

Effect of 2,4,6-Triamino-3,5-Dinitropyridine-1-Oxide on the Properties of 1,3,5-Trinitro-1,3,5-Triazinane-based PBX Explosives

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Abstract: In the design and preparation of polymer-bonded explosives (PBX), the energetic systems possessing the characteristics of high energy output and low sensitivity are always preferred for researchers. The integration of high explosives and an energetic passivator is considered to be the most useful method in achieving this object. Herein, to explore the effect of 2,4,6-triamino-3,5-dinitropyridine-1-oxide (TANPyO) on the detonation properties and mechanical safety of 1,3,5-trinitro-1,3,5-triazinane (RDX)-based PBX, two methods including direct mixing and crystallization coating were utilized to prepare main explosives, followed by the solvent suspension distillation method to obtain relevant composites. The morphology, mechanical sensitivity, and detonation velocity of as-prepared samples were tested and characterized, as well as armor-piercing ability. Results

show that TANPyO plays a positive role in increasing the mechanical safety of RDX-based PBX and is reflected by the 4%-56% reduction in explosion probability than that of pure RDX. Moreover, the prepared samples using the crystallization coating method exhibit better performances in detonation velocity and perforation ability than the direct mixing method. These differences reach the peak when the mass ratio of RDX and TANPyO is 1:1. In that situation, the reduced values of explosion probability are 20% (impact sensitivity) and 8% (friction sensitivity) and the increasing detonation velocity is $309 \text{ m} \cdot \text{s}^{-1}$. These results demonstrate that the integration of TANPyO and the crystallization coating method is a promising way of adjusting the properties of RDX-based PBX.

Keywords: TANPyO · PBX · Crystallization coating · Mechanical sensitivity · Detonation velocity · Perforation

1 Introduction

In the domain of energetic materials, 1,3,5-trinitro-1,3,5-triazinane (RDX) is a representative compound of nitroamine explosives with the characteristics of low-cost, high detonation velocity, and strong power capability[1]. To date, RDX has been widely used in many fields including ammunition charge[2], propellants[3], and civil mining[4]. However, the high mechanical sensitivity and powder state characters of RDX should be noticed, which has been a stumbling block for its applications. The key to solve these problems lies in the assembly manners that could integrate RDX powders to the desired shape with proper explosion property and safety[5].

Polymer bonded explosive (PBX) mainly consists of explosive powders, binders, plasticizers, and other modified agents. Assisted by these agents, explosive powders in PBX could be assembled to desired shapes with uniform distribution[6,7]. This simple, cheap, and flexible method attracts the focus of many scientists and presents as an excellent way for the application of RDX[8,9]. Ideally, PBX could also achieve a relative equilibrium between energy output and safety under the synergistic effect of all components. Nevertheless, plenty of inert materials such as stearic acid and paraffin are commonly used in actual preparing

PBX, making a reduction in detonation properties. Fortunately, the introduction of energetic desensitizers into PBX has been proved to be an efficient way to balance it. The typical insensitive explosive, TATB, seems to be a suitable partner for the main explosive except for its high cost and pollution to the environment[10].

2,4,6-triamino-3,5-dinitropyridine-1-oxide (TANPyO) belongs to polyaminopolynitropyridine materials whose molecular formula is shown in Figure 1[11]. Structurally, TANPyO is relatively symmetrical where the electrons on substituents can form conjugated systems to keep a low molecular energy level. Compared with TATB, TANPyO performs similarly in comprehensive properties but stands out at low cost, which makes it a promising candidate for TATB[12]. To date, TANPyO is commonly deemed as excellent li-

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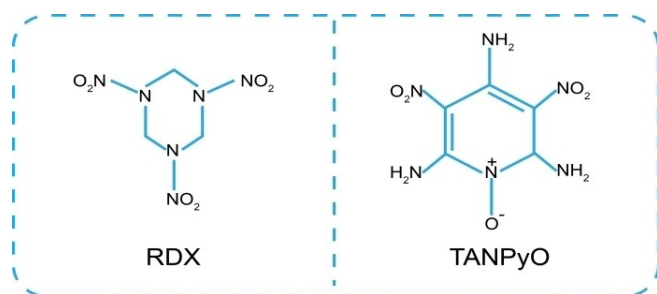


Figure 1. Molecular formula of RDX and TANPyO.

gands in loading metal ions to form energetic catalysts[13,14]. Plenty of efforts have been devoted to explore the thermal decomposition behaviors of TANPyO based compounds[15,16]. However, the energetic desensitizer role of TANPyO in PBX systems has not attracted any attention. Herein, to explore the sensitivity reduction effects of TANPyO in RDX based PBX system, two integration methods covering crystallization coating and direct mixing were first selected to prepare corresponding composites. Whereafter, RDX/TANPyO based PBXs were born by means of suspension distillation. The morphology, detonation velocity, and mechanical sensitivity of as-prepared samples were observed and tested, as well as the nail-breaking ability.

