NPSCA-containing fertilizers based on ammonium nitrate melt and powder Suprefos-NS

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Abstract. Methods for improving the composition and properties of ammonium nitrate (AN) using various inorganic additives are considered. It has been shown that the employing Suprefos-NS, a product of JSC "Ammofos-Maxam", as an additive in the AN melt allows obtaining a stabilized fertilizer containing, except nitrogen and phosphorus, macroelements such as calcium and sulfur, exclusively in assimilable forms. In this case, the mass ratio AN: Suprefos-NS varied in the range from 100: 5 to 100: 35. It was found that any amount of Suprefos-NS additives significantly improves the composition and properties of the AN.

1. Introduction

Ammonium nitrate (AN) is the most widespread and demanded fertilizer in the agricultural sector around the world. In Uzbekistan, AN is produced by three largest chemical enterprises (JSC “Maxam-Chirchiq”, “Navoiyazot” and “Farg’onaazot”) in the amount of over 1 million 700 thousand tons per year. It is used both domestically and sent for export. The volume of AN output is constantly increasing. The main disadvantages of the fertilizer are its high caking ability due to hygroscopicity, solubility, modification transitions; thermal instability [1]. In addition, the presence of only one nutrient (nitrogen) in the AN reduces the agrochemical value of this fertilizer. A more radical solution is the transition to the production of complex fertilizers based on AN, which retains agrochemical efficiency, with significantly greater resistance to external influences and, accordingly, less explosiveness. For this, research is underway to select highly effective additives that improve the strength of the granules and increase the thermal stability of the fertilizer. Calcium carbonates, dolomite, potassium chloride, ammonium sulfate, gypsum or phosphogypsum are used as additives that reduce the level of potential hazard and improve the commercial qualities of nuclear power plants [2-8]. In this respect, phosphorus-containing additives are promising [9]. Namely their application can positively solve the problem of improving the quality of nitrate in terms of enriching its composition with a phosphorus component with a simultaneous decrease in its detonation ability. In Russia, nitrogen fertilizers are already produced on the basis of AN with a nitrogen content of no more than 27% with the addition of a liquid complex fertilizer of the composition 11% N and 37% P₂O₅ based on superphosphoric acid [10]. However, there is occurred the inlay process on the wall apparatus that requires preparation each month in AN producing factory. In that case, it would be better to apply ammonium phosphate purified free from sulphate, iron, aluminum and other impurities ions [11].
In [12-15], a stabilized AN technology was developed called NPF (nitrogen-phosphorus fertilizer) based on ammonium nitrate melt and high-carbonate powder phosphorite of Kyzyl Kum. For that there are solved two goals at once. Firstly, to obtain a stabilized AN, and secondly, to convert the phosphate raw material into an effective phosphoric fertilizer. The second goal is achieved by transferring the indigestible form of P\textsubscript{2}O\textsubscript{5} into the raw material into a assimilable form for plants. It was found that with the introduction of phosphorite powder (17-18% P\textsubscript{2}O\textsubscript{5}, 14-16% CO\textsubscript{2}) into the ammonium nitrate melt in the amount of 3.52-5.04% P\textsubscript{2}O\textsubscript{5} in the products, the nitrogen content ranges from 25.24 to 28.09%. The main result is that if in the initial phosphate raw material the relative content of the assimilable form of P\textsubscript{2}O\textsubscript{5} in relation to the total is 17.5%, then this indicator in the resulting NPF will increase from 88.29 to 94.6%. This suggests that the AN melt activates phosphorite, that is, it converts the indigestible form of P\textsubscript{2}O\textsubscript{5} into an assimilable form. In the products, the strength of the granules is in the range of 7.33-7.80 MPa, which is 4.5-5 fold higher than the strength of the standard AN (1.6 MPa).

