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RHEOLOGICAL PROPERTIES OF AMMOPHOSPHATE PULPS OBTAINED USING PHOSPHORITE POWDER OF THE KHODJAKUL DEPOSIT

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Abstract:

Objective. The rheological characteristics (density, viscosity) of acidic and ammoniated phosphate slurries obtained from the decomposition of Khodjakul phosphate powder with extraction phosphoric acid (EPA) in a wide range of acid : phosphorite mass ratio and temperatures were studied. It has been shown that both acidic and ammoniated pulps have high fluidity and can be transported by pumping devices without any restrictions.

Methods. Experiments on the decomposition of phosphorite powder (PP) with phosphoric acid were carried out in a tubular glass reactor equipped with a screw stirrer driven by a motor. The required amount of EPA was placed in the reactor and heated to 65°C. Phosphate raw materials were introduced into the reactor with an EPA attachment. The mass ratio of EPA :PP was chosen in the range of 100: (5-30). The density of acid phosphate pulps was determined by the pycnometric method with a measurement accuracy of 0.05 rel. %, and kinematic viscosity - using a glass capillary viscometer VPJ-1 with an error of 0.2 relative to % in the temperature range of 20-90 °C.

Results. Laboratory experiments have established that the most optimal mass ratios of EPA :PP is 100:20 and 100:25. Under these conditions, the ammonium phosphate pulp at a temperature of 60°C has a density and viscosity of 1.32-1.34 g/cm³ and 20.06-24.13 centipoise, respectively.

Conclusion. In the range of 20-90°C, the density and viscosity of acid phosphate pulps obtained from the decomposition of phosphorite powder Khojakul with phosphoric acid in a wide range of initial components (EPA :PP = 100:5-30). It is shown that the pulps have high fluidity (no more than 1.37 g/cm³ and 7.08 centipoise).

Keywords: nodular phosphate powder, extraction phosphoric acid, acidic and ammoniated phosphate pulps, density, viscosity.

Introduction. There are 25.73 million hectares of agricultural land in Uzbekistan, of which 3.73 million hectares are irrigated [1]. It is on irrigated lands that over 97% of the agricultural products of the republic are obtained.

One of the main problems is the shortage of water resources, which does not allow a sharp increase in the area of irrigated land. Therefore, the chemicalization of agriculture provides for an increase in the production of mineral fertilizers, including phosphorus-containing ones.

In 2018. the chemical industry produced 138.2 thousand tons of phosphoric fertilizers in terms of 100% P₂O₅. Whereas, the demand for agriculture was 688.4 thousand tons of phosphoric fertilizers. Based on this situation, it can be seen that the demand is provided only by 20%.

As for the assortment of phosphorus-containing fertilizers, it consists of ammophos (10% N; 46% P₂O₅), suprephos-NS (8-15% N; 20-24% P₂O₅), PS-Agro (4-6% N; 34-41% P₂O₅), feed ammonium phosphate (12% N; 53-55% P₂O₅), simple (1.5% N; 13.5% P₂O₅) and enriched superphosphate (2.5% N; 18-

26% P₂O₅) [2]. The main phosphate raw material for their production is washed and calcinated concentrate (26% P₂O₅), produced at LLC "Kyzylkum phosphorite complex". The complex annually produces 716 thousand tons of concentrate, processing phosphorite ore (17% P₂O₅) of the Kyzylkum deposit. However, the volume of concentrate is not enough for the production of the above-mentioned mineral fertilizers, which makes it necessary to involve other local deposits in the production. Phosphorite manifestations are present in many regions of Uzbekistan. These are Ferghana, Surkhandarya, Pritashkent, Navoi (Penjikent), Central Kyzylkum, Bukhara-Khiva and Karakalpak [3].

There are a number of deposits of nodular phosphorites in Karakalpakstan such as Khodzhakul, SultanUzidag, Khojeyli, Nazarkhan, Chukai-Tukai, Porlitau, Beshtyube and others [4].

The deposits belong to the carbonate-sandy type, represented by a phosphate mineral with an admixture of clay and carbonate substances with abundant gangues of quartz grains, feldspar, mica, glauconite, zircon and iron hydroxides cemented with carbonate cement [5].