2 Experimental Section

2.1 Materials

Pure TANPyO with a crystal density of $1.876 \text{ g}\cdot\text{cm}^{-3}$ was synthesized in the laboratory. Nitrile butadiene rubber (NBR) was purchased from Changzhou Yurong Chemical Co., Ltd. Industrial product of pure RDX was supplied by Gansu Yingguang Chemical Industry Group Co., Ltd. The organic solvents (AR grade) and deionized water were obtained from Sigma-Aldrich Co., Ltd.

2.2 Preparation of PBX Samples

Crystallization coating method: A certain amount of TANPyO was added to a single round-bottom flask followed by a suitable amount of CF_3COOH . To promote the dissolution, the flask was placed in a 70°C water bath for 20 minutes. Whereafter, the solution was cooled at room temperature to obtain the TANPyO based precursor. Moreover, a certain amount of RDX, as well as toluene, were weighed and added to a three-port round-bottom flask with an agitator. Then, the TANPyO/ CF_3COOH and NBR/ethyl acetate solution were added slowly with a constant pressure funnel in order. Subsequently, the flask was placed in a constant temperature water bath in 50°C accompanied with vacuum distillation. The target product was born after the process of

10 min water bath at 75°C , cooling down, static position, filtration, washing, and drying (60°C). Among the as-prepared samples, the ratio of RDX to TANPyO was 5:1, 2:1, 1:1, 1:2, and 1:5, which were recorded as C_1 , C_2 , C_3 , C_4 , and C_5 , respectively.

Direct mixing method: First of all, a certain amount of TANPyO and RDX were weighed separately and added to a 250 mL round bottom flask equipped with an agitator. Next, an appropriate amount of water was added to obtain explosive suspensions. Then, a small amount of NBR/ethyl acetate solution was added, heated, and stirred in a water bath for 20 minutes at a constant temperature. The vacuum distillation method was selected to accelerate the NBR separation from the solution. After that, the mixture was filtered in a sand core hopper and dried to constant weight in a water bath oven at 60°C . Among them, the ratio of RDX to TANPyO was 5:1, 2:1, 1:1, 1:2, and 1:5, which were denoted by D_1 , D_2 , D_3 , D_4 , and D_5 , respectively.

2.3 Particle Size Distribution, Microcosmic Appearance, and X-ray Diffraction

MASTERSIZER2000 Laser Particle Size Tester with whose range ability 0.02 to $2000 \mu\text{m}$ was used for particle size analysis. The gas laser of He-Ne was used as a light source and the scanning speed was set as $1000 \text{ times}\cdot\text{s}^{-1}$. To observe the morphology of as-prepared samples, a scanning electron microscope (SEM, JEOLJSM-6380LV) with 30 kV electron beam energy was selected to finish this process. The grating scanning mode was used to irradiate the surface of samples. The crystal morphology of samples was examined through the DX-2700 X-ray diffraction (XRD) (Dandong HaoYuan Instrument Co., Ltd) instrument.

2.4 Mechanical Sensitivity, Detonation Velocity, and Steel Target Armor Breaking

The impact sensitivity was tested by explosion probability method referred to as GJB772 A-97 with the weight of drop hammer, the weight of samples, and drop height 10 kg, 50 $\pm 2 \text{ mg}$, and 250 $\pm 1 \text{ mm}$, respectively. The fraction sensitivity of samples was also determined by the explosion probability method referred to as GJB772A-97[17]. The weight of the pendulum, the weight of samples, and the gauge pressure were 1.5 kg, 30 $\pm 1 \text{ mg}$, and 4.9 MPa, respectively. The detonation velocity was measured by an electric probe method in GJB772A-97[17].

To test the steel target armor breaking ability of as-prepared samples, the DP3425-1 bullet was chosen to carry out corresponding experiments. The pressing process adopted two ways: viscous or belt cover and the liner was made up of bismuth alloy powder. The explosive charge was 25 g per shot with whose loaded pressure valued at 2 to 5.5 MPa, the pressure retention time 3 s, respectively. The armor

breaking test device was mainly composed of the detonating system (detonating cord and 8[#] electric detonator), perforating projectile, supporting tube, and 45[#] round steel target. The schematic diagram and optical image of the penetration test are shown in Figure 2.

