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DETONATION PROPERTIES OF AMMONIUM NITRATE CONTAINING CALCIUM CARBONATE, DOLOMITE, AND FLY ASH

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Because of its low cost and easy availability, ammonium nitrate (AN) is widely used in the agricultural industry; however, the explosive properties of this compound are often exploited for misuse in illegal activities. To minimize the use of AN outside the agricultural industry and to meet the nitrogen requirements of AN-based fertilizers, we attempted to use calcium carbonate, dolomite, and fly ash in different concentrations. These additives helped in reducing the explosion heat and detonation velocity of the resulting AN mixtures. According to the results of calorimetric tests, the AN mixture with a composition of 17.5% $CaCO_3$, 7.5% dolomite, and 20% fly ash dramatically decreased the reaction enthalpy, thereby eliminating the possibility of detonation problems.

KEY WORDS: *ammonium nitrate, calcium carbonate, dolomite, fly ash, detonation, explosive heat*

1. INTRODUCTION

Ammonium nitrate (AN) is widely known to be an important fertilizer. Increasing consumer requirements have triggered the production of AN as well as the modification of various properties of this compound. However, AN is inexpensive, and its explosive decomposition can be easily induced; hence, there is increased concern regarding the possible misuse of this compound in preparing explosives for antisocial activities. For this reason, the governments of many countries have imposed strict limitations on the industrial-scale production and use of AN, resulting in consumer demands for fertilizers not being met. This in turn has led to increased cost of AN-based fertilizers and restricted use of such fertilizers in agricultural fields.

Several formulations and processes aimed at suppressing the detonation probability of AN have been proposed by researchers. Although various chemical compositions have been designed and applied to degrade the detonation properties of this compound, these are not intended for meeting consumer requirements. In addition, there are several patented studies on the detonation properties of AN. Resende et al. (2014) attempted to degrade the detonation properties of AN by diluting AN solution with fillers such as calcium ammonium nitrate. They also replaced the fuel oil in ammonium nitrate/fuel oil (ANFO), which is a widely used bulk industrial explosive mixture, with rice straw, corn cob, sugarcane bagasse, and biodiesel (Resende et al., 2014).

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Zygmunt and Buczkowski (2007) investigated the effects of the porosity of AN, the level of oil adsorption on AN in ANFO, and the granulometric distribution on the detonation properties of ANFO. The results showed that the detonation velocity of ANFO is increased significantly with increasing porosity and decreasing size of AN prills/granules (Zygmunt and Buczkowski, 2007).

In another study, Buczkowski and Zygmunt (2011) attempted to suppress the detonation probability of AN by diluting it with different concentrations of dolomite and by coating its surface with diesel oil. No detonation occurred when 40% dolomite was used for dilution and the AN surface was coated with 3.5% diesel. Jackson et al. (2011) demonstrated that the addition of aluminum to ANFO mixtures, such that it filled the AN pores, led to a decrease in the detonation velocity. Miyake et al. (2001) successfully prevented the detonation of AN on the industrial scale. They prepared ANFO samples using the same AN pore diameter and pore volume and found that the detonation velocity (3.85 km/s) was the highest for the smallest particle diameter (<0.85 mm). After a 12 month aging period, the detonation velocities of ANFO changed, indicating that the reaction between ANFO was influenced by the physical and chemical properties of the AN particles and oil during the storage. In a similar study, Oyumi et al. (1996) investigated the combustion properties of a glycidyl azide polymer (GAP)–AN mixture and found that GAP addition reduced the heat of explosion to 1500 kJ/kg, which is insufficient to induce the explosion of AN. Voreck (1983) also attempted to mitigate the detonation of AN. Ornellas (1982) attempted to determine the calorimetric value for mixtures of AN-based propellants or explosives; however, this study could not be extended to the industrial scale. Miyake et al. (2007) developed different mixtures of AN with fluorine and sulfur in various compositions to suppress the detonation of AN; in particular, the use of pentafluorothioethylorthocarbonate gave good results (Der Van Steen et al., 1990). This research group also performed an industrial-scale study on the combustion and ignition properties of a mixture of AN and activated carbon (Miyake et al., 2007). In yet another study, it was found that ANFO containing prilled AN would show less probability for detonation when the pore size of AN was <0.85 mm (Kołaczkowski et al., 2000). Chan and Turner (1998) succeeded in minimizing the detonation ability of AN by using a mixture of 70% AN, 10% nitroguanidine, and 20% 3-nitro-1, 2, 4-triazol-5-one.

