Oxidant-Accelerated Polydopamine Modification Process for the Fast Fabrication of PDA on HMX with Improved Mechanical Stability

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Abstract: Polydopamine surface coating shows the capability to improve the adhesive properties as well as mechanical stabilities of energetic materials, which is highly desired for the development of insensitive munitions. However, the slow polymerization process of dopamine makes the coating process time-consuming and uneconomic. Herein, an oxidant-accelerated strategy was demonstrated to obtain fast polymerization kinetics of dopamine for the surface modification of HMX crystals. Upon the addition of oxidant, PDA was able to form surface layer of HMX particles within 5 min without changing the shape and polymorph of HMX crystals. The PDA coating showed the ability to improve the wettability of HMX, contributing to its abundant active groups. Moreover, the mechanical stability of PDA on HMX (HMX@PDA) samples was improved accompany with the increasing coating time, and the friction explosion probability and drop height of 300 s treated sample were 40% and 64 cm, respectively. The detonation velocity was also measured, which showed that the effect of PDA coating on the detonation velocity of HMX was not significant. More importantly, our protocol showed the capability to fabricate HMX@PDA with different particle sizes on a large scale, satisfying the application demands of insensitive high explosives.

Keywords: HMX particle · PDA coating · Fast kinetics · Insensitive · Large-scale fabrication

1 Introduction

Energetic materials with high energy density and improved safety properties are desirable for the development of insensitive munitions [1]. Constructing a core-shell structure with energetic core material and an insensitive coating layer is considered an effective way to reduce the mechanical sensitivity of explosive composites [2]. Plastic materials such as wax, thermal plastic polymers, and graphite/graphene are commonly employed as coating materials, which are able to form smooth and flexible membranes to prevent the formation and propagation of hot-spot [3]. Although these materials decrease the mechanical sensitivity of various energetic materials, their incomplete coverage, partial aggregation, and thick layers lead to a reduction of energy density. To solve these issues, explosives with relatively low mechanical sensitivities such as TATB are chosen to be protective shell, which supposes to be a highly effective way to reduce the mechanical sensitivity of core material as well as keeping its high energy density [4]. Furthermore, energetic materials including TATB and HMX usually exhibit relative low adhesion to polymer binders, resulting in poor mechanical properties when used in polymer bonded explosives (PBXs) [4c]. Therefore, the construction of thin, insensitive, and adhesive surface coating layer with a large-scale fabrication capability is of great importance to obtain in-

sensitive explosives as well as meet the needs of practical applications.

Inspired by the adhesive properties of mussels, polydopamine (PDA) has been demonstrated to construct thin, uniform, full-covered coating layer onto the surface of energetic particles [4c, 5]. The PDA coating layer is able to achieve full coverage of particle surface, eliminating irregularities [6]. The PDA outer layer enhances the interfacial interactions between explosive crystals and binders because of its diverse active groups such as $-OH$ and $-NH_2$ [7]. Our previous work reported that a PDA surface layer reduced the mechanical sensitivity of HMX particles and increased the water wettability of HMX surface [5a]. Also, it is reported that PBXs based on the core-double shelled HMX@TATB@PDA materials displayed improved mechanical properties compared to the pristine HMX, the compressive and tensile strength was increased by 31.9% and 42.4%, and the compressive and tensile fracture energy was in-

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creased by 38.4 % and 89.2 % [4c]. In this regard, multi-functional PDA surface modification brings new methods for insensitive energetic composite as well as high-quality PBXs. However, the formation of PDA layer usually takes 10–24 hours due to the slow polymerization process of dopamine. The long synthesis process makes all PDA-involved fabrication time-consuming and high cost, limiting its industrial application in various fields. Efforts have been made to resolve this issue. It is reported that the addition of metal ions and chemical oxidants accelerates the oxidation process of catechol, resulting in enhanced polymerization kinetics of catechol compounds [8].