In addition, activation process can be existed based acidulation Kyzyl Kum phosphorite by nitric acid followed by composting with bird litter to derive organomineral fertilizers [16]. But organic matter is not reliable in terms of AN due to its explosiveness. Organic matter is useful in terms of composting low-grade phosphorite [17]. Hereby we decided to obtain a stabilized AN by introducing nitrogen-phosphorus-sulfur-containing fertilizers into its melt under the name Suprefos-NS, a product produced by JSC “Ammofos-Maxam”. Its composition (wt.\%): N - 11.0; P\textsubscript{2}O\textsubscript{5 total} - 23.82; P\textsubscript{2}O\textsubscript{5 assim.} - 20.28; P\textsubscript{2}O\textsubscript{5 water} - 4.89; CaO\textsubscript{total} - 18.78; CaO\textsubscript{assim.} - 15.13; SO\textsubscript{3 total} - 26.72; SO\textsubscript{3 water} - 25.33; pH - 5.3; the strength of the granules is 3.5 MPa. Suprefos-NS consists of 45% precipitate, 40% ammonium sulfate and 15% mono- and di-ammonium phosphate. The product is produced by deep conversion of phosphoric acid gypsum pulp using gaseous ammonia (pH = 7.5-8), carrying out the reaction as follows [18-20]:

\[
\text{CaSO}_4 \cdot 2\text{H}_2\text{O} + \text{H}_2\text{PO}_4 + 2\text{NH}_3 \rightarrow (\text{NH}_4)_2\text{SO}_4 + \text{CaHPO}_4 \cdot 2\text{H}_2\text{O}
\]

This reaction was confirmed by studying the interaction of components in the systems \(\text{NH}_4\text{H}_2\text{PO}_4 - \text{CaSO}_4 \cdot 2\text{H}_2\text{O} \cdot \text{H}_2\text{O}\), \((\text{NH}_4)_2\text{HPO}_4 - \text{CaSO}_4 \cdot 2\text{H}_2\text{O} \cdot \text{H}_2\text{O}\) and \(\text{NH}_4\text{H}_2\text{PO}_4 - (\text{NH}_4)_2\text{HPO}_4 - \text{CaSO}_4 \cdot 2\text{H}_2\text{O} \cdot \text{H}_2\text{O}\) depending on time, temperature and their molar ratio [21].

The aim of this work is to study the composition and some properties of samples of NPSCa-containing fertilizers obtained by introducing Suprefos-NS powder into an ammonium nitrate melt at a wide range of NH\textsubscript{4}NO\textsubscript{3}: Suprefos-NS mass ratios.

2. Materials and Methods

The experiments were carried out as follows. Granulated AN (34.5% N) produced by JSC “Maxam-Chirchiq” was melted at 170 °C in a metal cup on an electric stove. Ground Suprefos-NS was introduced into the melt in such an amount that the final product contained from 1 to 6% P\textsubscript{2}O\textsubscript{5}. That is, an additive was introduced into the nitrate melt at the ratio AN: Suprefos-NS = 100: 5-35. The mixture was stirred at 180 ° C for 20 minutes. The melt granulation was carried out on a granulator, which is a metal cylindrical bowl with a perforated bottom, the diameter of the holes in which was equal to 1 mm. For this, the melt was poured into the granulator, then a pressure was created by a pump in the upper part of the bowl, and the melt was sprayed from a height of 25 meters onto a plastic film lying on the ground. Afterwards, the strength of the granules was measured.

The static strength was determined using the standard procedure [20] for granules with a diameter of 2-3 mm. The force required to break the granules was measured using an MIP-10-1 instrument.

Determination of caking of NPSCa-fertilizer samples was carried out in accordance with the method proposed by JSC “NIUIF” [22]. Fertilizer granules with a diameter of 2-3 mm were placed in perforated presses equipped with calibrated springs and kept for 8 hours in a climatic chamber with internal circulation Binder KBF-115 (made in Germany) at a temperature of 40 ° C, then for 2 hours at room temperature and humidity. The spring load of the presses was 2.8 kg. The value of caking was determined as the force required to destroy a cylindrical briquette formed from granules, having a diameter of 33 mm and a height of 40 mm, referred to its cross-sectional area. The briquettes were tested for destruction using the MIP-10-1 device.
The hygroscopic characteristics of fertilizers pre-dried to constant weight with a granule size of 2-3 mm were determined by the desiccator method at a temperature of 25 °C and a relative humidity of 50; 62.5; 69.5; 74.5; 80% [23]. Determination of the gain or loss of moisture in the substance at a constant temperature and certain relative air humidity was carried out for 3 hours. The required relative air humidity was created in a closed desiccator above a layer of sulfuric acid of a known concentration poured into it.