3 Results and Discussion

3.1 Detonation Velocity

Detonation velocity is a critical parameter in evaluating the detonation performance of PBX samples. Herein, the PBX samples with the different mass ratios in main explosives were tested to obtain the mapping relationships of mass ratio in detonation velocity. As a contrast, the pure RDX and TANPyO were also prepared and examined. The details are exhibited in Table 1.

As seen in Table 1, the charge density values of samples prepared by two methods are close to each other and remain around 1.72 g cm^{-3} . Relatively, the charge density of samples prepared by the crystallization coating method is equal to or slightly larger than that of samples prepared by the direct mixing method. With the decrease of RDX content in samples, the detonation velocity of the samples prepared by two different methods decreases regularly. Under the same ratio of RDX and TANPyO, the detonation velocity of the samples prepared by the direct mixing method decreases more greatly, which means that the process of crystallization coating method has less effect on the detonation

velocity. The possible reason for these phenomena may be attributed to the interactions of RDX and TANPyO. During the crystallization coating process, the intermolecular distance of RDX and TANPyO is less than that in the direct mixing process, resulting in a higher interaction than the simple adsorptions in the direct mixing method. This stronger interfacial effect then brings a rising trend in charge density and corresponding detonation velocity. For instance, the C_4 (33.3% RDX-66.7% TANPyO) performs better than D_2 (66.7% RDX-33.3% TANPyO) in detonation velocity even at a lower charge density. Among the samples prepared through the crystallization method, C_3 (50% RDX-50% TANPyO) exhibits a higher charge density than that of C_2 and C_4 , which is different from the downtrend of RDX amount. Moreover, the difference value of $309 \text{ m} \cdot \text{s}^{-1}$ between C_3 and D_3 in detonation velocity is the biggest one at the same mass ratio of RDX and TANPyO. It indicates that the interfacial effect may reach the strongest when the ratio of RDX and TANPyO reaches 1:1. In this effect, the desired PBX samples with high density would be easier to obtain and exhibit higher detonation velocity[18]. The above results show that the crystallization coating method is superior to the direct mixing method in obtaining RDX/TANPyO based PBX samples with high detonation performance.

3.2 Mechanical Sensitivity

Impact sensitivity and friction sensitivity are two vital parameters in assessing safety performance of PBX. In order to compare the effect of different preparation methods on the mechanical sensitivity of samples, pure RDX, pure TANPyO, and PBX samples were tested and exhibited in Figure 3.

In Figure 3a, the explosion probability values of RDX, D_1 , D_2 , D_3 , D_4 , D_5 and TANPyO are 84%, 80%, 72%, 64%, 56%, 40% and 20%, respectively. The corresponding values from C_1 to C_5 are 72%, 60%, 44%, 40% and 28%, respectively. In Figure 3b, the explosion probability values of RDX, D_1 , D_2 , D_3 , D_4 , D_5 and TANPyO are 72%, 64%, 52%, 44%, 36%, 28% and 16%, respectively. The corresponding values from C_1 to C_5 are 60%, 48%, 36%, 32% and 24%, respectively. In general, the explosion probability of PBX samples in impact sensitivity and friction sensitivity tests decreases regularly with the increasing ratio of TANPyO, and the hypoallergenic

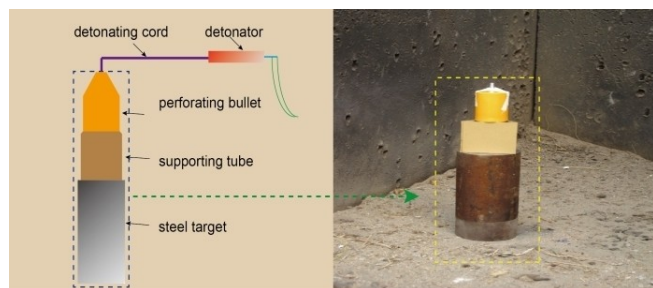


Figure 2. Schematic diagram and optical image of the penetration test.

Table 1. Detonation velocity of RDX, TANPyO, and PBX samples.

