

A new chemical approach for preventing the anti-social use of ammonium nitrate

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ABSTRACT

The aim of the present study is to limit the detonation properties of ammonium nitrate fertilizers used in agriculture. The results presented in this paper are obtained by using analytical methods. This study also evaluated the physiochemical properties of nitrogenous fertilizers such as the specific heat, explosive enthalpy, and the effects of dolomite and fly ash additives that have been suggested for increasing the nutritional content of ammonium nitrate fertilizers. The results obtained regarding additives differ from those of previous studies. The present study produced important results regarding additives, and efforts were made to limit certain physical properties such as the detonation enthalpy and detonation velocity.

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1. Introduction

There are several problems associated with the production and use of ammonium nitrate. Generally, these challenges relate to the specific heat and ignition temperature during combustion, as well as the detonation enthalpy of ammonium nitrate. Several studies have attempted to solve these problems. In the study performed by Babrauskas, a method was developed for solving the problem of detonation during the storage and transfer of ammonium nitrate. This method is now being applied industrially [1]. In another study, Anderson *et al.* measured the detonation velocity of a mixture of ammonium nitrate and aluminum with a cylinder test wall [2]. In the study, detonation velocities were measured for ammonium-nitrate fuel-oil (ANFO), which has been reported as having a limited specific heat [3]. Tan *et al.* limited the specific heat of ammonium nitrate through the addition of urea [4]. The same researchers investigated how mixtures of ammonium nitrate and other nitrogenous fertilizers have different detonation velocities [5]. Another study of the detonation properties of an ammonium nitrate mixture was performed by Yamamoto *et al.* [6]. The effect of sodium chloride on the detonation properties of ammonium nitrate was investigated by Tan *et al.* [7]. The determination of the effects of propellants on the detonation

properties and specific heat of ammonium nitrate is one of the most important developments affecting the industry [8].

Using several different testing methods, the thermal decomposition of ammonium nitrate was investigated by Chaturvedi [9]. To thermally decompose ammonium nitrate, the ReaxFF reactive force field method was proposed by Shan [10]. The specific heat of a mixture of carbonate salts and ammonium nitrate was investigated by Popławski *et al.* [11]. Dana *et al.* reported that mixing ammonium nitrate with liquid urea decreased the detonation property of the former [12]. Also, important observations regarding the limitation of the detonation properties of ammonium nitrate were made by Buczkowski [13], as well as its use in industry. To reduce the detonation velocity of ammonium nitrate, reactions between sodium salts and ammonium nitrate were investigated by Cagnina [14].

A calorimetric study of the detonation enthalpy of ammonium nitrate in the presence of an inhibitor was conducted by Han *et al.* [15]. The thermal decomposition of the ammonium nitrate with various additives was investigated and a less energetic ammonium nitrate material than a mixture with liquid urea was obtained [16]. Taulbee *et al.* conducted a study of fly ash mixtures to reduce the detonation property of ammonium nitrate [17].

$$125 \text{ kg CaMg(CO}_3)_2 \times \frac{1 \text{ kmol CaMg(CO}_3)_2}{184 \text{ kg CaMg(CO}_3)_2} \times \frac{1 \text{ kmol Mg}}{1 \text{ kmol CaMg(CO}_3)_2} \times \frac{24 \text{ kg Mg}}{1 \text{ kmol Mg}} = 16.3 \text{ kg Mg} \quad (1)$$

Lotspeich conducted another study of the detonation properties of ammonium nitrate prills [18]. In the study performed by Robbins, the detonation enthalpy was dramatically reduced, as a result of the addition of aluminum powder to the ammonium nitrate [19]. The study undertaken by Kweeder *et al.* on the thermal decomposition of ammonium nitrate is one of the most important patented industrial works [20]. In a study investigating the decreased detonation enthalpy of pure ammonium nitrate with no mineral additives, Araos *et al.* reported on the importance of the dilution temperature with carbonate salts during the ammonium nitrate production process [21]. The effects of the ammonium nitrate and tetrazoles on the detonation enthalpy of an ammonium nitrate solution were investigated by Hasue *et al.* [22]. Buczkowski *et al.* used dolomite and diesel oil to prevent the detonation of ammonium nitrate [23].