Herein, an oxidant-accelerated method to obtain fast polymerization kinetics of dopamine has been developed, which is suitable for the surface modification of energetic materials. Taking HMX as an example, PDA was deposited onto the surface of HMX crystals after 5 min addition of $NaIO_4$. The morphology and structure of HMX particles with different coating time were systemically observed to have a deeper understanding of polymerization process. Furthermore, the PDA coated samples showed reduced mechanical sensitivities as well as better water wettability, suggesting that the oxidant-accelerated method was able to produce insensitive HMX particles within just 5 min. The PDA coating was very thin (0.78 % weight percentage for 300s coated sample), and showed insignificant effect on detonation velocity of HMX samples. Most importantly, this oxidant-accelerated process demonstrated the capability to fabricate PDA on HMX (HMX@PDA) with various particle sizes in kilogram scale, which satisfied the requirement of practical applications.

2 Experimental Section

2.1 Materials

Dopamine hydrochloride, Tris, $NaIO_4$ were provided by Aladdin Chemical Ltd (Shanghai, P.R. China) with analytically pure. All reagents were used as-received without further purification. HMX particles were used without any pre-treatment.

2.2 Experiments

10 g HMX particles were washed with 50 mL deionized water and 50 mL ethanol three times and dried for further modification. The reaction solution consisted of dopamine hydrochloride (8 mmol) and Tris (40 mmol) dissolved in deionised water (200 ml). To this was added $NaIO_4$ (16 mmol) as an oxidant. To this solution, HMX (10 g) was added and stirred using stirrer paddle with 100 rpm (30 s, 90 s, 150 s, 240 s, 300 s) to produce HMX@PDA particles with different coating thicknesses. The resultant particles were washed

with 50 mL deionized water three times and dried at 50 °C for 6 hours for further characterizations.

2.3 Characterizations

The morphologies of pristine HMX particles and PDA modified HMX particles were observed by an optical microscope and a scanning electron microscope (CamScan Apollo 300). The samples were randomly selected and taped to the sample table with conductive tape, then sputter coated with gold for 40s at a current of 20 mA for further analysis. Fourier transform infrared spectrometer (FT-IR) spectra were carried out on a BRUKER VERTEX70 instrument from 500 cm^{-1} to 4000 cm^{-1}, 32 scans were accumulated in all cases, with a spectral resolution of 4 cm^{-1}. HMX, PDA, and HMX@PDA particles were mixed with KBr powders, and then pressed at 10 MPa for 3–5 min. X-ray photoelectron spectroscope (XPS) spectra were performed on a Thermo ESCACAB250 instrument. Thermogravimetry (TG) measurements were recorded with a NETZSCH STA 448 C instrument from room temperature to 500 °C (10 °C/min, N_2 atmosphere). X-ray diffraction (XRD) patterns were collected on a BRUKER D8 instrument using Cu K_α source. Contact angles were measured by a DSA30s (KRÜSS), water and CH_2I_2 was used as indicators. The mechanical sensitivity tests were carried out according to classic methods [9]. All samples were tested without sieving, and the average size of particles was 450 μm. According to the explosion probability method, the friction sensitivity of the pristine HMX and PDA coated samples were tested with a friction tester (WM-1) at the standard of 90° swing angle, 1.5 kg pendulum-hammer, and 3.92 MPa pressure. The impact sensitivity of pristine HMX and PDA coated samples were tested by the drop height test, which tested the falling height of a drop hammer with a 50 % explosion probability of samples. The tests were carried out on a drop weight apparatus (WL-1) with 2 kg drop-hammer at room temperature. The PBXs based on pristine HMX and coated HMX were prepared to evaluate the detonation velocity. Firstly, molding powders were produced with polymer binder (fluoropolymer F2314), explosives, and insensitive agents (weight ratios: 5/92/3). Then the molding powders were pressed into cylinder grains (Φ14×14) for further analysis. The density of pellets was measured by Archimedes drainage method. The detonation velocity of PBXs was tested by electrometric method.

