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Minimizing the explosion enthalpy of ammonium nitrate with polyethylene glycol and carboxymethyl cellulose to prevent terrorist attacks

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The use of ammonium nitrate due to its high nitrogen content (> 26%) has made it the most utilized fertilizer in agricultural areas. However, being easily accessible with this feature encouraged its use for different purposes. Ammonium nitrate is usually produced with large tonnage (> 50 ton/h) and high cost (> $20 million) production processes. Therefore, any changes that can be made in the process must be applied in the process so that the result can be achieved easily without increasing the cost in any way. In this study, it is aimed to reduce the explosion sensitivity of ammonium nitrate used for explosive purposes in terrorist attacks. Thus, it was aimed to solve the problem by adding various chemicals to the ammonium nitrate production process so that it can only be used for agricultural purposes. For this purpose, the production process was examined by adding carboxymethyl cellulose and polyethylene glycol to the ammonium nitrate production process and the accuracy of the results was tested by instrumental analysis methods.

Keywords: carboxymethyl cellulose, polyethylene glycol, ammonium nitrate, detonation

1. INTRODUCTION

Considering that ammonium nitrate is an important nitrogen source for its use in agricultural areas, it is a generally accepted phenomenon in the agricultural industry that it is a commercially high value and most preferred nitrogen fertilizer. However, since ammonium nitrate is an inexpensive and easily available material, it paved the way for its use for different purposes than its various properties, such as its high thermal value. In order to prevent such problems, researchers have focused on various methods and processes. The methods and materials developed were mainly in the form of chemical additives to the production processes. In a research study conducted by Gezerman (2017), he added calcium carbonate, dolomite and fly ash additives to the ammonium nitrate production process and reduced the high thermal energy of ammonium nitrate. In another study, (Gezerman and Çorbacioglu, 2017) analyzed the thermal value of ammonium nitrate by combining dolomite additive with fly ash (FBC). Similar results were obtained in other studies in which the effect of fly ash on the thermal value of ammonium nitrate was analyzed (Gezerman and Çorbacıoğlu, 2020; Gezerman, 2019).

However, the heavy metal content of fly ash poses a threat to agricultural areas. Therefore, it should be included in the production process at a maximum of 5% of the total mass of the product. Therefore, the above mentioned papers are an important step towards minimizing the heavy metal problem.

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Researchers have made several attempts to reduce the reaction enthalpy of ammonium nitrate. Ammonium nitrate mixed with mineral oil and aluminum, up to 40% in particular, has been found to have an explosive property (Buczkowski and Zygmunt, 2011). Another study to limit the explosive properties of ammonium nitrate is the treatment of ammonium nitrate with fly ash. When applied on ammonium nitrate with the commercial classification of fly ash in itself, it has been found that ammonium nitrate significantly limits the explosive property (Taulbee, 2010). In order to ensure the thermal stabilization of ammonium nitrate and limit the detonation properties, it was mixed with 50% urea and significant results were obtained. However, it was not found to be commercially viable due to the limited storage life of ammonium nitrate mixed with urea (Tan et al., 2015). One of the studies carried out to limit the explosive properties of ammonium nitrate commercially without affecting the nitrogen value is the addition of sodium chloride (NaCl) salt to the fertilizer production process. Ammonium nitrate produced by processes in which sodium chloride (NaCl) salt is added has not shown explosion sensitivity when used with fuel oil during explosion (Tan et al., 2014). The effect of chloride salts on the explosion enthalpy of ammonium nitrate is an important focus of most researchers. For this purpose, the sensitivity of an explosive mixture consisting of mixing potassium chloride salt with ammonium nitrate was studied and it was concluded that the chloride radical triggered the explosion reaction of ammonium nitrate (Izato and Miyake, 2015). In order to measure the explosion sensitivity of ammonium nitrate, its mechanism must be defined. To describe the mechanism, the explosion reaction of ammonium nitrate treated with diesel fuel and mineral oil has been studied (Janesheskiet al., 2012). Another research study conducted to improve the explosion sensitivity of ammonium nitrate is treatment with polystyrene. It has been reported that such a reaction medium can limit its explosive properties (Dobrilović et al., 2013). Another study on the explosion sensitivity of ammonium nitrate has been done with potassium chloride and mono ammonium phosphate. The addition of potassium chloride and monoammonium phosphate has been reported to reduce the explosion rate of the ammonium nitrate mixture (Tan et al., 2016). However, in another study examining the effect of potassium chloride on ammonium nitrate, the addition of potassium chloride and sodium sulphate has been reported to affect the explosive properties of ammonium nitrate (Han et al., 2015). In another research study to reduce the explosion sensitivity of ammonium nitrate, calcium sulphate was added to the ammonium nitrate solution. In this way, it has been reported that the explosion sensitivity was reduced and it could be produced easily and reliably for the granulation process (Lylykangas et al., 2012). Another chemical additive that researchers are working on to reduce the reaction enthalpy of ammonium nitrate is urea and formaldehyde. When urea and formaldehyde are included in the reaction medium in which ammonium nitrate is produced, it has been reported to have an impact on the detonation enthalpy (Ouadday et al., 2017). Significant results have been reported regarding the reduction of explosion intensity in solutions using polymeric components and oil-soluble anionic surfactants to limit the explosion enthalpy of ammonium nitrate (Fedorov and Love, 2016). In another research study, ammonium nitrate, ammonium sulphate, ammonium phosphate was treated with ammonium molybdenum, ammonium hexafluorosilicate, calcium nitrate, calcium carbonate, magnesium nitrate, potassium nitrate and potassium phosphate. Thus, it was found that the reaction temperature of ammonium nitrate during explosion was significantly reduced (Levy et al., 2014). In another study in which the explosion enthalpy of ammonium nitrate is reduced, potassium perchlorate is included in the production reaction medium of ammonium nitrate. The explosion sensitivity of ammonium nitrate obtained in such a reaction medium has been reported to be significantly limited (Smith, 2019). In another study to reduce the explosion sensitivity of ammonium nitrate, sodium nitrate (16%), thiourea (2%) and urea (12%) were added to the reaction medium. It has been reported that the explosion sensitivity of the ammonium nitrate produced under these reaction conditions is greatly limited (Cordova and Lopez, 2012).

