it reached a maximum at approx. 55 N. For the samples containing 20% of this resin, the load at about half of the cycle reached the maximum at approx. 45 N and remained on a more or less constant level until the end of the test. However, in the samples with the lowest polymer content, *i.e.*

10%, the maximum load was achieved in the middle of the cycle, at a level of about 20 N, and then began to decrease to a value of about 15 N. From the course of the curve in the last mentioned sample, it can be seen that the cohesion forces responsible for maintaining the continuity of the sample structure decreased due to the applied load.

On the basis of the characteristics obtained, calculations were made of parameters such as sample hardness, work necessary to overcome the internal bonding forces of the sample, elasticity and cohesiveness.

The graphs of Figures 7 and 8 show the maximum hardness of the samples and the load at the target point for 20% and 40% deformation. Comparative analysis of the hardness of the samples obtained from the 1st compression cycle shows that the composition containing 90% of the explosive, both in mixtures with EFX-30 and EFX-50 resin, has the lowest hardness. Moreover, with this explosive content, the differences between the maximum hardness and the hardness at the target point are the largest. This is particularly evident in the samples containing the EFX-30 resin. This relationship is a measurable confirmation of a significant decrease in the cohesive forces in the sample, which is evidenced by the shape of the load-distance curve characteristics, discussed earlier (Figure 5).

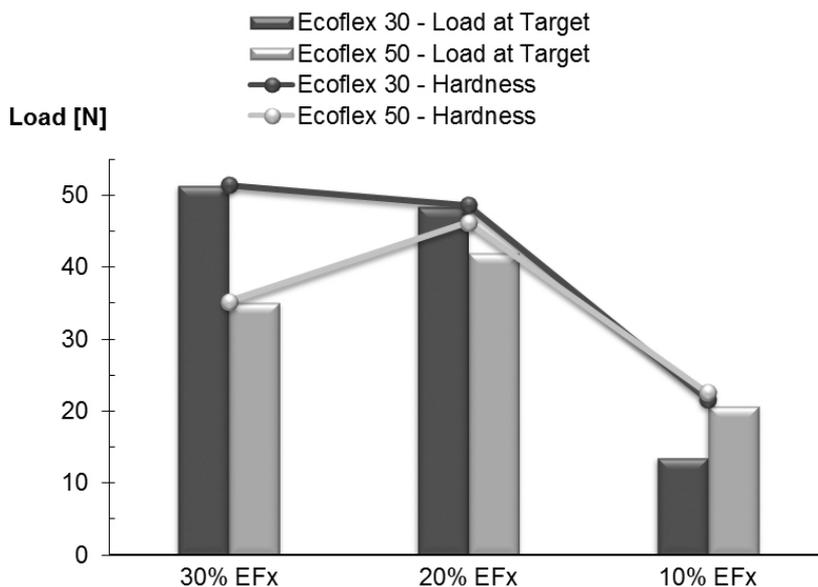


Figure 7. Load at target and hardness of the PETN/EFx compositions, (target point: 20% deformation)

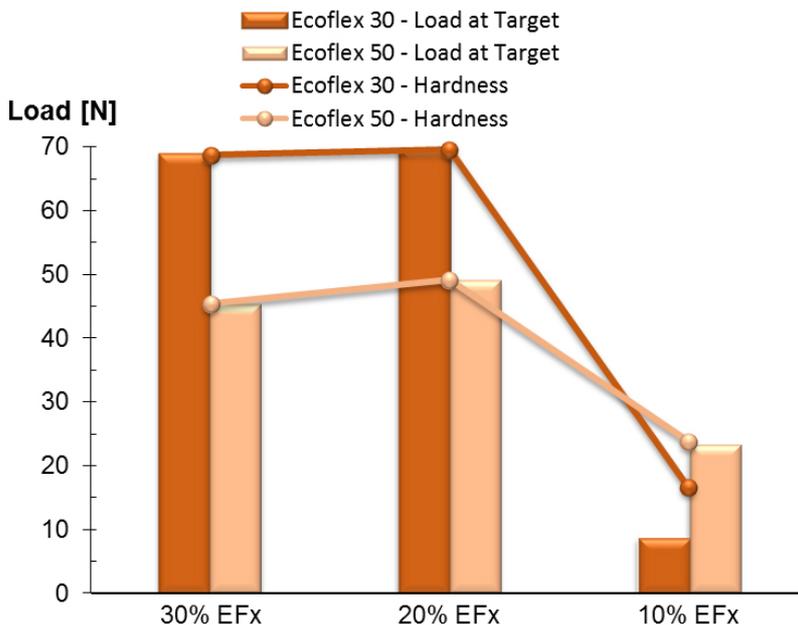


Figure 8. Load at target and hardness of the PETN/EFx compositions (target point: 40% deformation)

The maximum hardness may also be presented in relation to the surface of the sample subjected to compression. Figures 9 and 10 show the maximum stress obtained in the two measurement series, where the sample was compressed by 20 and 40% of its original height, respectively. The comparison shows that for the tested compositions, the use of only 20% compression may not be sufficient for a proper evaluation of the differences between the EFX-30 and EFX-50 polymer (small differences in the values for 10 and 20% of polymer content). The stress value obtained with 40% compression confirms that the strength of the sample containing 90% explosive is the lowest. Due to the above conclusion, in further considerations only the results where the target point was 40% of the change in the initial height of the samples are presented. Considering the differences between compositions containing 70 and 80% PETN with the EFX-30 and EFX-50 polymer, it can be concluded that the samples based on the EFX-30 resin have a higher compression resistance, which is confirmed by increases in hardness and maximum stress of 45% compared to samples based on EFX-50.