However, these low-grade phosphorites do not meet the requirements for sulfuric acid processing to produce extraction phosphoric acid (EPA) and ammophos. However, they can serve as secondary phosphate raw materials in the production of qualified phosphorus-containing fertilizers.

In the early 80st of the last century, specialists of the NGO "Minudobreniya", NGO "NIIHIMMASH, NIUIF, Mendeleev Moscow Art Institute developed the technology of nitrogen-phosphorus fertilizer-ammonium phosphate [6, 7]. Its production was mastered from Karatau phosphorites at the Dzhambul superphosphate plant in Kazakhstan, at the Chardzhou chemical plant in Turkmenistan, at Almalyk JSC "Ammophos" (now JSC "Ammophos-Maxam"), from apatite concentrate at the Balakovo "Mineral Fertilizers" in Russia. An important advantage of ammonium phosphate is the possibility of using almost any kind of phosphate raw materials.

The process of obtaining ammonium phosphate has also been studied on the example of various grades of phosphorites of Central Kyzylkums (phosphorite powder, thermoconcentrate, chemically enriched concentrate, pulverized fraction) [8]. At the same time, the mass ratio of EPA : PP is taken in the range of 100:(5-30).

However, there is no information in the literature about the use of nodular phosphorites of Karakalpakstan in ammophosphate fertilizer technology. In this connection, in this work, the process of decomposition of phosphate rock from the Khodzhakul deposit with extraction phosphoric acid was carried out and the rheological properties of acidic and ammoniated phosphate pulps were studied.

Research methods. For the experiments, phosphate rock from the Khodzhakul deposit was used as feedstock, composition (wt. %): P_2O_5 – 19.11; P_2O_5 acceptable by citric acid: P_2O_5

general. = 63.26; P_2O_5 acceptable. : P_2O_5 general. = 35.85; CaO – 32.83; MgO – 0.30; CO_2 – 4.03; Fe_2O_3 – 3.50; Al_2O_3 – 1.54; SO_3 – 1.10; F – 1.58; SiO_2 – 28.0; insoluble residue. – 1.64; H_2O – 2.62; $CaO_{total.}$: $P_2O_{5total.}$ = 1.72, extraction phosphoric acid (EPA) produced by JSC "Ammophos-Maxam" composition, mass%: 14.65 P_2O_5 ; 0.43 CaO; 0.57 MgO; 0.23 Fe_2O_3 ; 0.26 Al_2O_3 ; 0.73 F; 1.33 SO_3 , ρ = 1.20 g/cm³ and gaseous ammonia (100% NH_3). The dispersion of phosphorites is characterized as follows: class (-5+3) – 7.65%; (-3+2) – 24.43%; (-2+1 mm) – 18.29%; (-1+0.5 mm) – 3.0%; (-0.5+0.25 mm) – 22.73%; (-0.25+0.16 mm) – 10.07%; (-0.16+0.1 mm) – 4.11%; (-0.1+0.05 mm) – 6.20%; (-0.05 mm) – 3.51%.

Experiments on the decomposition of phosphorite powder (PP) with phosphoric acid were carried out in a tubular glass reactor equipped with a screw stirrer driven by a motor. The required amount of EPA was placed in the reactor and heated to 65°C. Phosphate raw materials were introduced into the reactor with an EPA attachment. The mass ratio of EPA : PP was chosen in the range of 100: (5-30). The duration of the decomposition process from the end of the loading of the raw material is 45 minutes. After that, the density and viscosity of the pulps were measured depending on the mass ratio of EPA: PP and temperature. The density of acid phosphate pulps was determined by the pycnometric method with a measurement accuracy of 0.05 relative to %, and the kinematic viscosity was determined using a glass capillary viscometer VPJ-1 with an error of 0.2 relative to% in the temperature range of 20-90°C. The results of the experiments are summarized in tables 1 and 2.