3 Results and Discussion

A dopamine-Tris solution was employed as a normal condition to obtain polydopamine (PDA) coating layer, $NaIO_4$ was added as oxidant to accelerate the polymerization of dopamine. The HMX particles were added into the above solution as substrate materials. After stirring for a certain time (30 s, 90 s, 150 s, 240 s, and 300 s), the PDA coated

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samples were obtained. The color of crystals gradually deepened with increasing stirring time, indicating that the coating process of PDA was time-related (Figure 1). The 300 s treated HMX sample displayed a dark brown appearance, showed that the polymerization process of dopamine was accelerated upon the addition of oxidant.

The morphology of pristine HMX and PDA coated HMX were observed by microscope (Figure 2), which demonstrated that the size and shape of HMX crystals did not change during the coating process. The images of coated samples indicated that PDA was completely deposited onto
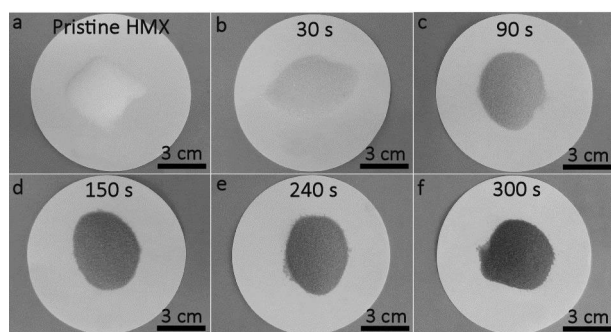

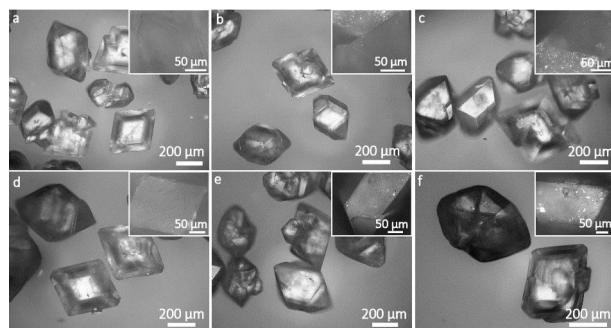**Figure 1.** Optical images of pristine HMX particles (a) and HMX@PDA particles with different coating time (b-f).

Figure 2. Microscope images of pristine HMX (a) and coated HMX (b-f). 30 s (b), 90 s (c), 150 s (d), 240 s (e), 300 s (f).
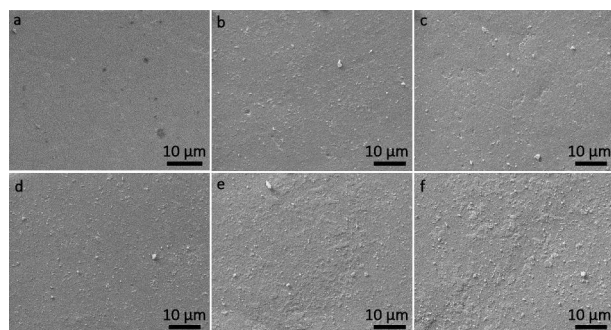

Figure 3. SEM images of pristine HMX surface (a) and coated HMX surface(b-f). 30 s (b), 90 s (c), 150 s (d), 240 s (e), 300 s (f).

the surface of HMX crystal forming a thin coating layer as the light could transmit the coated crystals. To further observe the surface condition of PDA coated sample, scanning electron microscopy (SEM) was carried out as shown in Figure 3. The surface of pristine HMX crystal was clean, while after 30 s coating process, some particles was deposited onto the surface (as shown in Figure 3b). The amount of PDA particles was increased with the increasing coating time, the particles have gradually covered the surface of crystal. All above characterizations demonstrated that the PDA coating layer was formed for 5 min, which was far faster than the traditional way, showing that the oxidant-accelerated strategy was suited for the surface modification of HMX.