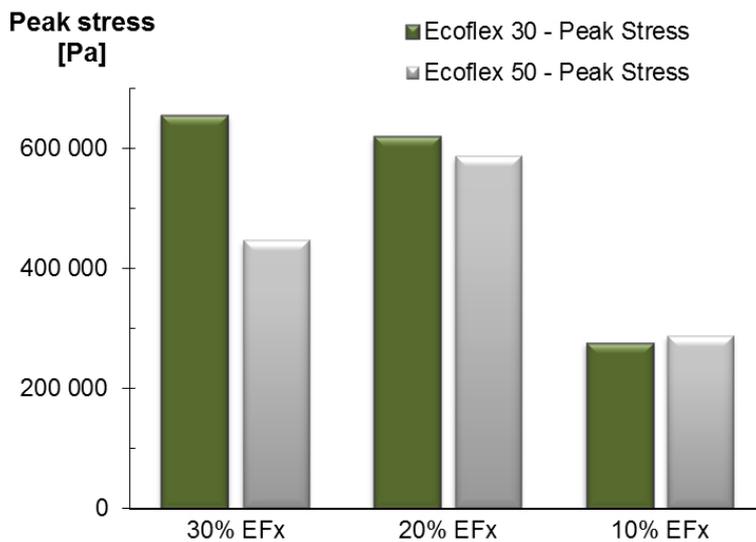


Figure 9. Peak stress of the PETN/EFx compositions (target point: 20% deformation)

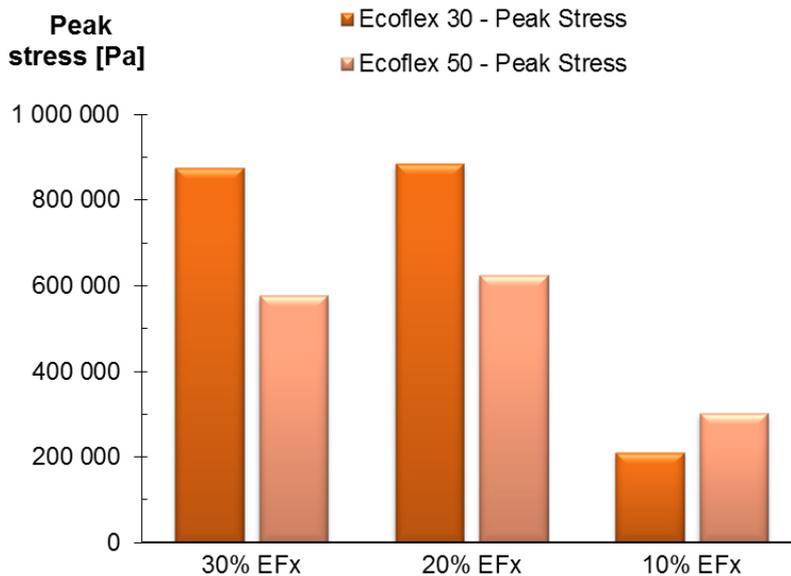


Figure 10. Peak stress of the PETN/EFx compositions (target point: 40% deformation)

Another parameter for assessing the suitability of the tested compositions is the amount of sample deformation at the point of its maximum hardness and the recoverable deformation, defined as the height recovered by the sample after the pressure force ceases. The deformation values at the point of maximum hardness (Figure 11), obtained for samples containing 70 and 80% PETN are the same for both polymers. On the other hand, in the sample containing 90% of the explosive, the values differ with the different polymers (EFx-30 and EFx-50). Moreover, for this composition, the deformation expressed as a percent of the initial sample height does not reach the value corresponding to the end point. In the sample containing EFx-30 this is 10% of the initial height, while in the sample with EFx-50 at maximum load, the deformation is 29%. This is another measurable confirmation that the sample containing 90% of explosive, regardless of the type of polymer used, is less durable to compression than the rest.

