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Research on the Ammonium Polyphosphate Fertilizer Production Process

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ABSTRACT: A series of experiments were conducted to study the influence of the molar ratios of raw materials, reaction temperature and time on the process of obtaining long-acting ammonium polyphosphate fertilizers based on monoammonium phosphate-urea. Based on the obtained scientific results, it was shown that the optimal conditions are a molar ratio of monoammonium phosphate-urea - 1.5:1, a process temperature of 160-170°C and a duration of 45 minutes. An X-ray diffraction pattern of ammonium polyphosphate obtained on the basis of monoammonium phosphate and urea is presented.

I. INTRODUCTION

Ammonium polyphosphates have long been widely used as fire retardants, fire extinguishing agents, food additives, etc [1]. In recent years, ammonium polyphosphates have been increasingly used as an alternative source of effective phosphate fertilizers in agriculture to increase soil phosphorus availability and improve the use efficiency of phosphorus fertilizers. Generally, when polyphosphate fertilizers are applied to the soil, they cannot be immediately absorbed by plants and are gradually hydrolyzed to orthophosphorus. The hydrolysis of ammonium polyphosphate fertilizers mainly depends on its chemical nature and soil factors, such as pH, soil composition and soil temperature. Some studies have shown that short-chain soluble polyphosphate fertilizers are superior to orthophosphate-based fertilizers (such as monoammonium phosphate, diammonium phosphate, and double superphosphate) in increasing the amount of P_2O_5 available in the soil and increasing yield and improving the efficiency of phosphorus fertilizer use. Since polyphosphate has a slow hydrolysis property, the application of ammonium polyphosphate can significantly reduce the amount of P_2O_5 in the soil and increase the availability of P_2O_5 in the soil. Therefore, the application of polyphosphate fertilizers in agriculture has attracted great attention [2-5].

Currently, one of the promising methods for increasing the concentration and efficiency of nutrient components in phosphorus fertilizers is to obtain long-acting phosphorus fertilizers containing polymerized P_2O_5 . As a result of the slow hydrolysis of P_2O_5 in the form of polymers under the influence of water, P_2O_5 has a long-term effect when used. One of the effective ways to solve this problem is to increase the efficiency of phosphorus fertilizers [5-8].

In the context of an acute shortage of high-quality phosphorus raw materials and a shortage of thermal phosphoric acid, the goal of the research was to obtain long-acting phosphorus fertilizers based on monoammonium phosphate and urea and alkaline polyphosphates for technical purposes [9].



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II. RESEARCH METHODS

Research objects and methods. Research was conducted on the production of ammonium polyphosphate fertilizer (NH₄H₂PO₄) obtained from extractive phosphoric acid based on Central Kyzylkum phosphorites in various molar ratios with urea. In conducting experimental research, monoammonium phosphate was used, which was isolated from a solution of extractive phosphoric acid neutralized with ammonia to pH=4.5. The chemical composition of monoammonium phosphate crystals is as follows: $P_2O_5 - 59.43\%$; N - 11.67%; CaO - 0.22%; MgO - 0.27\%; Fe₂O₃ - 0.038; Al₂O₃ - 0.034\%; SO₃ - 2.07; F - 0.026\%.

High-temperature experiments on the NH₄H₂PO₄:CO(NH₂)₂ interaction were conducted in a closed-loop laboratory setup.

The samples were identified based on diffraction patterns obtained on an XRD-6100 diffractometer (Shimadzu). CuK α radiation (β filter, Ni, 1.54178, tube current and voltage mode 30 mA, 30 kV) and a constant detector rotation speed of 4 deg/min with a step of 0.02 deg ($\omega/2\theta$ coupling) were used, and the scanning angle varied from 4 to 80°.

III. EXPERIMENTAL RESULTS

The effect of the molar ratio of reagents and process temperature on the process of obtaining ammonium polyphosphate fertilizer based on different molar ratios of monoammonium phosphate and urea (NH₄H₂PO₄:CO(NH₂)₂) obtained from extractive phosphoric acid based on Central Kyzylkum phosphorites was studied.