In order to limit the thermal properties of ammonium nitrate, reaction mechanisms were followed by adding boron, manganese and copper to calcium and magnesium carbonate salts and it was determined that these additives did not affect reaction enthalpy by adding up to 0.5 mol concentration (Klimova et al., 2011).

In another research study in which the reaction enthalpy of ammonium nitrate was limited, it was reported that the contribution of methylnitrotetrazole (MNT) and glycidyl azide polymer (GAP) at concentrations of up to 15% had an effect on increasing the explosion enthalpy of ammonium nitrate (Sinditskii et al., 2005).

Similar to all these research studies, in another study examining the thermal decomposition of ammonium nitrate, the moisture absorption and thermal degradation processes of ammonium nitrate at different humidity values were followed with the adsorption model of Brunauer – Emmett – Teller (BET). According to this model, it is reported that the moisture absorption potential of ammonium nitrate increases as the moisture content of the air increases (Videla et al., 2017). In another study examining the thermal degradation of ammonium nitrate, the effect of calcium carbonate and calcium sulfate on the thermal degradation and reaction enthalpy of ammonium nitrate was investigated, and significant results were obtained on dilution of ammonium nitrate with calcium carbonate and calcium sulfate, reducing reaction enthalpy and slowing thermal degradation (Menicacci et al., 2020).

In this study, in order to limit the explosion enthalpy of ammonium nitrate, encapsulation of ammonium nitrate particles was included by adding to the dilution medium of chemicals such as polyethylene glycol and carboxymethyl cellulose, and the separation of ammonium nitrate was significantly limited after the granulation process.

2. MATERIAL AND METHODS

2.1. Ammonium nitrate production process

As the raw materials used in this study, anhydrous ammonia (CAS No: 7664-41-7) and 55%, wt Nitric acid (CAS No: 7697-37-2) were obtained from Toros Agri Industry. Other process chemicals, polyethylene glycol (CAS No: 25322-68-2) and carboxymethyl cellulose (CAS No: 9004-32-4) were obtained from Acarchemicals.

In this study, ammonium nitrate was produced using a two-stage vacuum concentration method. For the production of ammonium nitrate, anhydrous ammonia (99.9% wt.) at 5 °C and 6.1 kg/cm^2 reacts with 50% nitric acid at 50 °C to form ammonium nitrate in a pipe reactor. Ammonium nitrate produced under industrial conditions with this process has a maximum concentration of 80%. However, since it is not possible to granulate ammonium nitrate in this concentration, the water in it is removed by vacuum method in order to obtain ammonium nitrate with a minimum concentration of 99% before granulation.

For this purpose, using environmentally heated (serpentine) evaporators, excess water in ammonium nitrate solution having 80% concentration is removed and 99% concentration is obtained before granulation. In order to improve the storage conditions of ammonium nitrate, chemical additives are added to the ammonium nitrate solution, which is brought to a concentration of 99% and above before granulation. Therefore, in this study, 0.01% polyethylene glycol and 0.01% carboxymethyl cellulose chemicals, which are studied to reduce the enthalpy value of ammonium nitrate, were added at this stage (Fig. 1).

2.2. Electron microscopy analysis

The surface pore structures of the particles obtained by the ammonium nitrate production process are an important factor on the explosion sensitivity of ammonium nitrate.

Scanning electron microscopy (SEM) was used to examine the effect of additives such as polyethylene glycol and carboxymethyl cellulose added to the reaction medium to reduce the explosion sensitivity on