The detonation properties of AN in pyrotechnic reactions, which form the basis of fireworks, were also studied (Chan and Turner, 1998; Brown and Rugunanan, 1988), where the detonation heat was decreased to 1500 kJ/kg, which is the permitted limit in the explosives industry. The detonation velocity is less than 1.5 m/s when the detonation heat is within the aforementioned limit, and thus the explosion risk of AN is mitigated. Brown and Rugunanan (1988) reported that mixing nitrate fertilizers with other fertilizers such as urea and potassium nitrate could limit the detonation of AN. For example, a mixture of AN with guanyl urea nitrate and perchlorate salts has been proven to be effective in preventing detonation (Klapötke and Sabaté, 2008). Deribas and Simonov (1999) investigated the detonation properties of AN in different physical tests carried out under firing range conditions by the d'Autriche method. In a preliminary series of laboratory experiments (with charges of 400 × 180 × 60 mm in size), the results obtained with the d'Autriche method were verified on the same test stand using ionization probes, and the response time was measured with a Ch3-34 A frequency meter. Sil'vestrov and Plastinin (2009) determined the detonation violence of AN in an emulsion solution. They used emulsion explosives (EEs) with a detonation velocity up to 2–3 km/s. The compositions contained a large amount of hollow glass microspheres. Low detonation velocity EEs were used to clad metal plates with foils up to 0.1 mm in thickness and to weld a small-diameter tube to a steel collar in a parallel explosive welding arrangement.

Brousseau et al. (2002) attempted to prevent detonation by adding nanometer-sized aluminum powder to an AN solution. Ostmark et al. (2002) investigated the detonation properties of a mixture of ammonium dinitramide and AN. A similar patented study was performed by Mueller and Cziesla (1993). Fleming (1996) reported a patented study on the detonation properties of AN mixtures stabilized with binders, plasticizers such as trimethylolethane trinitrate, or dessicants, and successfully reduced the explosion risk of AN. For producing explosives with low detonation velocity, Mousavi et al. (2005) devised a process based on the Williamsburg equation of state. Boyars (1978) showed that the risk of accidental detonation of large masses of commercial AN during handling and storage under normal climatic conditions can be reduced by replacing some of the AN with a solid solution of potassium nitrate (0–50 wt%). Kimura and Oyumi (1996) successfully improved the combustion properties of AN, which have a marked effect on the heat of detonation, by adding the necessary proportion of potassium nitrate.

However, the detonation problem has not been eliminated in any of these studies since none of the proposed additives or strategies were fully effective. Thus, in the present study, we attempted to decrease the detonation velocity and explosion heat of AN by adding calcium carbonate, dolomite, and fly ash, which are easy to handle and readily available. Calorimetric analysis was performed to confirm the effectiveness of the aforementioned chemicals in preventing the detonation of AN.

2. MATERIALS AND METHODS

2.1 Details of AN Production for Use as a Fertilizer

In a typical ammonium nitrate production process, hot (55°C) 50%–70% nitric acid solution and ammonia gas (6 bar 10°C) to afford AN (Fig. 1). Then, calcium carbonate or dolomite is added to dilute AN, and thus obtain the desired composition as per the consumer requirements in the agricultural industry. Among the various methods adopted to suppress 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, and then the surface of prilled AN is coated with fly ash. For 26% calcium AN production, 250 kg of $CaCO_3$ is required per 1000 kg AN. In this study, a calcium carbonate and dolomite ratio of 50:50 was used such that the nitrogen content of the resulting fertilizer was not decreased. During prilling, the fly ash content added to the rotating coating drum was in the range of 10%–50%.

A common industrial process for AN production is described here. According to this process, anhydrous ammonia (99.9% w/w, 10°C) is heated to 22°C at 8 kg/cm^2. Then, to produce AN in a tubular reactor, vaporized ammonia is heated and allowed to react with the produced nitric acid (55%w/w) at 50°C by oxidation of anhydrous ammonia. Thus, an AN solution with 70%–80% concentration is obtained. Before prilling, this solution is sent to an evaporator for the removal of water. Multiple evaporators can be used to remove water in the AN solution more effectively. In this study, to decrease the water content in the AN solution, two evaporators were used. The first evaporation was achieved at 125°C, and the second evaporation was realized at 175°C. Thus, water in the AN solution was removed gradually. As a result of this evaporation, the concentration of AN solution became approximately 97%. After obtaining the AN solution with this concentration, limestone ($CaCO_3$), dolomite [$CaMg(CO_3)_2$], and fly ash were added.

