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CHEMICAL ENGINEERING

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Development of a new ammonium nitrate composition: an attempt to prevent misuse of explosives for antisocial activities

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ABSTRACT

The importance of ammonium nitrate (AN) in the production of explosive materials is similar to that in the agricultural industry. However, its use as in explosives has led to it being used for destructive purposes. Therefore, efforts are being made to develop the means to suppress the detonation potential of AN. In this study, AN is diluted with calcium carbonate and dolomite, which serves to increase its nutrient content and decrease its detonation enthalpy. Applying a coating of fluidized bed combustion ash can prevent its application in areas other than the agricultural industry.

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Ammonium nitrate; calcium carbonate; dolomite; FBC ash

1. Introduction

Ammonium nitrate (AN) is the most commonly used nitrogenous fertilizers. In its purest form, however, AN presents several problems. The nitrate content exhibits physicochemical properties that lead to the use of AN in various applications. Compounded with its ready availability and low cost, AN is, unfortunately, attractive for application in areas other than agriculture and, in particular, its high level of enthalpy has led to its application in antisocial endeavors, notably bomb making.

Therefore, researchers have been striving to devise means of preventing the use of AN for such destructive activities. The developed methods have been devised mainly to reduce the detonation enthalpy. However, these problems have not yet been solved, even though the provision of government incentives has ensured that researchers remain dedicated to this area.

AN is produced in several different forms. Therefore, each of these forms requires a different method to prevent the AN from being used as an explosive. Zygmunt and Buczkowski (2007) examined the addition of fuel oil to the spherical (prill) form of AN to suppress its explosive characteristics.

Zygmunt and Buczkowski (2007) examined the reactions between different fuel materials and AN compounds, and their effects on the detonation velocity of AN. In an additional study, the detonation velocity was calculated using the Gurney energy equation. It was found that the detonation velocity of AN was dramatically reduced by adding 50 wt.% of dolomite to an AN mixture (Buczkowski and Zygmunt 2011).

Oommen and Jain (1999) examined the detonation enthalpy of AN, as it is regarded as one of the most important detonation parameters. In their study, they examined the detonation properties of AN mixed with rocket fuel. In another study, the detonation properties of AN mixed with fuel oil (ANFO) were determined (Miyake et al. 2001).

An additional study to investigate the detonation properties of AN was performed by Oxley et al. (2002). They found that the addition of nitrogen compounds, such as urea and oxalate, significantly enhanced the detonation properties of the AN.

The development of techniques to measure detonation parameters, such as the detonation velocity of explosives produced using AN, are vital to understanding the detonation properties of AN. Paszula, Trzciński, and Sprzątczak (2008) used piezoelectric gauges for these measurements.

Another important study was undertaken by Jackson, Kiyanda, and Short (2011), who examined the detonation properties of AN when mixed with aluminum powder. For use in the mining industry, Schoch et al. (2013) devised a means of increasing the detonation properties of AN, using it to form an emulsion.

In a study by Zygmunt (2009), the effects of aluminum powder on the detonation properties of AN were addressed, and the detonation velocity was measured using the Dremin method.

In industry, the effects of various chemical additives on the detonation properties of AN have been investigated. One such study addressed the effects of pyride on the detonation velocity. The pyride was found to decrease the detonation velocity of the AN (Gunawan and Zhang 2009).

The enthalpy of azotetrazolate, another chemical additive, was investigated by Hiskey, Goldman, and Stine (1998). They noted the degradation of the detonation velocity of the AN due to the presence of low-energy chemicals.

A mixture of guanidine nitrate, AN, fuel oil, and aluminum powder exhibited a significantly reduced detonation enthalpy (Maranda 1990).

Benson et al. (2009) studied the detonation features of AN with mass spectrometry, determining the materials that were produced as a result of the detonation.

Recently, the problems resulting from these properties of AN were investigated by Babrauskas (2016).

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Djerdjev et al. (2018) used chemicals such as urea to minimize the presence of NO and HNO_2, which are produced by AN detonation. Furthermore, they investigated the mechanisms of these reactions.

To date, however, an industrial-scale chemical composition that can prevent the detonation of AN has not been devised. With the process described herein, the detonation enthalpy is reduced without compromising the nitrogen content that plants require; meanwhile, the nutrient content is increased with the addition of fluidized bed combustion (FBC) ash, which is used as a coating. The further addition of calcium carbonate and dolomite both decrease the detonation enthalpy. This approach eliminates the detonation problem. As part of our study, calorimetry analyses were undertaken.

2. Materials and methods

2.1. Details of ammonium nitrate (AN) production for use as a fertilizer

In a typical AN production process, 80 ton/h of hot (55°C, 1300 kg/m^3) 50%–70% nitric acid solution and 12 ton/h of ammonia gas (6.1 kg/cm^2, 15°C, 4.3 kg/m^3) are reacted for 1 h in a continuous tubular reactor to afford AN (Figure 1).

Thereafter, calcium carbonate or dolomite is added to dilute AN to obtain the desired composition according to the consumer requirements in the agricultural industry. Among the various methods adopted for suppressing the explosive nature of AN, dilution with calcium carbonate is the most commonly used.

Typically, calcium carbonate is used in the dilution stage, followed by the addition of dolomite; thereafter, the surface of granulated AN is coated with FBC ash. For the production of 26% calcium ammonium nitrate, 250 kg of $CaCO_3$ is required per 1000 kg of AN. In this study, a 50:50 ratio of calcium carbonate and dolomite is used, such that the nitrogen content of the resulting fertilizer does not decrease. During granulation, the amount of FBC ash added to the rotating coating drum is in the range 5%–20% (Figure 1).

2.2. Analysis of detonation properties

The detonation properties, that is, detonation velocity and heat of explosion of the AN fertilizer–fuel mixture were calculated. The detonation velocities of such explosive mixtures were studied in steel tubes with a wall thickness of 3 mm and inner diameter of 36 mm. The theoretical energy of detonation and energy of expansion (Gurney constants of the explosive mixtures; Xie et al. 2007) were determined based on the detonation velocity (Table 1).

2.3. Detonation velocity

Detonation velocity is an important parameter that serves as a measure of the detonation performance of an explosive. In this study, the detonation velocities of various AN mixtures were determined according to the Dautriche method (Tete, Deshmukh, and Yerpude 2013), using cylindrical tubes with 250-mm length, 3-mm wall thickness, and 36-mm inner diameter. Commercial AN mixtures were used, that is, AN mixtures of various compositions were prepared by dilution with calcium carbonate or dolomite, and the addition of FBC ash. The samples were placed in the cylindrical tubes, and detonation was induced by an electrical fuse. In the detonator, the tubes were separated from each other by a distance of 100 mm to protect them from the detonation explosion.

The compositions of AN mixtures and detonation measurement results for different dilution ratios are shown in Table 1.

