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***Title:** *Max. 20 words.* • *A good title should contain the fewest possible words that adequately describe the content of a paper.*	*Facile production of $NaIO_4$-encapsulated nanoAl microsphere as green primary explosive and its thermodynamic research*
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***Keywords:** *At least 3 keywords.* • *There is no limit on the no. of keywords you can list.* • *Please remember that effective keywords should not repeat words appearing in your title, and should be neither too general nor too narrow.*	*Encapsulation;* *Spray drying;* *Sodium periodate;* *Primary explosive;* *Detonation*
***SECTION:** • *Agricultural and Biological Sciences* • *Biochemistry, Genetics and Molecular Biology* • *Chemical Engineering* • *Chemistry* • *Computer Science* • *Earth and Planetary Sciences* • *Energy* • *Engineering* • *Environmental Science* • *Immunology and Microbiology* • *Materials Science* • *Mathematics* • *Medicine and Dentistry* • *Neuroscience* • *Pharmacology, Toxicology and Pharmaceutical Science* • *Physics and Astronomy* • *Psychology* • *Social Sciences* • *Veterinary Science and Veterinary Medicine*	*Chemistry* *Energy*

Method Article

*** Title: Facile production of $NaIO_4$-encapsulated nanoAl microsphere as green primary explosive and its thermodynamic research**

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*** Keywords:** ***Encapsulation; Spray drying; One-step preparation; Sodium periodate; Primary explosive; Detonation***

ABSTRACT

***Abstract:** *Max. 200 words, include up to 3 bullet points.*

• *Suspend nanoaluminum powder in an aqueous solution of $NaIO_4$*

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• *$NaIO_4$-encapsulated nanoAl can directly initiate the detonation of high explosives*

• *The $NaIO_4$-encapsulated nanoAl is more safer than common energetic materials*

SPECIFICATIONS TABLE

Subject Area *Chemical Engineering* *Materials Science*	*Select one of the following subject areas:* • *Agricultural and Biological Sciences* • *Biochemistry, Genetics and Molecular Biology* • *Chemical Engineering* • *Chemistry* • *Computer Science* • *Earth and Planetary Sciences* • *Energy* • *Engineering* • *Environmental Science* • *Immunology and Microbiology* • *Materials Science* • *Mathematics* • *Medicine and Dentistry* • *Neuroscience* • *Pharmacology, Toxicology and Pharmaceutical Science* • *Physics and Astronomy* • *Psychology* • *Social Sciences* • *Veterinary Science and Veterinary Medicine*
More specific subject area:	*Nanoenergetic materials*
Method name:	
Name and reference of original method	
Resource availability	

Facile production of $NaIO_4$-encapsulated nanoAl microsphere as green primary explosive and its thermodynamic research

Zaoyan Wan[a], Arcani T. Mary Cruz[a], Yan Li[a], Yanchun Li[a], Yinghua Ye[a], Shunguan Zhu[a,]*, Lin Zhang[a,]*

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Highlights

• Suspend nanoaluminum powder in an aqueous solution of $NaIO_4$

• $NaIO_4$-encapsulated nanoAl is fabricated using a one-step preparation method

• $NaIO_4$-encapsulated nanoAl can directly initiate the detonation of high explosives

• The $NaIO_4$-encapsulated nanoAl is more safer than common energetic materials

Abstract

$NaIO_4$-encapsulated nanoAl nanothermites without additives were produced by a one-step spray-drying method under the temperature of 80 ^{o}C. This approach enables the $NaIO_4$ matrix to completely cover Al nanoparticles, which are finally fabricated into $NaIO_4$-encapsulated Al nanothermite. Differential scanning calorimetry revealed that the obtained $NaIO_4$-encapsulated nanoAl released a heat of 1415.7 J g^{-1}, which is more than that of Al/CuO (1069 J g^{-1}) nanothermite. The burning rate of the $NaIO_4$-encapsulated nanoAl nanothermite was found to be almost six times higher than that of Al/CuO. At the same time, the new nanothermite system also demonstrated a high pressurization rate of 2.94 GPa s^{-1} with a transient peak pressure of 5.66 MPa, as well as a rapid release of gases which was nearly three times higher than that of Al/CuO and two times higher than that of Al/$NaIO_4$ (by physical mixing) and Al/Fe_2O_3 nanothermite. More importantly, the $NaIO_4$-encapsulated nanoAl obtained by spray drying exhibits lower activity energy compared to Al/$NaIO_4$ prepared by physically mixing method due to the higher degree of intermixing between fuel and oxidizer. The electrostatic sensitivity threshold of the $NaIO_4$-encapsulated nanoAl is obviously higher than that of n-Al/CuO and Al/Fe_2O_3. In this paper, $NaIO_4$-encapsulated nanoAl was used to directly initiate pentaerythritol tetranitrate, the initiating ability is greater than lead styphnate and close to lead azide. These results provide a feasible design scheme for replacing lead-based primary explosives because of the initiation power and process simplicity of thermites.

Keywords: Encapsulation; Spray drying; One-step preparation; Sodium periodate; Primary explosive; Detonation

1. Introduction

Primary explosives are substances that can rapidly change from deflagration to detonation and generate shock waves to detonate secondary explosives, which are extensively used in both military and commercial explosion fields [1-3]. At present, lead styphnate (LS) and lead azide (LA) are still the most widely used primary explosives. However, the long-term use of primary explosives which contains lead has caused considerable contamination in the environment and public health [4]. The environmental concerns have pushed development towards green energetic materials and environment-friendly process.

New organic primary explosives have been synthesized by large organic solvents usage and complex process [3, 5, 6]. Nanothermites usually composed by a metal fuel (e.g., Al) and an oxidizer, show a hopeful possibility of being an alternative as primary explosives through the scientist's researches and theoretical calculation. Yang [7] and Marc Comet [8] reported a series of composites comprised of nanothermite and cyclotrimethylene trinitramine (RDX) and proved these composites could be used as primary explosive substitutes to initiate high explosives. Our group introduced $Al/CuSO_4·5H_2O$ nanothermite that was prepared by electrostatic spraying (5 wt% nitrocellulose is required as a binder) as a candidate for replacing lead-containing primary explosive [9]. However, Marc Comet pointed out that the combustion of nanothermite can reach the detonation threshold as long as the pressure released by the nanothermite reaction is sufficient to permit the diffusion of the gaseous species through porosity [10]. Similarly, as reported by Zachariah's group, the nanothermite reactions have twice the energy density of 2,4,6-trinitrotoluene (TNT), and higher pressure-volume work if more gas generated [11]. Additionally, a comparison of the energy densities per volume shows that the energy of thermite-based materials can easily exceed the best existing molecular explosives. We calculated the energy density of four nanothermite reactions shown in supporting information (S0) and found that the energy densities per volume of periodate-based thermite can even reach 22.26 kJ cm^{-3}.

However, there are few reports on the usage of pure nanothermites as primary explosives. In order to fully utilize and tailor the performance of nanothermites, various powerful oxidizers, novel manufacturing technologies have been developed in recent years [12-14]. Some pioneering works were about nanothermites formulations [15-20], that can be classified into two general approaches. One is the selection for different oxidizers, which should be easy to handle and decompose to allow the gases (such as O_2, I_2) to react with the fuels. In the most commonly studied nanoenergetic formulations, metal oxide nanoparticles serve as oxidizers (Fe_2O_3 [21], CuO [22], Bi_2O_3 [23], $KMnO_4$ [24], I_2O_5 [25], $CuSO_4{\cdot}5H_2O$ [26], Bi $(ON)_3$ [27]. etc.). In a recent report, Zachariah's group prepared periodate salt nanoparticles by using an aerosol spray drying method, then combining oxidizer nanoparticles with nanoAl in a stoichiometric ratio utilizing the mixing method [28]. Their study shows that the gas phase oxygen release from the oxidizer decomposition is critical to the ignition and combustion. Also, Jared D. Moretti pointed out that the periodate salts can be uesd as pyrotechnic oxidizers [29]. Based on the consideration above and the calculation of energy densities of thermite seen in supporting information, $NaIO_4$ shows a hopeful possibility of being an indispensable ingredient for nanothermite, as it yields high energy density and high producing gas, more importantly, it is friendly to the environment [30]. So $NaIO_4$ is used as an oxidizer to fabricate high reactive nanothermite in this paper.

