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SCIENTIFIC PAPER

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EFFECTS OF SODIUM SILICATE, CALCIUM CARBONATE AND SILICIC ACID ON AMMONIUM NITRATE DEGRADATION AND ANALYTICAL INVESTIGATIONS OF THE DEGRADATION PROCESS ON AN INDUSTRIAL SCALE

Article Highlights

- Different compounds were added to prevent the degradation of ammonium nitrate
- Multiple instrumental analyses were used to monitor the degradation process
- We describe a method to limit the degradation properties of sulfuric acid

Abstract

Ammonium nitrate is an inorganic chemical that has numerous applications in different industries. However, various problems are associated with both the production and subsequent storage of ammonium nitrate, including caking, degradation, unwanted phase transition, and recrystallization. Although several methods have been developed to attempt to solve these problems, many of them fail to work in practice. In this study, different compounds including silicic acid and sodium silicate were added to slow the progress of or to prevent the degradation of ammonium nitrate. Multiple instrumental analyses such as ion chromatography and scanning electron microscopy were used to monitor the degradation process.

Keywords: ammonium nitrate, caking, degradation, ion chromatography, downstream process.

The fertilizer industry has developed in many ways according to the requirements of the agricultural industry in the 20th and 21st centuries. However, these advances have created additional problems owing to increased usage conditions, including reactions of chemicals used during production processes and technical problems associated with fertilizer storage and manufacturing methods intended to reduce costs. Currently, many universities, institutes, and research and development centers are working to find solutions to these problems of fertilizer usage.

Simultaneously, various chemical compositions have been developed to improve the chemical properties of fertilizers, including their chemical stability. For example, the reaction of calcium silicate, which is obtained from an aqueous solution of sodium silicate and calcium chloride, with ammonium nitrate produced a fertilizer salt with good chemical stability [1]. In other studies, the addition of calcium silicate was observed to increase the nutriment properties of ammonium nitrate fertilizer [2]. In a similar study, C1--C6 alcanoic acid or sodium silicate salts of this acid were sprayed on ammonium nitrate fertilizer [3], and in another study, Joseph *et al.* used sodium silicate salts to solve the degradation problem by adding 20% of a sodium silicate solution to ammonium nitrate during the production process [4]. Rinkenbach also applied sodium silicate to prevent the degradation of ammonium nitrate by treating 100 parts ammonium nitrate with 0.5-5 parts sodium silicate at 130 °C [5]. Adam investigated methods for increasing the nutrient value of soil by using an ammonium nitrate solution and calcium silicate [6]. Another study applied a polymeric solution containing sodium silicate to coat the surface of ammonium nitrate particles to improve the degradation properties [7].

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The most commonly used chemical reducing agents in fertilizer production are chrome, zinc, iron(II) sulfate, salicylic acid, and silicic acid [8]. Silicic acid ($Si(OH)_4$) is a weak acid employed in the fertilizer production process to increase the nutrient value and as a reducing agent [8]. The Kjeldahl manufacturing method is an example of the use of this reagent. In this method, sodium thiosulfate ($Na_2S_2O_3$), salicylic acid, or silicic acid reagents are added during analysis to account for nitrite and nitrate. Gezerman et al. added silicic acid and a calcium lignosulfonate solution to an ammonium nitrate melt to test the degradation process [9].

The reducing agent $Na_2S_2O_3$ reacts with H_2SO_4 to generate sulfur dioxide (SO_2), preventing the formation of double salts. In this process, 1 g $Na_2S_2O_3.5H_2O$ consumes 0.5 mL H_2SO_4. If silicic acid and salicylic acid are used, these reagents also act as reducing agents for H_2SO_4, generating CO_2 and water. During this process, 1 g salicylic acid or 1 mL silicic acid consumes 5.6 mL H_2SO_4.

Known patented processes for the dilution of fertilizer solution involve the addition of calcium carbonate [10]. However, we are not aware of any patented or otherwise published processes for limiting the degradation properties of sulfuric acid, which is a required processing component. In this paper, we describe a method for the dilution of fertilizer solution with a mixture of calcium carbonate, sodium silicate, and silicic acid to limit the degradation properties of sulfuric acid and produce a more stable ammonium nitrate fertilizer solution.