2. Ammonium nitrate production process

Using the pressurized neutralization process for ammonium nitrate, calcium ammonium nitrate was produced at a rate of 100 ton/h. This contained 26% N. A total of 99.5% of the anhydrous ammonia was gasified in the ammonia vessels and the ammonia gas was passed through a neutralizer. In addition, nitric acid (55%, w:w) and sulfuric acid (98%, max 1 ppm) were added to the neutralizer. In the neutralizer, under pressure, the nitric acid and ammonia reacted to produce 76% ammonium nitrate. When this ammonium nitrate was heated in a vacuum in a heat exchanger, it vaporized and was then separated, with the obtained ammonium nitrate solution (95%) being stored in the process vessel. Thereafter, the solution was re-heated in a heat exchanger, and its temperature was raised to 169 °C, the freezing point of ammonium nitrate, in a second vaporizer under a vacuum to attain a 99.5% concentration. This solution was mixed with calcium carbonate to produce an ammonium nitrate solution containing 26% nitrogen. This mixture was sprayed from the prilling tower using a prilling bucket and was cooled by air circulation in the prilling tower. The resulting cooled prills were sent to a fluid drum for further cooling. Subsequently, to prevent the caking and degradation of the prills, the surfaces of the prills were coated with an anticaking agent and calcium carbonate [24].

3. Experimental

3.1. Ammonium nitrate production method

The present study was based on the ammonium nitrate production process with the main reaction being between nitric acid (55%) and anhydrous ammonia at a pressure of 5.6 bar, producing an ammonium nitrate solution with a minimum concentration of 80%. Nutrition materials and chemical additives that support the chemical and physical strength can be added to the obtained ammonium nitrate solution, according to the desired requirements. Since this study is based on a pressurized process, without changing the freezing point of the ammonium nitrate, calcium carbonate, calcium, and magnesium carbonate were added in the dilution step of the process and fly ash was added in the surface-coating step.

3.2. Progress analysis

In the experimental study, samples were coated with 20% (average value) fly ash at the coating level with 10-50% fly ash, as described in the literature [17]. For each experiment,

500 g of 26% calcium ammonium nitrate and 20% fly ash (100 g) were weighed, and then heated to 100 °C using a lab-scale oven. A process ambience for the calcium ammonium nitrate and its coating material were studied and created [17].

4. Results and discussion

In the first step of the experimental study, a 500 g sample of fertilizer and 100 g of fly ash were heated to 100 °C and were then fed into a rotating drum at 1400 rpm for 15 min. However, because the fly ash adheres to the wall of the rotating drum, the cycle time of the rotating drum fell to 5 min at the wall of the rotating drum, due to the temperature, so adhesion to the wall of the rotating drum was not observed.

The heat of the combustion reaction of the fly-ash-coated ammonium nitrate was measured using a calorimetric bomb. The aim was to measure the heat reaction between the ammonium nitrate and fuel derivatives, as would occur in antisocial use, and to measure how these additives affect the heat produced by the combustion reaction.

In addition to the above, an analysis of the combustion velocity of the ammonium nitrate was also performed under atmospheric conditions. For the investigation of the combustion velocities of the ammonium nitrate samples under atmospheric conditions (the experimental materials), diesel as a fuel derivative for the combustion reaction, aluminum foil to assure the maximum contact with the reaction heat, and the use of an open flame was selected. For this study, ammonium nitrate and fly ash, containing 26% nitrogen, were utilized as the main experimental material. As a result, the burning speed of a mixture of calcium ammonium nitrate and diesel is markedly affected when it burns with an open flame. A smaller amount of burned material was detected at the end of the 30 s reaction between the fly-ash-coated calcium ammonium nitrate (another sample) and the diesel. In this experiment, the burning speed of the diesel was unaffected, but the 50 g ammonium nitrate/fly ash sample did not burn completely, such that a large proportion (20 g) of the sample became waste material.