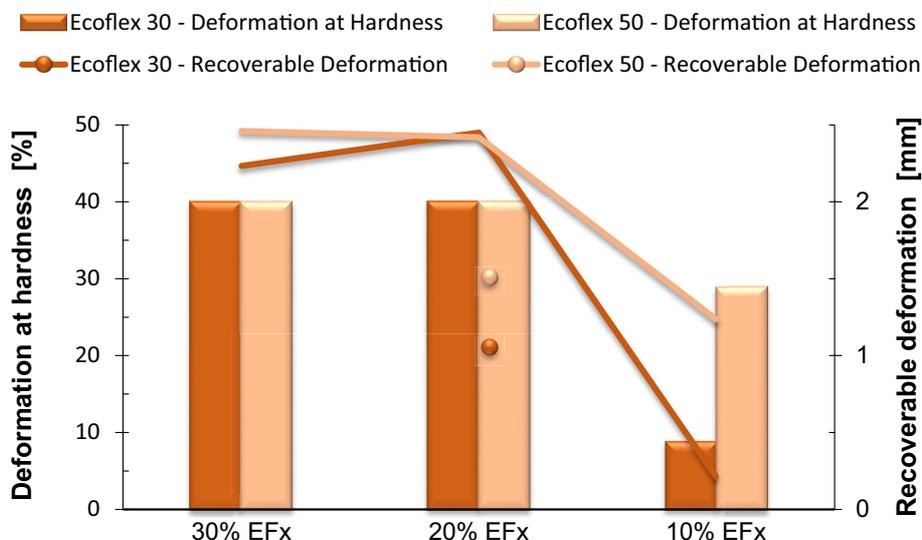


Figure 11 Deformation at maximum hardness and recoverable deformation of the PETN/EFx compositions

The height recovered by the sample after removing the load from its surface is the parameter that allows a significant differentiation only for the samples containing 90% PETN. The recoverable deformation for the highly-packed composition is lower for the EFx-30 polymer than for the composition containing 80% PETN. For the EFx-30 polymer this is approximately 90%, and for the EFx-50 polymer about 50%.

The recoverable deformation for the high-packed composition is lower for the EFX-30 polymer than for the composition containing 80% PETN. For the EFX-30 polymer this is approximately 90%, and for the EFX-50 polymer about 50%.

The analysis of the 1st compression cycle was completed by determining the work (energy) needed to overcome the internal bonding forces in the sample (Figure 12). The value of the work was determined by calculating the area under the load-distance curve. Regarding the series of samples with contents of 90, 80 and 70% PETN, the comparison of the calculated work values makes sense only for compositions containing 70% and 80% of explosive. This is due to the fact that the area is defined under the entire curve, *i.e.* from the contact point to the target point. Thus, for a sample containing 90% of the explosive, in which the maximum hardness was recorded at 10% of sample deformation, further compression of the sample begins to decrease rapidly, and the total area determined does not reflect the real work required to overcome the cohesion forces. Therefore, this result has no physical meaning.

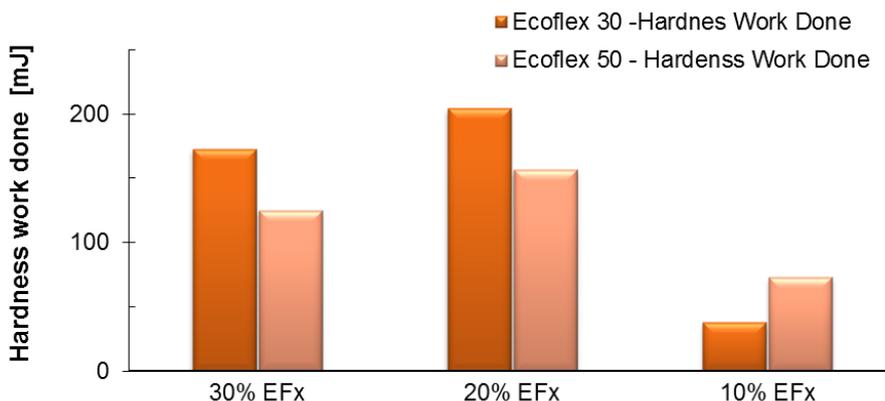


Figure 12. Hardness work done on the PETN/EFx compositions

The value of the work calculated in the 1st compression cycle is a parameter that allows differentiation between compositions containing 70 and 80% of explosive. Both of the samples based on EFX-30 and EFX-50 with 80% PETN in the composition are characterized by a higher value of the work necessary to overcome the cohesion forces in the sample compared to samples containing 70% PETN. A summary of the TPA compression first cycle allows two conclusions to be drawn. The first is elimination of the composition containing 90% explosive regardless of the type of polymer used. The second conclusion

leads to the statement, that the use of EFX-30 resin in the sample provides better mechanical properties.

In order to further optimize the sample composition, the results recorded in the second compression cycle of the TPA test were used. Figure 13 presents the results of calculating the sample cohesiveness depending on the PETN content in the explosive composition. The cohesiveness parameter was defined as the ratio of work in the 2nd compression cycle to work in the 1st cycle. The cohesiveness of the tested samples is influenced by two factors, one being the type of silicone resin used, while the other is the solid content (PETN). As can be seen from the Figure 13, the cohesiveness of samples containing the EFX-30 polymer depends to a lesser extent on the explosive content in the mixture. Only at 90% of PETN in the composition does the cohesion of the elastic mass decrease rapidly. Another relationship was found for samples containing the EFX-50 polymer, where at 80% PETN, the cohesiveness is reduced by about 25%.