In this study, ASTM C618-85 Class F fly ash (CAS No: 68131-74-8) was used, which is produced and supplied by the PG&E Generating Company (Kennerdell, Pennsylvania, USA). Limestone ($CaCO_3$, CAS No: 1317-65-3) was supplied by Lafarge North America Inc. (Reston,

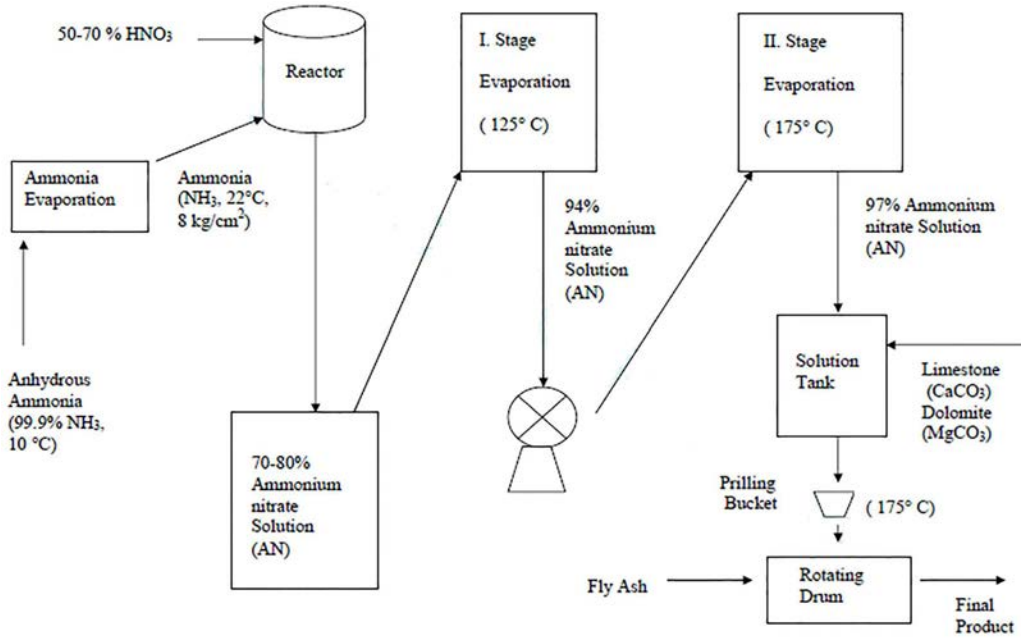


FIG. 1: Fertilizer manufacturing process involving two evaporation stages

Virginia, USA) Dolomite [$CaMg(CO_3)_2$; CAS No: 16389-88-1] was supplied by Longcliffe Quarries Ltd. (Hopton, Wirksworth, UK). Anhydrous ammonia was supplied by the Qatar Fertilizer Company (Mesaieed, Qatar).

2.2 Analysis of the Detonation Properties

The detonation properties, i.e., the detonation velocity and heat of explosion of a mixture of AN fertilizer and fuel, were calculated. The detonation velocities of such explosive mixtures were studied in steel tubes having a wall thickness of 3 mm and inner diameter of 36 mm. Theoretical energies of detonation and energy of expansion (Gurney constants of the explosive mixtures) (Xie et al., 2005) were determined based on the detonation velocity (Table 1).

2.3 Detonation Velocity

The 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 (Kosanke and Kosanke, 1992) using cylindrical tubes (50 mm in length, 3 mm wall thickness, and 125 mm inner diameter). In the Dautriche method, a 125-mm-diameter and 50-cm-long detonation column is used. Typically, in this method, 10–50 g samples can be used (Kosanke and Kosanke, 1992); however, in this study 50 g AN mixtures were employed.