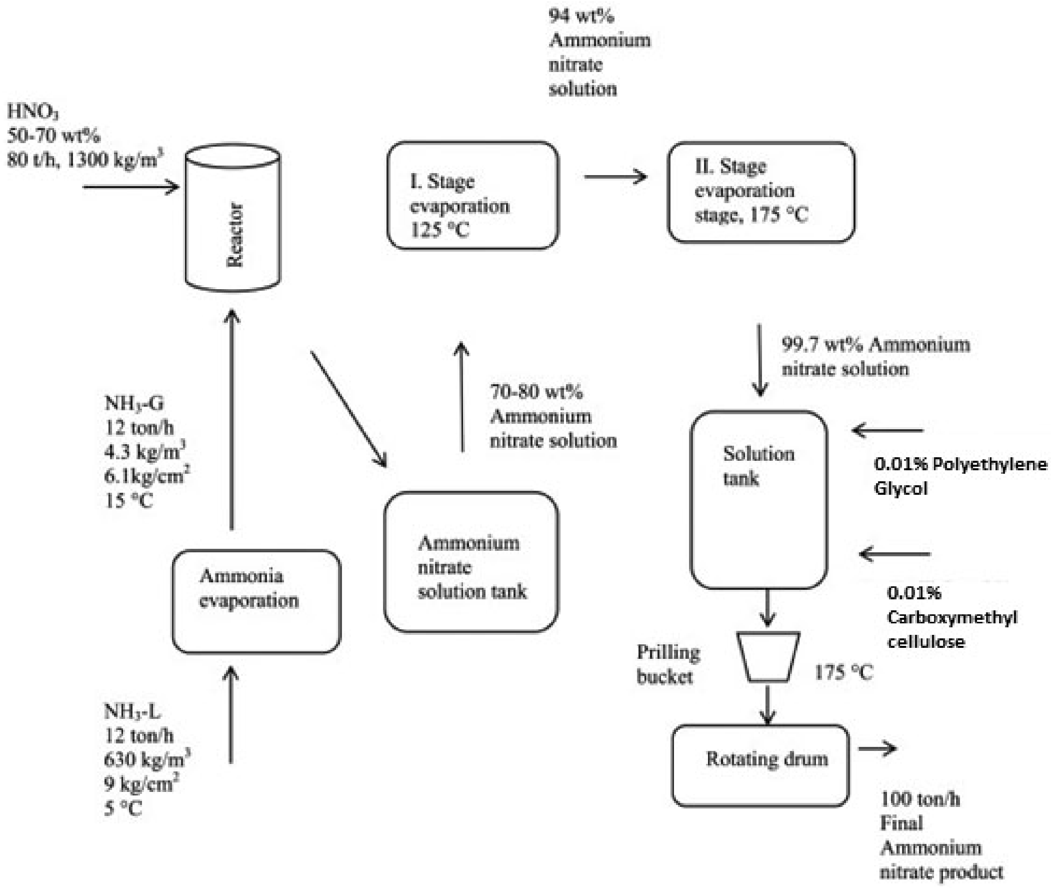

Fig. 1. Ammonium nitrate production with two-stage evaporation method (Gezerman, 2020)

the particle surface. Surface analysis made by electron microscopy was performed using a Carl Zeiss DSM-960A device according to ASTM E986-97 standards. While electron microscope was used during operation, accelerating voltage, 1–30 kV; useful magnification, 10 – 30000 ×; resolution was set to 70 Å.

2.3. Ion chromatography analysis

In this study, chemicals such as polyethylene glycol and carboxymethyl cellulose were used while producing a non-explosive ammonium nitrate fertilizer. These chemicals should not decrease the minimum nitrogen and nitrate content of the ammonium nitrate produced.

Ammonium nitrate is mostly used in the agricultural industry. Therefore, nitrogen and nitrate needed by the plant is obtained from ammonium nitrate fertilizer. Hence, in order to determine the nitrogen and nitrate content produced, ion chromatography was studied. Nitrate analysis by ion chromatography was performed using a Shimadzu Prominence HIC – NSn system device with ASTM E1151-93 (Gezerman, 2020) standard. During operation, device parameters were determined as follows. conductivity 0.01–51200 µS cm^{-1}, and flow rate of 0.001–5 ml min^{-1}.

2.4. Screen sieve analysis

In the ammonium nitrate production process, the effect of polyethylene glycol and carboxymethyl cellulose on the particle structure is important for determining the suitability for the 2 mm particle diameter which is accepted as standard by the European fertilizer manufacturers association.

For this purpose, the effect of polyethylene glycol and carboxymethyl cellulose on the particle structure in the ammonium nitrate production process was investigated using a Vibratory Sieve Shaker AS 200 (manufactured by Retsch, Germany) according to the ASTM E11-09 (Gezerman, 2017) standard. After the production by sieve analysis, percentage distribution of 3.35 mm, 2.5 mm, 2.0 mm, 1.0 mm, 0.5 mm and under-sieve particle sizes were determined.

2.5. Crushing strength analysis

In terms of agricultural use, another parameter followed for particle analysis in the production of ammonium nitrate is the crushing strength of the particles obtained in the granulation process. Instron Test System (Model 4411) crushing strength test device was used for the crushing strength of ammonium nitrate particles with an average diameter of 2 mm obtained as a result of the process in which polyethylene glycol and carboxymethyl cellulose were added.

2.6. Analysis of nitrogen

The nitrogen content of ammonium nitrate particles is the biggest requirement that must be provided commercially. Any chemical additives to be used during production should not affect the minimum nitrogen value that ammonium nitrate should provide.

For this purpose, nitrogen amount analysis is carried out continuously during the production period. For this purpose, 7 g ammonium nitrate sample is diluted to 500 ml. Then, 50 ml of 20% NaOH is mixed with 10 ml sample taken from this solution and titrated with 98% sulfuric acid.

$$\text{Calculations:}\ \ \text{N, \%} = (50 - A) \times F \tag{1}$$

where A is the volume of NaOH during titration and F is the dilution factor.