Besides the selection of different oxidizers, another approach is the design and synthesis of novel nanothermites employing different preparation methods in order to obtain the unique desired structures, which would increase the interfacial contact of oxidizers and fuels effectively to enhance the reactivity. Numerous studies show that the contact area plays an important role more than the particle size for enhancing the reactivity [22, 31, 32]. Some traditional preparation methods include physical mixing which is well known for its simplicity but inhomogeneity [33], sol-gel synthesis method is safe but low impurity of the final products [34] and vapor deposition, regarded as the easier method to fabricate some nanothermites with novel structures but is costly [35-37]. Other novel methods include self-assembly which can improve the homogeneity efficiently but additives would be inevitable [38], electrophoretic deposition which is facile and surfactant-free method but high-cost and spray pyrolysis [22, 24] which would all the physical and chemical process occur in the confined aerosol droplet at high temperature (even up to 300 ^{o}C) due to the decomposition of salt. Spray-drying is a mature technology that has been widely deployed in many industrial sectors [39-41]. However, to our knowledge, one-step preparations of nanothermites by spray-drying are seldom reported. Moreover, $NaIO_4$ is well soluble in water which water is cheaper and safer than any other organic solvents, thus it is advantageous for us to use spray drying method to construct nanothermites with excellent properties environmentally friendly characteristics.

In this work, we utilize spray drying method for aqueous preparation of $NaIO_4$-encapsulated nanoAl nanothermites, which has only one-step, mild temperature (~ 80 ^{o}C), without additives and easily to be scaled-up. The method established a strong intimacy between nanoAl fuel and $NaIO_4$ oxidizer for the formation of core-shell morphology, which is helpful for enhancing the combustion properties. The size, morphology, and composition were characterized and the performance of $NaIO_4$-encapsulated nanoAl nanothermites was also studied. The thermodynamic properties were investigated for the illumination of high reactivity. Finally, the detonation performance of $NaIO_4$-encapsulated nanoAl nanothermites was compared with primary explosives LS and LA.

2. Experimental section

2.1. Chemicals and materials

Al nanoparticles (approximately 100 nm) were commercially available from Jiaozuo Nanomaterial Co., Ltd. (China) with an active Al content of 70 wt%. The sodium periodate ($NaIO_4$) (99.5%) was purchased from Meryer Chemical Technology Co., Ltd. (China). CuO nanoparticles

(50 nm, 99.9%, sphere), Fe_2O_3 (30 nm, 99.9%, sphere), Hexane (C_6H_{14}, 98%), and Deionized water were from the laboratory.

2.2. Synthesis of $NaIO_4$-encapsulated nanoAl by continuous-flow Spray Drying

The composite energetic materials were prepared in a stoichiometric ratio that is shown in Eq. (1).

$$3\ NaIO_4 + 8\ Al \rightarrow 3\ NaI + 4\ Al_2O_3 + \triangle H\ (-22.26\ kJ\ cm^{-3}) \quad (1)$$

First, 1.98 g of $NaIO_4$ were dissolved in 150 mL of deionized water to form a uniform solution, which was followed by the addition of 0.66 g of nanoAl to form a suspension. The suspension was continuously stirred for 30 min to prevent the precipitation of nanoAl. About the reactivity and stability of nano Al with the aqueous solution during the preparation process be tested (Supporting Information, Fig. S1). The sodium periodate-containing thermite formulation contains 25 wt% Al nanoparticles and 75 wt% $NaIO_4$. Spray drying process includes two steps: atomization and volatilization of solvent (Fig. 1). The suspension was spray dried using a Spray Dryer with an inlet gas temperature of 80 ^oC, an air flow rate of 38 m^3/h (aspirator setting of 100%), a pump rate of 3 mL min^{-1} (10% of the maximum rate), and a nozzle air flow rate of 500 L h^{-1}. Because of other experimental conditions as well as the solution properties, the outlet temperature was around 40 ^oC. The operating conditions have been selected based on preliminary studies. The same procedure was followed for three different formations of $NaIO_4$-encapsulated nanoAl content (30 wt%, 35 wt% of nanoAl), and $NaIO_4$ nanoparticles (for details see the Supporting Information, Fig. S2).

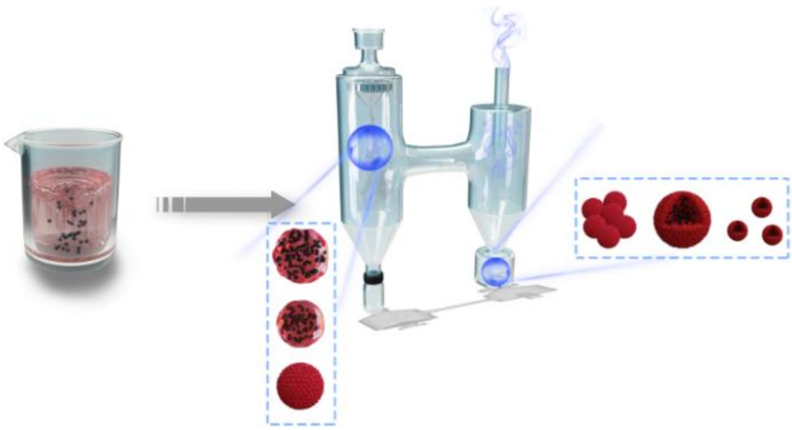


Fig. 1. Schematic illustration for the synthesis of $NaIO_4$-encapsulated nanoAl by continuous-flow spray drying.

2.3. Synthesis of Al/$NaIO_4$, Al/CuO, Al/Fe_2O_3 nanothermites

For comparison, we prepared Al/$NaIO_4$, Al/CuO and Al/Fe_2O_3 nanothermites by using a sonication mixing process. The as-prepared $NaIO_4$ nanoparticles, CuO, Fe_2O_3, and nanoAl with the ratio of Al : $NaIO_4$ = 25 : 75 wt%, Al : CuO = 36 : 64 wt%, Al : Fe_2O_3 = 34 : 66 wt% were dispersed in approximately 30 mL of hexane respectively. The mixtures were ultrasonicated for 30 min to ensure intimate mixing. After the evaporation of hexane in vacuum oven at 60 ^oC for 4 hours, the powders were gently collected.