When an explosive detonates, shock waves are distributed through the length of the explosive in the cylindrical Dautriche blasting system. Because of this detonation effect, in the Dautriche method, shock waves move from one measurement point to the next. The detonating

TABLE 1: PDV probe position parameters during detonation

Probe	Cylinder (50.8 mm)		Cylinder (76.2 mm)	
	Distance, z (mm)	Distance, r (mm)	Distance, z (mm)	Distance, r (mm)
1	100	145	150	150
2	100	145	150	155
3	200	145	300	145
4	200	145	300	145
5	300	145	450	160
6	300	145	450	160
7	500	125	600	158
8	500	125	600	145

fuse generates two shock waves at both points. During detonation, the point at which the two shock waves collide on the lead plate is considered the deformation point. In the Dautriche method, a detonation column is filled with the AN mixture, and a detonating fuse links this column from two different points and the lead plate. After the AN mixture detonates, the strain caused by the two shock waves on the lead plate by the detonating fuse that is linked to the column is recorded. The detonation velocity is calculated from the distance between the point of collision of the shock waves and the mid-point of the detonating fuse (Kosanke and Kosanke, 1992):

$$D_u = D_f \left(\frac{d_1}{2d_2} \right)$$

where D_u = detonation velocity of the detonating column; D_f = detonation velocity of the detonating fuse; d_1 = linked point of the detonating fuse; and d_2 = distance between the meeting point of the shock waves and the mid-point of the detonating fuse.

The Dautriche method is not suitable for all explosives. Pyrotechnic materials do not produce shock pressures; therefore, their detonation velocity cannot be measured by this method. For comparison of the calculated detonation velocities, the Dautriche method test and cylindrical tube test were performed. Commercial AN mixtures were used, i.e., AN mixtures of various compositions were prepared by dilution with calcium carbonate or dolomite and the addition of fly ash. The samples were placed in the cylindrical tubes, and detonation was induced by an electrical fuse. The tubes were separated by a distance of 100 mm from one another in the detonator in order to protect them from detonation violence. The compositions of the AN mixtures and the detonation measurement results for different dilution ratios are given in Table 1. The physical aspects of an explosive material, such as the 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 improve the detonation properties of AN and AN-based materials are prill formation and particle size. AN-based materials are crushed to the desired sizes to enhance their detonation properties.

Figure 2 shows the surface of granulated AN after the addition of each chemical additive such as limestone, dolomite, and fly ash. Under similar granulation conditions, as per the surface analysis, AN that contains $CaCO_3$ has the smallest particle and the largest surface area and detonation enthalpy. With the addition of fly ash to AN containing $CaCO_3$ and dolomite, the surface area increases but the detonability decreases. Increasing the particle surface area and minimizing

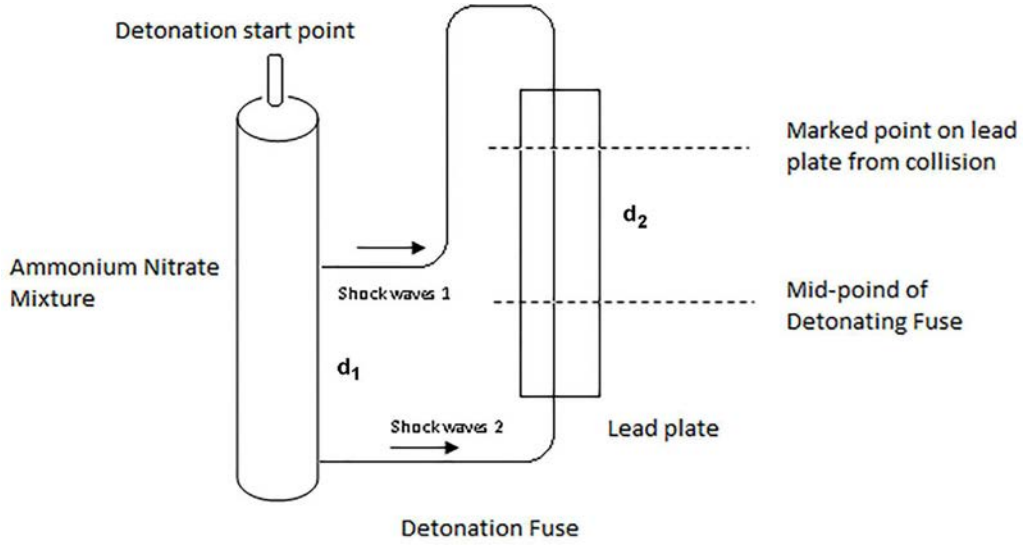


FIG. 2: Measurement of detonation velocity by the Dautriche method

the detonation enthalpy of AN suppresses the detonation tendency (Chaturvedi and Dave, 2013). Figure 3 shows fertilizer particles that have an irregular surface shape 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.