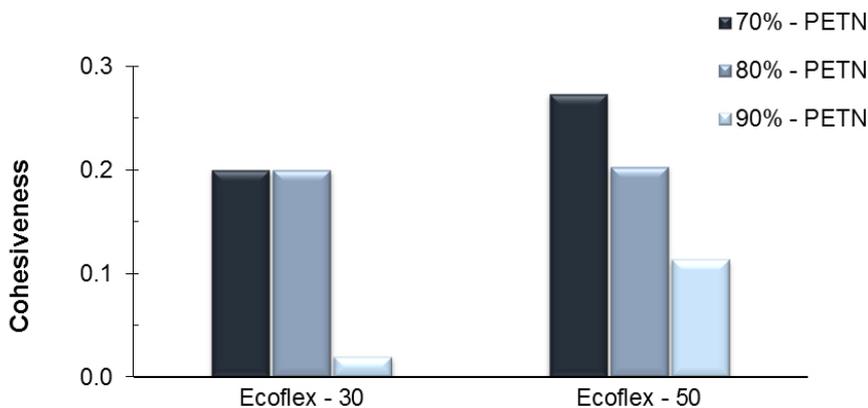


Figure 13. Cohesiveness of the different PETN/EFx compositions depending on the PETN content

On the basis of the results obtained in the second compression cycle, a further parameter was determined, *i.e.* springiness. This value determines the height that a sample recovers to between the first and second compression cycles. Figure 14 shows a comparison of the calculated springiness values depending on the PETN content in the explosive composition. The values obtained indicated higher springiness in samples based on the EFX-30 polymer. Moreover, it should be noted that the springiness of the samples with EFX-30 differ slightly from each other (about 4%) for compositions containing 70 and 80% of the explosive.

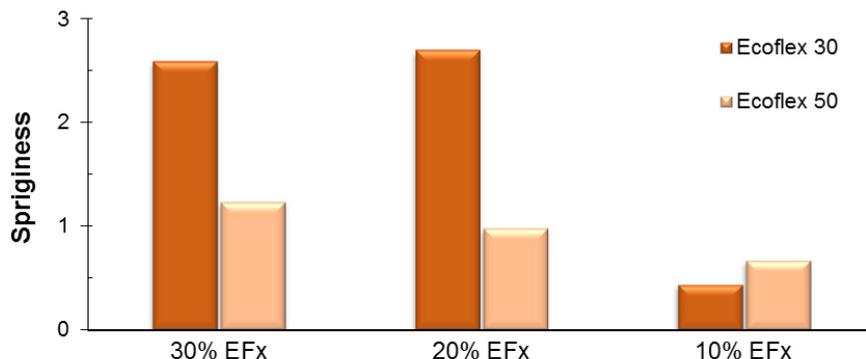


Figure 14. Springiness of the PETN/EFx compositions

A summary of the 2nd compression cycle of the TPA test allowed the optimal PETN/EFx composition to be finally selected. The tests were complemented by the determination of the springiness parameter of the tested samples. Due to the fact that the springiness parameter of the composition containing of 80% PETN does not differ from the value obtained for 70% PETN content, this is the composition that should be considered as the optimal in terms of all the mechanical properties.

3.2 Assessment of the stability and compatibility of PBX-Si compositions

The compatibility of the components of the PETN/EFx mixture was assessed based on the results of DTA/TG analysis performed in accordance with NATO standards STANAG 4147. The characteristic temperatures, which were used to calculate the mass losses, were determined based on the thermograms obtained. Single components were analysed, *i.e.* PETN and EFx-30, and two mixtures in the weight ratios of PETN/EFx 53/47 (according to STANAG recommendations) and 80/20. Figures 15-18 show the thermograms obtained from the TG/DTG analyses. The temperature value selected for compatibility assessment, *i.e.* 184 °C, corresponds to the maximum of the decomposition of the PETN/EFx 53/47 mixture.