The nitrogen content in the products was determined according to Kjeldahl – distillation of ammonia in an alkaline medium with Devard’s alloy followed by titration [24], SO₄²⁻ by weight ion – by precipitation in the form of barium sulfate [25]. The content of phosphorus as a P₂O₅ in the general, assimilable and water-soluble forms was determined by a differential photometric method using a phosphorus – vanadium – molybdenum complex [26]. The determination of the CaO content was carried out by the volumetric complexometric method: titration with 0.05 N solution of Trilon B in the presence of the fluoroexon indicator. The assimilable forms of P₂O₅ and CaO were determined by solubility in 2% citric acid.

The density of NPSCa melts was determined by the pycnometric method with a measurement accuracy of 0.05 %, and the kinematic viscosity - using a glass capillary viscometer VPJ-1 with an uncertainty of 0.2 % in the temperature range 175-185°C.

The buffering effect of Suprefos-NS on the pH of nitrate as a function of time was studied as follows. AN or the test sample was introduced into the reactor, which was placed in a thermostat. The temperature in the thermostat was raised to a value (180°C) at which the sample was completely melted. Then the stopwatch was turned on, and every 20; 40; 60; 80; For 100 and 120 min, samples were taken from the melt using a glass spoon. From the cooled products, 10% aqueous solutions were prepared and their pH was measured on a laboratory I-130M ion meter with an electrode system of ESL 63-07, EVL-1M3.1, and TKA-7 electrodes with an accuracy of 0.05 pH units.

As a sample for comparison with the studied samples, the granular AN (with the addition of 0.28% MgO and the strength of the granules of 1.6 MPa) was chosen.

3. Results and Discussion

The rheological properties of melts obtained by the interaction of AN’s melt with Suprefos-NS is an important parameter that determines the possibility of their processing into granular NPSCa fertilizers. In this regard, the primary task was to study the density and viscosity of melts at the above mentioned ratios AN: Suprefos-NS in the temperature range 175-185 °C. The results are shown in Figure 1.

![Figure 1. Density (a) and viscosity (b) of NPSCa-melt against of the amount of Suprefos-NS additive and temperature](image-url)
As can be seen from these Figure 1, Suprefos-NS has a significant effect on both the density and the viscosity of the AN’s melt. So, if at 180 °C the density and viscosity of the melt of a conventional AN is 1.439 kg / cm³ and 5.09 cps, then with a 15% addition of Suprefos-NS these indicators increase to 1.478 kg / cm³ and 7.85 cps, and at 24% additive up to 1.502 kg / cm³ and 12.2 cps, respectively. The more Suprefos-NS is introduced into the AN’s melt, the higher the density and viscosity of the latter. The highest values of density (1.53 kg / cm³) and viscosity (21.35 cps) are obtained when the ratio of AN: Suprefos-NS = 100: 35. Moreover, when the temperature drops to 175 °C, the melt begins to solidify. However, in any case, the rheological properties of the AN’s melt with the addition of a complex fertilizer remain quite acceptable for pumping the melt and its granulation by the prilling method. The results showed that with an increase in the amount of Suprefos-NS additive to the melt of AN significantly increases the crystallization temperature, as a result of which the melt of the mixture begins to solidify (Figure 2). Therefore, we kept the resulting mixture at higher than 175 °C and constant stirring, after which we carried out the granulation process.

The strength of granules of NPSCa-fertilizer samples based on AN’s melt and Suprefos-NS powder are given in Table 1. From the data in the Table 1 can be seen that the strength of fertilizer granules increases with an increase in the amount of additive. So, with the addition of Suprefos-NS to the AN’s melt at a mass ratio of 100: 5, the strength of the product granules is 7.05 MPa, at 100: 15 - 9.34 MPa, at 100: 24 - 10.79 MPa, and at 100: 35 is already 11.7 MPa. That is, with an increase in the amount of the additive in AN’s melt leads to an increase in the strength of the NPSCa-fertilizer granules in comparison with the strength of the standard AN granules (1.6 MPa) by 4.4-7.3 fold.

