



PROPERTIES OF AMMONIUM NITRATE AND METHODS FOR ELIMINATING ITS CAKING

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Annotation. Ammonium nitrate (AN) is a universal and agro chemically valuable nitrogen fertilizer. It is effective on all types of soils and for almost all crops. However, this fertilizer has two serious drawbacks - its caking during storage and increased explosion hazard. The theory of the caking process of granular AN is discussed in some detail in the literature and possible ways to eliminate it are shown. To eliminate the disadvantages inherent in AN, methods have been developed by introducing various additives into its composition. These additives can be divided into the following main groups: additives that bind free moisture; additives affecting the process of polymorphic transformations; dusting granules and treating them with surfactants and additives that form crystallization centers.

Key words: ammonium nitrate (AN), caking, non-caking AN, additives that bind free moisture, additives that affect the process of polymorphic transformations and additives that form crystallization centers.

Аннотация. Аммиачная селитра (АС) – универсальное и агрохимически ценное азотное удобрение. Она эффективна на всех типах почв и практически под все сельскохозяйственные культуры. Однако данное удобрение имеет два серьезных недостатка – это её слеживаемость при хранении и повышенная взрывоопасность. В литературе довольно подробно рассмотрена теория процесса слеживания гранулированной АС и показаны возможные пути её устранения. Для устранения недостатков, свойственных АС разработаны способы путем введения в её состав различных добавок. Эти добавки можно разделить на следующие основные группы: добавки, связывающие свободную влагу; добавки, влияющие на процесс полиморфных превращений; опудривание гранул и обработка их поверхностно-активными веществами и добавки, образующие центры кристаллизации.

Ключевая слова: аммиачная селитра (АС), слеживаемость, неслеживающаяся АС, добавки, связывающие свободную влагу, добавки, влияющие на процесс полиморфных превращений и добавки, образующие центры кристаллизации.

Annotatsiya. Ammiakli selitra (AS) universal va agrokimyoviy xossasi yuqori bo'lgan azotli o'g'itdir. U barcha turdagi tuproqlarda va deyarli barcha qishloq xo'jaligi ekinlari uchun samarali. Biroq, ushbu o'g'itning ikkita jiddiy kamchiliklari bor – saqlash vaqtida uning donalari o'zaro bir birlari bilan yopishib qolishi va portlash xavfining yuqoriligi. Donador AS ning yopishqoqlik xossasi ilmiy adabiyotlarda batafsil muhokama kilingan va uni bartaraf etishning mumkin bo'lgan usullari ko'rsatilgan. Uning tarkibiga turli noorganik qo'shimchalarni kiritish orqali AS kamchiliklarini bartaraf etish yo'llari ishlab chiqilgan. Ushbu ko'shimchalarni quyidagi asosiy guruhlarga bo'lish mumkin: erkin namlikni bog'laydigan; polimorf o'zgarishlar jarayoniga ta'sir qiluvchi; donalarni yuzasini qoplovchi va ularni kristallanish markazlarini hosil qiluvchi sirt faol moddalar kabi qo'shimchalardir.

Kalit so'zlar: ammiakli selitra (AS), yopishqoqlik, yopishqoqligi bo'lmagan AS, erkin namlikni bog'lovchi qo'shimchalar, polimorf o'zgarishlar jarayoniga ta'sir qiluvchi qo'shimchalar va kristallanish markazlarini hosil qiluvchi qo'shimchalar.

Introduction

Ammonium nitrate (AN – NH_4NO_3) is a colorless crystalline substance with a molecular weight of 80.05 cu., contains 35% N in ammonium and nitrate forms. AC is highly soluble in water. Dissolution occurs with significant heat absorption. AC is a strong oxidizing agent for



a number of organic and inorganic compounds. Ammonium nitrate in the molten state decomposes noticeably according to the equation: $\text{NH}_4\text{NO}_3 = \text{NH}_3 + \text{HNO}_3 - 41,7 \text{ kilo cal.}$, and the acidity of the melt gradually increases.

The main disadvantage of AN is its hygroscopicity and high solubility in water, which varies greatly with temperature. The ability of AN granules to easily moisten and dry with changes in temperature and relative humidity of the air with which they come into contact leads to caking of the mass of granules. Under unfavorable storage conditions (poor condition of the container, frequent fluctuations and air humidity), loose AN at the beginning of granulation can turn after some time into a solid mass [1].