The vacuum concentration method [10], which is the most commonly used method in the fertilizer industry, was used to investigate the effects of the different chemical additives. According to this method, 70–85% concentrated ammonium nitrate solution, formed through the reaction between 55% HNO3 and gaseous anhydrous ammonia, can be concentrated up to 97% (Figure 1). Upon obtaining the maximum concentration of ammonium nitrate, calcium carbonate, sodium silicate, and silicic acid were added to the bulk solution. In fertilizer manufacture processes in the literature, the addition of calcium carbonate is a known method for the dilution of ammonium nitrate solution.

On an industrial scale, sodium silicate, which is readily available and can be used as an alternative to or together with calcium carbonate, is used in fertilizer production. The addition of silicic acid to a fertilizer solution with a dilution material such as calcium carbonate and sodium silicate, and its effects on the amount of sulfuric acid, are investigated analytically in this study for the first time.

Main causes of fertilizer caking and degradation

The mechanism of fertilizer caking involves the formation of contact points between particles in the bulk fertilizer. These contact points are formed by three contact forces: phase contact, adhesive contact, and surface diffusion [11]. Phase contact refers to the formation of crystal bridges between fertilizer particles, and is commonly believed to be the main cause of fertilizer caking problems [11]. Crystal bridges occur through processes such as recrystallization, dissolution, and heating during the reaction to produce the product. Adhesive contact arises from molecular movements between contacting surfaces, and is

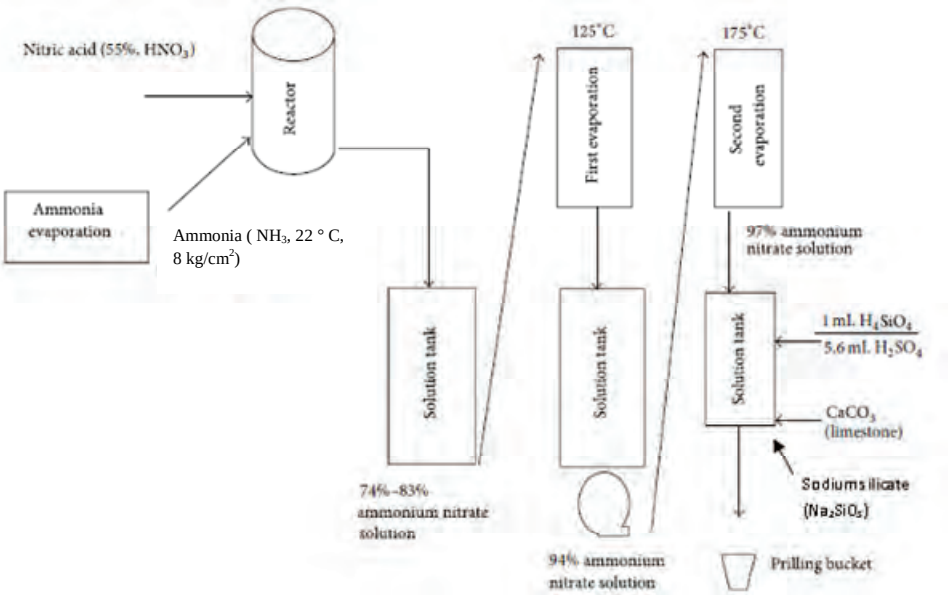


Figure 1. Ammonium nitrate production process (concentrated method by vacuum).

more commonly referred to as the van der Waals force. This type of contact typically occurs because of the plasticity of fertilizer particles and pressure in the bulk. Fertilizer particles participating in adhesive contact can be destroyed by small forces during handling. Surface diffusion occurs because of the formation of salt complexes from ion hydrates, which results in surface diffusion processes that promote the self-adhesion of fertilizer particles [11].

Besides these, another factor that causes caking of fertilizer particles is the water activity between the particles. Water activity or deliquescence refers to the dissolution of a material by water absorption at high water activity. If the relative humidity under the storage conditions increases, water condensates around the solid particles, causing them to dissolve. Because of the increased surface contact, cohesion between the particles is initiated, and consequently, caking progresses. Caking, which continues to proceed by the absorption of water, results in deliquescence [12].