The density of the samples after treatment with calcium carbonate, dolomite, and fly ash was 1020 kg/m^3 and the kinematic viscosity was 3.03 mm^2/s^{-1} at 40°C. The surface areas of finely ground calcium carbonate, dolomite, and fly ash were 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 as follows based on Gurney's energy (E_G) (Xie et al., 2005):

$$\phi = NM^{1/2}Q^{1/2}$$

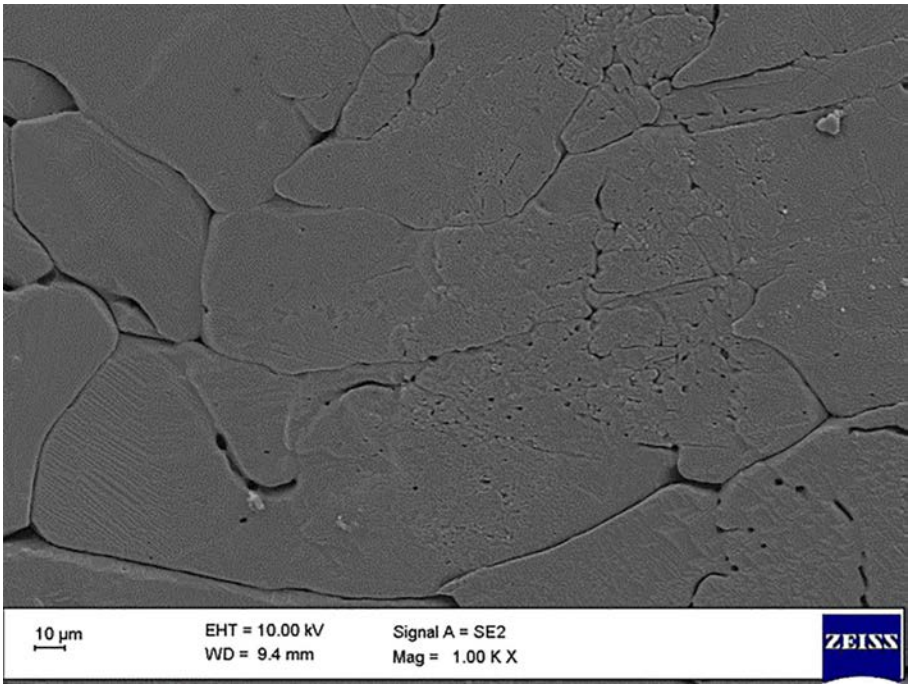
$$D = 1.01\phi^{1/2}\,(1 + 1.3\rho_0)$$

where N is the gaseous detonation product per gram of explosive (in mol); 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 et al., 2002):

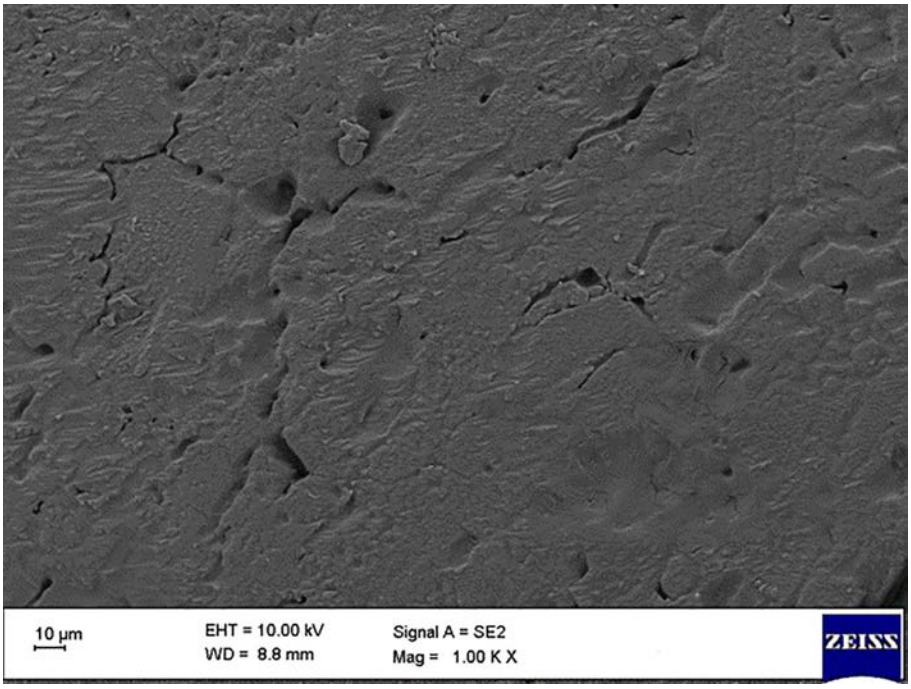
$$E_{\mathrm{G}} = \frac{D}{3}$$

As per the test results, the detonation velocities of all of the AN samples continued to decrease. The highest detonation velocity of 3.3 km/s and Gurney's energy were observed for pure AN. With an increase in the amounts of dolomite, calcium carbonate, and fly ash, the Gurney's energy and detonation velocity decreased. For AN fertilizer mixtures containing 25% calcium carbonate, 7.5%–12.5% dolomite, and 10%–20% fly ash the explosion heat and detonation velocity dramatically decreased to less than 540 kJ/kg and 1322 m/s, respectively.

