



Investigation of ammonium nitrate based emulsion ignition characteristic



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ARTICLE INFO

Article history:

Received 25 January 2013

Received in revised form

27 February 2013

Accepted 9 October 2013

Keywords:

Ammonium nitrate

Emulsion explosives

Accelerating Rate Calorimeter

Combustion

Pressure

ABSTRACT

This paper presents an analysis of the observed ignition behaviors of ammonium nitrate (AN) and emulsion explosives (EE). Pure AN and EE locally ignited by a heat source do not undergo sustained combustion when the pressure is lower than some threshold value usually called the Minimum Burning Pressure (MBP). A reason for pure AN's and EE's incapable of burning was suggested, and the roles of sample water, inorganic salts, oil phase, and sensitizer were discussed. Accelerating rate calorimeter (ARC) was used to study the thermal stability of EE and AN. The results also showed that MBP is of vital importance and useful for the manufacture and application of AN and EE in terms of accident prevention.

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1. Introduction

Ammonium nitrate (AN), one of the major chemical products, exhibits explosive properties under some conditions. Infrequently, its large-scale production and transportation are fraught threatened with explosions that can cause much harm. AN is an important component of emulsion explosives (EE) and has become the main product of the explosives industry in the world. The water-phase material used in the production of EE is the oxidizer mainly composed of AN. Recently, quite a few accidents have occurred during the production, especially in the pumping process, raising much concerns about the safety of EE production (Perlid, 1996). It has been acknowledged that pumping EE during the manufacture, transfer and handling operations is the most hazardous procedure among all. For instance, in 2009, 22 workers in India were killed on spot and three explosive plants were completely demolished in an accident. Later, it was found that the explosion occurred when the emulsion matrix was being pumped, and hot flying fragments hit the explosive van packed in another explosive plant, which as a result aggravated the explosion severity and increased the damage (Pingua et al., 2012). Therefore, investigating the explosion characteristics of EE is important for the safety of EE production.

AN finds extensive application as a component of a variety of energetic compositions, mainly industrial explosives and propellants. Despite the fact that AN combustion had been studied for a long time since 1950s and the society gained renewed interest in AN nowadays, the scientific understanding of the mechanism of AN ignition is far from satisfactory. A.P. Glazkova (1976) was one of the scientists who first extensively investigated the burning of AN and AN-based composition. The results show that pure AN does not burn at all under atmosphere pressure. So far, many endeavors have been pooled to investigate the combustion characteristics of AN with additives (Greiner, Frederick, & Moser, 2003; Kondrikov, Annikov, & DeLuca, 1998; Kondrikov et al., 1996; Sinditskii et al., 2005). Also, EE does not burn at all under atmosphere pressure. However, when it came to the question why AN and EE do not undergo sustained combustion when the pressure is lower than some threshold value, which is usually called the Minimum Burning Pressure (MBP), the answer was not provided. It has been found that, once locally ignited EE requires a Minimum Burning Pressure to sustain the spreading of the combustion wave (Chan & Kirchnerova, 1978). There might be a pumping accident if the pressure exceeds the MBP value. Therefore, the concept of MBP is adopted in the estimation of safe operating pressures for pumping equipment. The Canadian Explosives Research Laboratory (CERL) has conducted many researches on MBP (Chan & Turcotte, 2009; Goldthorp et al., 2008; Turcotte, Goldthorp, & Badeen, 2008; Turcotte et al., 2009). This study was carried out on the basis of CERL's experiment results.

In the present study, AN and EE were selected based on the expected influence of their respective ingredients on their ignition

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and combustion characteristics in a pressure vessel. The influence of additives (including water, inorganic salts, wax, emulsifier and sensitivity agent) on the MBP value of EE formation was studied.

2. Experiment

2.1. Materials

Pure AN and other inorganic salts, both of which are industry products (bought from Nanjing Chemical Industry Co., Ltd.), were used to prepare the samples. The formation of EE was the oxidizer solution composed of AN (83%, mass percent), water (10%, mass percent) and oil phase made up of wax and surfactant (7%, mass percent).

2.2. Preparation of emulsion matrix

The procedure involves two steps. First, the oxidizer solution was prepared (oxidizer, 83%; water, 10% mass percent) by dissolving the ingredients in a large stainless steel beaker heated at a temperature of approximately 120 °C. The oil phase comprised of oil, wax, and surfactant (7% mass percent) was then introduced to the mixer bowl and heated until all the wax melted; the bowl was heated at 110 °C. At this point, the mixer was stirred at a proper speed (approximately 200 rpm), and the hot oxidizer solution was slowly poured into the bowl while the speed was maintained. After the pouring, the speed of mixing was accelerated (approximately 200 rpm) and continued for a few seconds to achieve the final refinement.