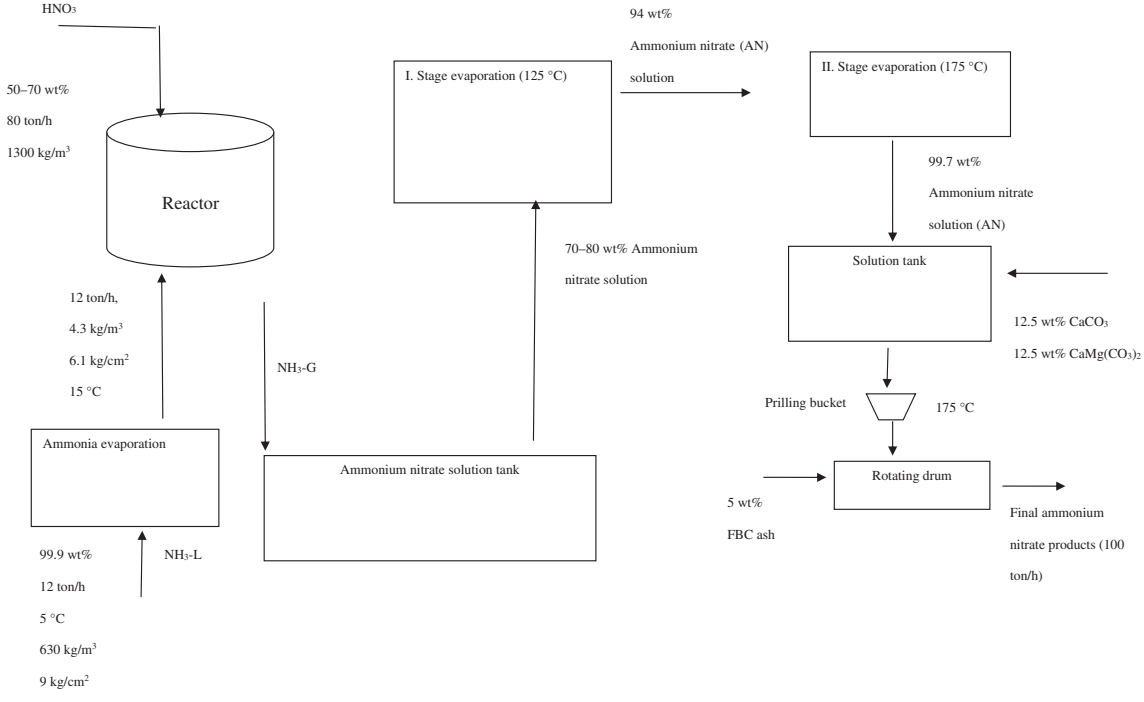


Figure 1. Manufacture of fertilizer involving two evaporation stages.

Table 1. Experimental vs. calculated explosion heats of ammonium nitrate mixtures (containing calcium carbonate, dolomite, and FBC ash) for five experiments.

Calcium carbonate (kg $CaCO_3$/ton AN)	Dolomite (kg dolomite/ton AN)	FBC ash (%)	Heat of explosion (kJ/kg) (Experimental)	Gurney's energy-heat of explosion (kJ/kg) (Calculated)	Detonation velocity (km/s)	Density (kg/m^3)
250	0	0	1480	966	2.900	910
175	75	5	440	330	1.322	899
175	75	10	290	200	0.752	891
125	125	5	195	150	0.503	883
125	125	10	0	0	0	879

The physical aspects of an explosive material, such as porosity, significantly affect its detonation properties. These effects are especially pronounced for AN and its non-explosive mixtures. The most important parameters that can help control the detonation properties of AN and AN-based materials are prill formation and particle size. AN-based mixtures are crushed to the desired sizes to enhance their detonation properties.

Figure 2 shows that fertilizer particles contain an irregular surface after grinding.

The particle sizes are about 1 mm or smaller. Some of the particles have smooth surfaces, while others have rough, non-uniform surfaces.

Surface images of ground AN are shown in the SEM analysis in Figure 2.

Figure 2 shows the surfaces of ground AN after the addition of each chemical additive. The ground AN containing calcium carbonate was shown to have the smallest surface area, while the ground AN containing the mixture of 12.5% calcium carbonate, 12.5% dolomite, and 5% FBC ash exhibited the maximum surface area. Increasing the particle surface area and minimizing the detonation enthalpy of AN corresponds to a decrease in the detonation velocity.

The density of the samples after treatment with calcium carbonate, dolomite, and FBC ash is 1020 kg/m^3 and the kinematic viscosity is 3.03 mm^2 s^{-1} at 40°C. The surface areas of finely ground calcium carbonate, dolomite, and FBC ash are 10–70, 50–100, and 28 cm^2/g, respectively.

The ground AN samples were mixed with fuel oil for the detonation tests. The detonation velocity is determined from Gurney's energy (EG) (Xie et al. 2007).

$$\emptyset = NM^{1/2}Q^{1/2} \tag{1}$$

$$D = 1.01\emptyset^{1/2}(1 + 1.3\rho_0), \tag{2}$$

where N is the moles of gaseous detonation product per gram of explosive, M is the average molecular weight of the gaseous detonation product, Q is the chemical energy of the detonation reaction (kJ/kg), and D is the detonation velocity (Koch, Arnold, and Estermann 2002):

$$E_G = \frac{D}{3} \tag{3}$$

Based on the test results, the detonation velocities of all AN samples continue to decrease. The highest detonation velocity of 3.3 km/s and Gurney's energy of 1500 kJ/kg are observed for pure AN. With an increase in the amounts of dolomite, calcium carbonate, and FBC ash, Gurney's energy and the detonation velocity decrease. For AN fertilizer mixtures containing 25% calcium carbonate, 7.5%–12.5% dolomite, and 5%–20% FBC ash, the explosion heat and detonation velocity dramatically decreased to less than 540 kJ/kg and 1322 m/s, respectively.

2.4. Heat of explosion

The reaction heats for different concentrations of FBC ash, calcium carbonate, and dolomite in AN solution are shown in Table 1. A steel vessel with an inner volume of 5.6 dm^3 was used for the calorimetric tests. The spherical volume of the bomb is filled with 2.0 MPa compressed Ar gas, and the detonation velocity and heat of explosion are determined. However, the gaseous products formed by AN decomposition can react with the FBC ash. For the detonation test, 80 mg of the AN sample was fired by using a standard fuse and 670 mg-RDX (Electric Detonator DYNAWELL, 0015 FDB RDX 1.4S). Two trials were performed for each mixture. The average of the obtained explosion heats is shown in Table 1. Shock waves that are reflected toward the bomb wall can trigger a heat explosion to increase the reaction heat. The final heat measured in the calorimetric system decreased with an increase in the FBC ash content in the AN mixture.

3. Results and discussion

The obtained results can be evaluated as follows. Minerals such as calcium carbonate and dolomite are added to dilute the AN solutions. When the concentration of explosives decreases, the reaction heat of AN containing the calcium carbonate–dolomite–FBC ash mixture also decreases. The addition of dolomite and FBC ash plays an important role in the absorption of heat during the reaction, causing a decrease in the detonation velocity and reaction heat. As a result, when the concentrations of dolomite and FBC ash increase, the detonation explosion decreases.