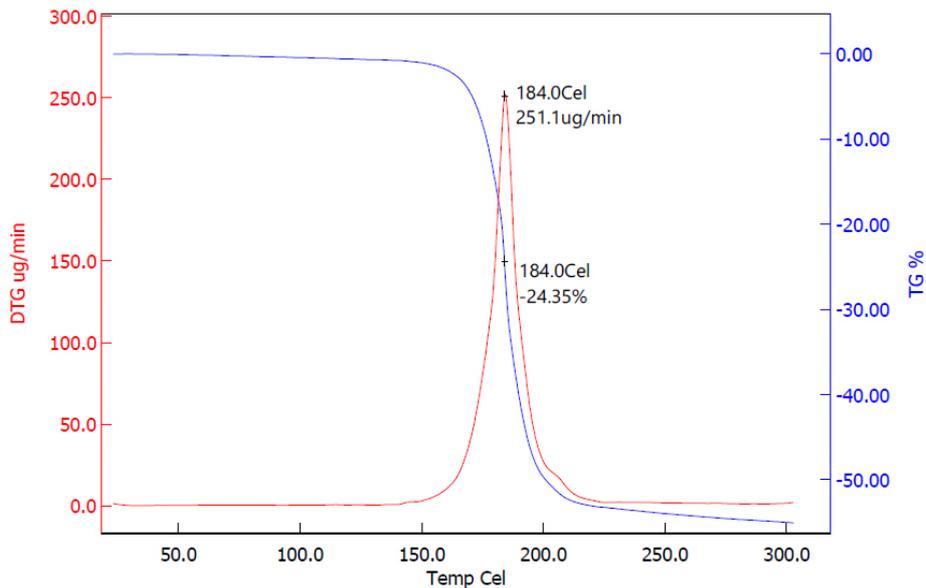


Figure 15. TG/DTG curves of the PETN/EFx-30 (53/47) composition

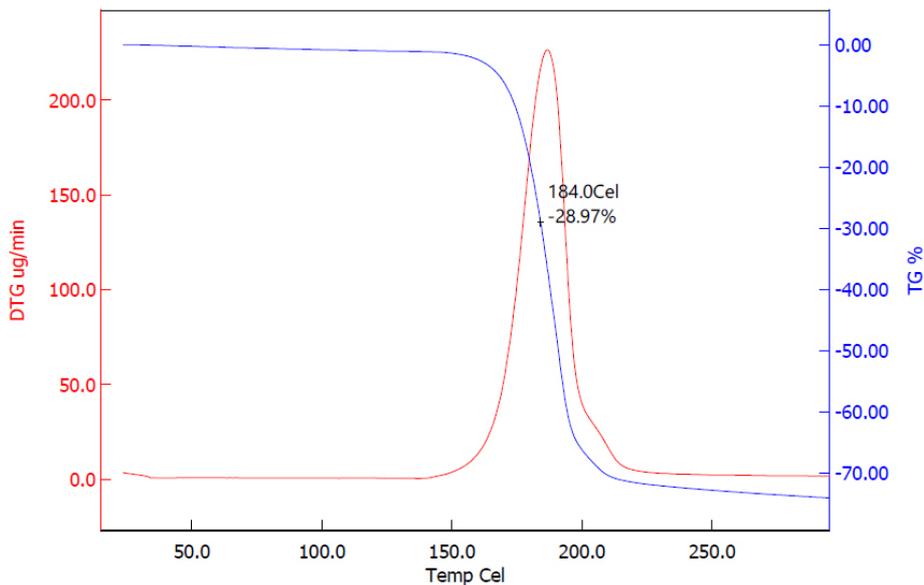


Figure 16. TG/DTG curves of the PETN/EFx-30 (80/20)