In this study, two detonation tests were performed, a cylindrical test (Anderson et al., 2017) and a Dautriche test. The cylindrical test is a well-known method for evaluating explosive performance, and it is used to measure the detonation energy. In this test, AN is filled in an oxygen-free

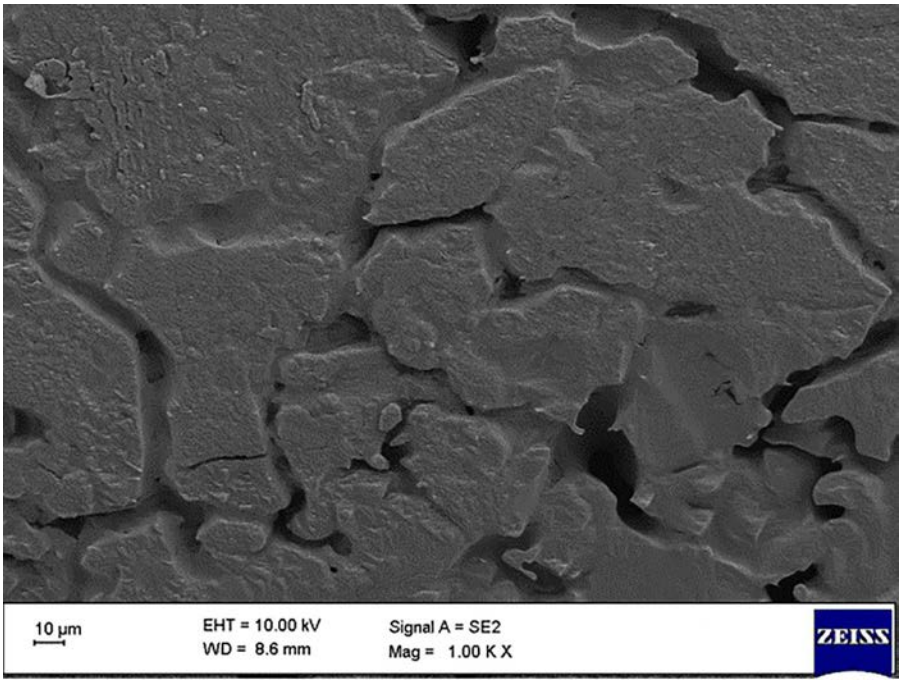

10 µm
EHT = 10.00 kV
WD = 9.4 mm
Signal A = SE2
Mag = 1.00 K X
ZEISS

(a)


10 µm
EHT = 10.00 kV
WD = 8.8 mm
Signal A = SE2
Mag = 1.00 K X
ZEISS

(b)

FIG. 3



(c)

FIG. 3: Scanning electron microscope images of granulated AN containing (a) limestone, (b) limestone + dolomite, and (c) limestone + dolomite + fly ash

high-conductivity copper cylindrical tube. During detonation by a photonic Doppler velocimetry (PDV) probe, the cylindrical tube wall expands due to deformation and the wall movement before and after the explosion is recorded. For comparison, measurements are made using two different tubes (50.8 and 72.6 mm) over eight different points. Here, r denotes the displacement of the cylindrical wall and z is the distance from the bottom of the cylindrical wall (Fig. 4). As shown in Fig. 4, the PDV probe is positioned 90°to the cylindrical tube. For this test, the AN samples are granulated in a Jaw Crusher BB 50 (Zhengzhou Yifan Machinery Co., Zhengzhou, China) grinding mill for 10 min to achieve a particle size of 850 µm.