Materials and methods

A significant disadvantage is the polymorphism inherent in ammonium nitrate [2]. The structural prerequisite for this is flat NO_3^- ions, which occupy different positions in crystals of different modifications and, within certain temperature limits, change their rotationally symmetric state. NH_4NO_3 has five different modifications. The centers of the nascent phase are always present in the mother phase and initiate the transformation. The mechanism of the transformation itself consists of the dissolution of one phase and the crystallization of another. The transition from one form of crystals to another is accompanied by a change in volume, crystal density, refractive index, specific heat capacity and other properties, as well as the release of a certain amount of heat [3,4]. Temperature 32°C is the transition point of crystalline modification $\text{III} \leftrightarrow \text{IV}$. Modification III is stable within the range of $32-84^\circ\text{C}$. The transition of modification IV to III is accompanied by a thermal effect and an increase in the volume of AN by 0.00221 cm^3 per 1 gram of salt. The reverse transition is accompanied by a reduction in volume by the same amount. This is why AN, when stored in warehouses or in the sun, increases in volume, taking on the appearance of a fluffy mass with a low volumetric weight (up to 0.41 g/cm^3).

In the second half of the last century, the formation and rapid development of the nitrogen industry led to extensive study of the crystal structures and thermodynamic properties of NH_4NO_3 [5-7]. These studies were carried out using physicochemical methods. Studies [4, 7] show that when heating and cooling NH_4NO_3 , along with successive transitions $\text{I} \leftrightarrow \text{II} \leftrightarrow \text{III} \leftrightarrow \text{IV} \leftrightarrow \text{V}$, at $47-55^\circ\text{C}$ a metastable transition $\text{II} \leftrightarrow \text{IV}$ is observed, bypassing modification III, and with the introduction in NH_4NO_3 of some additives – metastable transformation $\text{II} \leftrightarrow \text{V}$ [6].

Fundamental studies of the rate of transformation $\text{IV} \leftrightarrow \text{III}$ and $\text{II} \leftrightarrow \text{III}$ were carried out by B.V. Erofeev and N.E. Mitskevich [5], who showed that the kinetics of these processes is described by the topo kinetic equation: where: α is the fraction of the reacted substance; k , n – constants characterizing the rate of transformation; t – time. They showed that the rate of overall transformation depends on the rates of emergence of nuclei of the new phase and their growth. The conditions of preliminary thermal treatment of the sample under study and its history strongly influence the rate of modification transformations of NH_4NO_3 . The applicability of the above equation or its various modifications to describe the kinetics of polymorphic transformations $\text{IV} \leftrightarrow \text{III}$ and $\text{II} \leftrightarrow \text{III}$ was established in [5].

Many works have been devoted to studying the effect of moisture on the temperature of modification transformations of NH_4NO_3 [4]. In these works, a pattern was identified where transformations $\text{IV} \leftrightarrow \text{II}$ occur with substitution, and $\text{IV} \leftrightarrow \text{III}$ and $\text{III} \leftrightarrow \text{II}$ occur with structural restructuring. It was shown that the $\text{III} \leftrightarrow \text{II}$ transformation occurs easily and completely in a dried sample. The authors suggest that in the presence of moisture, nuclei of a new phase form on the surface of particles that have adsorbed moisture, then they begin to grow, as a result of which the transformation occurs. In dry samples, the centers of growth of the new phase are residual nuclei. The presence of moisture does not affect the change in the structure of NH_4NO_3 crystals, but increases the rate of transformation. AN humidity



decreases, the IV \leftrightarrow III transformation temperature increases to 51°C, and the transition temperature drops to 47°C. At low humidity, the metastable transformation IV \leftrightarrow II occurs at 47°C. The results of physicochemical research methods are confirmed by practical experience in the use of NH₄NO₃, which shows that the volume of AN changes only in cases where the salt contains a certain amount of moisture - 0.5% and above. Dry speakers, even with a sharp change in temperature, do not change volume for a long time.

Caking of AN lead to large losses and additional labor costs for grinding the product before use [8]. It has been shown to be caused by low mechanical strength of granules, high hygroscopicity of salt, polymorphic transformations, as well as technological shortcomings [8, 9].