Furthermore, Fourier transforms infrared spectrometer (FT-IR) and X-ray photoelectron spectroscope (XPS) was carried out to demonstrate the presence of PDA on the HMX surface. FT-IR spectra of pristine HMX and coated HMX samples were shown in Figure S1. For pristine HMX, the absorption band around $1280 \, cm^{-1}$ could be attributed to the N—N stretching, and the absorption band around $1562 \, cm^{-1}$ could be attributed to the NO_2 asymmetric stretching [10]. As for PDA, the absorption band around $1280 \, cm^{-1}$ could be attributed to the C—O asymmetric stretching and bending vibrations [11], and the band around $1562 \, cm^{-1}$ could be attributed to the –NH– bending vibrations [12]. The band around $1510 \, cm^{-1}$ and $1615 \, cm^{-1}$ were consisted with the indole or indoline structure [13]. The relevant intensity of the absorption bands around $1280/1562 \, cm^{-1}$ of HMX@PDA samples was much stronger than the pristine HMX sample. Thus, it was reasonable to infer that the increased peak intensity for the coated samples was originated form the existence of PDA.

XPS results showed the changes in surface chemical composition during the coating process. As shown in Table 1, the average atomic percentage of C, N, O on the surface of pristine HMX sample were about 26.74%, 39.37%, and 33.89%, respectively, which were similar to the 30 s treated HMX sample. The average atomic percentage of C increased with increasing coating time. After 300 s coating, the amount of carbon on the surface of sample increased to 48.56%, this is attributed to the higher carbon content of PDA, indicating that PDA was coated onto the surface of HMX sample and changed the surface chemical compositions. XPS spectra of 30 s coated sample and 240 s coated

Table 1. Atomic percentage (calculated from XPS peaks) of various elements on the surface of pristine HMX and coated HMX samples.

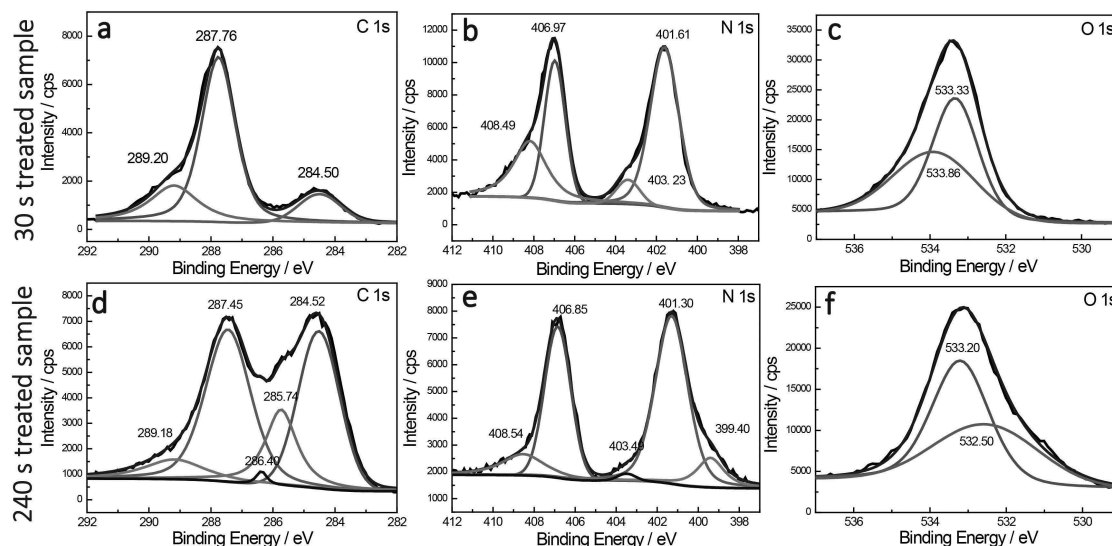| Sample	C (atom%)	N (atom%)	O (atom%)
HMX	26.74	39.37	33.89
30 s	25.1	38.86	36.04
90 s	31.22	34.44	34.33
150 s	37.96	26.04	36
240 s	44.46	24.45	31.09

Figure 4. XPS spectra of 30 s HMX@PDA sample (a–c) and 240 s HMX@PDA sample (d-f).

sample was shown in Figure 4, Figure S2-S3. Peaks located at 284.5 eV in C 1s spectrum, 399.4 eV in N 1s spectrum, and 532.5 eV in O 1s spectrum were corresponding to C−C, −NH$_2$, and C=O of PDA, respectively [14]. Compared to 30 s treated sample, the area of C−C, −NH$_2$, and C=O peaks showed an increased trend in 240 s treated sample, suggesting the PDA was deposited onto the surface of HMX crystals gradually.