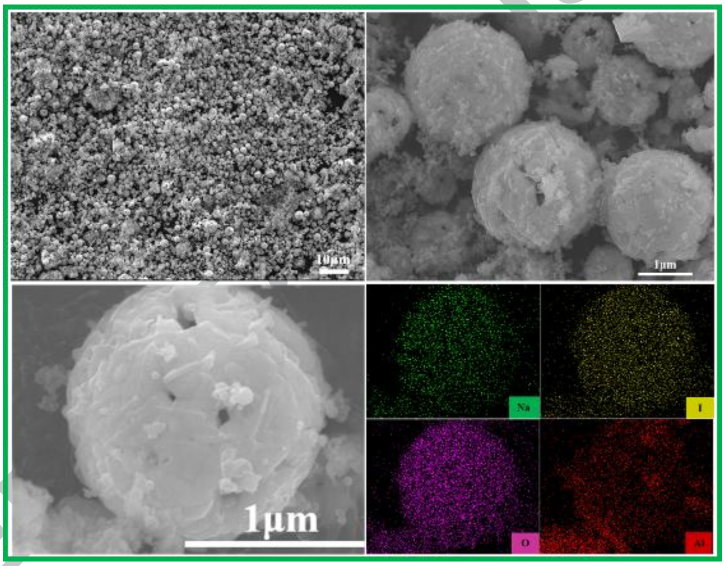
2.4. Characterizations

Field emission scanning electron microscopy (FESEM, S-4800, Hitachi), scanning transmission electron microscopy (STEM; JEOL, JEM-2100) operated at 300 kV and high-resolution transmission electron microscopy (HRTEM) were used for the morphology characterization of the samples. The structural and compositional information of the prepared samples are obtained by X-ray diffraction (PXRD, λ=1.5406 Å for Cu $K\alpha$-radiation, Bruker D8-ADVANCE, Germany) and X-ray photoelectron spectroscopy (XPS, PHI Quantera SXM). XPS was also performed to analyse further the combustion products of $NaIO_4$-encapsulated nanoAl. To evaluate the heat release and products of the reaction, we characterized the $NaIO_4$-encapsulated nanoAl, Al/$NaIO_4$ and $NaIO_4$ nanoparticles with differential scanning calorimetry (DSC) and thermogravimetric (TG) that were performed on a NETZSCH STA 449C Simultaneous Thermal Analysis (NETZSCH Group, Germany). The samples with a weight of 1~3 mg were loaded into an 85 μL alumina crucible inside the apparatus and analysis was conducted under flowing nitrogen (N_2, 30 mL min^{-1}). The temperature increased from 30 to 1000 ^oC at a typical heating rate of 10 ^oC min^{-1}. Additionally, in order to explore the kinetics of thermal decomposition behavior of $NaIO_4$-encapsulated nanoAl and Al/$NaIO_4$ nanothermites, the samples were conducted under the same conditions mentioned above at the heating rate of 2, 5, 10, 15, and 20 ^oC min^{-1}, respectively. The burning process was recorded by a high-speed camera (FASTCAM, Photron) with recording rates of 36000 frames per second. The powders were loaded into 2 mm inner diameter, 150 mm outer diameter, and length of 200 mm Poly methyl methacrylate (PMMA) tubes containing 300 mg of powder each (Support Information S3). The pressure of the nanoAl thermite reactions was conducted in a confined combustion cell with a constant volume (25 mL). In this study, 300 mg of loose powder was loaded inside the combustion cell. After igniting the sample with a heated nichrome coil, the temporal pressure from the thermite reaction was recorded by an attached piezoelectric pressure sensor together with an oscilloscope. The explosion temperature test was used for determining the minimum temperature at which an explosive will ignite or explode in five seconds, which is a general approach (Support Information S4, Fig.S3). Approximately 20 mg of the samples were heated on a molten metal bath (Wood’s metal) set at a particular temperature. And the activation energy of the thermite reaction was determined according to the Five-second explosion temperature (FET) results. The electrostatic sensitivity of $NaIO_4$-encapsulated nanoAl was measured with an electric spark tester JGY-50III, the charge capacitance which is 500 pF and the electrode gap length is 0.12 mm between the electrodes (Supprot Information S5, Fig.S4). Approximately 20 mg $NaIO_4$-encapsulated nanoAl powder was loaded on the steel plate and tested using Neyer Doptimal method for each condition. For comparison, Al/$NaIO_4$ (by physical mixing), Al/CuO, Al/Fe_2O_3 thermite systems were tested under the same conditions.

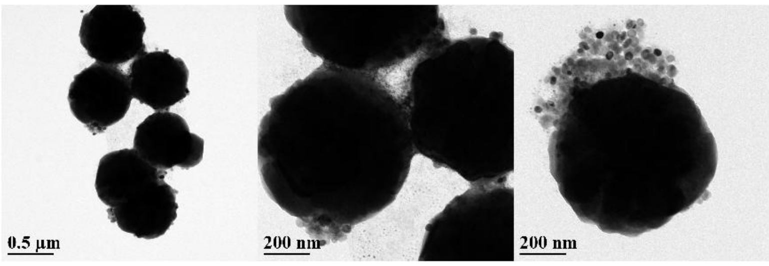
3. Results and discussions

3.1. Phase and Morphology characterizations

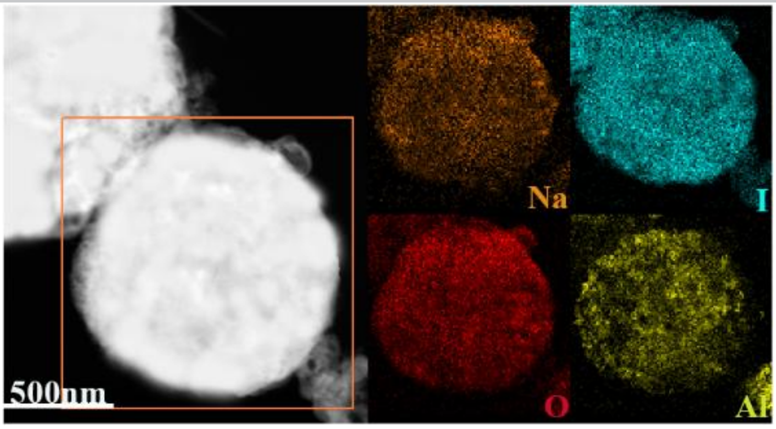
Having successfully synthesised $NaIO_4$-encapsulated nanoAl by continuous flow spray drying, we then performed SEM with elemental mapping and TEM analysis to evaluate the new morphology of $NaIO_4$-encapsulated nanoAl as shown in Fig. 2 (a) and (b). The SEM images indicate the morphology of $NaIO_4$-encapsulated nanoAl were uniformly spherical shaped structure with a diameter of 800 nm ~ 2 μm. From the elemental mapping images, the Na (green), I (yellow) and O (purple) were the predominant elements, while Al (red) showed a little amount in the $NaIO_4$-encapsulated nanoAl. This confirms that nanoAl is uniformly dispersed in the $NaIO_4$ matrix by spray drying and this was also verified by the STEM analysis as shown in Fig. 2 (c) and the XRD. For comparision, Fig. 2 (d) shows the HRTEM images for the Al/$NaIO_4$ by physical mixing which nanoAl and $NaIO_4$ were observed to be homogeneously mixed and the nanoAl particles agglomerates around the $NaIO_4$ particle. Otherwise, the HRTEM of $NaIO_4$-encapsulated nanoAl was further carried out to determine the exact composition and the crystal lattice parameters (for details see the Support Information S6, Fig. S5).



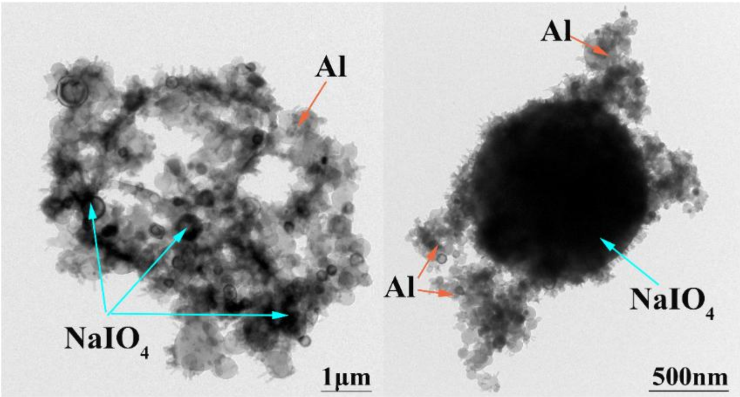
(a)



(b)



(c)



(d)

Fig. 2. (a) SEM with the Elemental mapping, (b) TEM images of $NaIO_4$-encapsulated nanoAl, (c) STEM elemental map showing the distribution of Na, I, O and Al in a single particle of $NaIO_4$-encapsulated nanoAl, (d) HRTEM images of a single particle of Al/$NaIO_4$.