To reduce the caking of saltpeter, it is advisable to obtain it in the form of granules of the same size, and pack it in multi-layer paper bags, already dried and cooled. Saltpeter containing 0.8-1% moisture, loaded into paper bags at a temperature of 60°C, strongly cakes within several days. The work explains why a phase transition at 32.3°C can lead to caking of granular nitrate, and in work [10] this is confirmed experimentally.

To eliminate caking of AN, methods have been developed to improve its physical properties, aimed at solving the following problems:

- reduce the moisture content in the AN to a minimum by increasing the concentration of the initial melt and using water-removing additives;
- protect the surface of the granules from moisture by covering them with hydrophobic substances;
- eliminate direct contact between the surface of the granules by covering them with various dusting substances;
- change the nature of polymorphic transformations occurring in AN granule by introducing additives into its solutions or melts so that when stored in warehouses, AN is always in a stable modification;
- create a condition for volumetric crystallization in a melt drop by using additives that form crystallization centers [11].

Experience in the production of granular AN indicates that to obtain a product with good physical properties, it is necessary that the moisture content in it be no higher than 0.2%. However, producing AN with such humidity requires bringing the melt evaporation temperature to critical (200°C), which sharply increases the explosiveness of the process.

To eliminate the disadvantages inherent in AN, methods have been developed by introducing various additives into its composition. These additives can be divided into the following main groups [1].

Additives that bind free moisture. Magnesium additive is used at several enterprises in the CIS countries. This is one of the best additives to speakers that eliminate caking. It is a caustic magnesite obtained by firing the natural mineral magnesite. To decompose it, 35% nitric acid is used, thereby obtaining an approximately 40% solution of magnesium nitrate. The magnesium nitrate present in the AN solution is gradually dehydrated in the process of obtaining a 99.8% AN melt. One part by weight of anhydrous magnesium nitrate can bind about 0.7 parts by weight of water.

Caustic magnesite is used by two factories in Uzbekistan - Maksam-Chirchik JSC and Ferganaazot. Navoiazot JSC also uses magnesite additive, but in the form of the natural mineral brucite Mg (OH)₂.

The same group of additives that bind water in the AN melt includes the so-called dolomite additive. But compared to the magnesium additive, it gives the desired effect at a lower moisture content in the product. It is obtained by decomposing dolomites containing 32-33% CaO, 16-19% MgO and 43-44% CO₂ with nitric acid. Dolomite additive was widely used in the Soviet Union for a number of years, but recently it has been gradually replaced by magnesium additive [1]. But despite this, work continues to improve the process of producing AS with dolomite additive.



Such additives include calcined gypsum, which can add 2 molecules of water to form solid crystalline hydrate $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ [12-14]. One of the conditions for the reliable action of such solid additives is their high degree of dispersion, which ensures uniform distribution of additives in the granule, greater completeness of use of additives and a high rate of absorption of moisture from the mother liquor. A finely ground additive heated to melt temperature should be added to the melt before granulation. A technically uniform supply of solid additives can be easily achieved, for example, using a small screw feeder connected to the pipe section that supplies the melt to the spray basket. The uniform distribution of the additive in the melt is achieved due to intensive mixing of the melt in the basket itself during its rotation or the simultaneous action of the basket and a simple propeller-type mixer.

Powdering granules and treating them with surfactants. In [15], the influence of highly dispersed additions of glauconite, pyrophyllite and clinoptilolite on the physicochemical and mechanical properties of NH_4NO_3 was studied. In [16], it is recommended to use the mineral pyrophyllite in an amount of 0.05-2.5 wt.% as a powdering additive to AN granule. At the same time, the caking ability of speakers decreases by 17-40 times. And when glauconite is used in an amount of 0.5-5 wt.%, caking decreases by 5-12 times [17]. After six months of storage, saltpeter with glauconite retains 100% friability.

In [18], a method was proposed for eliminating the caking of nitrate by dusting it with a powdered silicon-containing substance, characterized in that, in order to completely eliminate the caking of nitrate, aerosil coated with an organosilicon layer containing Si-R bonds is used as a powdered silicon-containing substance, where R is one from organic radicals. Modified Aerosil is introduced in an amount of 0.1-0.3% of the weight of the AN.