The density (ρ) of a sample is measured by density bottle method which was defined as the relation between the mass (m) and the volume (v) of a specific amount of material. The density of a sample can therefore be determined by dividing the mass of the sample by its volume: $\rho = m/v$.

The water content of emulsion explosives is measured by solvent extraction method. It is preferable to use a two-stage analysis to calculate the water content:

- i) Sample was disrupted with a mass sample of a 50:50 mixture of anhydrous toluene in a round bottom flask.
- ii) Water was extracted from the sample and the water content was calculated.

2.3. Pressure vessel and ignition apparatus

The main part of the apparatus designed for this experiment is a high-pressure vessel with an internal volume of 12.5 L rated for a 20 MPa maximum operating pressure. Considering the weight of the vessel itself, opening and closing operations were assisted by a vessel cart and a mechanical body lifting mechanism. Preliminary experiments on EE were performed using the ignition of various sizes with varying hot wire voltages. The chosen ignition geometry was a straight-line hot wire to make the measured MBP values independent of the source size (Turcotte et al., 2008). According to the characteristics mentioned above, hot wire voltage of 6–12 V was necessary to achieve the induction time of a few seconds. In the actual operation process, voltage of 12 V is used. The explosive sample was contained in a paper tube vertically placed in the high pressure vessel. The inner diameter of the paper tube is 32 mm and the length about 85 mm. The paper tube was fixed to the pressure vessel pedestal, where there is a hole through which the hot wire was passed. The wire used to prepare the ignition sources is made up of commonly generally available Ni–Cr wire with a diameter of 0.8 mm. The length of the wire used for each assembly is 110 mm, resulting in a total resistance of 0.4 Ω .

2.4. Data acquisition

The data acquisition/control system consists of a main acquisition device (JV5201B). Coupled with the main device are a sensor (Kistler211B), a low-noise cable (STY), and a specialized amplifier (5124A) designed to acquire and communicate specific signals, all of which are interfaced with a personal computer (PC). The hardware is interfaced with a PC using the software to control the sequence of the experimental procedures and acquire the signals. When the sample was equilibrated at the desired initial pressure, a constant voltage (12 V) was supplied to the hot wire. This voltage was provided by an alternating current supply controlled by a power transformer. In the upper part of the pressure vessel, a pressure gage was also prepared. Besides, the pressure transducer was also measured when the intake system was inflated. The initial pressure in the vessel was determined in accordance with two systems.

2.5. Criterion of the experimental results

The results of the small-scale cook off test showed that the amount of energetic materials was about 1–10% with a runaway reaction (Kaneshige, Renlund, & Schmitt, 2000). It could be obtained from the reference (Kaneshige et al., 2000) that about 1–10% explosives were undergoing chemical reactions when the explosion took place. Therefore, in the test process, we selected 5%, 10%, 20%, 40% and 50% of the total pressure (all samples' burning pressure in the pressure vessel) as the criteria. If the pressure exceeds the criteria, the hot wire would stop heating. In fact, when 5% was selected as the criterion, emulsion explosives were not completely ignited, while 15% and 20% were taken as the criterion respectively, emulsion explosives burned a little bit when the sample was taken out. At last, we obtained the result that the 50% criterion was suitable for our experiment.

To show the MBP experiment results correctly, a new and simple method was used according to the small-scale cook off test results. The heating of the sample was stopped when the inner pressure of the vessel rose to about 50% of the total pressure. If the pressure continued to increase, the result would be a “go” otherwise it was a “no go”.

2.6. Accelerating Rate Calorimeter (ARC)

AN (0.5 g) and AN-based emulsion explosive (0.2 g) were placed in a spherical vessel which was closed to maintain the pressure resulting from vaporization or decomposition of the sample. For each ARC experiment, 0.5–2 g of sample materials was placed in a lightweight spherical titanium vessel, which was subsequently attached to the apparatus to form a part of a closed, leak-tight system. The leak-tightness of the system was verified before each experiment. Experiments were started at ambient air pressure, and the standard ARC procedure of “heat-wait-search” was used. During this procedure, the temperature of the vessel was raised from an initial temperature in increments (heat period) of 5 °C for 0.5 g samples or 2 °C for a larger amount of samples. The vessel was maintained adiabatic during both the wait period (which enables the thermal transients to dissipate) and the search period. During the search period, the ARC system searched for exothermic behavior in the vessel. The system recorded an exotherm whenever the self-heating rate of the sample exceeded a chosen threshold value of 0.02 K min^{−1}. The temperature at which the self-heating rate first exceeded 0.02 K min^{−1} was recorded as the onset temperature. A “true” onset temperature was obtained by extrapolating self-heating rate data to a value of zero. The initial temperature for each “heat-wait-search” experiment was 30 °C. The instrument would terminate each experiment when the following conditions

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