TG results (shown in Figure 5) provided a rough estimation of the PDA content in composite explosives. The mass losses were 100 % and 99.22 % for the pristine HMX and 300 s coated HMX. The mass loading of PDA was 0.78 % after a 300 s coating process, indicating that the formed PDA layer was thin.
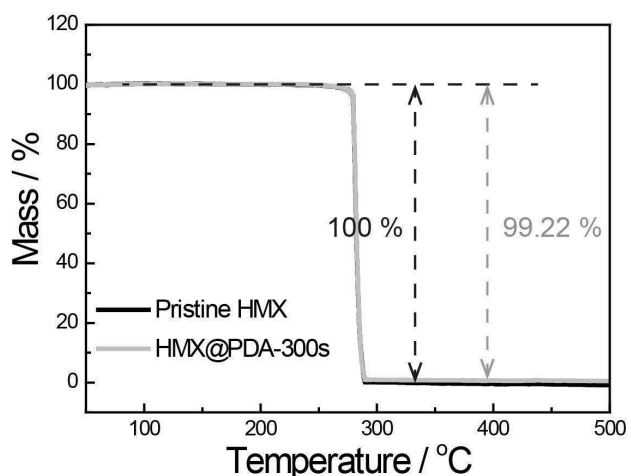

Figure 5. TG-DSC results of pristine HMX and HMX@PDA-300 s.

To have a further understanding of the structure of PDA coated sample, x-ray diffraction (XRD) patterns of pristine HMX and HMX@PDA samples with different coating time are present (Figure 6), which fits well with β-HMX (JCPDS No. 42–1768). The peaks located at 14.7°, 16.0°, 20.5°, 23.0°, 26.2°, 29.6°, and 31.9° were assigned the (0 1 1), (0 2 0), (−1 0 2), (−1 2 0), (−1 2 2), (0 2 2), and (−1 3 2) planes of β-HMX, showing that the coating process did not change the polymorph of HMX crystals. And the XRD pattern of PDA exhibited no obvious peaks from 10° to 50°, indicating that the obtained PDA was amorphous.

The oxidant-accelerated coating process also improves the water wettability of coated HMX particles. The optical images of a water droplet on the pristine HMX and coated HMX are shown in Figure 7. The water contact angle was about 83° for the surface of pristine HMX crystals. The contact angle decreased with increasing PDA deposition time, with a contact angle of 0° for 150 s, 240 s, and 300 s. Suggesting the water wettability of HMX crystals was improved because of the PDA coating layer. Furthermore, the contact angles between HMX samples and CH$_2$I$_2$ were tested as shown in Table 2, from which the surface energy, polar part, and disperse part of HMX samples were calculated (due to OWRK method) and listed in Table 2. The polar part of pristine HMX was 0.97 mJ/m^2, the polar part increased with the increasing treatment time, which suggested that the polar group was gradually modified onto the surface of HMX crystals. Contributed to the abundant active groups, including −OH and −NH$_2$ in PDA, the coated HMX samples exhibited higher surface energy, and improved water wettability.