In [19], in order to increase the adhesion of dusting materials to the surface of AN granules and reduce the cost of the conditioning method, crushed hydrolytic lignin is used as a dusting material in an amount of 0.1-5.0%. In [20], it is proposed to dust AN granule with a mixture of modified aerosil with highly dispersed ammophos in the ratio (1-6): (94-96) in an amount of 0.1-0.5% of the weight of the fertilizer.

In [21, 22], chemical production wastes or products synthesized on their basis were used as additives to granular AN.

In [23, 24], two surfactants were successfully used to eliminate the caking of AN: a sulphonic product - a waste from the electrode industry and a tributylamine salt of the sulphonic product of this waste, which is a mixture of 4, 5, 6 - nuclear aromatic hydrocarbons and heterocyclic compounds containing N, S atoms and O.

In [25], the caking of AN is eliminated by spraying its granules with a surfactant, which is polyethylene glycol ester of phosphoric acid (oxyphos) in an amount of 0.02-0.10% by weight of nitrate. In [26], the caking of AN is eliminated by spraying the granules with a 3-5% solution of sodium ethyl silicone in an amount of 0.05-0.1% by weight of nitrate. In [27], an organosilicon water-repellent liquid is used. Nitrate granules are sprayed with a 50-20% aqueous emulsion of polyethyl hydro siloxane in an amount of 0.05-0.1% by weight of nitrate.

In [28], AN granule is sprayed with a surfactant, which is a melt of nonionic surfactant OP-7 at 40-50°C in an amount of 0.04-0.1 wt.%. OP-7 is a product of the interaction of alkyl phenols with ethylene oxide (a mixture of polymer homologues of mono- and dialkyl phenols).

Additives that form crystallization centers. The introduction of solid insoluble impurities into the AN melt promotes the formation of granules with a fine-crystalline structure that have high density and strength.

According to the author's certificate [29], a non-caking nitrogen fertilizer with increased granule strength is obtained by introducing 1-2% salt into the AN melt with the general formula $3\text{K}_2\text{SO}_4 \cdot \text{Al}_2(\text{SO}_4)_3$, isolated from dehydrated alunite.

The strength and thermal stability of AN granules is increased by introducing into its melt 0.1-0.75% modified copper and sulfide, copper aerosol [30].



An increase in the mechanical strength of granules up to 1710 g/granule, resistance to modification transition and a decrease in the dissolution rate can be achieved if 2 g per 100 g of borogypsum nitrate, a waste from the enrichment of datolite ore (mass %): $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ 76.73, is added to the AN melt before granulation.; SiO_2 22.18; B_2O_3 1.09; pre-heat-treated at 130-180°C for 2 hours [31].

According to the author's certificate [32], 0.24-0.76% calcium or magnesium borate containing 0.05-0.1% boron, 0.19-0.49% magnesium oxide or 0.05-0.16% calcium oxide, and the rest is impurities and pre-crushed to 200 microns and dehydrated at 200-250°C. The strength of the product increases by 1.3-2.8 times.

Substances that promote the formation of crystallization centers in the NH_4NO_3 melt are vermiculite, talc, diatomite, perlite, aerosol, kaolin, bentonite, etc. It is known that the introduction of solid insoluble impurities into the NH_4NO_3 melt promotes the formation of granules with a fine-crystalline structure, which have a higher density and strength. It is proposed to use clays, various silicates, talc, silica gel, diatomite, aerosol, etc. as such substances. Of the numerous additives proposed in the literature that form crystallization centers, only bentonites have found industrial application [33].

In order to reduce the caking of AN, in the author's certificate [33], a structure-forming additive is introduced into its melt, for which bentonite is used in an amount of 0.5-2.0 wt.%, and the granules are then sprayed with a dispersant NF (sodium or ammonium salt of dinaphthyl methane disulfonic acid) in amount 0.5-3.0%. After applying the surfactant film, the granules are dusted with vermiculite in an amount of 1% weight. After storing the AN for six months in the open air, the samples crumbled without any effort.