Some materials such as nitrogenous fertilizers absorb a small amount of water *via* hydrogen bonding at low relative humidity, although they exist as liquid solutions. When the relative humidity increases, the solid particles in the condensate layer are dissolved to result in a saturated solution. If relative humidity increases, because of vapor condensation, a greater amount of melt solution is formed. This sequence of processes continues until complete deliquescence. Deliquescence can accelerate physical changes in the particles and chemical reactions between the particles [12].

Several methods for addressing caking problems have been developed thus far. Some reports describe improvements in only the physical properties, whereas others suggest improvements in fertilizer nutrient values. The purpose of the present study was to develop methods to prevent caking by changing the physicochemical properties of fertilizer particles to inhibit the contact phenomena described above. Through the addition sodium silicate, the viscosity and surface tension of NH_4NO_3 was expected to decrease, reducing the van der Waals forces between NH_4NO_3 molecules, and thus, preventing adhesive contact. Furthermore, the addition of silicic acid was expected to prevent salt complex formation, thereby inhibiting surface diffusion processes and preventing the caking of the fertilizer.

EXPERIMENTAL

Materials

Anhydrous ammonia (99.9%), nitric acid (55%), sodium silicate, silicic acid, calcium carbonate and sulfuric acid were obtained from Merck, Istanbul, of the highest grade available, and used without further purification.

Ammonium nitrate production process

Anhydrous NH_3 is pressurized in a vaporizer to 5.6 bar, and is then allowed to react with 55% HNO_3. The resulting solution in the reactor is 80% NH_4NO_3. This melt is brought to the required concentration by evaporation exchangers. Sodium silicate solution can be added during the production process in one of the two evaporation stages. In the present study, sodium silicate was added in the first evaporation stage, as described in the literature [9]. Calcium ammonium nitrate (CAN) containing 26% nitrogen was prepared by using 250 kg $CaCO_3$/t NH_4NO_3 and NH_4NO_3 containing 33% nitrogen was prepared using 60 kg $CaCO_3$/t NH_4NO_3 [10]. In this process, the reaction between NH_4NO_3 and $CaCO_3$ produced foam, which was removed by adding H_2SO_4.

Diluted solutions of sodium silicate were added together with calcium carbonate during the ammonium nitrate production process. Sodium silicate, in addition to its dilution properties, changes the prilling process, which operates at a rotation speed of 300 rpm. This was different from the effects of $CaCO_3$, as seen from the sieving analysis results (Tables 1 and 2), and was attributed to the increased prill particle sizes. For the minimization of production costs, as reported by patented studies [4], 20% sodium silicate was added into the reaction mixture.

Experimental methods

Sodium silicate was added to a solution of 84% NH_4NO_3. Silicic acid was first added to concentrated H_2SO_4 (1 mL silicic acid/5.6 mL H_2SO_4), and this solution was then added to the prepared fertilizer solution. Sulfuric acid is used to inhibit the formation of CO_2 during dilution of the fertilizer with $CaCO_3$; however, SO_4^{2-} promotes the formation of double salt complexes in solution, so silicic acid is added to H_2SO_4 to minimize this. Without further production processes, the NH_4NO_3 fertilizer was mixed with sodium silicate. This was followed by crystallization at a relative humidity of 75–95% and a fertilizer/water ratio of 2:1. The materials used for examination of the effects of internal additives were sodium silicate, silicic acid, H_2SO_4 (98%), anhydrous NH_3 (99.9%), HNO_3 (55%) and $CaCO_3$.