2.7. Calorimetric test and TGA analysis

The effect of polyethylene glycol and carboxymethyl cellulose, which was added to the ammonium nitrate production process to reduce detonation enthalpy, was measured with the IKA C 4000 brand / model calorimeter device. In addition, TGA analysis was performed with SII6000 Extar TG / DTA 6300 model device.

3. RESULTS AND DISCUSSION

The tendency of ammonium nitrate to oxidation and rapid reaction is one of the important problems studied today. However, there are many studies on reducing explosion severity. All chemicals added to the process must be added at the dilution stage that determines the reaction conditions of ammonium nitrate. Because in high pressure (15 Pa) ammonium nitrate processes, in case of any temperature change, ammonium nitrate can crystallize and block the lines with sudden temperature change (< 169 °C). Therefore, the chemicals to be added to the process should not have a significant effect on this temperature change, and this addition should be carried out at a process stage that would have minimal impact on physical conditions. Therefore, in this study, carboxymethyl cellulose and polyethylene glycol chemicals were studied to minimize the heat released during the reaction. However, due to their high molecular weights (> 80000 g/mol) and tendency to foam, both chemicals are expected to be added at a minimum concentration (< 0.01%). Due to the process conditions, minimizing the risk of foaming is only possible at the dilution stage.

In the study, various physical parameters of ammonium nitrate such as porosity, density that affect explosion sensitivity were also investigated. However, as another feature, nitrate concentration analysis was performed by ion chromatography and quality parameters to limit the use of ammonium nitrate in non-agricultural areas were examined since, in attempts to reduce explosion sensitivity, the protection of commercial quality is another important focus in this study.

According to the ion chromatography used in the amount analysis, it was determined that the amount of ammonium nitrate in the addition of polyethylene glycol (Fig. 3) and carboxymethyl cellulose (Fig. 4) is a negligible concentration loss (0.1%) compared to the concentration of nitrate ion in pure ammonium nitrate content (Fig. 2), which is not particularly commercial and agricultural risk.

Peak No.	Component Name	Retention Time	Area µS*min	Height µS	Amount ppm	Relative Amount %
1	Nitrate	5.21	5.632	16.912	962.3214	72,44

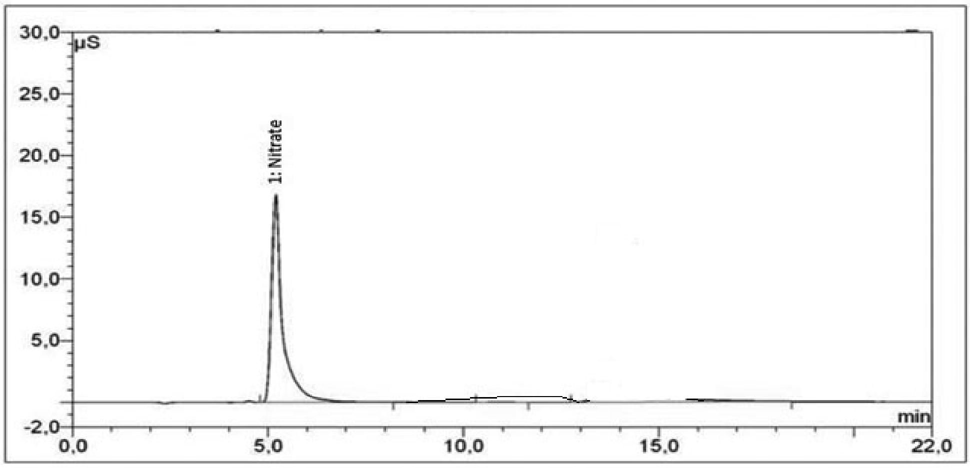

Fig. 2. Anion analysis in ion chromatography of pure ammonium nitrate

Peak No.	Component Name	Retention Time	Area µS*min	Height µS	Amount ppm	Relative Amount %
1	Nitrate	5,19	5,529	16,828	962,9863	72,12

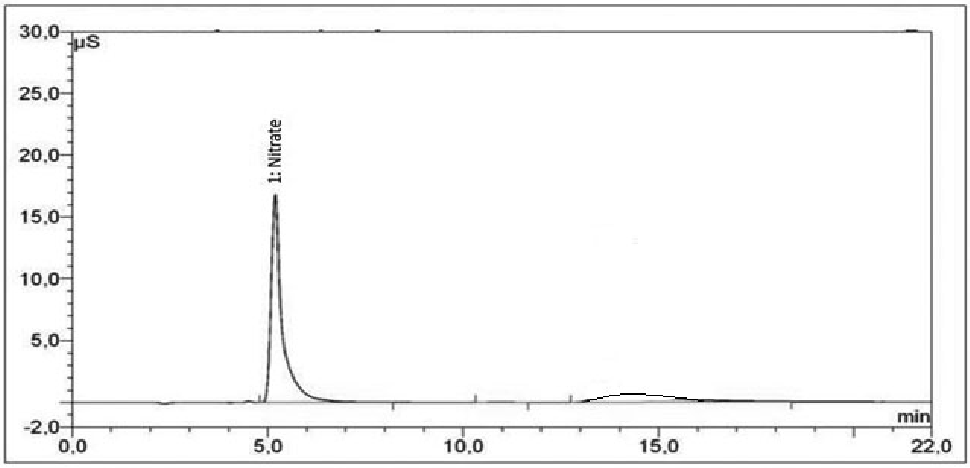

Fig. 3. Ion chromatography of ammonium nitrate with 0.01% polyethylene glycol additive