Among the parameters that affect the burning speed in the reaction between ammonium nitrate and fly ash, the melting point of the ammonium nitrate is regarded as being important. This study analyzed the melting point of ammonium nitrate, as was the effect of flammable materials on the burning progress during contact with diesel. The storage conditions were also addressed. It is expected that this parameter will affect the burning speed.

In the studied process, the effect of dolomite that is detected as an important chemical additive to the specific heat of ammonium nitrate was determined by detecting the material concentration in the ammonium nitrate samples. With this aim, the concentrations of chemical additives were analyzed by ion chromatography for certain results. According to the results of ion chromatography, the magnesium concentrations of samples that have two samples of material coated with kaolin and fly ash, as well as two other samples of materials coated only with fly ash, are given as 1) 448 mg/kg; 2) 401 mg/kg; 3) 461 mg/kg and 4) 368 mg/kg.

Based on these results, to determine the dolomite concentration, the obtained results can be expressed when the ammonium nitrate solution is diluted with 125 kg of calcium carbonate and 125 kg of dolomite. The amount of dolomite that should be added to ammonium nitrate with 26% nitrogen content is 125 kg. Therefore, to evaluate the obtained results from ion chromatography, the amount of magnesium in 125 kg of dolomite must be calculated (Equation 1).

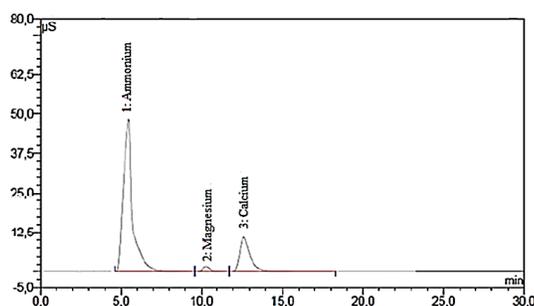
$$15472 \frac{\text{mg}}{\text{L}} \text{NO}_3 \times \frac{1 \text{mmolNO}_3}{62 \text{mgNO}_3} \times \frac{1 \text{mmolN}}{1 \text{mmolNO}_3} \times \frac{14 \text{mgN}}{1 \text{mmolN}} = 3493.67 \frac{\text{mg}}{\text{L}} \text{N} \quad (2)$$

$$622 \frac{\text{mg}}{\text{L}} \text{NH}_4 \times \frac{1 \text{mmolNH}_4}{18 \text{mgNH}_4} \times \frac{1 \text{mmolN}}{1 \text{mmolNH}_4} \times \frac{14 \text{mgN}}{1 \text{mmolN}} = 483.78 \frac{\text{mg}}{\text{L}} \text{N} \quad (3)$$

$$3493.67 \frac{\text{mg}}{\text{L}} \text{N} + 483.78 \frac{\text{mg}}{\text{L}} \text{N} = 3977.44 \frac{\text{mg}}{\text{L}} \text{N} \quad (4)$$

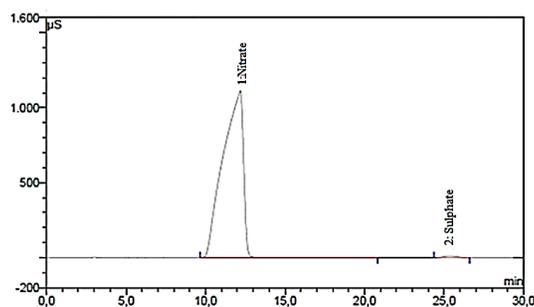
$$15472 \frac{\text{mg}}{\text{L}} + 622 \frac{\text{mg}}{\text{L}} = 16094 \frac{\text{mg}}{\text{L}} \text{NH}_4 \text{NO}_3 \quad (5)$$

$$\frac{3972.44 \times 100}{16.094} = 24.71\% \text{ (Nitrogen concentration for no. 1 calcium ammonium nitrate)} \quad (6)$$