Electron microscopy analysis

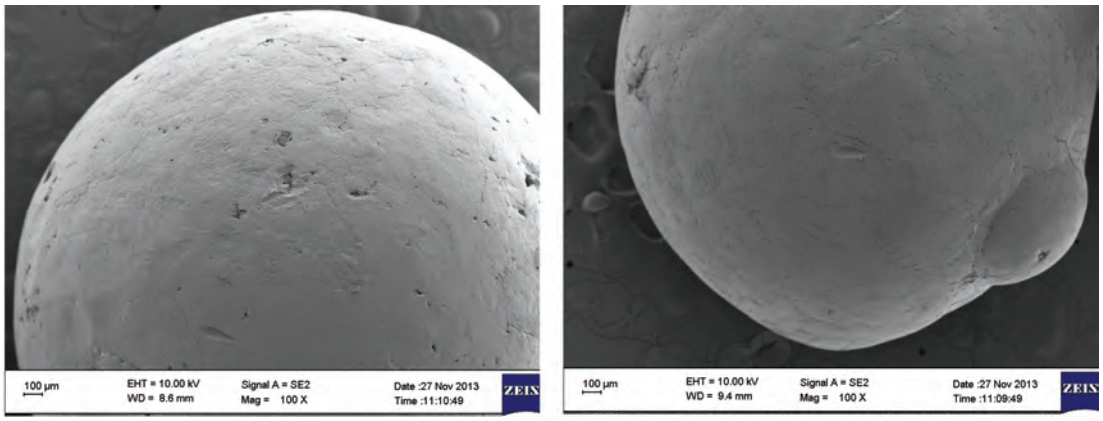
Electron microscopy was performed to investigate how sodium silicate, silicic acid, and sulfuric acid affected the fertilizer surface according to ASTM E986-97 [13] (Figures 2–4). For this analysis, a Carl

Table 1. Screened analysis of final production and final production after one year for ammonium nitrate prill particles, which contain calcium carbonate

Period, months	3.35 mm, %	2.5 mm, %	2.0 mm, %	1.0 mm, %	0.5 mm, %	E.A. mm, %	Crushing Strength	SO_4^{2-} ppm	N %
Final production	3.7	42.1	34.6	18.9	0.7	0	2.11	6.6028	26.52
Final production after one year	4.5	43.0	37.5	13.8	1.2	0	1.96	6.4317	26.33

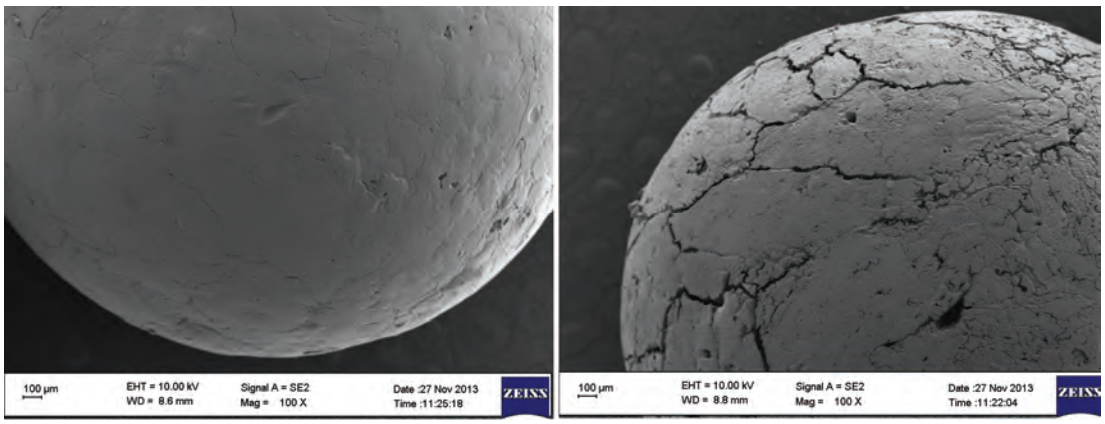
Table 2. Screened analysis of final production and final production after one year for ammonium nitrate prill particles, which contain calcium carbonate, sodium silicate, and silicic acid

Period, months	3.35 mm, %	2.5 mm, %	2.0 mm, %	1.0 mm, %	0.5 mm, %	E.A. mm, %	Crushing Strength	SO_4^{2-} ppm	N %	SiO_2 ppm
Final production	7.4	50.4	39.5	2.3	0.4	0	2.35	1.0004	26.49	0.1786
Final production after one year	7.1	50.1	38.4	3.7	0.7	0	2.12	0.9987	26.31	0.1783