Studies have shown that the thermal dehydration reaction of a mixture of $NH_4H_2PO_4$ and $CO(NH_2)_2$ has a significant effect on the chemical composition of ammonium polyphosphate fertilizer (Figure 1). The effect of the molar ratios of $NH_4H_2PO_4$ and $CO(NH_2)_2$ on the degree of polymerization of P_2O_5 in ammonium polyphosphate was studied. It was shown that with an increase in the molar ratio of $NH_4H_2PO_4$: $CO(NH_2)_2$ from 1.5:1 to 1.9:1, the degree of polymerization of P_2O_5 decreases from 83.68% to 71.39%.





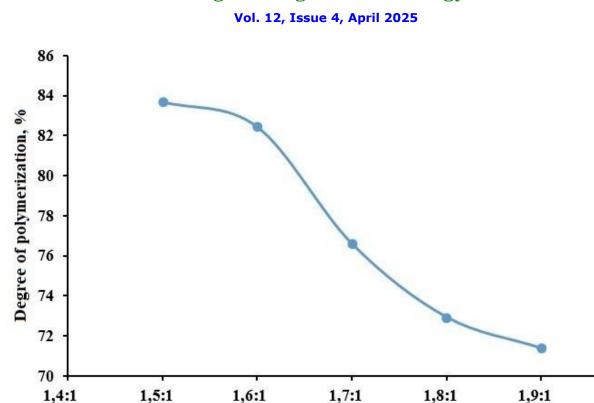


Fig. 1. Effect of NH₄H₂PO₄:CO(NH₂)₂ molar ratios on the degree of polymerization of P₂O₅ in ammonium polyphosphate

NH4H2PO4:CO(NH2)2 molar ratios

Analysis of the data presented in the table shows that the chemical composition of ammonium polyphosphates varies slightly depending on the molar ratio $NH_4H_2PO_4:CO(NH_2)_2 = 1.5:1$, the process temperature (150-190°C) and the reaction time. With an increase in the process temperature from 150°C to 190°C in 45 minutes, the total P_2O_5 content in ammonium polyphosphate products changed from 49.77% to 50.35%, the water-soluble P_2O_5 content changed from 10.64% to 7.73%, the polymerized P_2O_5 content changed from 38.48% to 42.10%, and the nitrogen content increased from 18.85% to 19.06%.

Nº	Process duration, minute	Process temperature, °C	Chemical composition of the product, %									
			P ₂ O ₅ total	P ₂ O ₅ water	P ₂ O ₅ poly	CaO	MgO	Fe ₂ O ₃	Al ₂ O ₃	SO ₃	F	Ν
1	45	150	49,77	11,29	38,48	0,184	0,226	0,032	0,0285	1,733	0,0217	18,85
2	45	160	49,98	10,16	39,82	0,185	0,2266	0,0319	0,0285	1,737	0,0218	18,93
3	45	170	50,19	9,13	41,06	0,186	0,228	0,032	0,0287	1,748	0,022	19,01
4	45	180	50,30	8,42	41,88	0,186	0,228	0,032	0,0288	1,752	0,022	19,05

Table. Parameters affecting the process of obtaining ammonium polyphosphate based on NH4H2PO4:CO(NH2)2

2:1



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Ι		100										
5	45	190	50,35	8,25	42,10	0,186	0,229	0,0322	0,0288	1,754	0,220	19,06
6	60	150	51,07	10,64	40,43	0,189	0,232	0,0327	0,0292	1,779	0,0223	19,34
7	60	160	51,34	9,56	41,78	0,190	0,233	0,0328	0,0294	1,788	0,0220	19,45
8	60	170	51,52	8,55	42,97	0,191	0,234	0,032	0,0300	1,795	0,0250	19,52
9	60	180	51,55	7,90	43,65	0,191	0,234	0,033	0,0300	1,796	0,0226	19,53
10	60	190	51,58	7,73	43,85	0,191	0,234	0,033	0,0295	1,197	0,0226	19,54
11	75	150	51,65	10,13	41,52	0,191	0,235	0,033	0,0295	1,799	0,0226	19,56
12	75	160	51,93	9,06	42,87	0,192	0,236	0,033	0,0297	1,809	0,0227	19,67
13	75	170	52,09	8,18	43,91	0,193	0,236	0,033	0,0298	1,814	0,0228	19,73
14	75	180	52,13	7,52	44,61	0,193	0,237	0,033	0,0298	1,816	0,0228	19,75
15	75	190	52,16	7,37	44,79	0,193	0,237	0,033	0,0298	1,817	0,0228	19,76