Peak no	Component name	Retention time, min	Area, µs.min	Height, µS	Amount, ppm	Relative amount, %
1	Ammonium	5.48	32.489	48.407	622.8895	82.88
2	Magnesium	10.29	1.083	1.702	10.0015	1.33
3	Calcium	12.63	8.718	10.718	118.6390	15.79

Figure 1. Cation analysis in the absence of kaolin and fly-ash-coated ammonium nitrate.



Peak no	Component name	Retention time, min	Area, µs.min	Height, µS	Amount, ppm	Relative amount, %
1	Nitrate	12.19	1648.834	1113.097	15472.2364	99.46
2	Sulphate	25.36	11.580	11.527	84.1731	0.54

Figure 2. Anion analysis in the absence of kaolin and fly-ash-coated ammonium nitrate.

Using this calculation, the amount of added dolomite was evaluated, as the amount of magnesium ions in the ambient reaction can be neglected (0.1304 kg/kg). On the other hand, before the start of the study, the nitrogen concentration of the calcium ammonium nitrate to be coated with fly ash was measured and determined to be 25.64%. According to the results obtained using ion chromatography, the nitrogen concentration was determined after coating with fly ash.

The nitrogen concentration of the first sample, which was coated with fly ash but without kaolin, was determined by ion chromatography analysis (Figure 1 and 2) (Equation 2-6).

The nitrogen concentration of the second sample, which was coated with fly ash but which had no kaolin, was deter-

mined by ion chromatography analysis (Figure 3 and 4) (Equation 7-11).

The nitrogen concentration of the third sample, coated with fly ash and kaolin, was determined by ion chromatography analysis (Figures 5 and 6) (Equation 12-16).

The nitrogen concentration of the fourth sample, which was coated with both fly ash and kaolin, was determined by ion chromatography analysis (Figures 7 and 8) (Equation 17-21).

As illustrated by the calculations above, the nitrogen concentrations of the fly-ash-coated ammonium nitrate are close to 25% and are acceptable for the nitrogen concentration of the ammonium nitrate production process, which normally demands 26%.

$$17102.31 \frac{\text{mg}}{\text{L}} \text{NO}_3 \times \frac{1 \text{mmolNO}_3}{62 \text{mgNO}_3} \times \frac{1 \text{mmolN}}{1 \text{mmolNO}_3} \times \frac{14 \text{mgN}}{1 \text{mmolN}} = 3861.81 \frac{\text{mg}}{\text{L}} \text{N} \quad (7)$$

$$676.08 \frac{\text{mg}}{\text{L}} \text{NH}_4 \times \frac{1 \text{mmolNH}_4}{18 \text{mgNH}_4} \times \frac{1 \text{mmolN}}{1 \text{mmolNH}_4} \times \frac{14 \text{mgN}}{1 \text{mmolN}} = 525.84 \frac{\text{mg}}{\text{L}} \text{N} \quad (8)$$

$$17102.31 \frac{\text{mg}}{\text{L}} + 676.08 \frac{\text{mg}}{\text{L}} = 17778.39 \frac{\text{mg}}{\text{L}} \text{NH}_4\text{NO}_3 \quad (9)$$

$$3861.81 \frac{\text{mg}}{\text{L}} \text{N} + 525.84 \frac{\text{mg}}{\text{L}} \text{N} = 4387.65 \frac{\text{mg}}{\text{L}} \text{N} \quad (10)$$

$$\frac{4387.65 \times 100}{17778.39} = 24.67\% \text{ (Nitrogen concentration for no. 2 calcium ammonium nitrate)} \quad (11)$$