The phase of the components is identified by XRD. As shown in Fig. 3(a), the diffraction peaks of the purchased nanoAl exhibit a strong peak at 38.47°, and other three peaks at 44.74°, 65.13°, 78.19°, which are exactly indexed to Al (ICDD 85-1327). Simultaneously, the characteristic peaks of $NaIO_4$-encapsulated nanoAl are kept in the same places as shown in Fig. 3(b), however, the signal that originated from Al is very weak due to the encapsulation of nanoAl by the $NaIO_4$ matrix.

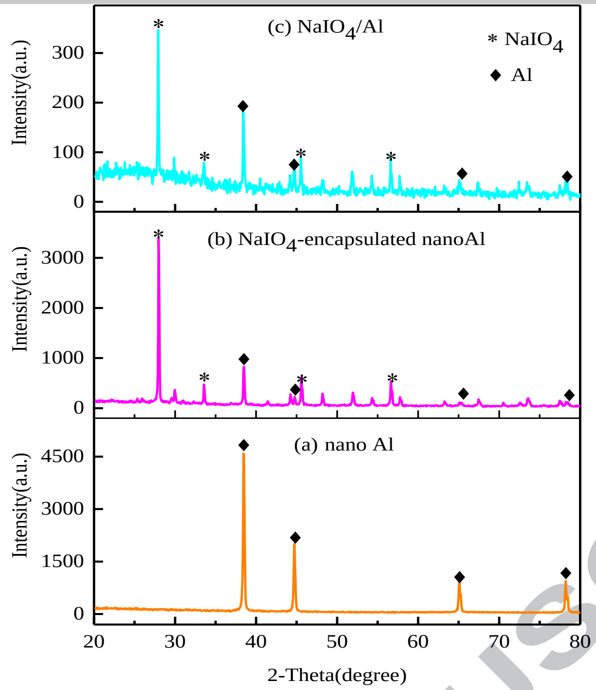


Fig. 3. XRD analysis for (a) nanoAl, (b) $NaIO_4$-encapsulated nanoAl and (c) Al/$NaIO_4$ (by physical mixing).

For comparison, Fig. 3(c) shows the XRD measurement for the Al/$NaIO_4$ prepared by simple sonication mixing process, in which nanoAl and nano-$NaIO_4$ were observed to be mixed. Among the samples, $NaIO_4$-encapsulated nanoAl were formed through a spray drying process. It was clearly observed that a strong signal was originated from both Al and $NaIO_4$ when Al and $NaIO_4$ were physically mixed. Fig. 4 shows XPS spectrum of the $NaIO_4$-encapsulated nanoAl. The survey-scan XPS spectrum shows the signals of Na, I, O, and Al elements. The XPS spectrum of Al 2p and 2s are not obvious, which a possibility is that the encapsulation of nanoAl by the $NaIO_4$ matrix.

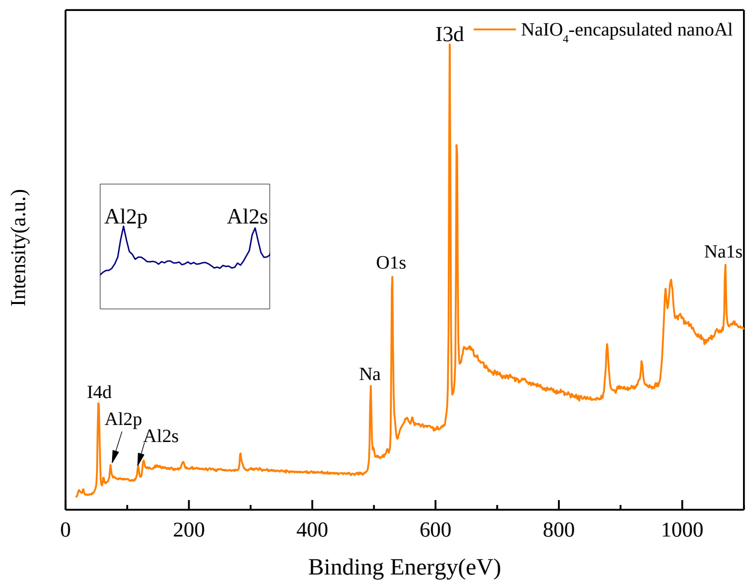


Fig.4. XPS analysis for $NaIO_4$-encapsulated nanoAl.

3.2. Thermal analyses and XPS test

As shown in Fig. 5(a), there are three exothermic peaks in the DSC curve, the first exotherm peak rises slowly with a peak temperature at 290.0 °C. This means that the $NaIO_4$-encapsulated nanoAl can react mildly below the melting point of Al (660°C). The mass of samples does not obviously change until 263°C, which is caused by decomposition of $NaIO_4$ into $NaIO_3$ and O_2 at this temperature (Support Information S7, Fig. S6). An endothermic peak is seen when the temperature rises from 260 °C to 310 °C which is accompanied by a decrease in the weight of the $NaIO_4$-encapsulated nanoAl. Due to the lower decomposition temperature of $NaIO_4$, the samples may exhibit a lower ignition temperature. This exothermal reaction attributed to the decomposition of $NaIO_4$ as shown in Eq. (2):

$$NaIO_4\ (s) \rightarrow NaIO_3\ (s) + O_2\ (g) \tag{2}$$

The second exotherm peak rises more sharply with a peak temperature of 533.3 °C and a mass reduction of 17.17%. This is consistent with the second decomposition phase of $NaIO_3$. In this stage, $NaIO_3$ decomposes exothermically between 465°C and 545°C to give NaI and O_2. This exothermal reaction is due to the partial decomposition of $NaIO_3$ as shown in Eq. (3):

$$NaIO_3(l) \rightarrow NaI(s) + O_2\ (g) \tag{3}$$

The last step is located between 650 °C and 690 °C with a peak temperature at 667.5 °C. The last exotherm peak rises slowly, but the mass reduction is plunged by 37.41 %. In this step, Al nanoparticles oxidize corresponding to the solid-state oxidation of Al by the diffusion of oxygen through the Al_2O_3 shell to the Al core, and the oxidation of liquid Al exuding from the cracks produced in the shell by the molten Al expansion. Because of the rest of $NaIO_3$ decomposition, a lot of gases coming out. About 26.8% of the $NaIO_3$ decomposes to oxygen and iodine, simultaneously the temperature is high enough to reach the melting point of NaI as shown in Eq. (4) and (5).

$$NaIO_3(l) \rightarrow Na_2O(s) + O_2(g) + I_2(g) \tag{4}$$

$$NaI(l) \rightarrow NaI(g) \tag{5}$$

The heat release from the decomposition of $NaIO_4$-encapsulated nanoAl is calculated to be 1415.7 J g^{-1}, which is higher than that of self-assembled aluminum-copper oxide nanoflower (1069 J g^{-1}) [42], but lower than Fe_2O_3 nanoring/Al nanoparticles (2039 J g^{-1}) [42] reported by Wang et al. During the whole decomposition process, large volumes of gases are emitted due to the decomposition of $NaIO_4$. It is also in good agreement with a high pressure as the Constant-volume combustion test mentioned below.