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When the process duration was 60 minutes, the total P₂O₅ content in ammonium polyphosphate products changed from 51.07% to 51.58%, the water-soluble P₂O₅ content from 11.29% to 8.25%, the polymerized P₂O₅ content from 40.43% to 43.85%, and the nitrogen content increased from 19.34% to 19.54%. When the process duration was 75 minutes, the total P2O5 content in ammonium polyphosphate products changed from 51.65% to 52.16%, the water-soluble P2O5 content from 10.13% to 7.37%, the polymerized P₂O₅ content from 41.52% to 44.79%, and the nitrogen content increased from 19.56% to 19.76%.

In the X-ray diffraction patterns of ammonium polyphosphate melt samples, intense peaks at Å 5.20-5.24; 4.267-4.303; 3.692-3.709; 3.037-3.0495; 2.62-2.64 and 1.99, characteristic of ammonium dihydrogen monophosphate, are very clearly visible. Moreover, at low temperatures of 160-180°C (Fig. 2), the intense lines inherent in pyrophosphates are weakly visible in the X-ray diffraction pattern due to their partial X-ray ammorphism. The diffraction maxima at 4.307; 3.037 and 1.99 can also be attributed to ammonium sulfate. Peaks with low intensity at 4.757; 2.94 and 1.657 Å are characteristic of magnesium ammonium phosphate monohydrate. Increasing the temperature to 190°C or increasing the duration of isothermal holding of the ammonium polyphosphate melt at 190°C leads to significant complications in the X-ray patterns of the samples.



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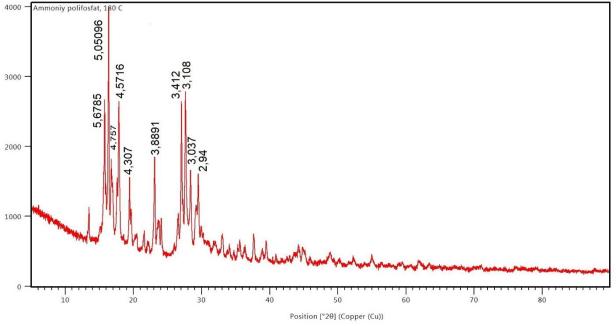


Fig. 2 - X-ray diffraction pattern of ammonium polyphosphates

The X-ray diffraction pattern of ammonium polyphosphate melt obtained by obtaining monoammonium phosphate and urea, in addition to the intense peaks characteristic of the above-mentioned salts, has a number of new maxima Å: 5.05096; 3.8891; 3.4750; 3.4120; 3.108; 2.7889 – (NH₄)₃HP₂O₇; 5.6785; 4.5716; 5.5096; 5.7669 for γ and β (NH₄)₂H₂P₂O₇.

The isolation of individual components from ammonium polyphosphate melts and complete identification with the establishment of the complete phase composition is a very difficult task, both in physicochemical and experimental terms.

IV. CONCLUSION

Thus, the results of the study led to the conclusion that the optimal conditions for the degree of polymerization in the process of dehydration of monoammonium phosphate and urea obtained from extraction phosphoric acid based on Central Kyzylkum phosphorites are the molar ratio $NH_4H_2PO_4:CO(NH_2)_2 = 1.5:1$, temperature 170-180°C and process time 45-60 minutes, the degree of polymerization is 83.40-84.68%. In this case, the maximum values of the degree of polymerization are achieved.

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