PHYSICO-CHEMICAL PROPERTIES OF NPCa SUSPENSION FOR NITRIC ACID PROCESSING OF PHOSPHORITES IN THE CENTRAL KYZYL KUM

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Abstract:*Results of investigation of physic-chemical properties (rheological properties, pressure of vapor saturation, boiling temperature of evaporated and crystallization) nitroammoniumcalcium solution, which is minor product of chemical enrichment of calcareous phosphorites of Central Kyzylkum have been given.*

Key words: *nitrogen-phosphorus-calcium solution, density, viscosity, pressure of vapor saturation, boiling temperature of evaporated and crystallization.*

The method of chemical processing of carbonate phosphorites of the Central Kyzylkum developed by us consists in their treatment with concentrated nitric acid The advantage of nitric acid processing is that nitric acid is used bilaterally: as a source of active hydrogen ion for the extraction of carbonates from phosphate raw materials and as a nitrogen carrier, a useful component of liquid fertilizers. The rate of nitric acid was taken stoichiometric based on the decomposition of calcium oxide. This rate assumes the decarbonization of calcium carbonate in phosphate raw materials: $CaCO_3 + 2HNO_3 \rightarrow Ca(NO_3)_2 + H_2O + CO_2$

During the decarbonization of phosphate raw materials, along with carbonates, at least to a lesser extent, the phosphate mineral also dissolves with the formation of water-soluble monocalcium phosphate according to the reaction:

$$2Ca_5F(PO_4)_3 + 14HNO_3 \rightarrow 3Ca(H_2PO_4)_2 + 7Ca(NO_3)_2 + 2HF$$

It is desirable to transfer it to the solid phase, from which concentrated phosphorus-containing fertilizers are obtained. To do this, before separating the acidic nitrocalcium phosphate pulp into liquid and solid phases. we subjected it to neutralization with monoethanolamine to pH=3.2. In the process of ammonization, an interaction occurs between monocalcium phosphate, calcium nitrate and monoethanolamine with the formation of liquid nitrogen-phosphorus-calcium fertilizer and ammonium nitrate according to the reaction:

$$Ca(H_2PO_4)_2 + Ca(NO_3)_2 + 2HOCH_2CH_2NH_2 + H_2O \rightarrow 2CaHPO_4 + (HOCH_2CH_2)_2 + 2NH_4NO_3$$

At least three parallel measurements were carried out for each potential

value. The obtained dependences are approximated in the Excel 2010 software environment by the straight line equation y = kx + b, where y is the potential value, mV; k and b are the coefficients of the approximation equation.

The values of the coefficients k and b and the square of the correlation coefficient are presented in Table 1.

Table 1.

Temperature,	*298*	*303*	*313*	*323*
k	***-58,007***	***-60,082***	***-61,673***	***-63,294***
b	***385,03***	***385,03***	***396,78***	***408,62***
R^2	***0,9999***	***0,9998***	***0,9998***	***0,9998***

The calculation of the chemical composition of the system at the initial and final stages of decomposition of phosphate raw materials allows us to conclude that there is a slight change in the ionic strength of the solution during the reaction, during which the decrease in the concentration of strong (nitric) acid is compensated by an increase in the concentrations of phosphoric acid and calcium and magnesium nitrates, which, in turn compensates for the ionic strength. In this case, the change in the activity of the solution, recorded by the electrode, adequately describes the change in the [H+] concentration:

$C_{H^+} = 10^{-pa_{H^+}} \approx 10^{-pH}/\gamma$

Where γ is the activity coefficient.

In this work, we used the ionometric method for determining ammonium nitrogen using an ammonium selective electrode.

The advantage of the ionometric method lies in the wide range of measurements (pNH4+ = 1.13÷5.0), the rapidity of the analysis, and the absence of the need for reagents.The measurements were carried out using an ELIS-121NH$_4$ ion-selective film electrode, GOST 22261-94 and TU 4215-015-35918409-2007, as an indicator and silver chloride reference electrode EVL-1M3.1.

The calibration dependence constructed from standard solutions prepared in accordance with the electrode certificate is shown in Fig.1.

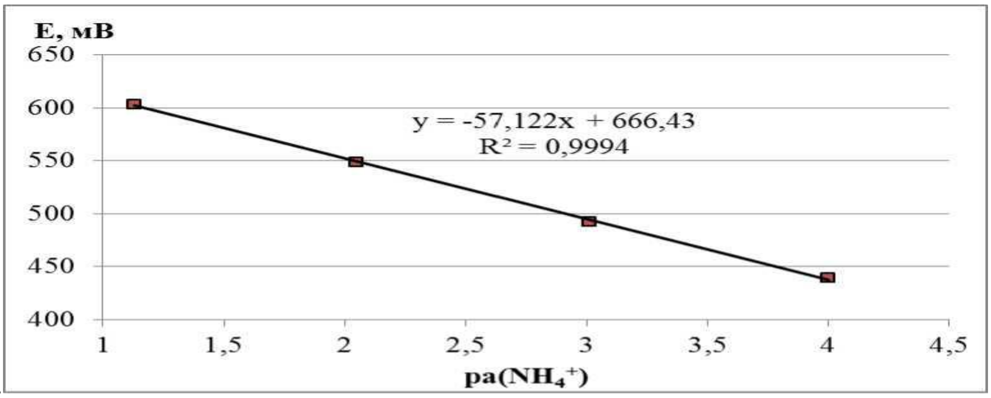
1. 