Table 1 shows the average detonation of different mixtures used in the experiment. As expected, AN mixtures with calcium carbonate, dolomite, FBC ash, and fuel have a lower detonation velocity than those of the mixtures of AN and fuel oil. The detonation velocity of AN and calcium carbonate was 2900 m/s. In this study, the detonation velocity of 100% AN was used as reference. For instance, the AN mixture with 25% calcium carbonate shows 60% detonation velocity; however, the AN mixture containing 17.5% $CaCO_3$, 7.5% dolomite, and 10% FBC ash shows 45% detonation velocity. The experimental energy values and calculated detonation velocities for each concentration of chemicals used are included in Table 1.

The AN, $CaCO_3$, dolomite, and FBC ash mixtures gave better results than those reported in the literature. Thus, AN mixtures with low detonation velocity were successfully obtained.

Moreover, the detonation velocity was found to increase with decreasing particle size (Miyake et al. 2001). This study

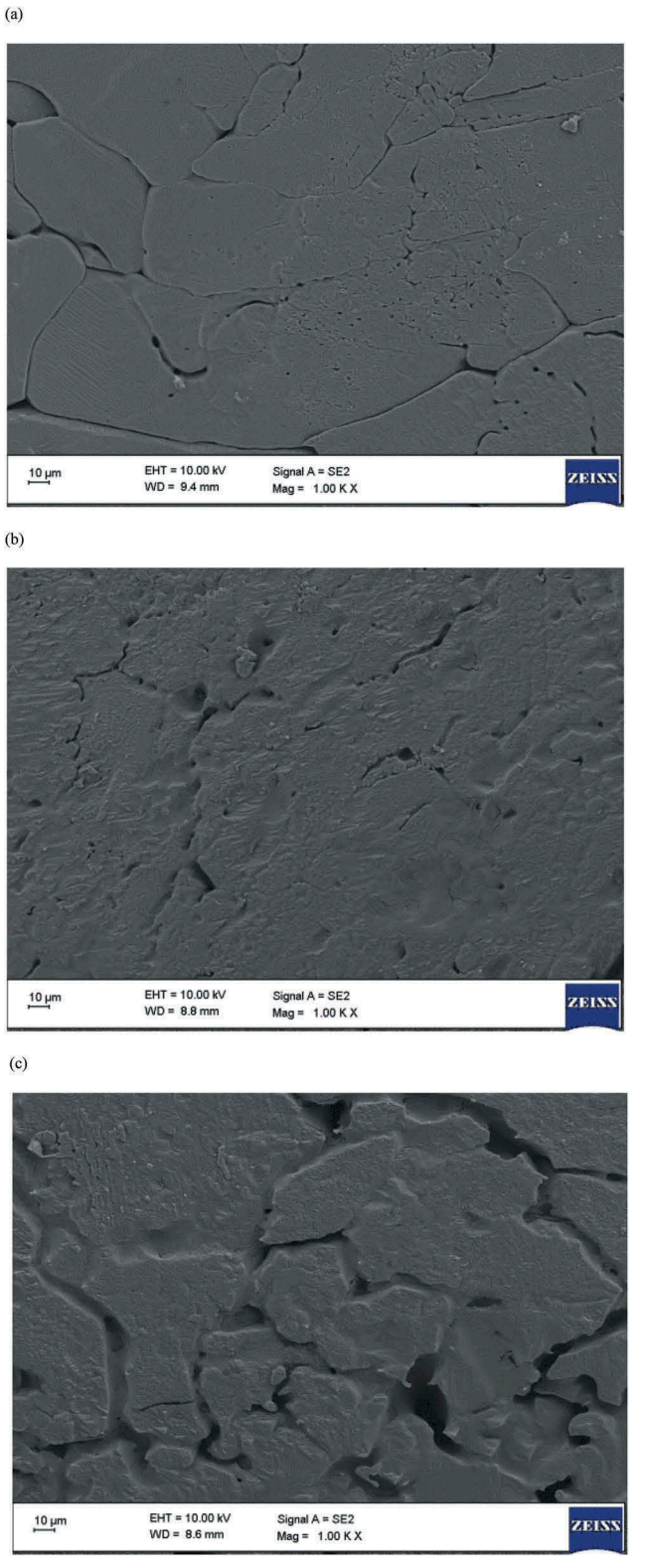


Figure 2. SEM images of ground ammonium nitrate containing (a) limestone ($CaCO_3$), (b) limestone and dolomite mixture, and (c) limestone, dolomite, and FBC ash mixture.

proposes a method that can be applied for the preparation of AN mixtures and to determine the detonation velocity of different AN mixtures. The tests show that the detonation velocity and reaction heat can be used to investigate various explosive mixtures of AN.

For comparison, the explosion heat and volume of gases during the explosion were calculated using the reaction heats of AN, fuel oil, FBC ash, calcium carbonate, and dolomite, as summarized in Table 1.

As shown in the table, the experimentally calculated explosion heat for AN mixtures containing $CaCO_3$ is 1480 kJ/kg. However, upon adding dolomite and FBC ash, the explosion heat decreases significantly (<540 kJ/kg).

The addition of more than 30% $CaCO_3$, dolomite, and FBC ash decreases the explosion enthalpy of AN. However, when using inert materials, the explosive properties can be degraded. Similar to studies performed on fertilizer solutions, this work shows that the addition of a maximum 40% dolomite significantly minimizes the detonation properties of fertilizers. If the amount of dolomite and AN in the fertilizer solution is equal, detonation can be completely prevented. Pure AN mixtures have a higher detonation velocity (3.0 km/s).

The addition of 40% dolomite to the AN solution decreases the detonation velocity to 1.9 km/s. These results are compatible with those reported in the literature (Zygmunt and Buczkowski 2007).

The addition of calcium carbonate (limestone) with dolomite to dilute AN during the AN production process decreases the enthalpy of detonation. However, to decrease the detonation enthalpy and, thus, prevent the misuse of AN for antisocial activities, the surface of granulated AN particles with calcium carbonate and dolomite had to be coated with FBC ash. The presence of these additives led to the formation of various compounds in the reaction environment. Their detonation enthalpies are shown below:

Compound	Percentage (%)
CaO	33
MgO	9
Na_2O	14
$CaSO_3$	16
B_2O_3	3
Fe_2O_3	8
Mn_2O_3	2
ZnO	2
K_2O	8
CuO	5

Below, it is calculated the enthalpies of the reaction between AN and the calcium carbonate/dolomite added in the production process.