$$16682.31 \frac{\text{mg}}{\text{L}} \text{NO}_3 \times \frac{1 \text{mmolNO}_3}{62 \text{mgNO}_3} \times \frac{1 \text{mmolN}}{1 \text{mmolNO}_3} \times \frac{14 \text{mgN}}{1 \text{mmolN}} = 3766.97 \frac{\text{mg}}{\text{L}} \text{N} \quad (12)$$

$$671.64 \frac{\text{mg}}{\text{L}} \text{NH}_4 \times \frac{1 \text{mmolNH}_4}{18 \text{mgNH}_4} \times \frac{1 \text{mmolN}}{1 \text{mmolNH}_4} \times \frac{14 \text{mgN}}{1 \text{mmolN}} = 522.38 \frac{\text{mg}}{\text{L}} \text{N} \quad (13)$$

$$3766.97 \frac{\text{mg}}{\text{L}} \text{N} + 522.38 \frac{\text{mg}}{\text{L}} \text{N} = 4289.35 \frac{\text{mg}}{\text{L}} \text{N} \quad (14)$$

$$16682.31 \frac{\text{mg}}{\text{L}} + 671.64 \frac{\text{mg}}{\text{L}} = 17353.95 \frac{\text{mg}}{\text{L}} \text{NH}_4\text{NO}_3 \quad (15)$$

$$\frac{4289.35 \times 100}{17353.95} = 24.71\% \text{ (Nitrogen concentration for no. 3 calcium ammonium nitrate)} \quad (16)$$

$$18574.97 \frac{\text{mg}}{\text{L}} \text{NO}_3 \times \frac{1 \text{mmolNO}_3}{62 \text{mgNO}_3} \times \frac{1 \text{mmolN}}{1 \text{mmolNO}_3} \times \frac{14 \text{mgN}}{1 \text{mmolN}} = 4194.34 \frac{\text{mg}}{\text{L}} \text{N} \quad (17)$$

$$723.67 \frac{\text{mg}}{\text{L}} \text{NH}_4 \times \frac{1 \text{mmolNH}_4}{18 \text{mgNH}_4} \times \frac{1 \text{mmolN}}{1 \text{mmolNH}_4} \times \frac{14 \text{mgN}}{1 \text{mmolN}} = 562.85 \frac{\text{mg}}{\text{L}} \text{N} \quad (18)$$

$$4194.34 \frac{\text{mg}}{\text{L}} \text{N} + 562.85 \frac{\text{mg}}{\text{L}} \text{N} = 4757.19 \frac{\text{mg}}{\text{L}} \text{N} \quad (19)$$

$$18574.97 \frac{\text{mg}}{\text{L}} + 723.67 \frac{\text{mg}}{\text{L}} = 19298.64 \frac{\text{mg}}{\text{L}} \text{NH}_4\text{NO}_3 \quad (20)$$

$$\frac{4757.19 \times 100}{19298.64} = 24.65\% \text{ (Nitrogen concentration for no. 4 calcium ammonium nitrate)} \quad (21)$$

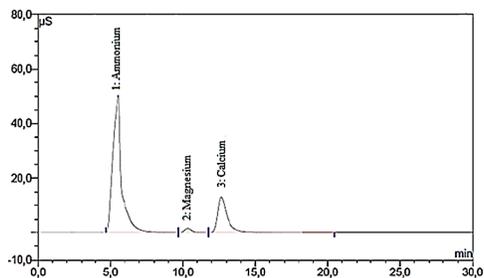
4.1. Detection of reaction heat by bomb calorimetry

For this analysis, an IKA C 4000 calorimetric bomb device was utilized. Four ammonium nitrate samples were prepared. From the results of the analysis, it appeared that these four samples did not burn. Therefore, a TG-DTA analysis was performed for these four samples. The results of the TG-DTA analysis indicate that the samples did not burn. Detailed results of the TG-DTA analysis are presented in [Figure 9](#).