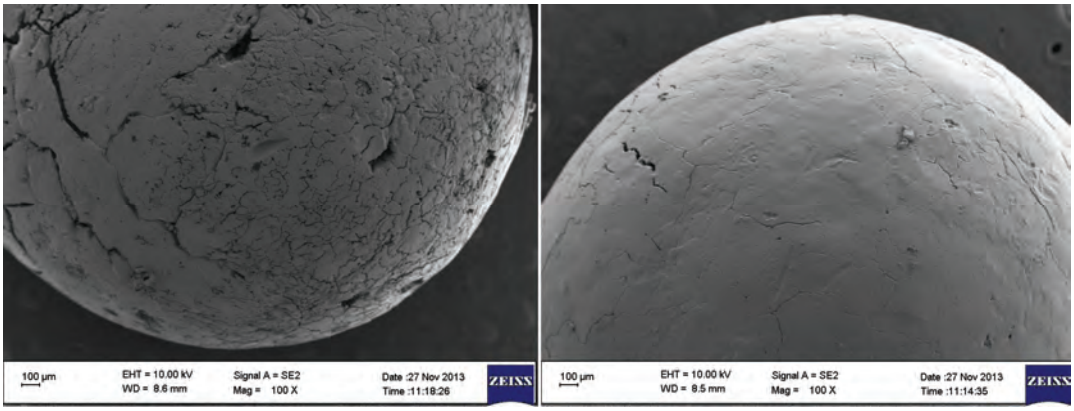
(a) (b)

Figure 2. SEM image of ammonium nitrate prill particle in which: a) calcium carbonate is added (after production) and b) calcium carbonate and sodium silicate are added (after production).



(a) (b)

Figure 3. SEM image of ammonium nitrate prill particle in which: a) silicic acid, calcium carbonate and sodium silicate are added (after production) and b) calcium carbonate is added (one year after production, under storage condition).



(a) (b)

Figure 4. SEM image of ammonium nitrate prill particle in which: a) calcium carbonate and sodium silicate are added (one year after production, under storage condition) and b) silicic acid, calcium carbonate and sodium silicate are added (one year after production, under storage condition).

Zeiss DSM-960A scanning electron microscope was used. The technical properties of this electron microscope are as follows: accelerating voltage, 1–30 kV; useful magnification, 10–30000×; resolution, 70 Å.

Ion chromatography analysis

For detection of the numbers of anions and cations of ammonium nitrate, ion chromatography was performed according to ASTM E1151-93 [14] (Figure 5) with a Shimadzu Prominence HIC-NS instrument, which has a measuring range of 0.01–51200 µS/cm and a flow rate control range of 0.001–5 mL/min.

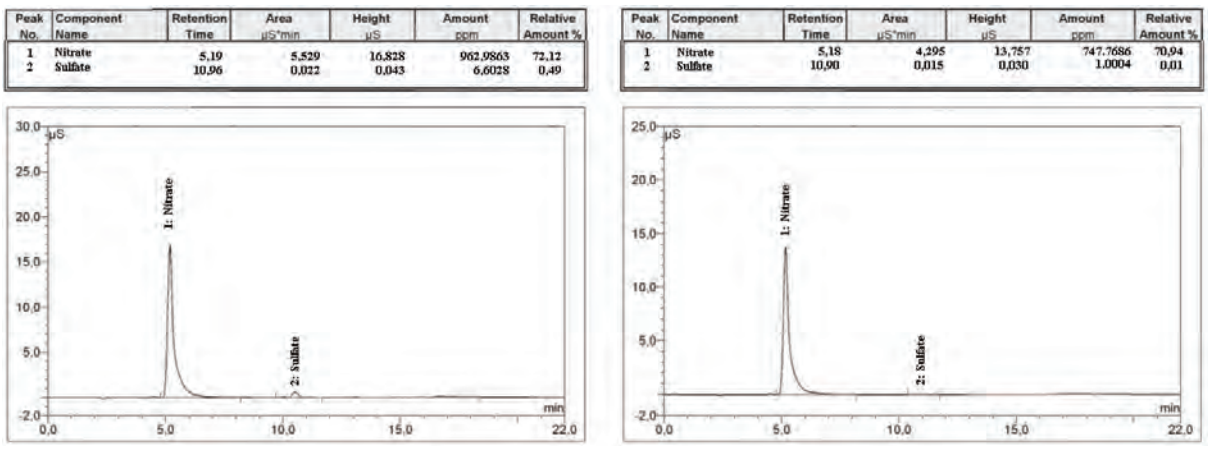
Screen sieve analysis

Fertilizer prills containing silicic acid, sulfuric acid, and sodium silicate were stored for one year (Table 1) and then subjected to screening analysis with a Vibratory Sieve Shaker AS 200 according to the ASTM E11-09 standard [15].