Reaction between calcium carbonate and AN:

$$2CaCO_3 + 4NH_4NO_3 \rightarrow Ca(NO_3)_2 + Ca(NH_4NO_3)_2 + 2NH_3 + 2CO_2 + H_2O + {}^1/_2O_2$$

$$\begin{aligned}\Delta H_{reaction} &= \sum \Delta H_{out} - \sum \Delta H_{in} \\ &= \sum (\Delta H_{Ca(NH_4NO_3)_2} + 2\Delta H_{NH_3} \\ &\quad + 2\Delta H_{CO_2} + 2\Delta H_{H_2O} + {}^1/_2\,\Delta H_{O_2}) \\ &\quad - \sum (2\Delta H_{CaCO_3} + 4\Delta H_{NH_4NO_3} \\ &= -295.58 kJ/mol\end{aligned} \quad (4)$$

The reaction between dolomite and AN can be expressed as follows:

$$2CaMg(CO_3)_2 + 8NH_4NO_3 \rightarrow Ca(NO_3)_2 + Mg(NO_3)_2 + Ca(NH_4NO_3)_2 + Mg(NH_4NO_3)_2 + 4NH_3 + 4CO_2 + 2H_2O$$

$$H_{CaMg(CO_3)_2} = \Delta H_{Ca} + \Delta H_{Mg} + 2\Delta H_{CO_2} = -2357.6 kJ/mol, \quad (5)$$

$$\Delta H_{Ca(NO_3)_2} = \Delta H_{Ca} + 2\Delta H_{NO_3} = -1418.2 kJ/mol, \quad (6)$$

$$\Delta H_{Mg(NO_3)_2} = \Delta H_{Mg} + 2\Delta H_{NO_3} = -875.2 kJ/mol. \quad (7)$$

$$\Delta H_{Ca(NH_4NO_3)_2} = \Delta H_{Ca} + 2\Delta H_{NH_4NO_3} = -1273.2 kJ/mol, \quad (8)$$

$$\Delta H_{Mg(NH_4NO_3)_2} = \Delta H_{Mg} + 2\Delta H_{NH_4NO_3} = -1192 kJ/mol, \quad (9)$$

$$\Delta H_{CO_2} = -393.5 \frac{kJ}{mol}; \ \Delta H_{H_2O} = 0 \frac{kJ}{mol}; \ \Delta H_{NH_4} = -1328 kJ/mol, \quad (10)$$

$$H_{\text{reaction between } CaMg(CO_3)_2 \text{ and } NH_4NO_3} = \sum \Delta H_{out} - \sum \Delta H_{in} = \left(\Delta H_{Ca(NO_3)_2} + \Delta H_{Mg(NO_3)_2} + \Delta H_{Ca(NH_4NO_3)_2} + \Delta H_{Mg(NH_4NO_3)_2} + 4\Delta H_{CO_2} + 4\Delta H_{NH_4}\right) - \left(\Delta H_{CaMg(CO_3)_2} + \Delta H_{NH_4NO_3}\right) = +1585.4 kJ/kmol. \quad (11)$$

The compounds in FBC ash minimize the detonation enthalpy. The enthalpy is equal to that of the reaction with the calcium carbonate.

Reaction between CaO in FBC and calcium ammonium nitrate:

$$Ca(NH_4\ NO_3)_2 + CaO \rightarrow CaO + Ca(NH_4\ NO_3)_2$$

$$\Delta H_{\text{reaction between CaO and } Ca(NH_4NO_3)_2} = 0 kJ/kmol. \quad (12)$$

As can be seen from this reaction, CaO does not affect the enthalpy of the calcium ammonium nitrate.

Reaction between calcium ammonium nitrate and MgO in FBC ash:

$$Ca(NH_4\ NO_3)_2 + MgO \rightarrow CaO + Mg(NH_4\ NO_3)_2$$

$$\Delta H_{\text{reaction between MgO and } Ca(NH_4NO_3)_2} = \left(\Delta H_{Mg(NO_3)_2} + \Delta H_{CaO}\right) - \left(\Delta H_{MgO} + \Delta H_{Ca(NH_4NO_3)_2}\right) = 42.7 kJ/kmol. \quad (13)$$

As determined by the enthalpy calculations, the addition of MgO to FBC resulted in an endothermic difference of 42.7 kJ/mol from the detonation enthalpy of AN.

Reaction between calcium ammonium nitrate and Na_2O in FBC ash:

$$Na_2O + Ca(NH_4NO_3)_2 \rightarrow 2Na(NH_4NO_3) + CaO$$

$$\Delta H_{\text{reaction between } Na_2O \text{ and } Ca(NH_4NO_3)_2} = \left(2\Delta H_{Na(NH_4NO_3)_2} + \Delta H_{CaO}\right) - \left(\Delta H_{Na_2O} + \Delta H_{Ca(NH_4NO_3)_2}\right) = -156.58 kJ/kmol. \quad (14)$$

The Na_2O compound in FBC ash emits −156.58 kJ/mol in the reaction with calcium ammonium nitrate.

Reaction between calcium ammonium nitrate and K_2O in FBC ash:

$$K_2O + Ca(NH_4NO_3)_2 \rightarrow 2K(NH_4NO_3) + CaO$$

$$\Delta H_{\text{reaction between } K_2O \text{ and } Ca(NH_4NO_3)_2} = \left(2\Delta H_{K(NH_4NO_3)_2} + \Delta H_{CaO}\right) - \left(\Delta H_{K_2O} + \Delta H_{Ca(NH_4NO_3)_2}\right) = +385.6 kJ/kmol. \quad (15)$$

The addition of K_2O to FBC ash, with its endothermic effect, dramatically decreases the detonation enthalpy of calcium ammonium nitrate.

Reaction between calcium ammonium nitrate and $CaSO_3$ in FBC ash:

$$CaSO_3 + Ca(NH_4NO_3)_2 \rightarrow Ca(NH_4NO_3)_2 + CaSO_3$$

$$\Delta H_{\text{reaction between } CaSO_3 \text{ and } Ca(NH_4NO_3)_2} = \left(2\Delta H_{Ca(NH_4NO_3)_2} + \Delta H_{CaSO_3}\right) - \left(\Delta H_{CaSO_3} + \Delta H_{Ca(NH_4NO_3)_2}\right) = 0 kJ/kmol. \quad (16)$$

As shown in the reaction above, $CaSO_3$ does not exert a significant effect on the detonation enthalpy.

Reaction between calcium ammonium nitrate and B_2O_3 in FBC ash:

$$B_2O_3 + 3Ca(NH_4NO_3)_2 \rightarrow 2B(NH_4NO_3)_3 + 3CaO$$

$$\Delta H_{\text{reaction between } B_2O_3 \text{ and } Ca(NH_4NO_3)_2} = \left(2\Delta H_{B(NH_4NO_3)_2} + 3\Delta H_{CaO}\right) - \left(\Delta H_{B_2O_3} + 3\Delta H_{Ca(NH_4NO_3)_2}\right) = 2145.38 kJ/kmol. \quad (17)$$

The material that has the greatest effect on the calcium ammonium nitrate is the B_2O_3 compound in FBC.

Reaction between calcium ammonium nitrate and CuO in FBC ash:

$$CuO + Ca(NH_4NO_3)_2 \rightarrow Cu(NH_4NO_3)_2 + CaO$$

$$\Delta H_{\text{reaction between CuO and } Ca(NH_4NO_3)_2} = \left(\Delta H_{Cu(NH_4NO_3)_2} + 3\Delta H_{CaO}\right) - \left(\Delta H_{CaO} + 3\Delta H_{Ca(NH_4NO_3)_2}\right) = 1.16 kJ/kmol. \quad (18)$$

The CuO in FBC endothermically affects the detonation enthalpy of calcium ammonium nitrate, having a major effect on the reaction heat (+1.16 kJ/mol)

$$Fe_2O_3 + Ca(NH_4NO_3)_2 \rightarrow 2Fe(NH_4NO_3)_3 + 3CaO$$

$$\Delta H_{\text{reaction between } Fe_2O_3 \text{ and } Ca(NH_4NO_3)_2} = \left(2\Delta H_{Fe(NH_4NO_3)_2} + 3\Delta H_{CaO}\right) - \left(\Delta H_{Fe_2O_3} + \Delta H_{Ca(NH_4NO_3)_2}\right) = -1171.8\text{kJ/kmol}. \quad (19)$$

Fe_2O_3 endothermically affects the enthalpy of calcium ammonium nitrate and, when combined with the effects of other reaction compounds, the effect of Fe_2O_3 is minimized.