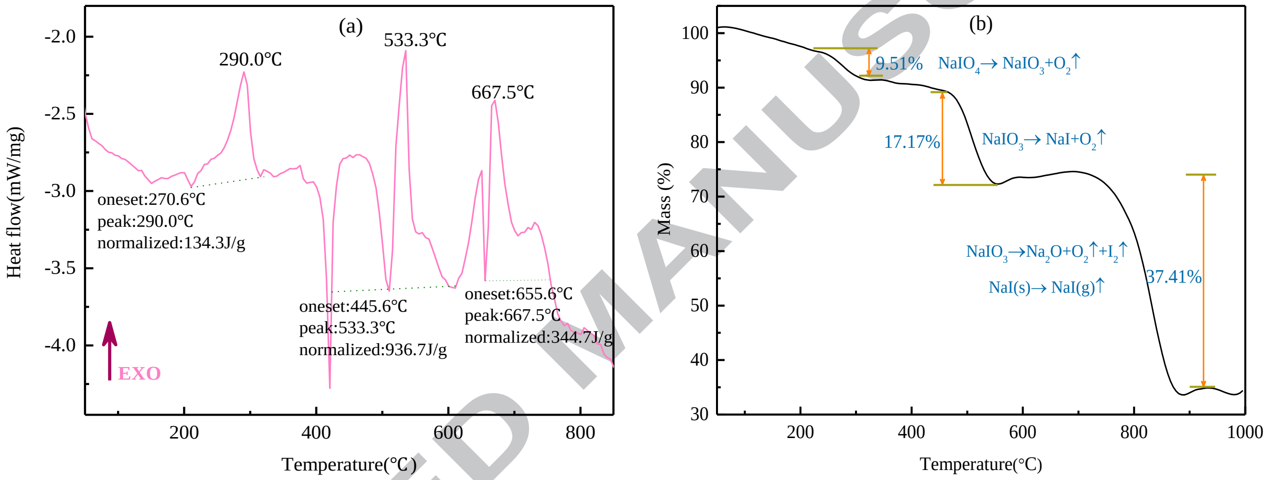


Fig. 5. Results of (a) DSC and (b) TG for $NaIO_4$-encapsulated nanoAl.

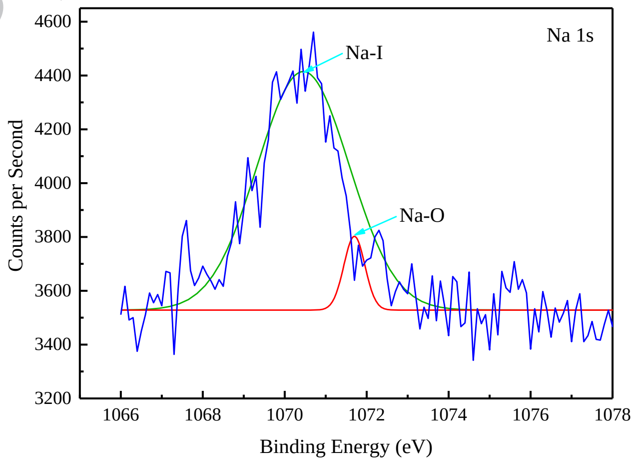


Fig. 6. XPS Na 1s spectra of reaction products of $NaIO_4$-encapsulated nanoAl.

To clarify the reaction mechanism of $NaIO_4$-encapsulated nanoAl, and detailed decomposition reactions process was done by using XPS to investigate the evolution of reaction products. The Na 1s spectrums of reaction products are shown in Fig. 6. It is observed that the binding energy of

1070.4 eV and 1071.1 eV are consistent with peaks of Na-I, Na-O, respectively. Thus, the results given by XPS confirm the existence of NaI and Na_2O from double decompositions of $NaIO_3$, which is consistent with the decomposition process of sodium periodate by DSC analysis.

3.3. High speed camera test

Flame speed results for the nanothermites formulations are shown in Fig. 7. The sodium periodate-containing thermites show a more violent reaction than that of Al/CuO and Al/Fe_2O_3 as obviously seen from the images. Their combustion events feature a large intense flame where the flames are brighter due to the rapid release of large amounts of gases. The flame cone front edge is used to calculate the burn velocity. Fig. 8 shows the position of the flame front as a function of time. The images clearly show that the maximum instantaneous burn velocity of $NaIO_4$-encapsulated nanoAl by Spray drying was 731 m s^{-1}, and the PMMA tube was blown to pieces, which is faster than Al/$NaIO_4$ of 561 m s^{-1}, Al/CuO of 128 m s^{-1}. In the case of charge, Al/Fe_2O_3 only ignites a thin layer and does not burn continuously. It makes impossible to calculate the flame propagation speed. According to Dr. T. Bazyn et al. [43] and Dr. D. Stamatis [44] previous works, the oxygen release rate of Al/ Fe_2O_3 is slower than that of Al/CuO, and the flame length of Al/ Fe_2O_3 increases rapidly at the moment of ignition. Consequently, excessive energy loss is generated in the burning zone and this cannot be sustained when the flame is far away from the ignition source. The reaction of Al/$NaIO_4$ is also very intense, nevertheless, the intimate contact between the fuel and oxidizer is not as close as that of $NaIO_4$-encapsulated nanoAl that lead to the lower burning rate than that of $NaIO_4$-encapsulated nanoAl. Whereas, for $NaIO_4$-encapsulated nanoAl, because of the production of a great amount of gases, sufficient pressure is formed, which is conducive to the formation of detonation. This also indicates a much faster energy release for the $NaIO_4$-encapsulated nanoAl thermite than Al/CuO and Al/Fe_2O_3 thermite systems.

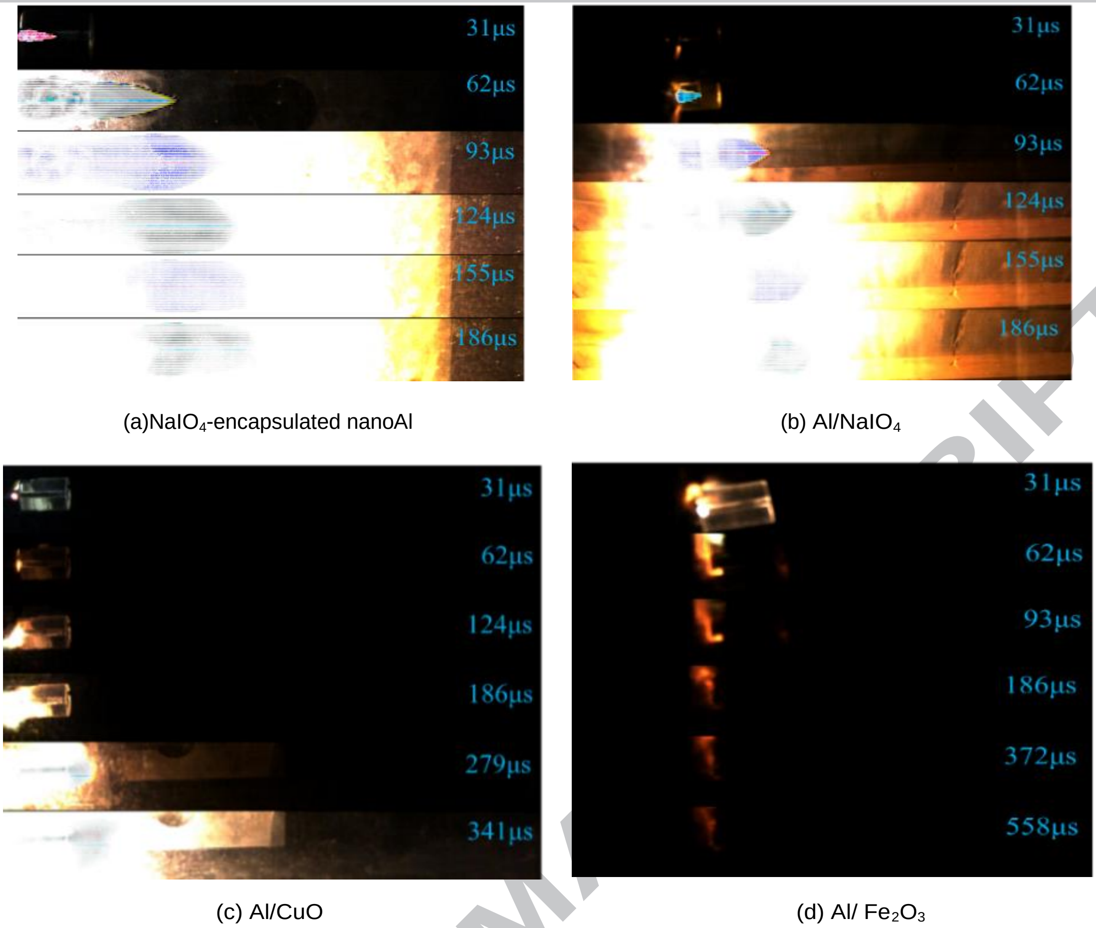


Fig. 7. Comparison of the reaction propagation in different proportions of (a)$NaIO_4$-encapsulated nanoAl and (b)Al/$NaIO_4$, (c)Al/CuO, (d)Al/Fe_2O_3.