Samples crystallization Method	Charge density / $\text{g} \cdot \text{cm}^{-3}$	Detonation velocity / $\text{m} \cdot \text{s}^{-1}$	Samples	Charge density / $\text{g} \cdot \text{cm}^{-3}$	Detonation velocity / $\text{m} \cdot \text{s}^{-1}$
RDX	1.7426	8456	–	–	–
C_1	1.7413	8315	D_1	1.7278	8245
C_2	1.7284	8243	D_2	1.7255	8014
C_3	1.7301	8184	D_3	1.7221	7875
C_4	1.7265	8021	D_4	1.7084	7762
C_5	1.7233	7642	D_5	1.7042	7458
TANPyO	1.7029	7146	–	–	–

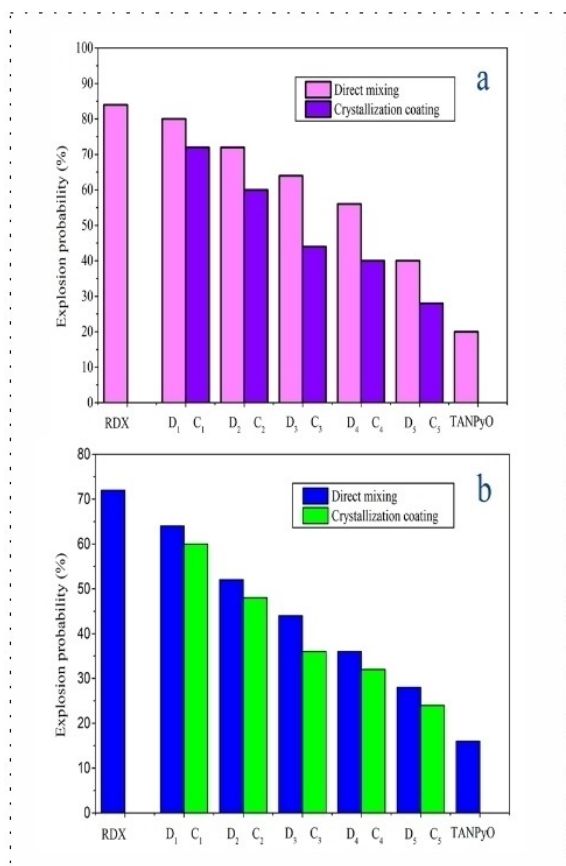


Figure 3. Impact sensitivity (a) and friction sensitivity (b) of RDX, TANPyO, and PBX samples.

performance on the crystallization coating method is better than that of the direct mixing method when the proportion of TANPyO and RDX keep same. The possible reason for these behaviors is the crystal assembly of RDX and TANPyO. The initiation mechanism of impact and friction initiation is mainly through the relative displacement between particles or hot spots generated by compressed air[19]. These tiny hot spots will trigger local ignition when enough energy is stored and gradually expanded to explode. The introduction of TANPyO to RDX based PBX systems may provide some buffer layers for RDX particles and then retard the formation of hot spots from external stimulus to some extent[20].

Among all PBX samples at the same mass ratio of RDX and TANPyO, the differences between C₃ and D₃ in explosion probability are the most and reach 20% (impact sensitivity) and 8% (friction sensitivity), respectively. This phenomenon behaves consistently with the change rules of detonation velocity, which may be also attributed to the strong interfacial effect between two main explosives.

3.3 Particle Size Distribution and SEM Photos of Samples

To observe the particle distribution of samples, the particle size of pure RDX, pure TANPyO, C₃, and D₃ were selected and analyzed by laser particle size analyzer. Results showed that average particle size (Dy, 0.5) of pure RDX, pure TANPyO, C₃, and D₃ were 97.632, 25.326, 57.614, and 47.262 μm , respectively. The corresponding specific surface areas were 0.245, 0.877, 0.569, and 0.613 $\text{m}^2 \cdot \text{g}^{-1}$ separately by analytical Multipoint BET Method with specific surface area and porosity. The particle size of C₃ and D₃ is between RDX and TANPyO, which is ascribed to the interactions between RDX and TANPyO crystals. During preparation, the assembly of new particles is finished with the noncovalent bonding forces among various components. However, the larger particle diameter of RDX weakens the bond strength and further leads to the easy falling of TANPyO crystals in the crystallization process. Meanwhile, it also results in a decrease in the number of tiny TANPyO particles and a significant increase in the number of medium particles. Thus, the average particle sizes of the as-prepared sample are all between those of TANPyO and RDX.