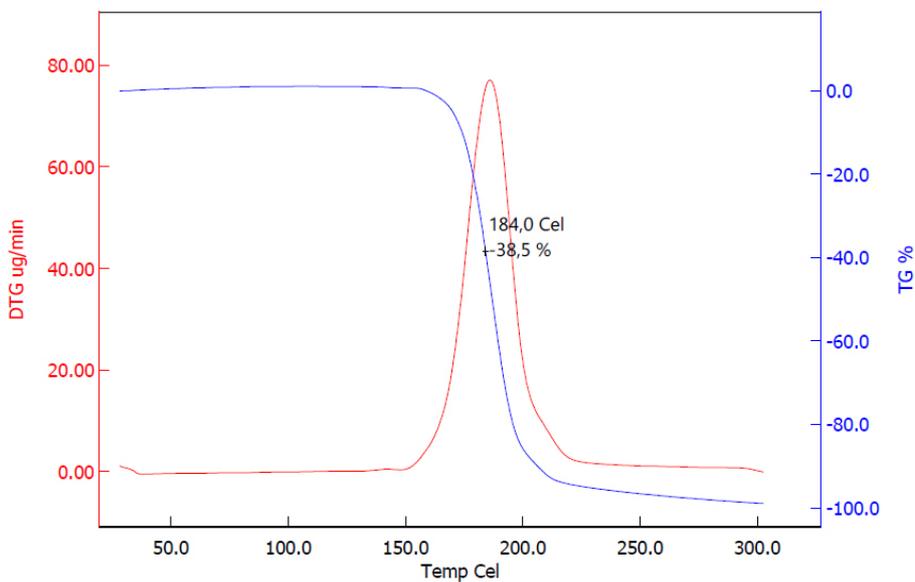


Figure 17. TG/DTG curves of the PETN

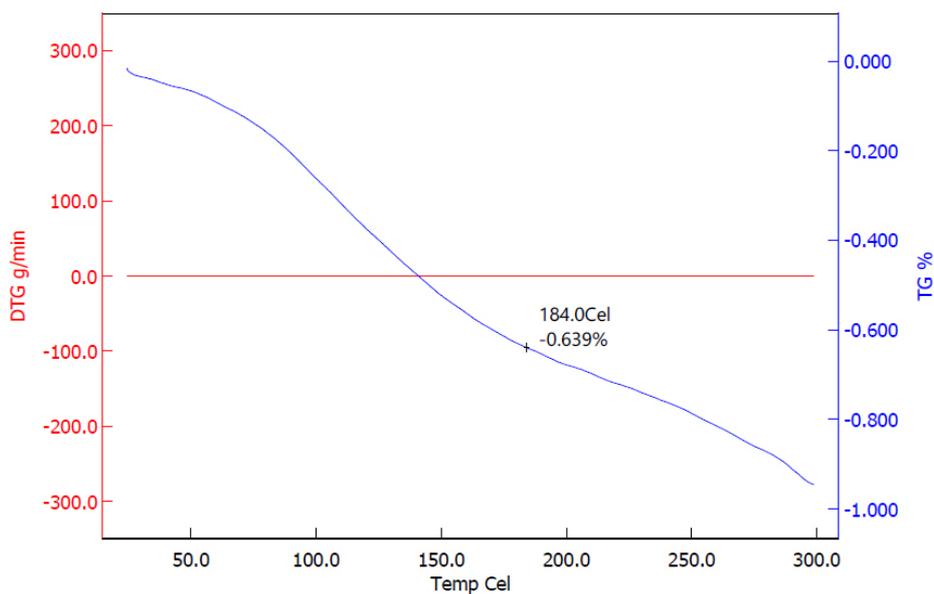


Figure 18. TG/DTG curves of the EFX-30

Table 4 summarizes the values of the weight losses of the mixtures and the individual components at the temperature of 184 °C obtained in the TG analysis. In accordance with the evaluation procedure contained in STANAG 4147, a mixture is considered as compatible if the difference between the registered weight loss of the mixture of the components and the calculated sum of the weight losses of the individual components does not exceed 4%. The values obtained in these tests allows confirmation that the components of the PETN/EFx composition are mutually compatible. The weight losses in the mixtures PETN/EFx 80/20 and 53/47 were 1.96 and 3.64%, respectively.

Table 4. Results of TG compatibility test (mass loss) at 184 °C for the PETN/EFx compositions

Sample	TG, Δm , observed mass loss [%] at 184 °C			
	Pure	Composition	Theoretical	Δm
PETN	38.5	–	–	–
EFx	0.639	–	–	–
PETN/EFx 80/20, g/g	–	28.97	30.93	1.96
PETN/EFx 53/47, g/g	–	24.35	20.71	3.64

DTA analysis was performed simultaneously with the TG analysis under the same conditions. Based on the thermograms, the values of the characteristic temperatures, such as the melting peak onset (T_{onset}), the melting peak maximum (T_m), and the decomposition peak maximum (T_d), were compared. The values obtained are summarized in Table 5.

Table 5. Characteristic temperatures of DTA/TG analysis for the PETN/EFx compositions

Sample	Melting		Decomposition
	T_{onset} [°C]	T_{max} [°C]	T_{max} [°C]
PETN	140.5	140.8	189.8
PETN/EFx 80/20, g/g	140.1	143.6	189.6
PETN/EFx 53/47, g/g	139.5	143.3	187.9

The characteristic temperature values obtained from this analysis confirmed the thermal stability of the PETN/EFx 80/20 composition. The beginning of the melting process of this composition occurred at 140.1 °C, thus slightly different from the value recorded for PETN itself, which is 140.5 °C. A comparison of the temperature at which the mixture decomposition process occurs with maximum speed (T_{max} of decomposition) also demonstrates that the use of the silicone resin

does not cause the composition to decompose at a lower temperature.

Table 5 also shows the values of the characteristic temperatures recorded for the PETN/EFx 53/47 composition. The values of T_{onset} and T_{max} of decomposition for this mixture are lower by 1.0 °C and 1.9 °C, respectively. This mixture was prepared only for the purpose of assessing the mutual compatibility of the components. With this mass ratio, a decrease in the melting point and the decomposition point is expected.

A compatibility assessment of the PETN/EFx 80/20 compositions using the DTA/TG technique was also made based on the Polish standard PN-V-04011-21: 1998. In this test, the masses of the samples of the individual components were increased, as well as the rate of heating and the flow of the carrier gas. This evaluation consisted in comparing the decomposition curves of pure PETN and its mixture with silicone resin in the mass ratio of 80/20. A comparison of the DTA/TG curves is shown in the Figure 19.

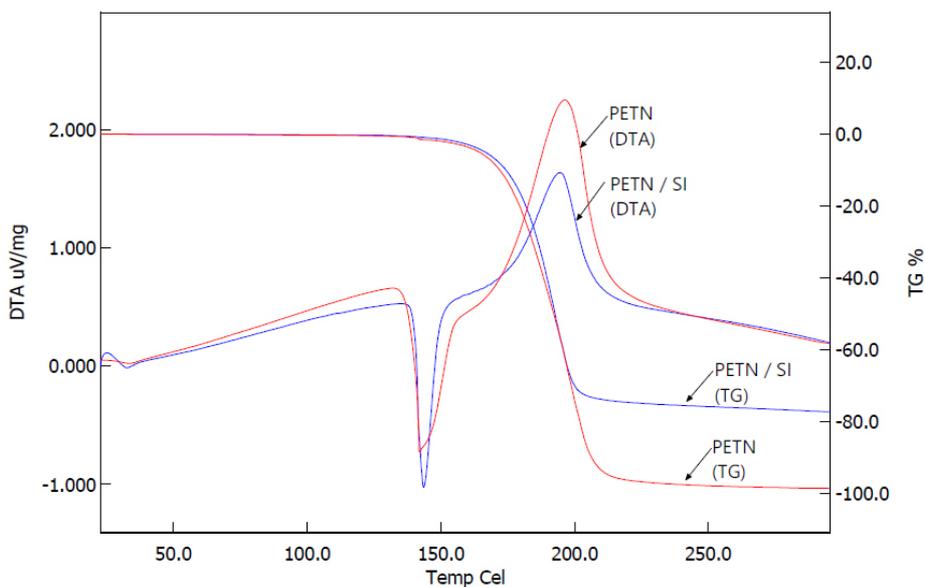


Figure 19. Comparison of the DTA/TG curves for PETN and PETN/EFx 80/20 (DTA/TG: heating rate: 5 K/min, mass of samples: 5 mg, N_2 : 50 mL/min)

A comparison of the decomposition curves (changes in sample mass – TG) clearly confirms that both samples start to decompose at about 140 °C. The decomposition is preceded by the melting of PETN at 142 °C (T_{max} melting point).