Furthermore, PDA surface coating layer was considered to have the capability to decrease the surface defect of HMX particles and improve the mechanical stability of HMX
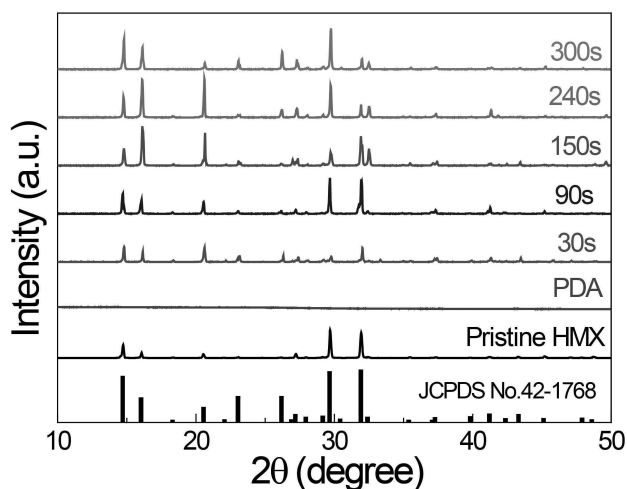
Figure 6. XRD patterns of pristine HMX, PDA, and HMX samples with different coating time.
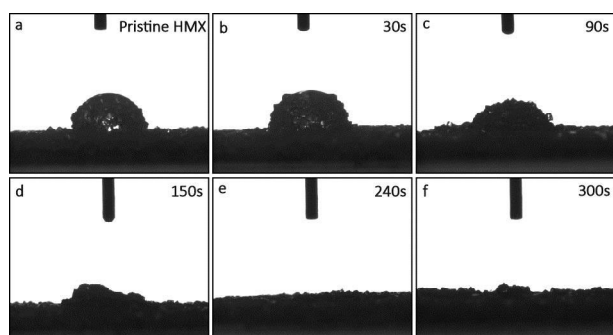

Figure 7. Optical images of a water droplet on the HMX sample (a) and HMX@PDA samples with different coating time (b-f). 30 s (b), 90 s (c), 150 s (d), 240 s (e), 300 s (f).

particles. To investigate the effect of PDA, the friction and impact sensitivities of pristine HMX particles and PDA coated HMX samples were carried out and results were listed in Table 3. The mean friction explosion probability of pristine HMX was 80%, while the drop height was 33.4 cm. With increasing PDA treatment time, the friction explosion proba-

Table 3. Friction and impact sensitivities of pristine HMX particles and HMX@PDA particles.

Samples	Friction sensitivities P (%)	Impact sensitivities H_{50} (cm)
Pristine HMX	80	33.4
30 s	70	32.5
90 s	50	40.9
150 s	50	46.3
240 s	50	55.2
300 s	40	64

bility showed a decreasing trend while the drop height increased. The mean friction explosion probability of 300 s treated sample was 40%, which was 40% lower than pristine sample, and the drop height of 300 s treated sample was 64 cm, which was increased by 30.4 cm compared to pristine HMX. The above results are convincing evidence that the PDA coating layer was able to improve the mechanical stability of HMX crystals.

To have a deep understanding of the effect of PDA coating on detonation performance, PBXs based on pristine HMX and HMX@PDA were prepared for further analysis (The photograph of HMX@PDA-300s based PBXs was shown in Figure 8a). A mass ratio of explosive (HMX or HMX@PDA), polymer binder (F2314), and insensitive agent was 92:5:3. The detonation velocity of PBXs based on pristine HMX and PDA coated HMX were carried out and listed in Table 4 (the photograph of the equipment for the detonation velocity measurement was shown in Figure 8b). The average detonation velocity of PBXs based on pristine HMX, 150 s, 240 s, and 300 s coated samples were 8381 m/s, 8423 m/s, 8404 m/s, and 8346 m/s, respectively. The deviation of detonation velocity was correlated to the density of PBXs. Therefore, the effect of PDA coating on detonation velocity of HMX crystal itself was not significant.

Most importantly, the oxidant-accelerate strategy was able to fabricate insensitive HMX particles with different sizes on a large scale. Figure 9 showed the photograph of PDA coated HMX samples with different particle sizes, 1 kg HMX@PDA with average particle size of 350 μm (Figure 9a) and 1 kg HMX@PDA with average particle size of 30 μm (Figure 9b). Indicated that this oxidant-accelerated strategy

Table 2. Contact angles between HMX samples and various liquids and calculated surface energy of HMX samples.