To provide a reasonable explanation for the different behaviors of C₃ and D₃ exhibited in detonation velocity and mechanical sensitivity, SEM was utilized to observe the microscopical appearance of samples. The results are shown in Figure 4. As shown in Figure 4 (a, b), the cube or sphere-like TANPyO crystals adhere uniformly and compactly to the surface of large RDX crystal particles. Unlike these neat arrangements, TANPyO particles in D₃ are flake-like and small-grained distributed around the larger rod-like RDX particles in Figure 4 (c–d). The significant differences are mainly due to the different surface interactions between TANPyO and

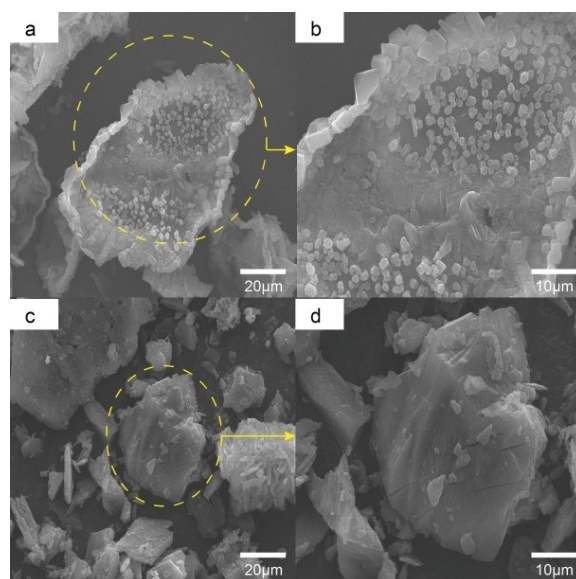


Figure 4. SEM pictures of C₃(a, b) and D₃ (c, d) at different magnifications.

RDX. From the view of the preparing method, mechanical stirring is the concrete form of direct mixing method, leading to a poor interface effect between components particles. Therefore, the contact probability of small crystal particles TANPyO to large RDX particles is low. Moreover, it is also difficult for RDX and TANPyO particles to form stable chemical adsorption and ideal coating effect. For the C_3 sample, the supersaturation of TANPyO/ CF_3COOH solution displays a rising trend at vacuum condition, which increases the driving force of crystallization and makes TANPyO crystals easy to form Van der Waals forces or H-bonds with RDX. As a result, a lot of small TANPyO crystal particles are deposited and uniformly coated on the surface of RDX. This phenomenon is consistent with our hypothesis in the detonation velocity and mechanical sensitivity tests.

3.4 XRD Patterns

Crystal morphology of pure RDX, pure TANPyO, C_3 , and D_3 were characterized by XRD with the diffraction angle ranging from 5 to 50. The measuring mode was step-in-step with whose step angle, tube voltage, and current 0.03°, 40 kV, and 30 mA, respectively. The detailed curves are shown in Figure 5.

It can be seen from Figure 5, C_3 and D_3 have the common positions of diffraction peaks, for example, 13.1°, 17.4°, and 29.3°. Compared with pure RDX and pure TANPyO curves, there are no new diffraction peaks appearing in the C_3 and D_3 . It means that no co-crystals between RDX and TANPyO are born[21].

3.5 Armor Breaking Ability

Armor breaking ability is a key reference index in determining the application direction of PBX samples. Penetrating depth, penetrating diameter, and regularity of holes are

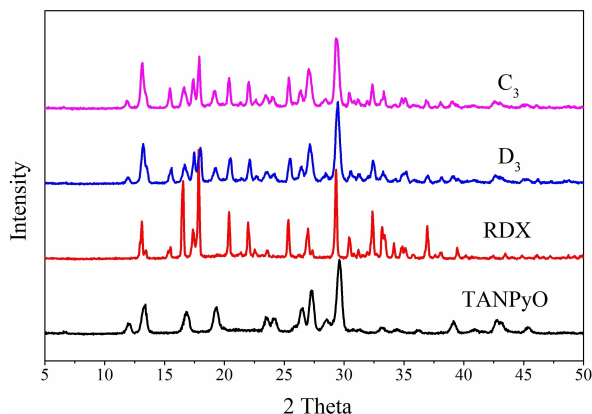


Figure 5. XRD patterns of pure RDX, pure TANPyO, and PBX samples.

concrete manifestations of it. Theoretically speaking, high perforating depth, proper perforating diameter, and uniform holes have aspired for energetic materials researchers. Herein, to have a clear understanding of RDX/TANPyO based PBX samples in armor breaking ability, three specimens including pure RDX, C_3 , and D_3 were selected to detect penetration effect. During experiments, each sample was tested two rounds to satisfy the demands of vicious and liner form. Thus, the total trials of steel targets armor penetration are 6 and the results are shown in Figure 6 where 1#, 2#, and 3# respectively represent pure RDX, D_3 , and C_3 at viscous form charge, 4#, 5#, and 6# are corresponding to pure RDX, D_3 , and C_3 at liner form charge.