Fig. 1. Calibration dependence of the electrode potential, E, mV, on the activity of

ammonium ions.

The steepness of the calibration characteristic of the electrode in the linear part at 25 °C lies in the range recommended for the electrode. Sodium, calcium cations have the order of the selectivity coefficient 10-. magnesium - 10-4. With respect to the K+ cation, the selectivity coefficient is 1.2 x 10-1. Due to the noticeable influence of potassium ions, the latter were removed before the determination of the ammonium ion by precipitation from solution using sodium tetraphenylborate.

For research, 4 types of phosphorites of the Central Committee were taken. For the complete splitting of phosphorites, nitrate acid in relation to CaO in the composition of phosphorite is calculated with a concentration of 57.8% at a rate of 100 - 120% nitrate acid. This process was carried out at a temperature of 25 - 30°C. After the process for separating the liquid and solid phases, water was added to the formed mass for liquefaction in a ratio of 1:1-2. This study was carried out at the Namangan Institute of Engineering and Technology at the Department of Chemical Technology using existing equipment, the resulting mass was separated using a centrifuge. The precipitate, that is, the undissolved residue, was washed with water 2 times. The liquid phase is neutralized while maintaining the liquid state to pH=4-5. The main purpose of neutralization is to keep the fertilizer liquid and to maintain an acidic environment.

NPCa can be directly applied as a finished liquid nitrogen-phosphorus-calcium fertilizer. Liquid nitrogen-phosphorus-calcium fertilizer is of great interest for agricultural production, it contains two of the six macronutrients most important for plant nutrition. These six macronutrients are: nitrogen, phosphorus, potassium, sulfur, calcium, and magnesium. Calcium nitrate is the only fertilizer completely absorbed by plants that replenishes the loss of water-soluble calcium in the soil.

№	Composition of raw materials	Type of raw material number of onents %			$CaO:P_2O_5$
		P_2O_5	CaO	CO_2	
1	Washed Calcined Phosphoconcentrate	25,62	52,17	2,1	2,03
2	Powder of simple phosphorite	18,70	47,52	17,23	2,54
3	Mineralized mass	14,35	42,31	12,08	2,95

It enjoys a great reputation among flower growers and is especially important for fast-growing crops. The main agrochemical features of calcium nitrate: stimulates the development of the root system, the growth and development of agricultural and ornamental crops; improves the formation of membranes and plant cell walls; activates the activity of enzymes and metabolism in plants; improves the processes of photosynthesis, the transport of carbohydrates and the absorption of nitrogen and other nutrients in plants; increases the resistance of plants to environmental stress factors, to fungal and bacterial diseases arising from calcium deficiency; improves the presentation and taste of fruits and vegetables.

To carry out the process of obtaining various concentrations based on nitrogen-phosphorus-calcium fertilizers, data on the physicochemical properties of the developed fertilizers are needed: density, viscosity, saturated vapor pressure and crystallization temperature, which determine the conditions for their production, storage, transportation and application to the soil.

This article presents the results of studying the saturated vapor pressure (kPa) over nitrogen-phosphorus-calcium fertilizers of various concentrations and their boiling points at various pressures. Determination of vapor pressure over nitrogen-phosphorus-calcium fertilizers was carried out by the dynamic method. The crystallization temperature of nitrogen-phosphorus-calcium fertilizers was determined by the visual-polythermal method.

Saturated vapor pressure (kPa) over nitrogen-phosphorus-calcium fertilizers was determined on a laboratory setup shown in fig.

Methodology for performing experiments. The investigated liquid in the amount of 75-100 ml is poured into the vessel (1). The vessel is closed with a ground stopper with a thermometer (3). The thermostat contains a stirrer (6), a contact thermometer (7) and a boiler (2). The vessel is connected to the vacuum system through a serpentine cooler (4), in which the vapors of the test liquid are trapped. This is necessary to prevent vapor condensation on the walls of the connecting pipes in the pressure gauge (11) and thermostat (12).