This indicates a decrease in the propensity of complex fertilizers to detonate, which is explained by a decrease in the size of the crystals, which ensure their denser packing, which is confirmed by the higher density of the new type of fertilizer in comparison with ordinary nitrate. The presence of Suprephos-NS in the mixture interrupts the homogeneity of the properties of the AN and the zone of propagation of the detonation wave. Thus, (NH₄)₂SO₄, NH₄H₂PO₄, and CaHPO₄ contained in Suprefos-NS when cooling the NPSCa fertilizer melt in the granulation tower by prilling acts as a structurant.
Table 1. Chemical composition of fertilizers obtained by introducing finely grounds Suprephos into the ammonium nitrate melt

| Mass ratio of AN: Suprephos-NS | Composition, mass. % | The strength of granule, MPa |
|-------------------------------|----------------------|----------------------------|
|                               | N  | P2O5_total | P2O5_assim | P2O5_water | CaO_total | CaO_assim | SO3_total | SO3_water |                      |
| Superfos-NS                  |    |            |            |            |            |            |            |            |                      |
| 100 : 5                      | 33.47 | 32.40 | 31.52 | 31.01 | 30.48 | 30.01 | 29.49 | 28.45 | 10.85 | 23.82 | 20.28 | 4.89 | 18.78 | 15.13 | 26.72 | 25.33 | 3.5 |
| 100 : 10                     | 32.40 | 2.12 | 2.03 | 1.08 | 1.20 | 1.20 | 1.59 | 1.90 | 6.16 | 10.04 | 0.62 | 0.90 | 0.62 | 1.28 | 1.55 | 2.44 | 2.36 | 4.51 |
| 100 : 12                     | 32.08 | 2.56 | 2.41 | 2.01 | 1.80 | 1.80 | 1.97 | 2.24 | 7.05 | 23.82 | 20.28 | 4.89 | 18.78 | 15.13 | 26.72 | 25.33 | 3.5 |
| 100 : 15                     | 31.52 | 3.11 | 3.23 | 3.32 | 3.49 | 4.24 | 4.63 | 5.47 | 6.16 | 10.04 | 0.62 | 0.90 | 0.62 | 1.28 | 1.55 | 2.44 | 2.36 | 4.51 |
| 100 : 18                     | 31.01 | 3.61 | 3.32 | 1.26 | 1.80 | 1.97 | 1.97 | 2.24 | 4.04 | 9.97 | 0.62 | 0.90 | 0.62 | 1.28 | 1.55 | 2.44 | 2.36 | 4.51 |
| 100 : 21                     | 30.48 | 4.12 | 1.20 | 1.80 | 2.44 | 2.44 | 2.44 | 3.50 | 6.95 | 23.82 | 20.28 | 4.89 | 18.78 | 15.13 | 26.72 | 25.33 | 3.5 |
| 100 : 24                     | 30.01 | 4.63 | 4.24 | 3.27 | 2.67 | 4.66 | 4.66 | 4.66 | 10.25 | 23.82 | 20.28 | 4.89 | 18.78 | 15.13 | 26.72 | 25.33 | 3.5 |
| 100 : 27.5                   | 29.49 | 5.14 | 4.63 | 3.51 | 3.51 | 5.77 | 5.77 | 5.77 | 11.26 | 23.82 | 20.28 | 4.89 | 18.78 | 15.13 | 26.72 | 25.33 | 3.5 |
| 100 : 35                     | 28.45 | 6.16 | 5.47 | 4.87 | 4.15 | 6.95 | 6.66 | 6.66 | 11.70 | 23.82 | 20.28 | 4.89 | 18.78 | 15.13 | 26.72 | 25.33 | 3.5 |