2.4 Heat of the Explosion

The reaction heats for different concentrations of fly ash, calcium carbonate, and dolomite in the AN solution are given 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 was filled with 2.0 MPa compressed Ar gas, and the detonation velocity and heat of the explosion were determined. However, the gaseous products formed by AN decomposition can react with the fly ash. For the detonation test, 80 mg of the AN samples was fired by using a standard fuse and 670 mg of cyclotrimethylenetrinitramine (RDX) (Electric Detonator DYNAWELL, 0015 FDB RDX 1.4S, DynaEnergetics, Troisdorf, Germany). Two trials were performed for each mixture. The average values of the obtained explosion heats are given in Table 1. Shock waves that are reflected to the bomb wall can trigger heat explosion to increase the reaction heat. The final heat

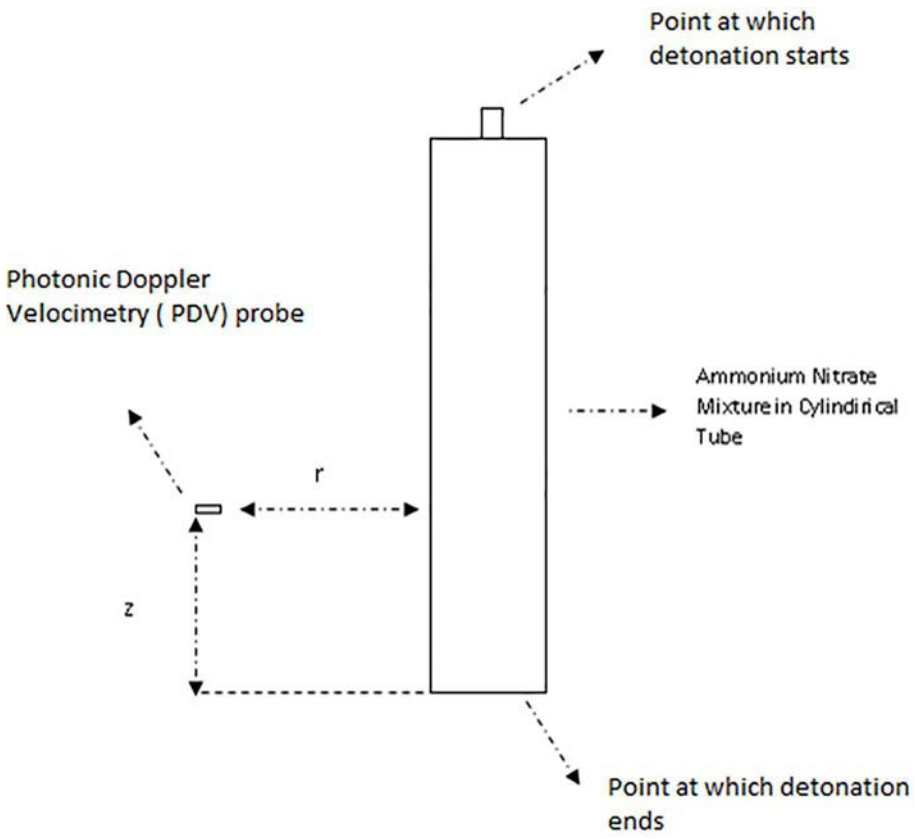


FIG. 4: Cylinder tube test performed for two different PDV probe positions (50.8 and 76.2 mm) for measurement of detonation velocity

measured in the calorimetric system decreased with an increase in the fly ash content in the AN mixture.

In the calorimetric tests, a bomb calorimeter was used, with 80 mg AN in which the particle size was 1.0–2.5 mm. The particle size of AN was reduced to 850 µm by granulation in a D50 ball mill. The aim of the test was to measure the amount of energy generated during AN combustion. For all of the prepared AN mixtures, combustion tests were carried out. The lowest combustion energy of 0 kJ/kmol was observed during the combustion of the AN mixture containing 12.5% $CaCO_3$, 12.5% dolomite, and 20% fly ash. The maximum combustion energy of 1480 kJ/kmol was generated during the combustion of the AN mixture containing $CaCO_3$. Unlike in the combustion energy calculations, the detonation velocities of the AN mixtures were calculated using the Gurney energy. The detonation velocity was investigated using the Dautriche method. The lowest detonation velocity was obtained for the AN mixture containing 12.5 $CaCO_3$, 12.5% dolomite, and 20% fly ash. In addition, the highest detonation velocity was obtained for the AN mixture containing 25% $CaCO_3$. All of the combustion energy values and detonation velocities are given in Table 2.

The combustion test is less expensive than the detonation test and requires simpler equipment and less sophisticated technology. For these reasons, it has become more practical to use.