4.2. TG-DTA analysis results

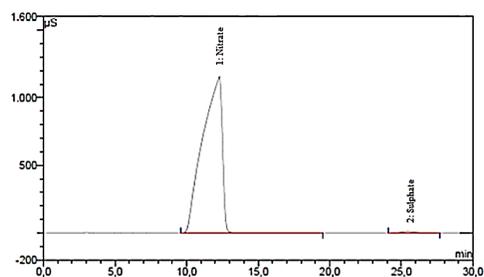
A thermal analysis of the fly-ash-coated ammonium nitrate samples was performed at 25-1035 °C at a 10 °C/min heating ratio with dry air, by using the SII Nanotechnology Mark SII6000Exstar TG/DTA 6300 device. According to the results

of the thermal analysis ([Figure 9](#)), the weight loss of the fertilizer samples was 33.4% between 27 and 304 °C. Thus, the weight loss of the fertilizer samples was recorded as 19.2% between 304 and 1000 °C. When the DTA/TG curves were investigated, endothermic peaks were observed at 241 and 547 °C, which corresponded to the weight loss. This decrease in weight loss can be expressed by the removal of gasses from the ammonium nitrate samples. Weight losses below 200 °C are caused by the presence of crystallized water in the ammonium nitrate. When the DTA (green lines) were investigated, two small peak points (endothermic peaks) and two large peak points (endothermic peaks) were obtained at 241 and 547 °C. Until the reaction was completed, no exothermic peak ([Figure 9](#)) was seen, such that the burning reaction was not realized.



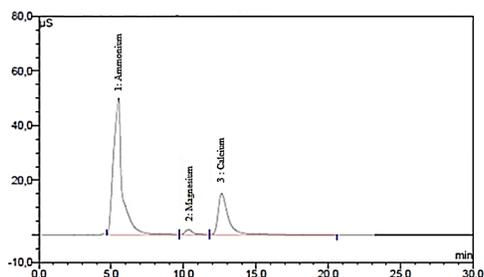
Peak no	Component name	Retention time, min	Area, $\mu\text{s}\cdot\text{min}$	Height, μS	Amount, ppm	Relative amount, %
1	Ammonium	5.52	35.252	50.249	676.0865	81.79
2	Magnesium	10.32	0.996	1.536	9.1601	1.11
3	Calcium	12.64	10.351	12.848	141.3413	17.10

Figure 3. Cation analysis in the absence of kaolin and fly-ash-coated ammonium nitrate sample.



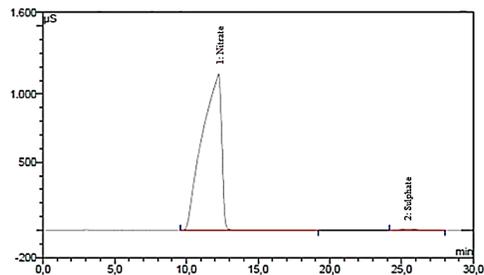
Peak no	Component name	Retention time, min	Area, $\mu\text{s}\cdot\text{min}$	Height, μS	Amount, ppm	Relative amount, %
1	Nitrate	12.30	1822.548	1157.196	17102.3169	99.43
2	Sulphate	25.45	13.540	12.672	98.4356	0.57

Figure 4. Anion analysis in the absence of kaolin and fly-ash-coated ammonium nitrate.



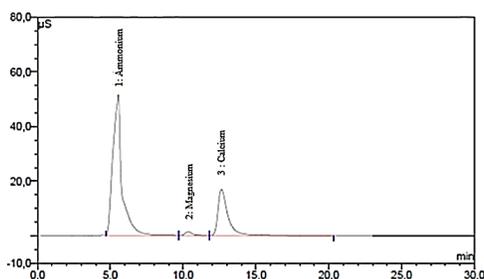
Peak no	Component name	Retention time, min	Area, $\mu\text{s}\cdot\text{min}$	Height, μS	Amount, ppm	Relative amount, %
1	Ammonium	5.52	35.021	50.066	671.6461	79.14
2	Magnesium	10.34	1.166	1.776	10.8042	1.27
3	Calcium	12.64	12.141	15.086	166.2141	19.59

Figure 5. Cation analysis in the presence of kaolin and fly-ash-coated ammonium nitrate.



