



# Effect of paraffin wax on combustion properties and surface protection of Al/CuO-based nanoenergetic composite pellets

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## ABSTRACT

We systematically investigated the effect of a polymer binder on various combustion properties and surface protection of nanoenergetic composite pellets containing Al and CuO nanoparticles (NPs) as the fuel and oxidizer, respectively. Al/CuO NP-based composite pellets were then fabricated by a pelletization process and the effect of paraffin wax (PW) binder concentration was investigated. The burn rate decreased with increasing PW content as the binder thermochemically interfered with the aluminothermic reaction between Al and CuO. However, the presence of a critical amount of PW (<20 vol% in the Al/CuO matrix) maintained, or even enhanced, the various combustion properties of Al/CuO composite pellets, including the total heat energy, maximum pressure, and pressurization rate, when they were ignited. Simultaneously, the presence of PW was also found to effectively protect Al/CuO pellets from severe oxidation under relatively high humidity conditions. This suggests that PW played key roles as an effective binder, versatile lubricant, and oxidation protection agent. In addition, it could also be used for controlling the combustion properties of nanoenergetic material-based pellets for various thermal engineering applications.

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## 1. Introduction

Energetic materials (EMs) consist of a fuel and an oxidizer and show an intense exothermic reaction due to a violent oxidation process. Aluminum is generally used as the fuel because it is relatively stable, abundant, and inexpensive. Various materials have been used as the oxidizer, such as NiO, TiO<sub>2</sub>, WO<sub>3</sub>, MoO<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, and KMnO<sub>4</sub>, which can vary the combustion properties of EMs by tailoring the degree of mixing, chemical composition, and physical structure [1–4]. The strong self-sustained exothermic reaction of EMs is accompanied by a relatively high temperature when they are ignited by external energy input. The typical aluminothermic reaction takes place as follows [5–7]:



where MO is a metal oxide, M is a metal, and  $\Delta\text{H}$  is the generated heat energy. Conventional EMs have some limitations for

practical thermal engineering applications, including their relatively low reaction rate and mass transfer between the fuel and oxidizer. Numerous studies have been performed that aimed to improve these properties using nanostructured materials, which increase the interfacial contact area between reacting materials [8–14].

Enhanced stability and insensitivity of EMs can be achieved by either passivating the surface of fuel particles or coating EMs with organic materials; however, this deteriorates their combustion properties to some extent [15–24]. Generally, Al particles have an oxidation layer on their surfaces after exposure to oxidizing atmospheres. The formation of this oxidation layer on Al particles plays a key role in their passivation [25–33]. In the case of Al NPs, a greater relative volume of oxidation product is formed due to the higher specific surface area compared to larger particles; hence, the relative volume of the reacting core is reduced, which eventually results in significant degradation of the combustion properties [34,35]. Jelliss et al. [36] reported a synthesis method for hydrogen-evolving polymer-capped Al NPs using LiAlH<sub>4</sub> (lithium aluminum hydride), which was designed to protect Al from oxidation. The polymeric binders coating the surface of Al NPs protected them from rapid oxidation; however, the energy re-

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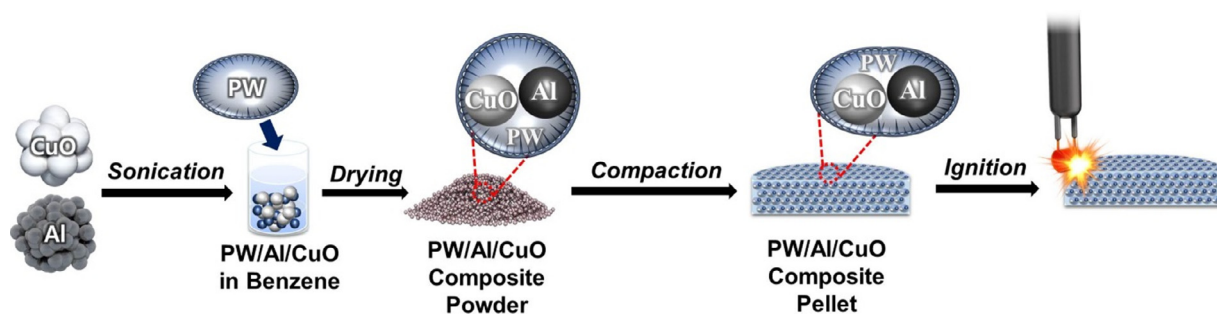


Fig. 1. Schematic diagram of the fabrication and ignition of PW/Al/CuO composite pellets.

lease rate was also significantly reduced. Gangopadhyay et al. [37] successfully synthesized nanoenergetic materials (nEMs) composed of self-assembled structures of poly (4-vinylpyridine)-coated Al NPs and CuO. They increased the energy release rate of nEMs using unique nanostructures of fuel, oxidizer, and polymeric binder; this was effective for powders, but not for preparation of pellets as the reactivity of the pellets was significantly reduced due to the lower specific surface area resulting from the compressive forces applied during pelletization. Since EM powders are not easy to handle, EM-based pellets are preferred for the various thermal engineering applications. Unlike EM powders, the fabrication and combustion properties of EM-based pellets are strongly affected by various parameters, including the theoretical density, particle size, and polymer additives.