The temperature where the decomposition rate of the samples is greatest does not change significantly. For PETN and its mixture with silicone resin it was 196.5 and 195.5 °C respectively. This comparison shows that the decomposition of pure PETN proceeds at a faster rate than in the mixture. It can be concluded that the silicone resin is a kind of decomposition inhibitor in the mixed sample. A comparison of the course of the decomposition curves and the characteristic temperatures allows the conclusion that the mixture of PETN and silicone resin is chemically compatible.

3.3 Results of detonation and sensitivity testing of PBX-Si compositions

The conducted measurements of the detonation parameters and sensitivity to mechanical stimuli confirmed the adopted assumptions. The composition with the mass ratio of PETN/EFx 80/20, optimized in terms of mechanical properties, detonated at a velocity above 6000 m/s at a density of 1.3 g/cm³, and its critical diameter exceeded the assumed 4 mm. Table 6 summarizes the results of the measurements of the detonation parameters, while Table 7 presents the results of the Plate-Dent Test and the detonation pressure estimated based on these results.

Table 6. The results of detonation and sensitivity testing of the PETN/EFx 80/20 composition

Sample	Detonation velocity [m/s]	Critical diameter [mm]	Friction sensitivity [N]	Impact sensitivity [J]
PETN/EFx	6320	5.5	180	7.8

Table 7. The results of Plate-Dent Test and the estimated value of the detonation pressure for the PETN/EFx 80/20 composition

Sample	Density [g/cm ³]	Average measured plate dent [mm]	Detonation pressure [GPa]
TNT*	1.63	–	21.2
TNT	1.58	4.19	20.5
PETN/EFx	1.42	3.72	18.2

*Literature Value [19]

4 Summary

- ◆ Summarizing the results of the research presented in this paper, it can be stated that it was possible to develop a new method to optimize the composition of flexible explosive compositions based on silicone resin and PETN, adapting the method of analysing texture properties for this purpose. Our own research methodology was developed to determine the basic mechanical properties of PETN/EFx compositions. The first tests performed confirmed that the applied method is a new tool that allows measurable characteristics of elastic PBX compositions to be achieved. The properties of the materials that can be determined using the analysis of the texture profile are particularly important from the point of view of designing technological processes. The new method also makes it possible to eliminate sensory evaluation from some technological operations, which reduces the risk of errors resulting from subjective assessment by the researcher. The analysis of texture properties, in addition to assessing the properties presented in the paper, *i.e.* hardness, cohesiveness, springiness etc., allows the values obtained to be indirectly related to the processes taking place in the sample, such as migration of liquid components, crystallization or evaporation. Therefore, the texture analysis method can be successfully used to test explosive compositions.
- ◆ The presented results are also a summary of preliminary research aimed at the development of an elastic explosive composition for special purposes. The essence of the research was to determine the optimal composition, primarily in terms of its mechanical properties. This process mainly involved the selection of the appropriate polymer and the selection of the quantitative constitution of the explosive in terms of energy and formulation. Tests of the mechanical properties showed that the optimal mass ratio of the components is 80% of explosive and 20% of Ecoflex-30 resin. However, it should be emphasized that the mixture containing the EFx-50 resin at the 20% content also has acceptable properties from the point of view of practical application. The main difference in the properties of the tested resins is their viscosity, the value of which at room temperature for the EFx-50 polymer is 2.6 times greater than that of the EFx-30. In addition, the cross-linking time for the composition is also much shorter for the EFx-50 resin (2.5 times shorter). These two factors make the effectiveness of homogenization of the components before cross-linking lower, which translates into the mechanical properties of the explosive formulation.

- ◆ This paper presents the basic physicochemical tests, as well as the detonation parameters of the composition indicated as optimal. The conducted tests confirmed that the components of the elastic explosive composition of 80% PETN and 20% of Ecoflex resin are mutually compatible. The explosive is stable, and its decomposition begins at a temperature of approx. 140 °C. The detonation parameters meet the requirements of a detonation velocity above 6000 m/s and a critical diameter in the range 4-10 mm.