Peak No.	Component Name	Retention Time	Area µS*min	Height µS	Amount ppm	Relative Amount %
1	Nitrate	5.09	5.496	16.852	963.1254	71.96

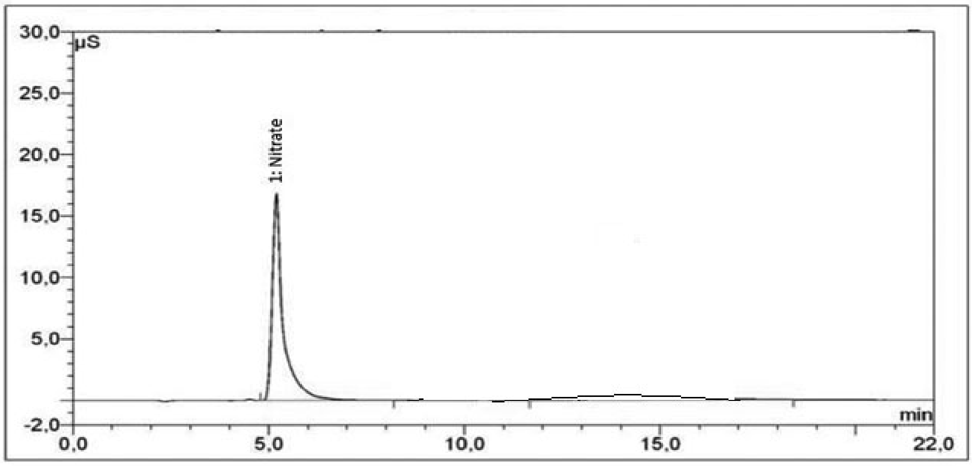

Fig. 4. Ion chromatography of ammonium nitrate with 0.01% carboxymethyl cellulose

Nitrate ion concentration is the most important factor affecting the nitrogen concentration of ammonium nitrate. While nitrate ion analysis is performed, the determined nitrogen concentration is important in terms of assessing the maximum nitrogen concentration that the plant will need during fertilization. Figures 3, 4 and 5 show ion chromatography results of ammonium nitrate particles, where 0.01% polyethylene glycol, 0.01% carboxymethyl cellulose and both were added, respectively. With ion chromatography analysis, nitrogen concentration can be calculated by performing nitrate ion analysis in ammonium nitrate content. Therefore, during the study, the effect of 0.01% polyethylene glycol and 0.01% carboxymethyl cellulose additives on nitrogen concentration in ammonium nitrate was investigated. However, nitrogen concentration of pure ammonium nitrate was 34.60% in nitrogen analysis, nitrogen concentration was 34.40% with carboxymethyl cellulose additive and 34.25% with polyethylene glycol additive. Since the content of pure ammonium nitrate guaranteed by the manufacturer is at least 34.20%, the nitrogen concentrations

Peak No.	Component Name	Retention Time	Area µS*min	Height µS	Amount ppm	Relative Amount %
1	Nitrate	4.96	5.236	16.784	962.7152	71.88

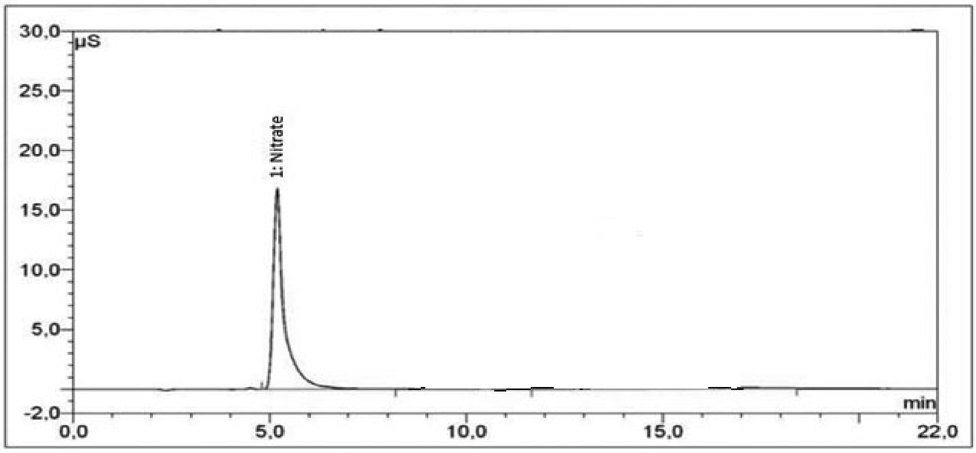

Fig. 5. Ion chromatography of 0.01% polyethylene glycol and 0.01% carboxymethyl cellulose added ammonium nitrate

obtained during this study were found to be not less than the nitrogen concentration required by the plant in agricultural areas.

However, to examine porosity from the physical properties of ammonium nitrate, surface scanning was done by electron microscopy. In electron microscopy examination, it was found that the additions of carboxymethyl cellulose (Fig. 8) and polyethylene glycol (Fig. 7) reduced the porous properties compared to pure ammonium nitrate (Fig. 6). Thus, additives such as polyethylene glycol and carboxymethyl cellulose have been found to reduce the pore structure for the explosion reaction. It has been determined that the porosity and surface roughness of ammonium nitrate decreased with the addition of both chemicals (Fig. 9).