Reactions between calcium ammonium nitrate and Mn_2O_3 in FBC ash:

$$Mn_2O_3 + 3Ca(NH_4NO_3)_2 \rightarrow 2Mn(NH_4NO_3)_3 + 3CaO$$

$$\Delta H_{\text{reaction between } Mn_2O_3 \text{ and } Ca(NH_4NO_3)_2} = \left(2\Delta H_{Mn(NH_4NO_3)_2} + 3\Delta H_{CaO}\right) - \left(\Delta H_{Mn_2O_3} + 3\Delta H_{Ca(NH_4NO_3)_2}\right) = -2303.0\text{kJ/kmol}. \quad (20)$$

In the same way as Fe_2O_3, Mn_2O_3 creates exothermic heat when it reacts with calcium ammonium nitrate. However, because it exists in low concentration in the FBC ash and considering the endothermic reactions of the other compounds, the effect of Mn_2O_3 is minimized.

Reaction between ZnO in FBC ash and calcium ammonium nitrate:

$$ZnO + Ca(NH_4NO_3)_2 \rightarrow Zn(NH_4NO_3)_2 + CaO$$

$$\Delta H_{\text{reaction between ZnO and } Ca(NH_4NO_3)_2} = \left(\Delta H_{Zn(NH_4NO_3)_2} + \Delta H_{CaO}\right) - \left(\Delta H_{ZnO} + \Delta H_{Ca(NH_4NO_3)_2}\right) = 105.21\text{kJ/kmol} \quad (21)$$

Another compound that decreases the enthalpy of calcium ammonium nitrate is ZnO in FBC. The presence of the ZnO compound produces an endothermic effect equal to 105.21 kJ/mol.

To determine the overall effect of the coating operation, the total enthalpy of reactions between the calcium ammonium nitrate and the FBC ash compounds can be calculated as follows:

CaO: 33%, MgO: 9%, Na_2O: 14%, SO_3: 29%, B_2O_3: 3%, Fe_2O_3: 8%, Mn_2O_3: 2%, ZnO: 2%

Total enthalpy of the reactions between the FBC compounds and calcium ammonium nitrate.

$$\begin{aligned}\sum \Delta H_{Ca(NH_4NO_3)_2 \text{ and FBCash}} = {} & 0.33\Delta H_{\text{reaction between CaO and } Ca(NH_4NO_3)_2} \\ & + 0.09\Delta H_{\text{reaction between MgO and } Ca(NH_4NO_3)_2} \\ & + 0.14\Delta H_{\text{reaction between } Na_2O \text{ and } Ca(NH_4NO_3)_2} \\ & + 0.08\Delta H_{\text{reaction between } K_2O \text{ and } Ca(NH_4NO_3)_2} \\ & + 0.21\Delta H_{\text{reaction between } CaSO_3 \text{ and } Ca(NH_4NO_3)_2} \\ & + 0.08\Delta H_{\text{reaction between } Fe_2O_3 \text{ and } Ca(NH_4NO_3)_2} \\ & + 0.03\Delta H_{\text{reaction between } B_2O_3 \text{ and } Ca(NH_4NO_3)_2} \\ & + 0.02\Delta H_{\text{reaction between } Mn_2O_3 \text{ and } Ca(NH_4NO_3)_2} \\ & + 0.05\Delta H_{\text{reaction between CuO and } Ca(NH_4NO_3)_2} \\ & + 0.02\Delta H_{\text{reaction between ZnO and } Ca(NH_4NO_3)_2} \\ = {} & -52.468\text{kJ/mol}\end{aligned} \quad (22)$$

As indicated by the calculations above, the detonation enthalpy decreases from −1500 kJ/mol to −52.468 kJ/mol.

Reaction products formed during the exothermic decomposition of AN, in the form of detonation, are shown below. The aim of this study is to prevent the generation of free N, CO_2, CO, and NO_x as detonation products, and to minimize the reaction heat.

As shown in the above calculations, all the reactions of AN particles coated with compounds of FBC ash and the formation of elemental by-products can be minimized or even prevented.

As observed from the results of ion chromatography, when FBC ash, $CaCO_3$, and dolomite are added to AN, detonation does not occur.

The detonation reaction of AN proceeds as follows.

$$(3n+1)NH_4NO_3 + C_nH_{2n+2} \rightarrow (3n+1)N_2 + nCO_2 + (7n+3)H_2O, \ (CO, \text{various } NO_x)$$

(Short 2009)

Therefore, the decomposition of AN coated with FBC ash can be realized by considering this reaction.

From the calculations above, when FBC ash and $CaCO_3$ are used, a small amount of exothermic energy is required for the decomposition of AN to N_2, CO_2, and H_2O (−52.468 kJ/mol, for the reaction of $CaCO_3$ with FBC ash)

Upon the decomposition of calcium ammonium nitrate coated with FBC ash, −52.468 kJ/mol of energy is emitted. Therefore, this reaction has a very low exothermic energy, and no detonation problems are encountered.

Furthermore, ion chromatography analysis was performed for both anions and cations (Figures 3–6). Fe, Zn, Cu, K, Na, Mg, Mn, and Ca ions are detected in the AN solution. With this surface coating, a large amount of CaO is generated, and the detonation of AN occurs, even if the reaction enthalpy is small.

When the detonation enthalpy and heat formed in the reactions between calcium ammonium nitrate, FBC ash, calcium carbonate, and dolomite were evaluated, it was revealed that the content of the compounds in FBC ash decreased dramatically at the detonation enthalpy of AN. The detonation heat of pure AN occurs at 1500 kJ/mol (Speight 2005). With the addition of FBC ash, the detonation heat decreases dramatically in the bomb calorimetry test (Table 2). Ion chromatography can be used to identify the cations and anions in the materials that react before and after detonation. Accordingly, after detonation, the nitrate compounds in the decomposition product do not have sufficiently high enthalpy for detonation. Because these reactions are only slightly exothermic, the addition of FBC ash and dolomite ensures that the detonation enthalpy of AN decreases; therefore, AN containing dolomite–calcium carbonate–FBC ash cannot be used outside the agricultural industry.

A mixture of AN and fuel oil (ANFO) is considered to be a strong explosive that can detonate with a detonation velocity higher than 3 km/s. ANFO is a third-class explosive, which is obtained from the mixture of a fuel and an oxidant with low sensitivity to detonation.