In order to study the composition of the products obtained with NPSCa fertilizers, we ground these products and analyzed them for the content of various components. The results are also shown in Table 1. From Table 1 it is shown that at the studied ratios AN: Suprephos-NS, the N content in the products decreases from 33.47% to 28.45%. But at the same time, the content of P2O5 increases from 1.08 to 6.16%, SO3 from 1.28 to 6.95%, and CaO from 0.90 to 4.87%. It should be noted that the AN’s melt is able to significantly increase the assimilable forms of CaO and P2O5 in Suprephos-NS. Thus, treatment AN’s melt at a ratio of AC: Suprephos-NS = 100: 15 led in Suprephos-NS to an increase in the relative content of the assimilable form of CaO and P2O5 from 80.56 and 85.13% to 88.89 and 93.54%, respectively (Fig. 3). Whereas, the treatment of Suprephos-NS with melting AN at the ratio AN: Suprephos-NS = 100: 5 increased these indicators to 92.02 and 96.29%, respectively. This means that the AN’s melt can convert the P2O5 form, indigestible in Suprephos-NS, into an assimilable and water-soluble form for plants. In products, sulfur is found exclusively in a water-soluble form.

It should also be noted that the content of the water-soluble form of P2O5 also increases in the samples of NPSCa-fertilizers. Thus, in the initial Suprephos-NS the content of the water-soluble form of P2O5 in relation to the total is 20.52%, then in the product with a 5% addition of Suprephos-NS it is 57.4% (Fig. 3). This indicates the occurrence of the following reactions between nitrate, ammonium sulfate and dicalcium phosphate at 180 °C:

\[2\text{NH}_4\text{NO}_3 + \text{CaHPO}_4 \rightarrow \text{Ca(NO}_3)_2 + (\text{NH}_4)_2\text{HPO}_4\]
\[\text{CaHPO}_4 + (\text{NH}_4)_2\text{SO}_4 \rightarrow \text{CaSO}_4 + (\text{NH}_4)_2\text{HPO}_4\]

It should also be noted that in this case a reaction is possible:
\[\text{Ca(NO}_3)_2 + (\text{NH}_4)_2\text{HPO}_4 \rightarrow \text{CaSO}_4 + 2\text{NH}_4\text{NO}_3\]

The latter reaction naturally serves to form strong product granules.

Figure 3. The relative content of water-soluble and assimilable forms of CaO and P2O5 of finished products, depending on the amount of Suprephos-NS additive.
Thus, the use of Suprefos-NS, a product of JSC “Ammofos-Maxam”, as an additive in the AN’s melt allows obtaining a stabilized fertilizer containing, in addition to nitrogen and phosphorus, such macroelements as calcium and sulfur, which are necessary for plant development. In the resulting product samples, the content of nutrient components, depending on the ratio of the AN: Suprefos-NS, ranges from 36.73-46.14%, and therefore their composition compares favorably with the composition of the traditional AN.

Caking is one of the most important indicators of the commercial properties of fertilizers. It determines the suitability of a granular product for long-term storage. The lower the caking of granules, the longer the product will be stored in a crumbly state. Caking of AN with a magnesian additive (0.28% MgO) - 4.67 kg / cm². With an increase in the amount of Suprefos-NS additive, the caking of nitrate decreases. Thus, the caking capacity of the product with a 5% addition of Suprefos-NS is 3.24 kg / cm², with a 15% addition - 2.78 kg / cm², with a 24% addition - 2.43 kg / cm², and with 35% additive - 2.35 kg / cm². The data obtained give reason to believe that these additives are very promising for the production of nitrate suitable for bulk transportation and storage.

The relative humidity of the air, at which the substance is not moistened or dried, is called the hygroscopic point of the substance. If the hygroscopic point is less than the relative humidity of the air, then the substance absorbs moisture from the air. If it is higher than the relative humidity of the air, the substance dries up. The values of hygroscopic points for our fertilizers with an initial moisture content of less than 0.5-1% turned out to be equal: for AN - 62%, for Suprefos-NS - 72%, for AN: Suprefos-NS = 100: 5 - 65%, for AN: Suprefos-NS = 100: 15 - 66%, for AN: Suprefos-NS = 100: 24 - 66.5% and for AN: Suprefos-NS = 100: 35 - 67. According to the hygroscopicity scale, NPSCa-fertilizer samples, although to hygroscopic substances, but they are less hygroscopic than AC. In addition, the moisture capacity of NPSCa-fertilizers obtained based on AN’s melt and powdered suprefos-NS is significantly higher than that of standard AN.