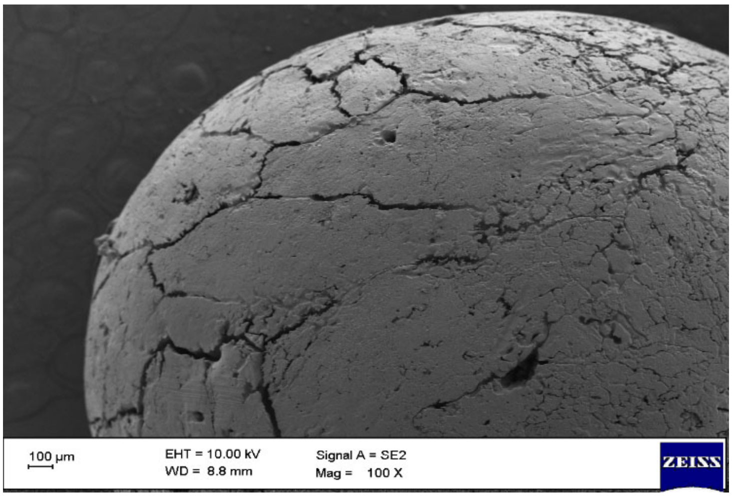

Fig. 6. SEM image of pure ammonium nitrate produced by two-stage evaporation

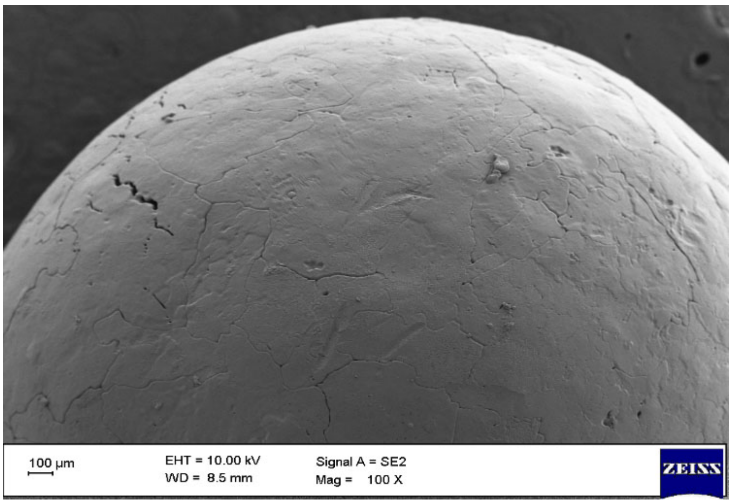

Fig. 7. SEM image of ammonium nitrate with 0.01% polyethylene glycol additive

In this study, density properties as another physical parameter for ammonium nitrate particles were investigated. Accordingly, the density of pure ammonium nitrate was measured as 1.42 g/cm^3, density of ammonium nitrate with 0.01% carboxymethyl cellulose, 1.32 g/cm^3, density of ammonium nitrate with 0.01% polyethylene glycol, 1.39 g/cm^3. The particle density of ammonium nitrate doped with polyethylene glycol and carboxymethyl cellulose was determined as 1.37 g/cm^3.

Furthermore, no clogging problems were observed in the granulation step of the process in the addition of carboxymethyl cellulose and polyethylene glycol. In this respect, it has been determined technically

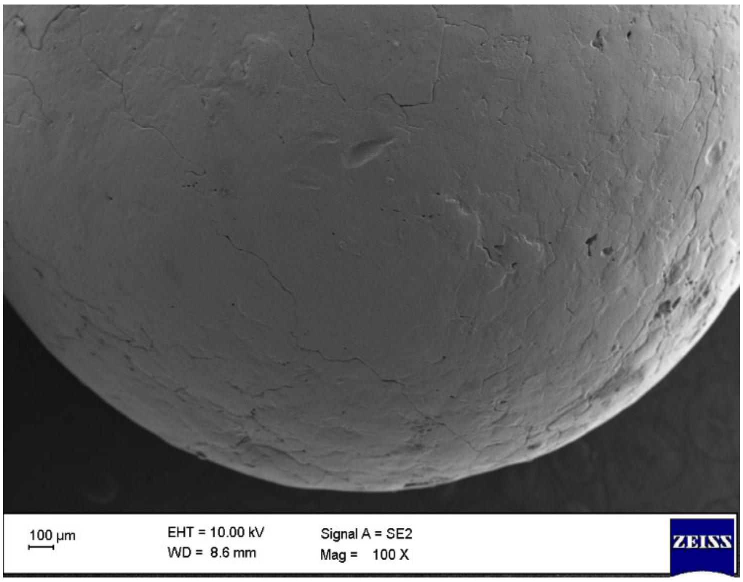

Fig. 8. SEM image of ammonium nitrate with 0.01% carboxymethyl cellulose additive