Analysis of sulfate

During fertilizer particle production, the amounts of sulfate salts require monitoring, since these species cause the degradation of NH_4NO_3. The detection method, based on the precipitation of barium sulfate ($BaSO_4$) in fertilizer solution consisted of: dilute HCl (d_{20} = 1.18 g/mL), $BaSO_4$ solution (122 g/L) and $AgNO_3$ solution (5 g/L).

Analysis samples were prepared by adding HCl (20 mL) to H_2SO_4 (50 mL); this solution was diluted with demineralized water to 300 mL. The prepared solution was then boiled. Barium chloride ($BaCl_2$; 20 mg) in water (20 mL) was added slowly, and the solution was boiled for a few minutes. The hot solution was allowed to stand for 1 h. Once this solution became clear, it was filtered, and the resulting precipitate was rinsed multiple times with hot water until no chloride was present in the filtrate, as determined



(a) (b)

Figure 5. Ion chromatogram of ammonium nitrate in which: a) calcium carbonate is added (after production on storage conditions) and b) calcium carbonate, sodium silicate, and silicic acid are added (after production, under storage condition).

by the use of $AgNO_3$. The filter paper and precipitate were placed in a porcelain cup in an oven at 500 °C for 1.5 h. The material was then left to cool in an isolated place.

Calculations: 1 mg $BaSO_4$ precipitate = 0.137 mg sulfur or 0.343 mg SO_3.

Sulfate in fertilizer was determined by:

$$S(\%) = W 0.0137\left(\frac{V_1}{V_2} M)\right) \quad (1)$$

$$SO_3(\%) = S(\%)2.5 \quad (2)$$

where W is the mass of $BaSO_4$ in the precipitate, V_1 the sample mass (volume), V_2 the total volume, and M the sample mass.

Analysis of silicic acid

The reagents used were dilute HCl (50%), concentrated H_2SO_4 (98%) and HF (48%).

A sample containing silicate (0.25 g) was placed in a platinum cup and fired in an oven at 500 °C for 1 h. The sample was rinsed twice with 50% HCl (10 mL). Demineralized hot water was then added to the solution to give a volume of 100 mL. Subsequently, all the water was evaporated in a water bath, and the cup was left in the bath. Next, 8–10 drops of HCl were added, and the sample was rinsed with water (50 mL).

The container with the solution was covered with a cover glass.This solution was then filtered through a filter paper (Whatman No. 42). The filter paper was placed in a platinum cup and 2–3 droplets of concentrated H_2SO_4 were added. This filter paper was fired slowly and kept in a muffle oven at 1000 °C for 0.5 h. The residue was cooled and weighed, and then two droplets of concentrated H_2SO_4 were added. Subsequently, 48% HF was added to a 25 mL portion of the original volume. Silicium fluoride was vaporized in a conventional oven at 250 °C for 1 h. This solution was kept in a muffle oven at 1000 °C for 0.5 h, and was then cooled and weighed.

Calculations:

$$SiO_2(\text{mg/L}) = 100 \text{lost weight} / \text{sample amount}(\text{mL}) \quad (3)$$

Analysis of nitrogen

Fertilizer samples of 7 g were used. The fertilizer was diluted to 500 mL with demineralized water. A 10 mL aliquot was taken from this solution, to which 20% NaOH (50 mL) was added. The solution was titrated with standard H_2SO_4.

Calculations:

$$N(\%) = (50 - A)F \quad (4)$$

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

RESULTS AND DISCUSSION

In the ammonium nitrate production process as supported by the EFMA (European Fertilizer Manufacturing Association), ammonium nitrate fertilizer is obtained as a result of the reaction between anhydrous ammonia (99.9%) and nitric acid (55%). According to commercial patents [10], various methods are used during production, and studies often focus on achieving the lowest costs and dilution rates depending on the manufacturers. The ammonium nitrate production process involves reaction parts that have several inputs and parameters. First, anhydrous ammonia is brought to process conditions through oxidation in the presence of a catalyst to form nitric oxide. This nitric oxide is then reacted with air to form nitrogen dioxide, which is mixed with water to give nitric acid. The acid is then reacted with gaseous anhydrous ammonia to yield ammonium nitrate salts. The other processes include the capturing of electrical energy and steam from the energy created during the production of nitric acid from ammonia.