The detonation of AN can be considered as a chemical reaction between a long-chain hydrocarbon (C_nH_{2n+2}) and AN. As a result of this reaction, nitrogen, carbon dioxide, and H_2O are generated. The strongest detonation is observed for a mixture of 94.3% AN and 5.7% fuel oil (Speight 2005). One can

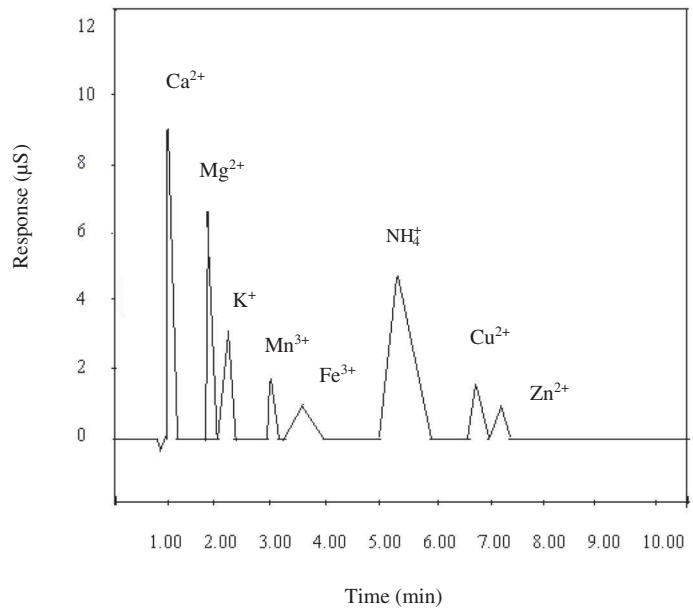


Figure 3. Cation analysis before coating with FBC ash.

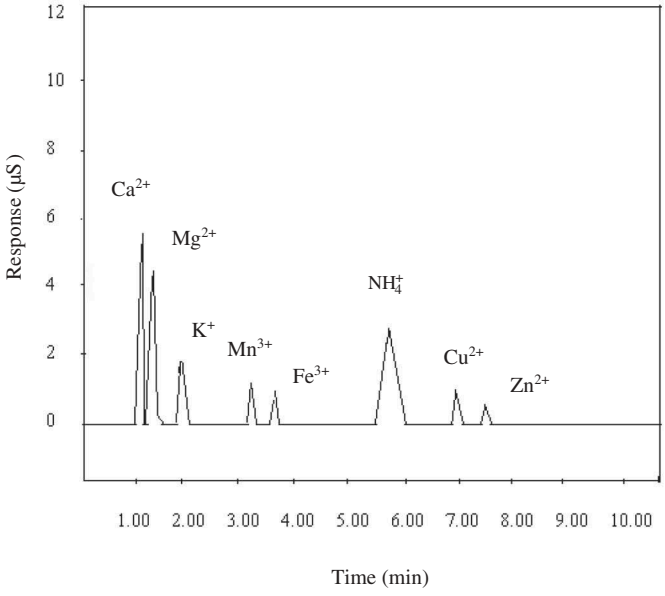


Figure 4. Cation analysis after coating with FBC ash.

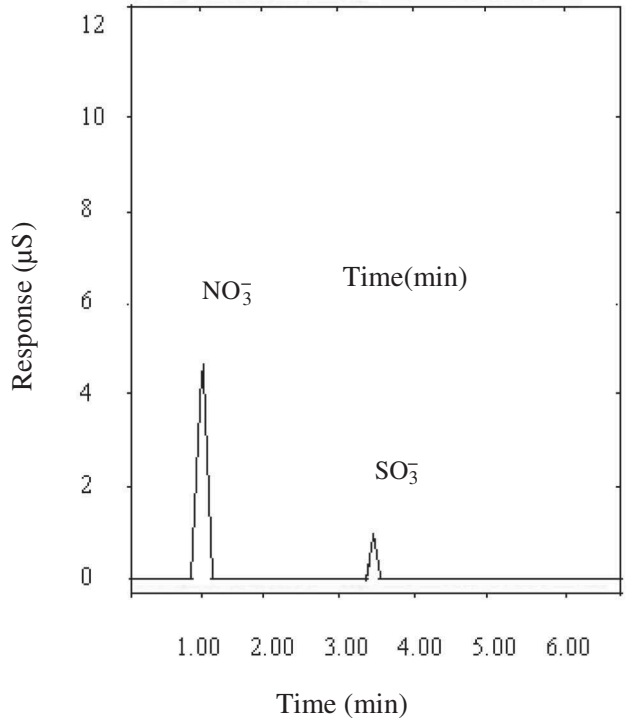


Figure 5. Anion analysis before coating with FBC ash.

expect the same gases as those mentioned above to be formed in the detonation reaction under ideal conditions. However, in practical applications, toxic gasses such as carbon monoxide and NO_x can be formed.

As stated earlier, the aim of the present study is to prevent the N_2, CO, CO_2, and NO_x gases that are created as a result of the detonation reaction of AN, while minimizing the reaction heat. Similar to the calculated reaction enthalpies of AN containing calcium carbonate, the reaction enthalpy of AN containing dolomite and FBC ash were calculated. The reaction enthalpies can be calculated as described below.

As observed in the reactions, upon the detonation of AN, the involvement of the materials that contain dolomite and that are coated with FBC ash is minimized.

On the other hand, another AN compound with which the FBC ash reacts is calcium magnesium ammonium nitrate, so-called dolomite-containing AN. The reactions between the FBC compounds and dolomite-containing AN, as well as their reaction enthalpies, are described below.

The CaO in the FBC ash reacts with the dolomite-containing AN as follows:

$$CaMg(NH_4NO_3)_2 + CaO \rightarrow Ca(NH_4NO_3)_2 + CaMgO$$

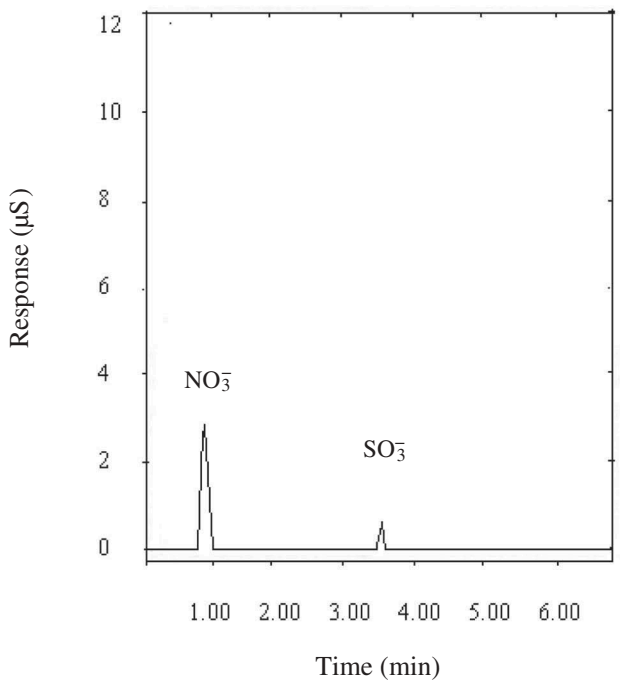


Figure 6. Anion analysis after coating with FBC ash.