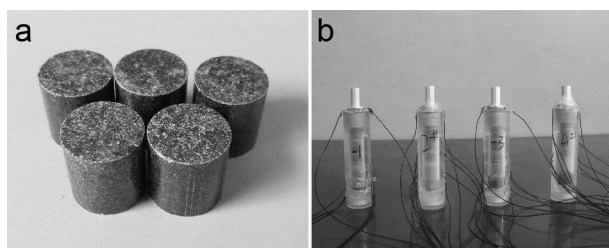| Sample	Contact angles (°)		Surface energy (mJ/m²)	Polar part (mJ/m²)	Disperse part (mJ/m²)
	CH_2I_2	H_2O			
Pristine HMX	0	82.8	53.76	0.97	52.79
30 s	0	75.6	51.46	3.35	48.12
90 s	0	58.4	52.26	13.78	38.47
150 s	0	0	72.9	48.56	24.34
240 s	0	0	72.9	48.56	24.34
300 s	0	0	72.9	48.56	24.34

Figure 8. The photograph of PBXs based on HMX@PDA-300 s (a), and equipment for the detonation velocity measurement (b).

Figure 9. Photographs of PDA coated HMX with different particle sizes. 1 kg 350 μm HMX@PDA (a), 1 kg 30 μm HMX@PDA (b).

was able to form PDA surface coating on different sized HMX crystals and showed ability to fabricate insensitive HMX@PDA particle on kilogram scale, which could satisfy the application requirement of insensitive high explosives.

4 Conclusions

In this study, an oxidant-accelerated method was demonstrated to obtain fast PDA surface modification of HMX particles. Upon the addition of $NaIO_4$ as oxidant, PDA coating layer was formed onto the surface of HMX crystals within 5 min. The shape and polymorph of HMX crystals did not change during the coating process. Along with the increase of coating time, the wettability of modified samples and the mechanical stability was improved. The above results revealed that PDA was gradually deposited onto the surface of HMX crystals and formed surface layer after a 5 min coating process. Furthermore, the effect of PDA coating on the detonation velocity of HMX samples was not significant. Most importantly, through our synthesis route, large-scale fabrication of HMX@PDA with various particle sizes was achieved. In conclusion, the oxidant-accelerated PDA coating process provide a fast, large-scale fabricate strategy for HMX@PDA particles, promoting the development of in-
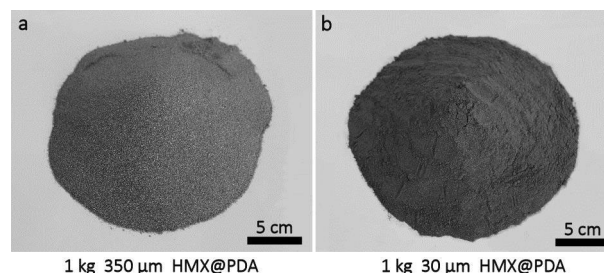
sensitive high explosives and satisfying its application demands.

Symbols and Abbreviations

HMX   cyclotetramethylenetetranitramine
PDA   polydopamine
TATB 1,3,5-triamino-2,4,6-trinitrobenzene
Tris 2-amino-2hydroxymethypropane-1,3-diol
SEM   scanning electron microscopy
FT-IR Fourier transform infrared spectrometer
XRD   X-ray diffraction
XPS   X-ray photoelectron spectroscope

Data Availability Statement

No data available.

Table 4. Detonation velocity of PBXs based on pristine HMX and HMX@PDA with different coating times.

Samples	Average density (g/cm³)	Standard deviation (g/cm³)	Detonation velocity (m/s)	Average Detonation velocity (m/s)
PBXs based on Pristine HMX	1.768	0.0145	8408 8371 8363	8381
PBXs based on HMX@PDA-150 s	1.780	0.0022	8419 8439 8412	8423
PBXs based on HMX@PDA-240 s	1.771	0.0087	8413 8370 8428	8404
PBXs based on HMX@PDA-300 s	1.758	0.0078	8341 8343 8355	8346

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