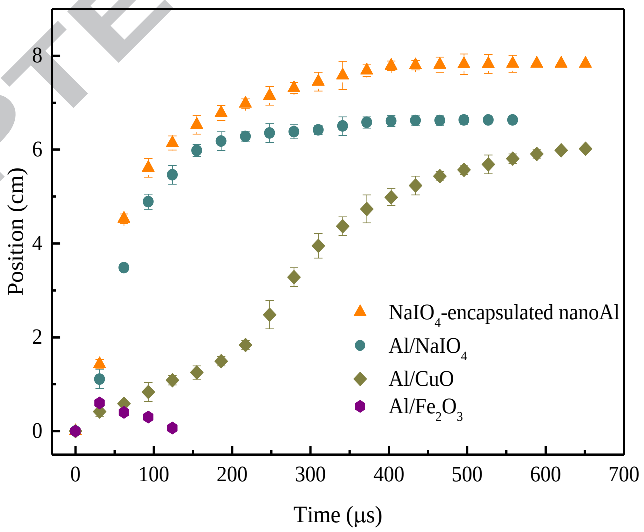


Fig.8. The position of the flame cone front as a function of time for $NaIO_4$-encapsulated nanoAl and Al/$NaIO_4$、Al/CuO and Al/Fe_2O_3.

3.4. Combustion cell test

The pressure rising of nanoenergetic reactions with different ratios of oxidants and fuel in the combustion cell is shown in Fig. 9. The peak pressure and pressurization rate are summarized in Table 1 along with Fe_2O_3 and CuO nanoparticles as the reference material. All pressure profiles show a rapid rise except Al/Fe_2O_3 nanoparticles, which occurs in a few milliseconds, with peak pressures as high as approximately 5.66 MPa, and average pressurization rate of 2.94 GPa s^{-1} for 75% $NaIO_4$-encapsulated 25% nanoAl. Both the maximum pressure and pressurization rate of $NaIO_4$-encapsulated nanoAl significantly outperforms Fe_2O_3 and CuO NPs (normally used as a standard), 1.01 GPa s^{-1} for Al/CuO and 0.07 GPa s^{-1} for Al/Fe_2O_3, $NaIO_4$-encapsulated Al nanoenergetic formulations deliver the highest values. In the sodium periodate systems, several gaseous species (O_2, I_2, etc.) can form during the decomposition, and the formation of these gaseous species can greatly enhance the peak pressure, while also aiding in the convective energy transport throughout the thermites. Furthermore, the gases of oxygen and iodine released from sodium periodate salt decomposition should enhance the burning rate of $NaIO_4$-encapsulated nanoAl. This point also was corroborated by High speed camera test. Additionally, we compare the results with energetic formulations comprising of different ratios of aluminum nanoparticles and $NaIO_4$. For the other three formulations with $NaIO_4$-encapsulated nanoAl, we found a very significant increase in the maximum peak pressure and pressurization rate with the decrease of nanoAl content. In comparison with the Pmax of 4.0 MPa as observed by Jian et al. [28], a larger pressure of 5.66 MPa with a 25 wt% content of nanoAl is shown in Table. 1. A comparison for the 75 wt% $NaIO_4$-encapsulated 25 wt% nanoAl (spray drying) and 25 wt% Al / 75 wt% $NaIO_4$ (physical mixing), the former shows greater peak pressure. The enhanced performance results from a more compact interfacial contact between $NaIO_4$ and Al by spray drying. Thus, the contact between oxidizer and fuel plays an improving role in the reactivity of nanothermites.

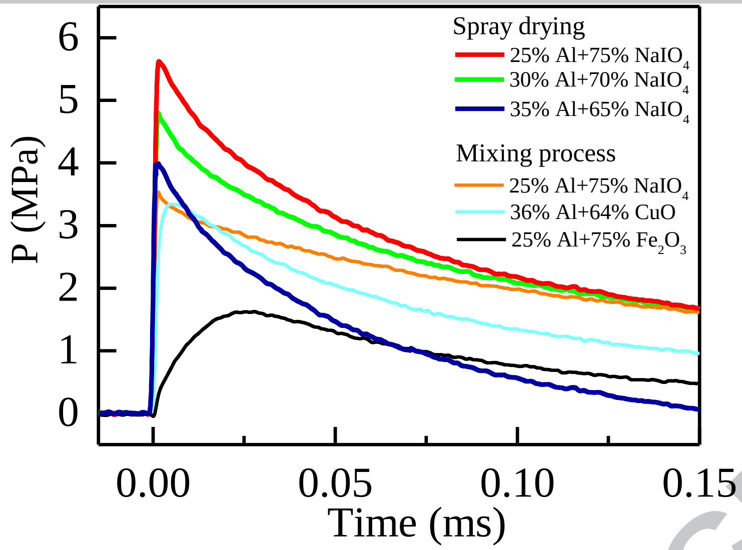


Fig. 9. Pressure trace of different proportions of $NaIO_4$-encapsulated nanoAl and Al/$NaIO_4$, Al/CuO and Al/Fe_2O_3.

Table 1 Results obtained in the combustion cell with different proportions of $NaIO_4$-encapsulated nanoAl and nanoenergetic formulations Al/$NaIO_4$, Al/CuO and Al/Fe_2O_3.

Preparation method	Formulations	P_{max} (Mpa)	Pressurization time (ms)	Average pressurization rate (GPa s^{-1})
Spray drying	25%Al+75%$NaIO_4$	5.66	1.92	2.94
	30%Al+70%$NaIO_4$	4.82	1.76	2.74
	35%Al+65%$NaIO_4$	4.00	1.82	2.19
Mixing process	25%Al+75%$NaIO_4$	3.55	1.86	1.91
	36% Al+64%CuO	3.37	3.35	1.01
	25%Al+75%Fe_2O_3	1.68	23.10	0.07

3.5. The thermodynamic research of $NaIO_4$-encapsulated nanoAl

The activation energy (Ea) is commonly defined as the minimum energy needed to initiate the reaction. Higher Ea value implies that more input energy is required to ignite the sample. In chemical kinetics, the pre-exponential factor (A) is the pre-exponential constant, an empirical relationship between temperature and rate coefficient. For obtaining Ea and A of periodate-based nanothermites, two methods are introduced for calculating. One conventional method is based on the DSC curves at various heating rates (from 2 to 20 °C min^{-1}) by utilizing the Friedman analysis. The change in Ea and A with different heat rate is shown in Fig. 10.