In this study, we systematically examined the effects of a polymer additive (paraffin wax; PW) as a binder, desensitizing agent, and oxygen/water protectant for EMs on the combustion properties of nEM-based pellets. Specifically, we used Al NPs and CuO NPs as the fuel and oxidizer, respectively. Various combustion properties of polymer-added nEMs pellets were systematically investigated, including heat energy, pressure trace, pressurization rate, burn rate, and total burning time.

## 2. Materials and methods

Figure 1 shows schematic diagrams of the fabrication and ignition processes of the PW/Al/CuO composite powders and pellets. Briefly, Al and CuO NPs were mixed with a ratio of 30:70 wt% in an ethanol solution, which was then sonicated (ultrasonic power = 170 W, ultrasonic frequency = 40 kHz) for 30 min. Al/CuO NP-based powders were obtained by drying the ethanol solution in a convection oven at 80 °C for 30 min. PW was dissolved in ~50 mL benzene solution with various mixing ratios (0, 10, 20, 30, 40, and 50 vol%), and then dispersed ultrasonically after addition of Al and CuO NPs.

To measure the heat energy generated by the aluminothermic reaction in the as-prepared PW/Al/CuO composite powders, differential scanning calorimeter (DSC; Setaram, Model No. LABSYS evo) analysis was performed under a nitrogen atmosphere with a heating rate of 10 °C min<sup>-1</sup> between 30 and 1000 °C. To measure the combustion properties of PW/Al/CuO composite pellets, 100 mg of the composite powder was first formed into a disk-shaped pellet with a diameter of 7 mm and height of 0.7 mm at a pressure of ~300 MPa. The surface of the composite pellets was then observed using field-emission scanning electron microscopy (FE-SEM; Hitachi, Model No. S4700) at ~15 kV. To observe the combustion reactivity of PW/Al/CuO composite pellets ignited in ambient air, a high-speed camera (Photron, FASTCAM SA3 120K) was used with a frame rate of 30 kHz. This camera had a maximum frame rate of 1,200,000 fps, minimum frame rate of 60 fps, CMOS image sensor size of 17.4 mm × 17.4 mm, and pixel size of 17 μm × 17 μm.

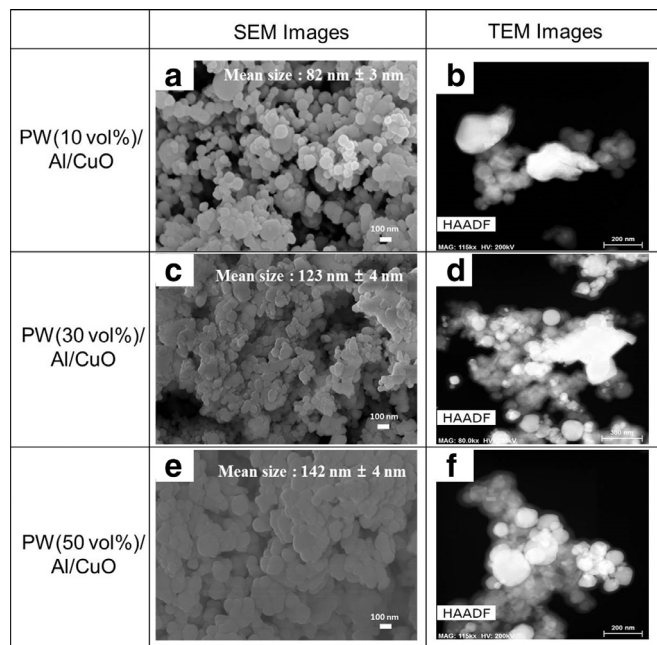


Fig. 2. SEM and TEM images of (a) & (b) PW(10 vol%)/Al/CuO, (c) & (d) PW(30 vol%)/Al/CuO, (e) and (f) PW(50 vol%)/Al/CuO composite powders.

In order to investigate the influence of the PW binder on the combustion properties of Al/CuO-based nEMs, the pressure curves and the pressurization rate were examined using a pressure cell tester (PCT). We placed ~26 mg of a PW/Al/CuO composite pellet in the PCT system, which was then ignited and exploded using a tungsten hot-wire. The pressure sensor system consisted of a pressure sensor (PCB piezotronics, Model No. 113A03) with a maximum detection pressure of ~15 kpsi, a signal amplifier (PCB piezotronics, Model No. 422E11) and an oscilloscope (Tektronix, TDS 2012B). The water permeability of the PW/Al/CuO composite pellets was estimated using a sessile drop method, where 10 μL of deionized water was dropped on the surface of the pellet and the contact angle of the droplet was measured using a CCD camera.

## 3. Results and discussion

FE-SEM analysis was performed to examine the size, shape, and homogeneity of PW/Al/CuO composites, as shown in Fig. 2. Figure 2a shows that highly aggregated Al/CuO composite NPs with an average diameter of ~82 ± 3 nm were formed. Al and CuO NPs were closely bound to each other in the PW matrix as shown in Fig. 2b. The elemental mappings were provided in Fig. S1 in the supporting information. The primary size of the Al/CuO composite powder increased with increasing the amount of PW in the

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