References

- [1] Kaye, S.M. *Encyclopedia of Explosives and Related Items*; Vol. 8, US Arradcom Dover- New Jersey, **1978**.
- [2] Liu, R.; Han, Y.; Li, M.; Jiang, Z.; He, S. Shock Ignition and Growth of HMX-Based PBXs under Different Temperature Conditions. *Cent. Eur. J. Energ. Mater.* **2019**, *16*(1): 21-32.
- [3] Zhang, Q.; Shu, Y.; Liu, N.; Lu, X.; Shu, Y.; Wang, X.; Mo, H.; Xu, M. Hydroxyl Terminated Polybutadiene: Chemical Modification and Application of These Modifiers in Propellants and Explosives; *Cent. Eur. J. Energ. Mater.* **2019**, *16*(2): 153-194.
- [4] Li, Y.; Wu, P.; Hua, C.; Wang, J.; Huang, B.; Chen, J.; Qiao, Z.; Yang, G. Determination of the Mechanical and Thermal Properties, and Impact Sensitivity of Pressed HMX-Based PBX. *Cent. Eur. J. Energ. Mater.* **2019**, *16*(2): 299-235.
- [5] Dai, X.; He, S.; Huang, X.; Yang, Z.; Wen, Y.; Li, M. Experimental Investigation of the Effect of Polymers and Crystalline Qualities on the Safety Performance of LLM-105-Based PBXs under Dynamic Compression and Shear. *Cent. Eur. J. Energ. Mater.* **2020**, *17*(2): 201-222.
- [6] Singh, A.; Sharma, T.C.; Kumar, M. Effect of the Molecular Structure and Molecular Weight of Poly (Vinylidene Fluoride-Chlorotrifluoroethylene) Copolymers on the Characteristic Properties of TATB-Based Composites. *Cent. Eur. J. Energ. Mater.* **2020**, *17*(3): 428-450.
- [7] Persson, I. *Water Resistant Elastic Explosive Mixture*. US Patent 5238512, **1993**.
- [8] Cooper, P. *Explosives Engineering*; Wiley-VCH, New York, **1996**.
- [9] Meyer, R.; Köhler, J.; Homburg, A. *Explosives*; Wiley-VCH, Weinheim, **2002**.
- [10] Anderson, P.E.; Cook, P.; Davis, A.; Mychajlonka, K.; Mileham, M. Silicon Fuel in High Performance Explosives. *Propellants Explos. Pyrotech.* **2014**, *39*(1): 74-78.
- [11] Chyłek, Z.; Jurkiewicz, R. Investigation of the Properties of Polymer Bonded Explosives Based on 1,1-Diamino-2,2-Dinitroethene (FOX-7) and 1,3,5,7-Tetranitro-1,3,5,7-Tetraazacyclooctane (HMX). *Cent. Eur. J. Energ. Mater.* **2016**, *13*(4): 859-870.
- [12] Elbeih, A.; Zeman, S.; Jungova, M.; Akstein, Z. Effect of Different Polymeric Matrices on the Sensitivity and Performance of Interesting Cyclic Nitramines.

- Cent. Eur. J. Energ. Mater.* **2012**, 9(2): 131-138.
- [13] Lee, J.S.; Jaw, K.S. Thermal Decomposition Properties and Compatibility of CL-20, NTO with Silicone Rubber. *J. Therm. Anal. Calorim.* **2006**, 2(85): 463-467.
- [14] Lee, J.S.; Hsu, C.K.; Chang, C.L. A Study on the Thermal Decomposition Behaviors of PETN, RDX, HNS and HMX. *Thermochim. Acta* **2002**, 392-393: 173-176.
- [15] Jaw, K.S.; Lee, J.S. Thermal Behaviors of PETN Based Polymer Bonded Explosives. *J. Therm. Anal. Calorim.* **2008**, 93(3): 953-957.
- [16] Tai, A.; Bianchini, R.; Jachowicz, J. Texture Analysis of Cosmetic/Pharmaceutical Raw Materials and Formulations. *Int. J. Cosmet. Sci.* **2014**, 36(4): 291-304.
- [17] McAteer, D.; Weaver, M.; Blair, L.H.; Flood, N.; Gaulter, S. Compatibility Assessment of Thermoplastic Formulations. *Proc. 19th Semin. New Trends Res. Energ. Mater.* **2016**, 401-409.
- [18] Stanković, M.; Dimić, M.; Blagojević, M.; Petrović, S.; Mijin, D. Compatibility Examination of Explosive and Polymer Materials by Thermal Methods. *Sci.-Tech. Rev* **2003**, 53: 25-29.
- [19] Singh, A.; Kumar, R.; Soni, P.K.; Singh, V. Compatibility and Thermal Decomposition Kinetics between HMX and Some Polyester-Based Polyurethanes. *J. Therm. Anal. Calorim.* **2020**: 1-13.
- [20] Myburgh, A. Standardization on Stanag Test Methods for Ease of Compatibility and Thermal Studies. *J. Therm. Anal. Calorim.* Springer, **2006**, 85(1): 135-139.
- [21] Vogelsanger, B. Chemical Stability, Compatibility and Shelf Life of Explosives. *Chim. Int. J. Chem.* **2004**, 58(6): 401-408.
- [22] NATO STANAG 4147: *Chemical Compatibility of Ammunition Components with Explosives (Non-Nuclear Applications)*. Edition 2, **2001**.
- [23] PN-V-04011-21:1998: *Military High Explosives – Methods of Testing – Determination of the Stability*. (in Polish) **1998**.
- [24] Jaffe, I.; Price, D. Determination of the Critical Diameter of Explosive Materials. *ARS J.* **1962**, 32(7): 1060-1065.
- [25] Smith, L.C. *On Brisance, and a Plate-Denting Test for the Estimation of Detonation Pressure*. Technical Raport. Los Alamos Scientific Lab., NM, **1963**.
- [26] EN-13631-4:2002: *High Explosives for Civil Uses – High Explosives Determination of Sensistiveness to Impact of Explosives*. **2002**.
- [27] EN-13631-3:2004: *High Explosives for Civil Uses – High Explosives Determination of Sensistiveness to Friction of Explosives*. **2004**.

Received: April 9, 2021

Revised: December 30, 2021

First published online: December 31, 2021