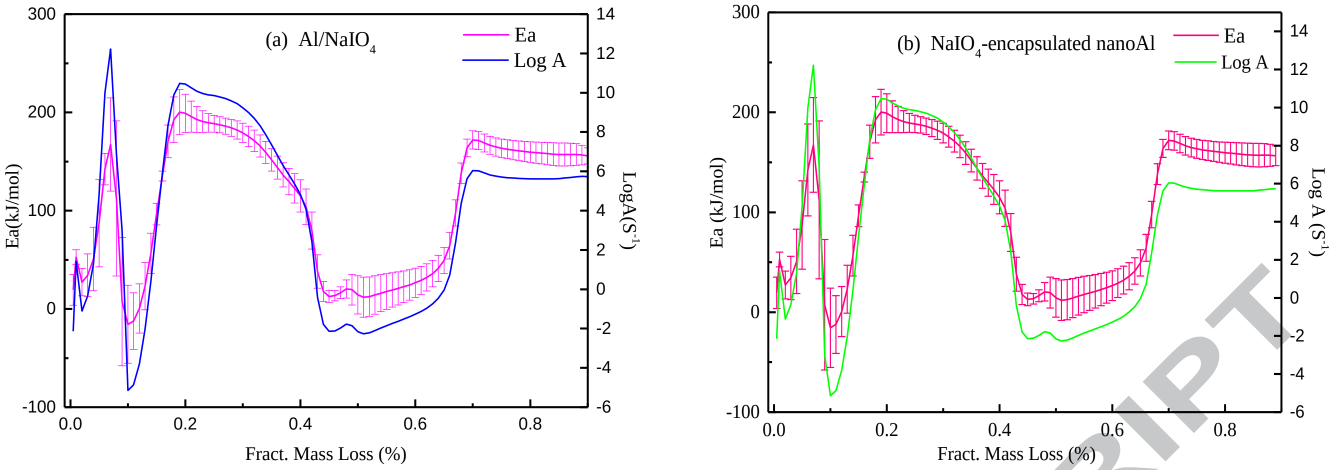


Fig.10. The fitting curves used for calculating the dynamic activation energy at different heating rates and by Friedman method: (a)Al/$NaIO_4$; (b) $NaIO_4$-encapsulated nanoAl.

According to Friedman method based on the DSC data, the value of calculated Ea and A are changeable gradually with the reaction process. However, the trend of change is almost same and the difference between the value is not obvious whether physical mixing (Al/$NaIO_4$) or spray drying ($NaIO_4$-encapsulated nanoAl). In fact, the experiments of perforation of lead plates mentioned below show that $NaIO_4$-encapsulated nanoAl exhibits better detonating performance than Al/$NaIO_4$. Accordingly, we can speculate a possible reaction mechanism. The decomposition temperature of $NaIO_4$ into $NaIO_3$ is about 263 ^oC, when the temperature is raised up to about 400 ^oC, $NaIO_3$ reaches its melting point where the solid becomes molten and liquidlike. At this moment, the nanoAl powders become mobile and begin to aggregate and coalesce at the within and around of the nano $NaIO_4$. The process mixes the nanoAl and $NaIO_4$ thoroughly, even if the different degree of intermixing between fuel and oxidizer resulted from the different preparation methods. More importantly, deflagration to detonation transition is a more violent process than simply thermal decomposition. The rate of DSC is comparatively slow, the method gives correct results only for the first-order reaction, otherwise we get formal kinetics [45], simultaneous evaporation often makes it difficult to obtain real kinetic parameters, which is not suitable for the periodate-based nanothermite. Especially, the reaction temperature and the heating rate play an important role in the thermal decomposition process of a complex molecule occurs. And for the nanothermites, high heating rate reaction is a near real reaction process. Considering these reasons, the five-second explosion temperature test [46-48] is more suitable for nanothermites. The original data of explosion temperature and time are processed by least square method. The fitting calculations obtained from Arrhenius equation are presented in Table.2.

Table 2 Calculation for the kinetic parameter of $NaIO_4$-encapsulated nanoAl and Al/$NaIO_4$.

Formulations	5s explosion temperature (^oC)	Ea (kJ/mol)	Ln A (s^{-1})
$NaIO_4$-encapsulated nanoAl	317	19.38	392.9
Al/$NaIO_4$	336	31.61	406.7

The results show that the Ea of $NaIO_4$-encapsulated nanoAl is estimated to be 19.38 kJ mol^{-1}, which is much lower than that of Al/$NaIO_4$. And the $NaIO_4$-encapsulated nanoAl has a lower 5s explosion temperature, that means the heating rate can influence evidently the thermal behaviour of periodate-based thermite. Under more violent conditions, rates of pressurization may be greater, and value of Ea of $NaIO_4$-encapsulated nanoAl is lower. Therefore, the $NaIO_4$-encapsulated nanoAl obtained by spray drying shows higher reactivity and energy output. Possibly this result is that the time of decomposition of $NaIO_4$ and molten of $NaIO_3$ is shortened, which makes the reaction of nanoAl and $NaIO_3$ still in solid phase.

3.6. Electrostatic Safety

The electrostatic sensitivity test is performed ensuring the stability and safety of $NaIO_4$-encapsulated nanoAl. The discharge energy at 50% of explosive (referred as E_{50}) is important for the evaluation of safety. The discharge energy at 50% of samples is measured and calculated with the formula $E = 0.5\ C\ U^2$, where C is the capacitance of the capacitor in Farads (F) and U is charge voltage in volts (V). High E_{50} values imply lower electrostatic sensitivity and higher safety concern. The application of nanoenergetic materials is strictly limited by their excessively low sensitivity thresholds to ESD, which are often below 0.14 mJ. Fig.11 compares the performance of $NaIO_4$-

encapsulated nanoAl with commonly used nanoenergetic materials, primary explosives, and some representative primary explosives from literatures also included.

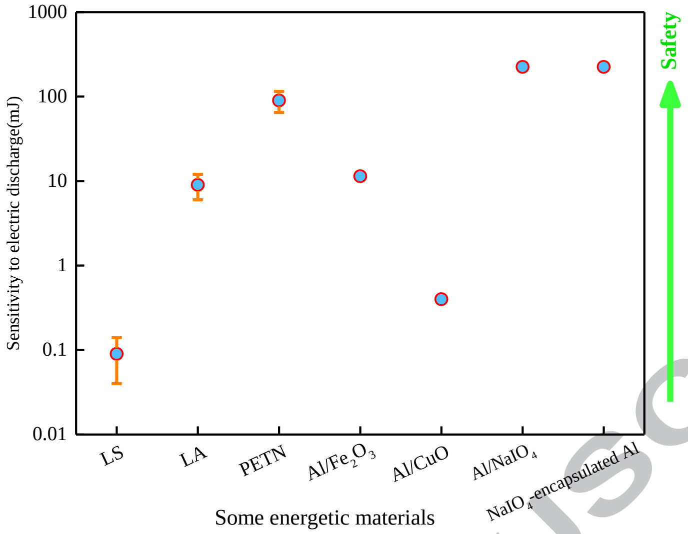


Fig.11. The electrostatic sensitivity of some energetic materials.

The electrostatic sensitivity of the $NaIO_4$-encapsulated nanoAl and Al/$NaIO_4$ are over 225 mJ. In other words, 25 $NaIO_4$-encapsulated nanoAl and 25 Al/$NaIO_4$ samples are not ignited at 30 kV (Maximum range of JGY-50III). The low electrostatic sensitivity of periodate-based thermite can ensure the safety in the fabrication process and application. The experimental results also show that 25 Al/CuO and 25 Al/Fe_2O_3 samples were ignited at 1.1 kV and 6.75 kV, which the electrostatic sensitivity of Al/CuO and Al/Fe_2O_3 are 0.3 mJ and 11.4 mJ, respectively. The electrostatic sensitivity threshold of the $NaIO_4$-encapsulated nanoAl is obviously higher than that of Al/CuO, Al/Fe_2O_3, and even pentaerythritol tetranitrate (PETN) [1], which means that the new nanothermite system is much safer than common explosives which makes them more widely available.

3.7. Detonation initiation of PETN without primary explosives

The perforation of lead plates is a simple qualitative method to evaluate the performance of primary explosives. As shown in Fig. 12 (a), the plates are placed in contact with the bottom of a tube. The diameter of the hole is an indicator of detonation ability. Several detonation tests are revealed defining the feasibility for $NaIO_4$-encapsulated nanoAl as primary explosive to detonate the PETN. In the configuration used for performing the detonation tests, the charge is divided into three sections: the first one is filled with $NaIO_4$-encapsulated nanoAl (96 mg, pressure of charge is 10 MPa) igniting composition; the second is loaded with the PETN (260 mg, press softly); the third comprises passivated RDX (450 mg, pressure of charge is 40 MPa).