Table 2. Chemical additives and their effects on detonation for all inorganic chemicals used in the study.

Compounds	Reaction enthalpy (kJ/mol)
CaO	−634.9
MgO	−601.6
Mn_2O_3	−962.3
ZnO	−350.5
$CaSO_3$	−277
B_2O_3	2017.28
CuO	−157.3
Na_2O	−416
$CaMg(NH_4NO_3)_2$	−1741
$Ca(NH_4NO_3)_2$	−1274
Fe_2O_3	−825.5
CaMgO	−1144.6
$Mg(NH_4NO_3)_2$	−1198
$MgSO_3$	−862.7
$Na(NH_4NO_3)$	−605.84
$K(NH_4NO_3)$	−1233.4
$B(NH_4NO_3)_3$	−522.46
$Fe(NH_4NO_3)_3$	−683.3
$Mn(NH_4NO_3)_3$	−1317.3
$Zn(NH_4NO_3)_2$	−884.39
$Cu(NH_4NO_3)_2$	−795.24
K_2O	−363.2
$CaMg(NH_4NO_3)_2$ and FBC ash	48.7744
$Ca(NH_4NO_3)_2$ and FBC ash	−52.468

$$\begin{aligned}\Delta H_{\text{reaction between CaO and } CaMg(NH_4NO_3)_2} &= \left(\Delta H_{CaMg(NH_4NO_3)_2} + \Delta H_{CaMgO}\right) \\ &\quad - \left(\Delta H_{CaO} + \Delta H_{CaMg(NH_4NO_3)_2}\right) \\ &= 42.7\text{kJ/mol}.\end{aligned} \tag{23}$$

As seen from this reaction, the CaO is exothermically affected to a lesser extent than the dolomite-containing AN.

Reaction between MgO in FBC ash and dolomite-containing AN:

$$CaMg(NH_4NO_3)_2 + MgO \rightarrow Mg(NH_4NO_3)_2 + CaMgO$$

$$\begin{aligned}\Delta H_{\text{reaction between MgO and } CaMg(NH_4NO_3)_2} &= \left(\Delta H_{Mg(NH_4NO_3)_2} + \Delta H_{CaMgO}\right) \\ &\quad - \left(\Delta H_{MgO} + \Delta H_{CaMg(NH_4NO_3)_2}\right) \\ &= 0\text{kJ/mol}.\end{aligned} \tag{24}$$

As shown in the calculations above, the MgO in the FBC ash has slight effect on dolomite-containing AN.

Reaction between dolomite-containing AN and Na_2O in FBC ash:

$$CaMg(NH_4NO_3)_2 + Na_2O \rightarrow 2Na(NH_4NO_3) + CaO + MgO$$

$$\begin{aligned}\Delta H_{\text{reaction between } Na_2O \text{ and } CaMg(NH_4NO_3)_2} &= \left(2\Delta H_{Mg(NH_4NO_3)_2} + \Delta H_{CaO} + \Delta H_{MgO}\right) \\ &\quad - \left(\Delta H_{Na_2O} + \Delta H_{CaMg(NH_4NO_3)_2}\right) \\ &= -291.18\text{kj/mol}.\end{aligned} \tag{25}$$

Na_2O affects the detonation enthalpy by an amount equal to –291 kJ/mol, having only a small amount of exothermic energy.

Reaction between K_2O in FBC and dolomite-containing AN:

$$CaMg(NH_4NO_3)_2 + K_2O \rightarrow 2K(NH_4NO_3) + CaO + MgO$$

$$\begin{aligned}\Delta H_{\text{reaction between } K_2O \text{ and } CaMg(NH_4NO_3)_2} &= \left(2\Delta H_{K(NH_4NO_3)_2} + \Delta H_{CaO} + \Delta H_{MgO}\right) \\ &\quad - \left(\Delta H_{K_2O} + \Delta H_{CaMg(NH_4NO_3)_2}\right) \\ &= -365.7\text{kJ/mol}.\end{aligned} \tag{26}$$

The K_2O in FBC exothermically affects the reaction environment. However, it has slight effect on the detonation enthalpy.

Reaction between $CaSO_3$ in FBC and AN:

$$CaMg(NH_4NO_3)_2 + CaSO_3 \rightarrow Ca(NH_4NO_3)_2 + CaMgSO_3$$

$$\begin{aligned}\Delta H_{\text{reaction between } CaSO_3 \text{ and } CaMg(NH_4NO_3)_2} &= \left(2\Delta H_{Ca(NH_4NO_3)_2} + \Delta H_{CaMgSO_3}\right) \\ &\quad - \left(\Delta H_{CaMg(NH_4NO_3)_2} + \Delta H_{CaSO_3}\right) \\ &= 0\text{kJ/mol}.\end{aligned} \tag{27}$$

$CaSO_3$ does not affect the detonation enthalpy of dolomite-containing AN.

Reaction between B_2O_3 in FBC ash and dolomite-containing AN.

$$3CaMg(NH_4NO_3)_2 + B_2O_3 \rightarrow 3CaO + MgO + 2Ba(NH_4NO_3)_3$$

$$\begin{aligned}\Delta H_{\text{reaction between } B_2O_3 \text{ and } CaMg(NH_4NO_3)_2} &= \left(2\Delta H_{Ba(NH_4NO_3)_3} + \Delta H_{MgO} + 3\Delta H_{CaO}\right) \\ &\quad - \left(\Delta H_{CaMg(NH_4NO_3)_2} + \Delta H_{B_2O_3}\right) \\ &= 2017.28\text{kJ/mol}.\end{aligned} \tag{28}$$

Despite the low concentration of B_2O_3 in FBC, it endothermically affects the dolomite-containing AN by 2017.28 kJ/mol.

Reaction between CuO in FBC ash and dolomite-containing AN:

$$CuO + CaMg(NH_4NO_3)_2 + {}^1\!/_2O_2 \rightarrow CaO + MgO + Cu(NH_4NO_3)_2,$$

$$\begin{aligned}&\Delta H_{\text{reaction between CuO and } CaMg(NH_4NO_3)_2}\\&= \left(\Delta H_{Cu(NH_4NO_3)_3} + \Delta H_{MgO} + \Delta H_{CaO}\right)\\&\quad - \left(\Delta H_{CaMg(NH_4NO_3)_2} + \Delta H_{\Delta H_{CuO}} + {}^1\!/_2\Delta H_{O_2}\right)\\&= -133.44\text{kJ/mol}\end{aligned} \tag{29}$$

The CuO in the FBC has minimal effect on the detonation enthalpy of the dolomite-containing AN, given that it has a very low enthalpy (−133.44 kJ/mol).