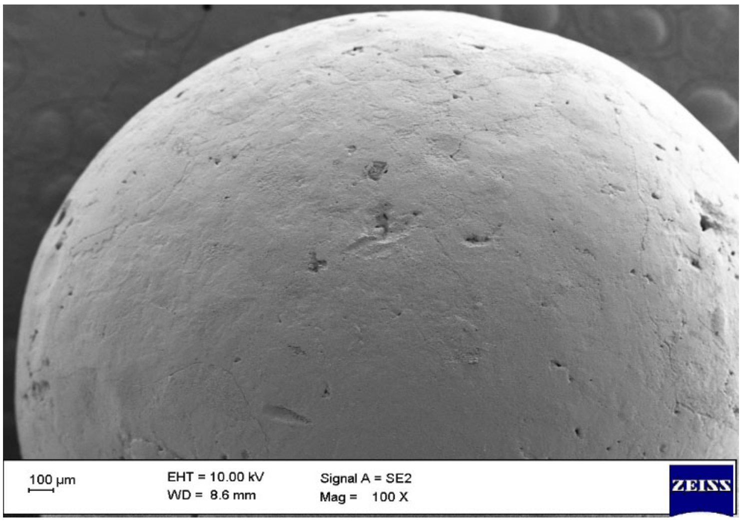

Fig. 9. SEM image of ammonium nitrate with 0.01% polyethylene glycol and 0.01% carboxymethyl cellulose additive

that both chemicals can be used in ammonium nitrate production processes. However, as a granulation system, a prilling system consists of a minimum height of 40 m (Gezerman, 2020). It is expected that ammonium nitrate particles are left to cool by means of a prill bucket from this height, to complete phase transformations of ammonium nitrate by the effect of cooling air. After the granulation process, it is necessary to complete these phase transformations and turn them into the solid phase.

Therefore, chemicals such as polyethylene glycol, carboxymethyl cellulose added in the dilution stage of the production process should contribute to the conversion of ammonium nitrate to the solid phase as a result of the solidification process of ammonium nitrate. In this process, it was determined that the temperature of leaving the prill tower of both polyethylene glycol and carboxymethyl cellulose added ammonium nitrate in industrial conditions was below 80 °C, so that ammonium nitrate turned into solid phase. In order to minimize the safety risk of ammonium nitrate, which is the main subject of this study, it was analyzed by calorimeter bomb in order to measure the thermal value of ammonium nitrate produced with polyethylene glycol and carboxymethyl cellulose additives.

In the calorimetric analysis with the calorimeter bomb, the calorimetric values of ammonium nitrate with 0.01% polyethylene glycol (Fig. 10) and 0.01% carboxymethyl cellulose (Fig. 11) were measured together with the thermal value of pure ammonium nitrate. According to the results obtained, the explosion reaction intensity was analyzed.

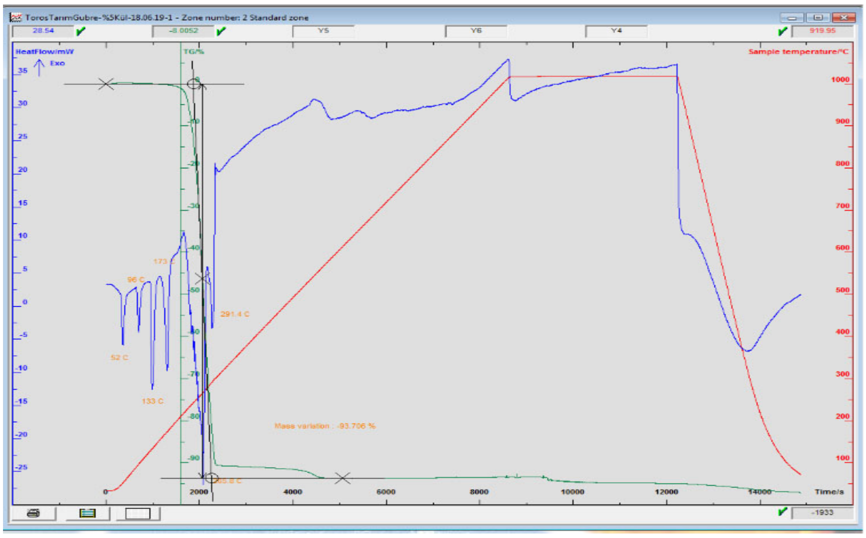

Fig. 10. TGA analysis of ammonium nitrate with 0.01% polyethylene glycol additive