Work [34] also studied the effect of adding bentonite on the strength of granules and caking properties of AN. In the experiments, bentonite powders were used, obtained from bentonite clays of the Gumbrinsky and Askansky (Georgia), Azkamarsky (Uzbekistan), Cherkasy and Krivorozhsky (Ukraine) deposits. Before using the clay, it was ground in a ball mill. The additive had particles with a size of 40 microns and a moisture content of 1-2%. The AN was melted in a reactor, then bentonite was added to the melt at 170-175°C and constant stirring. The resulting melt was granulated by pilling. Samples of granulated AN were obtained with the addition of 0.5-3.0% of various bentonites. The experimental results showed that the addition of bentonite in an amount of 1-3% can significantly increase the strength of AN granules, its resistance to III-IV modification transformations, and also reduce caking. The most effective additive is bentonite from the Cherkasy deposit. If the strength of AC granules without additive was 0.54 MPa, with 0.3% addition of this bentonite it was 0.97 MPa, then with 3% addition it was 2.2 MPa. The caking ability of nitrate without additive was 5.6 kg/cm², and with 3% additive – 2.53 kg/cm².

And in works [35-43], the process of obtaining non-caking and thermostable AS was studied by adding bentonite clays from the deposits of Uzbekistan (Navbakhorsky, Lagonsky, Kattakurgansky, Azkamarsky) to the NH_4NO_3 melt of the "pure" grade (99.85%) before its granulation. In this case, the amount of bentonite was changed from 0.5 to 3 g relative to 100 g of NH_4NO_3 melt. The composition and properties (strength of granules, caking ability, dissolution rate of granules, thermal stability) of the resulting products were determined. The rheological properties of bentonite-nitrate melts in the temperature range 165-185°C were studied. The results of the study are a reduction in caking, an increase in the strength and resistance of fertilizer granules to thermal heating-cooling cycles.

Additives affecting the process of polymorphic transformations. The sulfate additive is ammonium sulfate added to the AN solution. Adding a small amount of ammonium sulfate to the AN solution practically eliminates dust in the granite tower. At enterprises producing speakers with a sulfate additive, the consumption rate of ammonium sulfate is 4 kg per 1 ton of finished product.

Phosphate-sulfate-borate additive (called "Permalen-34") developed by the American company Mississippi Chemical Corporation [1;44] differs from phosphate-sulfate in that, in



addition to phosphate and ammonium sulfate, it contains orthoboric acid. These components are introduced into the AN solution in the form of aqueous solutions of orthoboric acid, pure diammonium phosphate and technical ammonium sulfate. In AN granule containing the additive "Permalen-34", the temperature of the polymorphic transformation IV→III shifts from 32°C to 43°C, and the point of polymorphic transformation II→III at 84°C, characteristic of pure NH_4NO_3 , is completely absent. Nitrate granules with the addition of "Permalen-34" withstand 1400 transitions of 20-60°C, while most samples with other additives are destroyed after 60-100 transitions. The presence of a phosphate-sulfate additive in the AN melt prevents the decomposition of the melt during deep boiling and thereby increases the safety of this process [45]. The production of AN with a phosphate-sulfate additive was discontinued due to a shortage of thermal phosphoric acid.

The phosphate-sulfate additive consists of ammonium phosphates and ammonium sulfate, introduced into the AN solution in the form of solutions of these salts, or in the form of phosphoric and sulfuric acids, then neutralized with ammonia gas to a solution pH of 5.5-6.8. At the same time, saltpeter granules have higher strength than pure saltpeter, less caking, and also, as already noted, higher resistance to variable temperatures. The presence of a phosphate-sulfate additive in the AN melt prevents the decomposition of the melt during deep boiling and thereby increases the safety of this process [45].

As can be seen from the above analysis of literature data, the physicochemical principles of AN conditioning are quite well developed, and if we produce nitrate strictly guided by these principles, we will obtain a non-caking product with durable granules and reduced sensitivity to polymorphic transformations.

Conclusion

Hundreds of anti-caking additives have been described in the literature, but only a few of them are used in industry. The best of them is magnesium supplement. Factories in Uzbekistan that produce AN purchase both caustic magnesite and brucine from Russia for foreign currency. It is very important for them to find a saltpeter additive based on local raw materials. In this regard, ammonium sulfate, mono- and diammonium phosphate (ammophos), calcium phosphates and phosphorites of the Central Kyzylkum are the most promising additives, the production volumes of which are sufficient in Uzbekistan.

Based on the above, conducting systematic research on the development of technologies for producing non-caking AN based-on ammonium nitrate melt, mono- and diammonium phosphate (ammophos), calcium phosphates and phosphorites of the Central Kyzylkum with the addition of ammonium sulfate is a very urgent task.

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