Reaction between Fe_2O_3 in FBC ash and dolomite-containing AN:

$$Fe_2O_3 + 3CaMg(NH_4NO_3)_2 + {}^3\!/_2O_2 \rightarrow 3CaO + 3MgO + 2Fe(NH_4NO_3)_3,$$

$$\begin{aligned}&\Delta H_{\text{reaction between } Fe_2O_3 \text{ and } CaMg(NH_4NO_3)_2}\\&= \left(2\Delta H_{Fe(NH_4NO_3)_3} + 3\Delta H_{MgO} + 3\Delta H_{CaO}\right)\\&\quad - \left(\Delta H_{CaMg(NH_4NO_3)_2} + \Delta H_{\Delta H_{Fe_2O_3}} + {}^3\!/_2\Delta H_{O_2}\right)\\&= 972.4\text{kJ/mol}\end{aligned} \tag{30}$$

This means that the compound that is most effective at minimizing the detonation enthalpy of dolomite-containing AN is Fe_2O_3 in FBC. To decompose the AN requires 972.3 kJ/mol of energy.

Reaction between Mn_2O_3 in FBC ash and dolomite-containing AN:

$$Mn_2O_3 + 3CaMg(NH_4NO_3)_2 + {}^3\!/_2O_2 \rightarrow 3CaO + 3MgO + 2Mn(NH_4NO_3)_3,$$

$$\begin{aligned}&\Delta H_{\text{reaction between } Mn_2O_3 \text{ and } CaMg(NH_4NO_3)_2}\\&= \left(2\Delta H_{Mn(NH_4NO_3)_3} + 3\Delta H_{MgO} + 3\Delta H_{CaO}\right)\\&\quad - \left(\Delta H_{CaMg(NH_4NO_3)_2} + \Delta H_{\Delta H_{Mn_2O_3}} + {}^3\!/_2\Delta H_{O_2}\right)\\&= -1203.8\text{kJ/mol}\end{aligned} \tag{31}$$

The Mn_2O_3 in FBC ash does not contribute to the detonation enthalpy of the dolomite-containing AN, as it is present in very low concentration.

Reaction between ZnO in FBC ash and dolomite-containing AN:

$$ZnO + CaMg(NH_4NO_3)_2 + {}^1\!/_2O_2 \rightarrow CaO + MgO + Zn(NH_4NO_3)_2$$

$$\begin{aligned}&\Delta H_{\text{reaction between ZnO and } CaMg(NH_4NO_3)_2}\\&= \left(\Delta H_{Zn(NH_4NO_3)_3} + \Delta H_{MgO} + \Delta H_{CaO}\right)\\&\quad - \left(\Delta H_{CaMg(NH_4NO_3)_2} + \Delta H_{\Delta HZnO} + {}^1\!/_2\Delta H_{O_2}\right)\\&= -29.39\text{kJ/mol}\end{aligned} \tag{32}$$

The ZnO in the FBC ash was found to decrease the detonation enthalpy of dolomite-containing AN.

The total enthalpies of reactions between compounds of FBC ash and dolomite-containing AN ($CaMg(NH_4NO_3)_2$) can be calculated as shown below:

CaO: 33%, MgO: 9%, Na_2O: 14%, $CaSO_3$: 29%, B_2O_3: 3%, Fe_2O_3: 8%, Mn_2O_3: 2%, ZnO: 2%

$$\begin{aligned}&\sum \Delta H_{CaMg(NH_4NO_3)_2 \text{ and FBCash}}\\&= 0.33\Delta H_{\text{reaction between CaO and } CaMg(NH_4NO_3)_2}\\&\quad + 0.09\Delta H_{\text{reaction between MgO and } CaMg(NH_4NO_3)_2}\\&\quad + 0.14\Delta H_{\text{reaction between } Na_2O \text{ and } CaMg(NH_4NO_3)_2}\\&\quad + 0.08\Delta H_{\text{reaction between } K_2O \text{ and } CaMg(NH_4NO_3)_2}\\&\quad + 0.21\Delta H_{\text{reaction between } CaSO_3 \text{ and } CaMg(NH_4NO_3)_2}\\&\quad + 0.08\Delta H_{\text{reaction between } Fe_2O_3 \text{ and } CaMg(NH_4NO_3)_2}\\&\quad + 0.03\Delta H_{\text{reaction between } B_2O_3 \text{ and } CaMg(NH_4NO_3)_2}\\&\quad + 0.02\Delta H_{\text{reaction between } Mn_2O_3 \text{ and } CaMg(NH_4NO_3)_2}\\&\quad + 0.05\Delta H_{\text{reaction between CuO and } CaMg(NH_4NO_3)_2}\\&\quad + 0.02\Delta H_{\text{reaction between ZnO and } CaMg(NH_4NO_3)_2}\\&= 8.7744\text{kJ/mol}\end{aligned} \tag{33}$$

In this manner, the detonation enthalpy of the AN decreases to a low exothermic value (48.7744 kJ/mol), and the detonation problem is minimized.

The use of a mixture of calcium ammonium nitrate and dolomite-containing AN affects the detonation enthalpy to such a degree that the detonation problem of the AN is completely prevented. This can be expressed as follows:

$$\begin{aligned}\sum \Delta H_{NH_4NO_3} &= \sum \Delta H_{Ca(NH_4NO_3)_2} + \sum \Delta H_{CaMg(NH_4NO_3)_2}\\&= 24.1562\text{kJ/mol}.\end{aligned} \tag{34}$$

When reactions of each material with calcium ammonium nitrate and calcium magnesium ammonium nitrate (AN containing dolomite) were investigated, it was found that more endothermic energy than that needed to decompose AN is required. Thus, the conditions required for detonation do not occur. Moreover, the reaction enthalpy decreases extremely rapidly in accordance with the reaction enthalpy of AN.

Finally, when calcium carbonate and dolomite are added to the AN solution, after which the AN is coated with FBC ash, the conditions required for the detonation of the AN do not occur. In the same manner, the calculations showed that the minimum energy required to decompose N_2, CO_2, and H_2O from AN could be realized at a low-energy level. Therefore, detonation does not occur.

The detonation of AN can be considered as a chemical reaction between a long chain hydrocarbon (C_nH_{2n+2}) and AN. As a result of this reaction, nitrogen, carbon dioxide, and H_2O are generated. The strongest detonation is observed for a mixture of 94.3% AN and 5.7% fuel oil (Speight 2005). The same gases as those mentioned above can be expected to be formed in the detonation reaction under ideal conditions. However, in practical applications, toxic gasses such as carbon monoxide and NO_x can be formed.

4. Conclusions

This study aimed at the use of additives such as calcium carbonate, dolomite, and FBC ash in the industrial production of AN to prevent detonation and its associated risks. With increasing concentrations of the additives, the detonation velocities of AN mixtures decreased gradually. The enthalpy decreased with the addition of FBC ash and dolomite to AN mixtures diluted using calcium carbonate. The systems with these three additives produced less heat than did AN to which only calcium carbonate was added. The explosion heat determined in bomb calorimetric tests was lower than the calculated value. This is because high concentrations of FBC ash and dolomite would react with the gaseous products obtained after the composition of AN in the bomb.

Nomenclature

$\emptyset$	Detonation constant
N	Moles of gaseous detonation products per g of explosive (mol gas/g explosive)
M	Average molecular weight of detonation product gas (g gas/mol gas)
Q	Chemical energy of detonation reaction (cal/g)
D	Detonation velocity (m/s)
ρ_0	Density (kg·m^{-3})
E_G	Gurney's energy
ΔH	Enthalpy

Disclosure statement

No potential conflict of interest was reported by the authors.

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