In the present study, the method of concentration by vacuum is applied, which is the preferred method in industrial ammonium nitrate production, and has the lowest cost and wastewater problems from an ecological viewpoint [10]. In this vacuum concentration method, all suggested chemical additives that are used for dilution and to decrease the caking problem are added to the fertilizer solution after a suitable concentration is reached (99%, Figure 1), to control the concentration levels of the ammonium nitrate solution (which is concentrated on two evaporation stages) and to protect against any corrosive effect or other process problems for the process pipeline and equipment. Calcium carbonate is the most commonly used material added for the dilution of fertilizer solution. It is added to the fertilizer solution to compensate for the lack of lime.

Silicate is another required chemical material. For addition to fertilizer solution, the reaction between carbonate and silicate must be known and taken into account. In the analysis conducted, no gas production or foaming reaction was observed upon the addition of sodium silicate, which is used to transform silicate material into fertilizer solution, and the electron microscopy images showed that the obtained fertilizer prills had a spherical shape. Another chemical used for investigating its behavior in fertilizer solution was silicic acid. There have been some works on silicic acid for fertilizer production [9]. Silicic acid is added to fer-

tilizer bulk solution to decrease the amount of sulfuric acid, which forms a double salt with nitrate ions. Sulfuric acid is added to the fertilizer solution to consume the carbon dioxide created by the addition of calcium carbonate (which is used for diluting the fertilizer solution). Electron microscopy (Figures 2–4) and screen sieve analysis (Tables 1 and 2) show that the degradation tendency decreases upon addition of silicic acid, while the nutriment value increases; the silicic acid also dilutes the solution and reduces the caking tendency. In terms of fertilizer solution, this work is the first investigation on an industrial scale using instrumental analyses such as ion chromatography, electron microscopy, and screen sieve analysis.

The storage of ammonium nitrate after production also presents several challenges, as it is a strong oxidant, and readily absorbs moisture. This process is related to the phase transition during ammonium nitrate production, and degradation can be limited through the addition of chemical additives such as sodium silicate. This compound may be mixed with calcium chloride to precipitate calcium silicate, which is often used in the nitrogenous fertilizer industry since it increases the $CaCO_3$ content in soils that do not contain $CaCO_3$ (according to market requirements). This compound is often prepared in the nitrogenous fertilizer industry through precipitation as calcium silicate; this increases the rate of formation of calcium silicate in soils that do not contain calcium silicate, according to market requirements. Calcium silicate can be prepared in many ways from sodium silicate, with one of the most commonly used preparation methods involving the reaction between calcium chloride and sodium silicate [16]. The main purpose of this reaction is to determine the calcium content and nutriment requirement of the soil.

In the current study, a better chemical composition is proposed that is different from that using the chemicals typically suggested for limiting the degradation of ammonium nitrate crystals in many patented works. Moreover, a detailed chemical analysis of the degradation progress was performed by using ion chromatography, electron microscopy and sieve analysis. Through sieve analysis, it was possible to observe the effects on the prill particle size of different compounds typically used to dilute the ammonium nitrate solution, including calcium carbonate, sodium silicate, and silicic acid. These results showed that dilution with calcium carbonate during ammonium nitrate production led to a prill particle size that was smaller than that with sodium silicate. The sieve rate increased with an increase in ammonium nitrate degradation in the dilution process with the addition of calcium carbonate (Table 1) than with both sodium silicate and calcium carbonate (Table 2). Thus, better results were obtained with the mixture of sodium silicate and calcium carbonate. Carbonate, which is formed during the addition of calcium carbonate, creates foam and causes the shape of the ammonium nitrate prill to break. The amount of carbonate should be decreased to reduce the foaming reaction. In many patented works, sulfuric acid is added to the reaction bulk to minimize the formation of carbonate during ammonium nitrate production. However, sulfuric acid can form double salts with nitrate after production, depending on the storage conditions, and crystal bridges, which accelerate degradation, may be formed between these double salts. The crystal bridges, in turn, lead to deliquescence through hydrogen bonding between the particles. In the current study, silicic acid was used instead of sulfuric acid to minimize the formation of sulfate. Silicic acid was applied to consume any excess sulfuric acid remaining from the production process. Our results show that 1 mL silicic acid is capable of removing 5.6 mL sulfuric acid.