TABLE 2: Experimental versus calculated explosion heats of AN mixtures that contain $CaCO_3$, dolomite, and fly ash

Calcium Carbonate (kg $CaCO_3$/ton AN)	Dolomite (kg dolomite/ton AN)	Fly ash (%)	Explosion Heat (kJ/kg) (Experiment)	Gurney's Energy/Explosion Heat (kJ/kg) (Calculated)	Detonation Velocity (km/s)	Density (kg/m^3)
250 (25%)	0	0	1480	966	2.900	910
175 (17.5%)	75 (7.5%)	10	540	440	1.322 (45%)	905
175 (17.5%)	75 (7.5%)	20	320	250	0.752 (30%)	900
125 (12.5%)	125 (12.5%)	10	270	167	0.503 (15%)	896
125 (12.5%)	125 (12.5%)	20	0	0	0 (0%)	891

Therefore, when detonation tests cannot be performed, the combustion energy values can give a good approximation of the energy used in AN mixtures.

3. RESULTS AND DISCUSSION

The obtained results can be evaluated as follows. Chemicals such as calcium carbonate and dolomite are added to dilute AN solutions. When the concentration of explosives is decreased, the reaction heat of AN mixtures containing calcium carbonate, dolomite, and fly ash also decreases. The addition of dolomite and fly ash plays an important role in the absorption heat during the reaction, causing a decrease in the detonation velocity and reaction heat. As a result, when the concentrations of dolomite and fly ash are increased, the detonation violence decreases.

Table 1 gives the average detonation results for the different mixtures used in the experiment. As expected, AN mixtures with calcium carbonate, dolomite, fly ash, and fuel have lower detonation velocities than those of mixtures with AN and fuel oil. The detonation velocity of the AN and calcium carbonate mixtures was obtained as 2900 m/s. In this study, the detonation velocity of the AN mixtures was taken as the reference when the mixtures contained 100% AN. For instance, the AN mixture with 25% calcium carbonate showed 60% detonation velocity; however, the AN mixture containing 17.5% $CaCO_3$, 7.5% dolomite, and 10% fly ash showed 45% detonation velocity. The other cases are depicted in Table 2.

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

Moreover, the detonation velocity was found to increase with decreasing particle size (Miyake et al., 2001). This study proposes a method to be applied as a reference 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 values of the explosion heat and volume of gasses during the explosion were calculated using the reaction heats of AN, fuel oil, fly ash, calcium carbonate, and dolomite, as summarized in Table 2. As can be seen from Table 2, the experimentally calculated explosion heat for AN mixtures containing $CaCO_3$ is 1480 kJ/kg. However, when adding dolomite and fly ash the explosion heat decreases significantly (<540 kJ/kg).

The addition of more than 30% $CaCO_3$, dolomite, and fly ash decreases the explosion enthalpy of AN. Thus, AN and fuel mixtures have excellent explosion properties. However, when

using inert materials, the explosive properties can be degraded. Similar to studies performed in the literature for fertilizer solutions, this work shows that the addition of a maximum of 40% dolomite significantly minimizes the detonation properties of fertilizers. If the amount of dolomite and AN in the fertilizer solution are equal, the detonation can be completely prevented. Pure AN mixtures have higher detonation velocity (3.0 km/s); 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).

To clarify the behavior of ammonium nitrate that is illegally mixed with aluminum, the detonation velocity and detonation enthalpy were measured for a mixture of ammonium nitrate containing 25% aluminum (Paszula et al., 2008) with the chemical additives used in this study, i.e., fly ash (20%), calcium carbonate (12.5%), and dolomite (12.5%). The measured detonation velocity and detonation enthalpy values were 0.128 km/h and 116 kJ/kg, respectively. Thus, the detonation ability of the mixture of ammonium nitrate, fuel oil, and aluminum that is used illegally was well mitigated. Moreover, the addition of aluminum did not notably increase the detonation velocity and detonation enthalpy of ammonium nitrate containing fly ash, calcium carbonate, and dolomite suggested for preventing detonation.

4. CONCLUSIONS

This study aimed to investigate the use of additives such as calcium carbonate, dolomite, and fly ash in industrial production of AN in order 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 fly ash and dolomite to AN mixtures diluted using calcium carbonate. The systems with these three additives produced less heat than did the AN mixture to which only calcium carbonate was added. The explosion heat determined in the bomb calorimetric tests was lower than the calculated value. This is because high concentrations of fly ash and dolomite reacted with the gaseous products obtained after the decomposition of AN in the bomb.

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