The presence of HNO₃ nitrate in the melt determines the autocatalytic nature of its thermal decomposition. Nitrogen dioxide, which is formed during the thermal decomposition of HNO₃, which is a product of the dissociation of the AN, has a noticeable effect on the rate of thermal decomposition of AN. This takes into account the proven position that one of the reasons for the decomposition of NH₄NO₃ is an increase in the acidity of its melt. In the course of studying the effect of Suprefos-NS samples on the mechanism of thermal decomposition of the NH₄NO₃ melt, it was found that they have a buffer effect on the acidity of the saltpeter melt. The buffering effect of Suprefos-NS on the pH of nitrate was studied depending on the residence time of the melt at 180 °C. The results are shown in Table. 2.

| Mass ratio of AN: Suprefos-NS | pH of a 10% solution of fertilizer melt depending on the residence time at the melting temperature |
|------------------------------|--------------------------------------------------------------------------------------------------|
|                              | without holding | 20 min | 40 min | 60 min | 80 min | 100 min | 120 min |
| AN                           | 6.13            | 5.98   | 5.70   | 5.31   | 4.79   | 4.18     | 3.76     |
| 100:5                        | 6.37            | 6.18   | 5.93   | 5.63   | 5.27   | 4.94     | 4.67     |
| 100:10                       | 6.13            | 5.97   | 5.79   | 5.53   | 5.28   | 4.69     | 4.75     |
| 100:12                       | 6.06            | 5.91   | 5.74   | 5.54   | 5.29   | 5.02     | 4.82     |
| 100:15                       | 5.99            | 5.86   | 5.72   | 5.54   | 5.32   | 5.07     | 4.87     |

Table 2 shows that with an increase in the residence time at 180 °C, the pH value of the AN’s melt with a magnesium additive decreases, and Suprefos-NS significantly slows down this process. So, with a 20-minute exposure of the AN’s melt, its pH value decreases from the initial 6.13 (without holding) to 5.98; at 60 minutes exposure up to 5.31, and at 120 minutes exposure up to 3.76. This confirms the fact that with prolonged exposure of the AN’s melt, the partial decomposition of NH₄NO₃ into NH₃ and HNO₃ occurs, the latter naturally forming a strongly acidic medium. A different picture was observed under the same conditions in the presence of Suprefos-NS. So, when the ratio of AN: Suprefos-NS = 100: 15, the pH of a 10% solution of NPSCa melt, kept for 120 minutes, decreases to 4.87, that is, when Suprefos-NS
is added to the AN’s melt, its acidification process is slow, so as the HNO₃ formed in the melt is partially neutralized by Suprefos-NS.

Based on the results obtained, it can be concluded that when the AN is heated, the studied additive has a favorable buffering effect on its melt, affecting the pH value of the melt towards a decrease in acidity. Consequently, Suprefos-NS contributes to an increase in the thermal stability of the speaker.

4. Conclusions
In laboratory conditions, by simulating the process of granulation in towers, NPSCₐ-fertilizer granules based on ammonium nitrate melt and powdered Suprefos-NS, a product manufactured by JSC “Ammofo Maxam”, were obtained. It is shown that the strength of nitrate granules increases from 1.60 MPa without the addition of Suprefos-NS to 11.7 MPa with the addition of Suprefos-NS in the amount of 6.16% P₂O₅, the hygroscopicity increases from 62 to 67%, and the caking of the granules decreases from 5.62 up to 2.35 kg / cm². It has been established that the AN’s melt is capable of converting the P₂O₅ form, indigestible in Suprephos-NS, into a form that is assimilable and water-soluble for plants. Sulfur is found in products in an exclusively water-soluble form. In the temperature range 175-185 °C, the obtained NPSCₐ melts have good fluidity, which makes it possible to granulate them in a tower by prilling without any special technological difficulties. Suprefos-NS has a favorable buffering effect on the nitrate melt, affecting the pH value of the melt in the direction of decreasing acidity.

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