The SEM images show that the formation breaking of ammonium nitrate with sodium silicate was less than that with calcium carbonate. Over one year, in bulk ammonium nitrate under 0.28 kg/cm^2 pressure [17], the ammonium nitrate surface was more deformed with calcium carbonate than with sodium silicate (Figures 3b and 4b). From these investigations, it was possible to observe the effects of adding only calcium carbonate for the dilution of ammonium nitrate (Figures 2a and 3b), adding calcium carbonate and sodium silicate to increase the nutriment value of ammonium nitrate (Figures 2b and 4a), and adding silicic acid to decrease the formation of double salts (Figures 3a and 4b) in the ammonium nitrate production process.

According to the results of these studies, the best surface properties were obtained when ammonium nitrate production involved sodium silicate, calcium carbonate, and silicic acid. When sodium silicate solution was added with calcium carbonate to the ambient reaction, the observed crushing strength of ammonium nitrate (Table 2) was higher than that of the ammonium nitrate produced when only calcium carbonate was used for dilution (Table 1).

In this study, ion chromatography was also used to investigate the ammonium nitrate degradation process. The main purpose here was to understand the effects of silicic acid on the sulfate salts. In the current work, the sulfate amount in the bulk ammonium nitrate was analyzed after one year, and was observed to have decreased. This was attributed to the consumption of excess sulfuric acid by the addi-

tion of silicic acid, as discussed above (Figures 5a and 5b). Note that the ion chromatography analysis presented in the current work was conducted for experiments involving the addition of calcium carbonate only, and for the mixture of calcium carbonate, sodium silicate, and silicic acid. It was not conducted for the experiments involving the addition of calcium carbonate and sodium silicate, since sodium silicate has an inorganic structure, and hence, will not produce a foaming reaction releasing CO_2 as a product. Moreover, CO_2 is produced during the reaction between sulfuric acid and silicic acid, which was not used in the experiments involving only calcium carbonate and sodium silicate.

CONCLUSIONS

In this study, the effects of the addition of calcium carbonate, sodium silicate, and silicic acid in the ammonium nitrate production process were investigated. The addition of sodium silicate to the reaction mixture resulted in increased particle sizes compared to those produced with the addition of calcium carbonate, which is typically used to dilute ammonium nitrate solutions. The addition of silicic acid resulted in less breaking of the surface morphology, and in the decreased production of double sulfate salts that cause crystal bridges, thus reducing eventual caking problems.

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NAUČNI RAD

EFEKTI NATRIJUM-SILIKATA, KALCIJUM-KARBONATA I SILICIJUMOVE KISELINE NA RAZGRADNJU AMONIJUM-NITRATA I ANALITIČKA ISPITIVANJA PROCESA RAZGRADNJE NA INDUSTRIJSKOM NIVOU

Amonijum nitrat je neorganska hemikalija sa brojnim primenama u različitim industrijama. Međutim, različiti problemi su povezani sa proizvodnjom i kasnijim skladištenjem amonijum--nitrata, uključujući koksovanje, razgradnju, neželjene fazne prelaze i rekristalizaciju. Iako je razvijeno nekoliko tehnika u cilju rešavanja ovih problema, mnoge od njih ne rade u praksi. U ovom radu su različita jedinjenja, uključujući silicijumovu kiselinu i natrijum-silikat dodata da bi usporila napredovanje ili sprečila razgradnju amonijum nitrata. Više instrumentalnih analiza, kao što su jonska hromatografija i skenirajuća elektronska mikroskopija, korišćene su za praćenje procesa razgradnje.

Ključne reči: amonijum-nitrat, koksovanje, razgradnja, jonska hromatografija, proces posle reaktora.