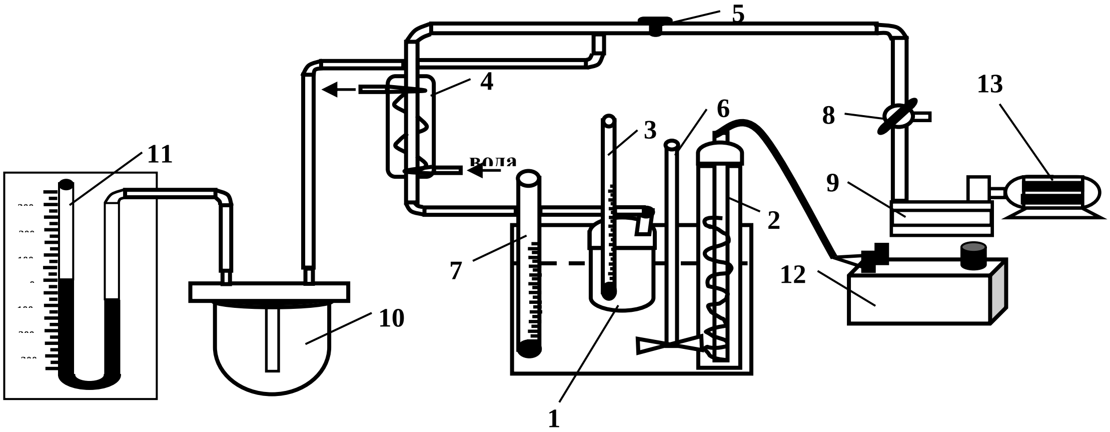


Fig. 2. Scheme of the installation for determining the pressure of saturated vapor over a liquid.

The refrigerator is connected to the vacuum pump (13) through taps (5, 8). To protect the latter from vapors, a column (9) is placed between the tap (8) and the vacuum pump (13). To measure the saturation vapor pressure, air is pumped out of the device until the liquid in the vessel (1) begins to boil rapidly. After the liquid in the vessel begins to boil, the boiler is turned off and the valve (5) is closed, and the valve (8) is connected to the atmosphere. 5 minutes after the valve (5) is closed, the thermometer readings are noted, and the pressure is measured using an open pressure gauge, which is connected to the refrigerator (4) through a trap (10). The vapor pressure will be equal, and ho is counted on an open pressure gauge (11) as the difference in the heights of mercury in two knees. This difference is made up of the height of the rise in the left knee and the amount of lowering in the right knee (counted from the level corresponding to the atmospheric pressure in the system). Measurements are started at room temperature, and then the temperature is successively increased by 4–6°C each time. Thus, by gradually increasing the temperature of the thermostat, the vapor pressure of the liquid is determined at several temperatures. Such measurements are repeated eight to ten times. After each measurement, gradually let air into the device. To do this, with the three-way valve (8) open to the atmosphere, carefully open the valve (5) and bring the pressure in the system to atmospheric.

Table 2.

Saturated vapor pressure (kPa) over nitrogen-calcium-phosphorus-calcium liquid fertilizer and crystallization temperatures of these solutions depending on their pH

pH solution	Type of equation lgP = A - B/T	Temperature, °C							Temperature crystal-zation, °C
		0	0	0	0	0	0	8 0	
Norms HNO_3 110%									
2	lgP=7,8727-							3	- 15
3	lgP=7,8311-							2	- 11
3	lgP=7,8130-							2	- 10
4	lgP=7,8001-							2	-7
Norms HNO_3 105%									
2	lgP=7,7971-							2	- 12
3	lgP=7,7711-							1	- 10
3	lgP= 7,7659-							1	- 8
4	lgP=7,7506-							1	-6

Saturated vapor pressure of NPCa in the range of 20-40°C is 0.44-6.38 kPa, which indicates their low volatility in the hot climate of Central Asia. In the range of NPCa concentrations of 53.02-65.71%, their crystallization temperature is in the range of (-15) ÷ (-6.0)°C, which allows them to be used in spring and summer as liquid nitrogen-phosphorus-calcium fertilizers. From Table. 2 shows that in the temperature range of 30-80°C NPCa with such concentrations has a density of 1.3873-1.6377 g/cm3 and a viscosity of 2.43-18.85 cps. That is, NPCa has good rheological properties. However, more concentrated NPCa 64-80% must be diluted with water before use, since their crystallization temperature is 29-60°C.

In subsequent experiments, we determined the boiling points of NPCa solutions at various pressures. The table shows that as the concentration of NPCa increases, the boiling point of these solutions increases. For example, at a concentration of NPCa - 73.02% at a pressure of 760 mm. rt. Art. its boiling point is 108°C, and at a concentration of NPCa of -88.77%, this figure rises to 152°C. Reducing the pressure also lowers the boiling point of the solutions. So, if at a pressure of 760 mm. rt. Art. the boiling point of 66.6% NPCa is 130□C, then at a pressure of 460 mm. rt. Art. it equals 112°C.

For the experiments, nitrogen-phosphorus-calcium fertilizers pH=3.2 were prepared. The results of measuring the pressure of saturated vapors over solutions of nitrogen-phosphorus-calcium fertilizers. Values A and B, depending on the pH of nitrogen-phosphorus-calcium fertilizers, range from 4.7818-4.8929 and 1201.3-1323.7, respectively.

Thus, the results of the studies performed show that with the content of nitrogen-phosphorus-calcium fertilizers, pH 4-5 have satisfactory physical and chemical properties, which make it possible to use them in spring and summer.

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