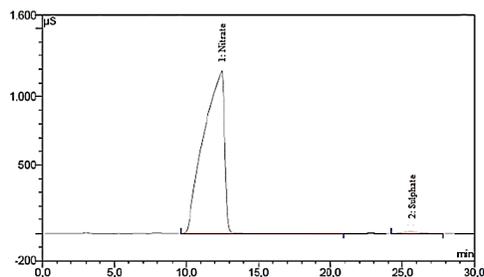
Peak no	Component name	Retention time, min	Area, $\mu\text{s}\cdot\text{min}$	Height, μS	Amount, ppm	Relative amount, %
1	Nitrate	12.25	1777.789	1146.281	16682.3106	99.43
2	Sulphate	25.47	13.160	12.264	95.6730	0.57

Figure 6. Anion analysis in the presence of kaolin and fly-ash-coated ammonium nitrate.



Peak no	Component name	Retention time, min	Area, µs.min	Height, µS	Amount, ppm	Relative amount, %
1	Ammonium	5.55	37.722	51.708	723.6571	78.69
2	Magnesium	10.35	0.923	1.375	8.4535	0.92
3	Calcium	12.63	13.671	16.946	187.4775	20.39

Figure 7. Cation analysis in the presence of kaolin and fly-ash-coated ammonium nitrate.



Peak no	Component name	Retention time, min	Area, µs.min	Height, µS	Amount, ppm	Relative amount, %
1	Nitrate	12.45	1979.485	1191.752	18574.9749	99.48
2	Sulphate	25.57	13.411	12.471	97.4959	0.52

Figure 8. Anion analysis in the presence of kaolin and fly-ash-coated ammonium nitrate.

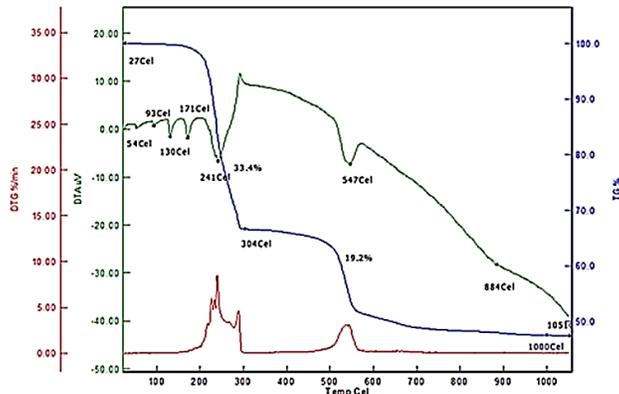


Figure 9. TG-DTA curves for fly-ash-coated calcium ammonium nitrate.

5. Conclusion

Based on the calcium ammonium nitrate production process, the nitrogen concentration of calcium ammonium nitrate was determined to be 26.64%. The nitrogen concentration of fly-ash-coated calcium ammonium nitrate was determined to be 25%, based on the results of ion chromatography analysis. If 10-50% fly ash is the proposed concentration for this operation before the conditions became less severe, it would be possible to compensate for the loss in the nitrogen concentration. In the present study, the reaction between the fly-ash-coated calcium ammonium nitrate and diesel was examined by bomb calorimetry, under atmospheric conditions. Based on the results of this analysis, it was determined that the use of fly-ash-coated calcium ammonium nitrate did not change the burning speed of the diesel. Since the gas outlet could not be seen, detonation conditions did not

arise. On the other hand, as a reference to the gas outlet and burning speed, after contact between the diesel and calcium ammonium nitrate without the fly ash, it was observed that the burning speed of calcium ammonium nitrate increased markedly.

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