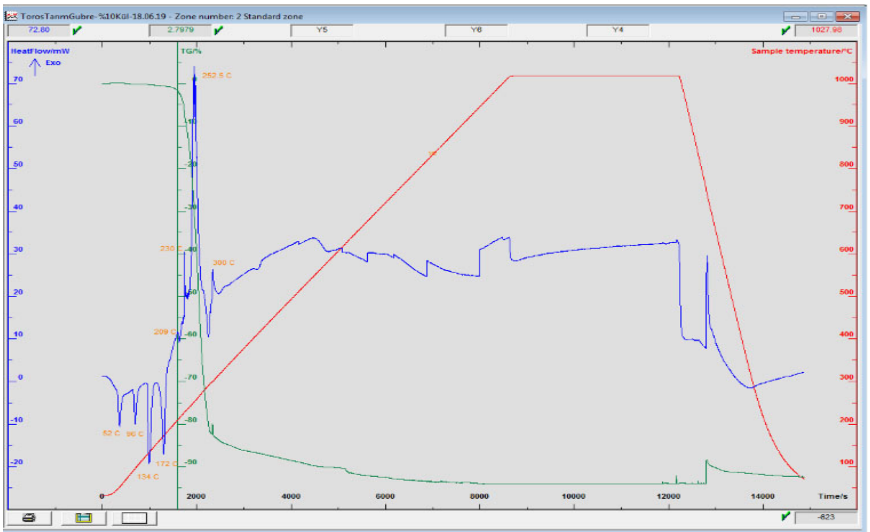

Fig. 11. TGA analysis of 0.01% carboxymethyl cellulose added ammonium nitrate

In the TGA analysis, it was found that the additive of polyethylene glycol reduced the explosion enthalpy of pure ammonium nitrate by 60% (Fig. 10), and the addition of carboxymethyl cellulose added as another additive material reduced the explosive enthalpy of pure ammonium nitrate by 55% (Fig. 11). In the case where carboxymethyl cellulose and polyethylene glycol were added together, it was found that the explosion enthalpy of pure ammonium nitrate decreased by 65% (Fig. 12).

The effect of carboxymethyl cellulose and polyethylene glycol, which is used to reduce the explosion enthalpy of ammonium nitrate, on the particle size and crushing strength of ammonium nitrate is important in terms of preserving the commercial quality of the ammonium nitrate produced. Therefore, as seen in the crushing strength and sieve analysis tests, it was found that the additive of polyethylene glycol and carboxymethyl cellulose increased the crushing strength of ammonium nitrate and increased the particle

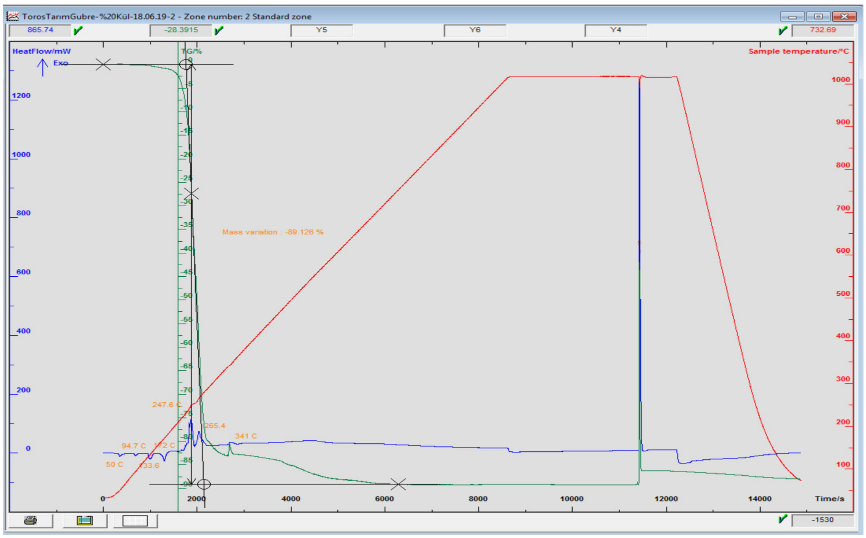

Fig. 12. TGA analysis of 0.01% polyethylene glycol and 0.01% carboxymethyl cellulose added ammonium nitrate

size (Table 1). When the effect of the added chemicals on the physical parameters of ammonium nitrate was examined, it was determined that the crushing strength of the ammonium nitrate with 0.01% polyethylene glycol was 2 kg/prill. However, the crushing strength of ammonium nitrate with 0.01% carboxymethyl cellulose additive was determined to be 2.05 kg/prill. The crushing strength of ammonium nitrate with 0.01% polyethylene glycol and 0.01% carboxymethyl cellulose additive was determined as 2.11 kg/prill (Table 1).

Table 1. Screen sieve analysis for ammonium nitrate containing

Chemical fertilizer composition	3.35 mm	2.5 mm	2.0 mm	1.0 mm	0.5 mm	U.S. [mm]	Crushing strength	N [%]
Pure Ammonium Nitrate	5.2	42.7	33.8	17.5	0.8	0	1.82	34.45
Ammonium nitrate with 0.01% Polyethylene glycol additive	5.4	41.8	38.1	13.8	0.9	0	2.00	34.33
Ammonium nitrate with 0.01% carboxymethyl cellulose additive	5.7	43.1	37.4	13.1	0.7	0	2.05	34.25
Ammonium nitrate with 0.01% Polyethylene glycol and 0.01% Carboxymethyl cellulose additive	5.1	42.9	36.5	15.4	0.1	0	2.11	34.32

4. CONCLUSIONS

The sensitivity of ammonium nitrate to explosion has diversified its use for yield in agricultural areas and has made it an easily accessible substance. However, the development of thermodynamic properties to prevent the use of it for anti-social purposes will be possible with the inclusion of chemicals that will affect the process properties. In this study, polyethylene glycol and carboxymethyl cellulose were added to limit the explosion properties of ammonium nitrate, and the thermal effect of polyethylene glycol and carboxymethyl cellulose additives on ammonium nitrate was observed. Since the inclusion of each chemical additive material in the process at a maximum of 0.01% does not significantly affect process operating conditions such as pressure and temperature, and its costs, it can be considered as an important alternative to the production of ammonium nitrate.

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