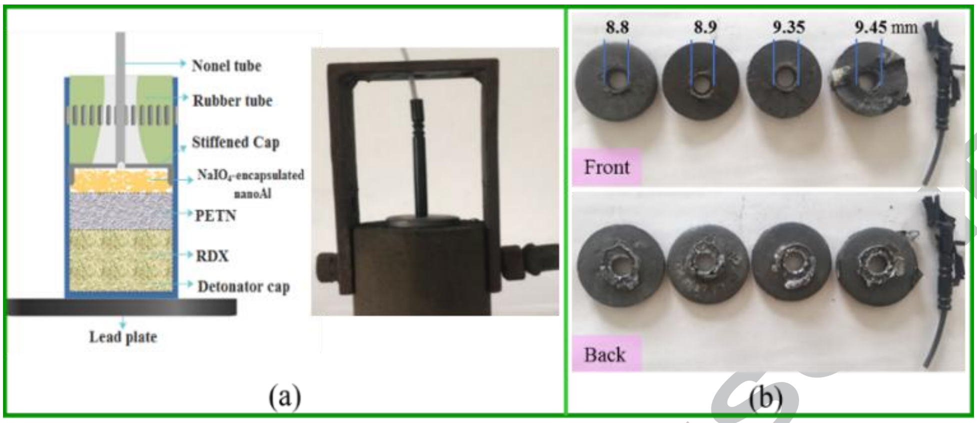


Fig. 12. Illustrations of (a)setup of explosive test and (b) results of the explosive test by lead plate method.

The experimental results for explosive reactions demonstrated shows that just 96 mg of $NaIO_4$-encapsulated nanoAl can detonate PETN and RDX to bore through the 5 mm thick lead plate, and the diameter of the hole ranges from 8.8 mm to 9.35 mm, as shown in Fig. 12 (b). The initial step corresponds to the $NaIO_4$-encapsulated nanoAl reaction, which initiates the PETN detonation. Because of the strong exothermic reaction, high rate and a great amount of generated gas products, the energetic nanocomposite reaction is explosive. Some comparative tests of lead azide (LA), Al/$NaIO_4$, Al/CuO, Al/Fe_2O_3 and lead styphnate (LS) as initiators are carried out in the same experimental conditions to further illustrate the excellent detonation capability of $NaIO_4$-encapsulated nanoAl.

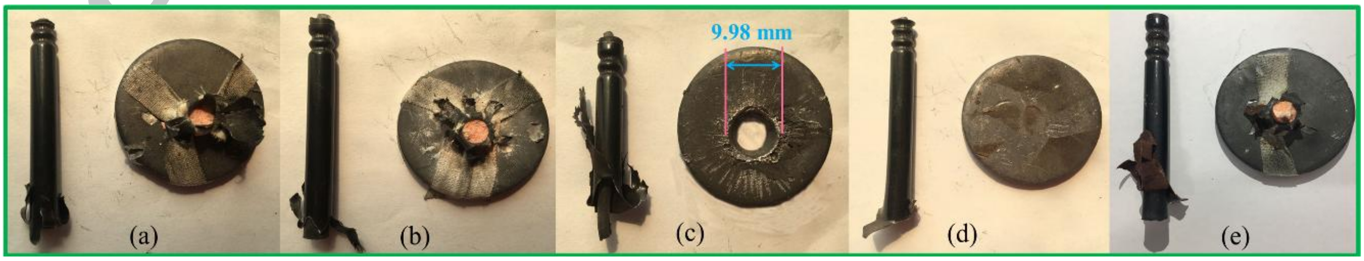


Fig.13. Illustrations of the results of detonation tests:(a) Al/ Fe_2O_3; (b)LS; (c)LA; (d) Al/CuO; (e)Al/$NaIO_4$.

As shown in Fig. 13, Al/$NaIO_4$, Al/CuO, LS, and Al/Fe_2O_3 could not be used as a primer owing to their poor initiating ability. Especially, the results show that neither LS, Al/$NaIO_4$ and Al/Fe_2O_3

cannot detonate RDX due to the LS, Al/$NaIO_4$ and Al/Fe_2O_3 have insufficient energy to reach the steady detonation of PETN. Although Al/CuO can initiate the PETN detonation, and make the burning rate of PETN reach the detonation threshold of RDX, it's still a low velocity detonation, in which several shallow traces are observed. Another comparative test of LA as initiator is carried out in the same experimental conditions, the 5 mm thickness lead plate is blown out of the hole of 9.98 mm, and further illustrates that the good detonation capability of $NaIO_4$-encapsulated nanoAl is closer to LA.

4. Conclusions

In summary, spray drying method was used to prepare $NaIO_4$-encapsulated nanoAl nanothermite as a primary explosive, which achieves low electrostatic sensitivity, high energy density, high producing gas, and excellent initiation ability; this means its overall performance outperformed most primary explosives. Importantly, 96 mg of $NaIO_4$-encapsulated nanoAl can directly detonate PETN to bore through the 5 mm thick lead plate. The new nanothermite system demonstrated highly reactive properties, and the exothermic decomposition of $NaIO_4$ contributes to the low ignition temperature. This spray drying method employed in this study is not only easy, cost-effective and safe (can avoid accidental explosion hazard unlike the other methods) , but also can enhance the reactive properties by increasing the degree of intermixing. These results demonstrate that $NaIO_4$-encapsulated Al nanothermite can satisfy the basic requirements for practical applications for explosive engineering.

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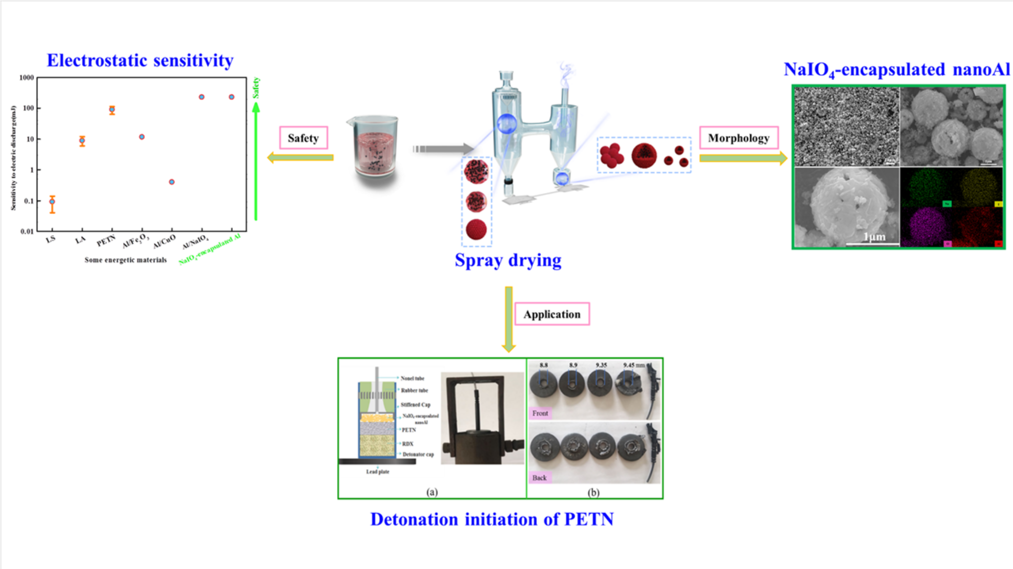
Electrostatic sensitivity
Safety
Some energetic materials
Spray drying
Morphology
NaIO$_4$-encapsulated nanoAl
1μm
Application
(a)
(b)
Front
Back
Detonation initiation of PETN

Highlights

- Suspend nanoaluminum powder in an aqueous solution of $NaIO_4$
- $NaIO_4$-encapsulated nanoAl is fabricated using a one-step preparation method
- $NaIO_4$-encapsulated nanoAl can directly initiate the detonation of high explosives
- The $NaIO_4$-encapsulated nanoAl is more safer than common energetic materials