Figure 6a shows the optical images of armor penetration of six samples where all steel targets have one big hole in the middle. The edge-edge distance representing the perforating accurate level for all samples can be seen in Figure 6b. Obviously, 1# and 4# perform best in all samples followed by 3# and 6#. This phenomenon illustrates that the energy output of RDX samples is the most accurate and concentrate, and C_3 displays relatively better performance than D_3 does in this regard. This inference is also proved by the perforation diameter illustrated in Figure 6c where the change roles of diameter values are consistent with edge-edge distance. In Figure 6d, the perforation depth of six samples all exceed 124 mm. The perforation depth of 1# and 4# is the largest among all samples with whose values exceeding 181 mm. For 3# and 6#, the perforation depths are 132 mm and 140 mm, which is 8 mm and 12 mm higher than that of 2# and 5#, respectively. It manifests that C_3 has a better armor breaking ability than D_3 , and the PBX samples prepared through the crystallization coating method are more beneficial to obtain higher impact kinetic energy and thermal energy utilization rate than the direct mixing method.

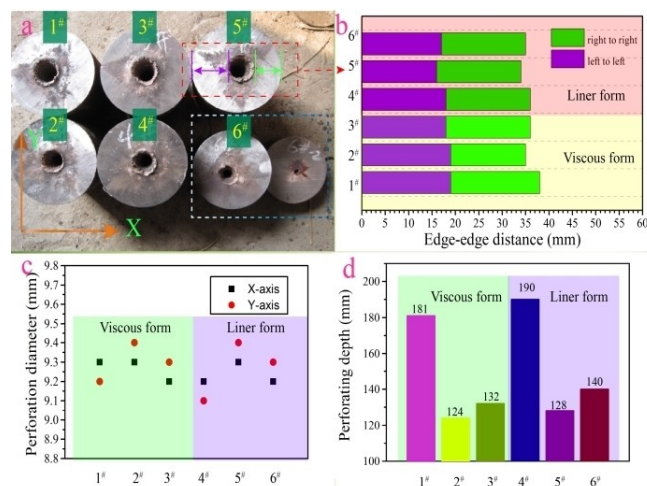


Figure 6. Optical images of armor penetration (a), edge-edge distance between holes and steel target (b), perforation diameter at different axis (c), and perforating depth (d).

In addition, the samples prepared in liner form exhibit outstandingly in creating regular penetration holes. The different values at two preparing forms of pure RDX, C₃, and D₃ samples in perforation depth are 9 mm, 4 mm, and 8 mm, respectively. The behaviors are attributed to the bismuth alloy powders used in liner form charge. The bismuth alloy has good fragility and produces small tension and shear stress during the liner collapsing process, making the jet more concentrated and higher kinetic energy. Moreover, the energy loss that can be neglected in the liner collapsing process prompts jet as an ideal fluid approximately, which also contributes to the improvement of perforation performance.

4 Conclusion

In this study, TANPyO was designed to be a partner of RDX to obtain the PBXs with high detonation performance and safety. To achieve this object, a series of RDX/TANPyO based PBXs with different mass ratios were prepared through the crystallization coating method and direct mixing method. Results show that the samples acquired from crystallization coating exhibit outstanding properties than those from direct mixing in detonation performances and safety. Such difference is the highest when the mass ratio is 1:1 for two main explosives where the detonation velocity increases around 4% and the explosion probability decreases by 31.25% (impact sensitivity) and 18.2% (friction sensitivity). SEM and XRD experiments exclude the formation of co-crystals for RDX and TANPyO. The TANPyO particles are uniformly coated on the surface of RDX crystals at the induction effect of intermolecular forces. This core-shell assembly structure in RDX/TANPyO based PBX not only provides pleasant behaviors in detonation velocity and mechanical sensitivity but also presents the delightful performance in the armor breaking tests with whose perforating depth reaching 132 mm (viscous form) and 140 mm (liner form), respectively. In conclusion, this research offers a feasible thought for modifying RDX based PBX. The corresponding method could also provide a guidance function for other high explosives such as PETN, HMX, and CL-20, which is very meaningful to the development of energetic materials.

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Data Availability Statement

No data available.

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