Results and discussion. As can be seen from the data in tables 1 and 2, both an increase in the proportion of phosphorite and an increase in temperature have a noticeable effect on the density and viscosity of the pulp. Thus, an increase in

the proportion of phosphorite in the system from 100:5 to 100:30 at 20°C leads to an increase in pulp density from 1.2104 to 1.370 g/cm³, that is 1.13 times, at 60°C from 1.1857 to 1.2714 g/cm³, or 1.07 times, and at 90°C from 1.1698 to 1.2124 g/cm³, or 1.04 times (table 1). An increase in pulp temperature from 20 to 90°C, depending on the EPA:PP ratio, contributes to a decrease in density.

For example, with EPA:PP = 100:10 this indicator decreases from 1.2253 to 1.1835 g/cm³, EPA:PP = 100:20 from

1.2559 to 1.2062 g/cm³ and at EPA:PP = 100:30 from 1.370 to 1.2124 g/cm³, that is, in 1.036, 1.04 and 1.13 times, respectively.

Table 2 shows that at a temperature of 20°C, the increase in the mass ratio of EPA:PP from 100 : 5 to 100 : 30 leads to an increase in viscosity from 3.28 to 7.08 centipoise, at 50°C from 2.03 to 3.79 centipoise, at 70°C from 1.68 to 2.89 centipoise and at 90°C from 1.52 to 2.68 centipoise, that is, 2.16, 1.87, 1.72 and 1.76 times, respectively.

Table 1

Density of acid phosphate pulps based on phosphoric acid decomposition of Khodzhakul phosphorite powder

Temperature, °C	Density (g/sm ³) at EPA:PP mass ratios					
	100:5	100:10	100:15	100:20	100:25	100:30
20	1.2104	1.2253	1.2403	1.2559	1.3123	1.370
30	1.2041	1.219	1.2331	1.2479	1.2949	1.3418
40	1.1979	1.2127	1.2237	1.2389	1.2779	1.3186
50	1.1918	1.2065	1.2144	1.230	1.2626	1.2961
60	1.1857	1.2004	1.2068	1.2234	1.2488	1.2714
70	1.1809	1.1943	1.2011	1.2168	1.2353	1.2506
80	1.1761	1.1895	1.1963	1.2115	1.221	1.2298
90	1.1698	1.1835	1.1907	1.2062	1.2092	1.2124

Table 2

Viscosity of acid phosphate pulps based on phosphoric acid decomposition of Khodzhakul phosphorite powder

Temperature, °C	Viscosity (centipoise) at EPA:PP mass ratios					
	100:5	100:10	100:15	100:20	100:25	100:30
20	3.28	3.99	4.70	5.43	6.26	7.08
30	2.69	3.28	3.88	4.34	5.04	5.78
40	2.29	2.74	3.19	3.52	3.98	4.54
50	2.03	2.36	2.71	2.96	3.31	3.79
60	1.82	2.10	2.37	2.58	2.93	3.22
70	1.68	1.89	2.12	2.33	2.66	2.89
80	1.58	1.77	1.96	2.15	2.44	2.73
90	1.52	1.66	1.80	2.00	2.34	2.68

An increase in temperature from 20 to 90°C at EPA:PP ratios = 100:10, 100:20 and 100:30 contributes to a decrease in

pulp viscosity by 2.40, 2.72 and 2.64 centipoise, respectively.

Thus, at the studied intervals of technological parameters, the values of the density and viscosity of the phosphoric acid pulp indicate that it has a high fluidity and can be transported by pumping devices without any restrictions.

According to the current technology of production of ammonium phosphate, acid phosphate pulp with a pH value of 2.0-2.5 and a moisture content of more than 60% is ammoniated to pH = 4.0-4.5 and then evaporated to a humidity of 35-40%, after which it is fed for granulation into a drum granulator dryer (BGS).

Based on this, acid phosphate pulps were neutralized with ammonia gas in a special reactor with intensive stirring to pH values = 4.0-4.5.

The pH value of the ammoniated pulps was measured using a laboratory ionomer of the I-130M brand with an electrode system of electrodes ESL 63-07, EVL-1M3.1 and TKA-7 with an accuracy of 0.05 pH units. The results of measuring the density and viscosity of ammoniated phosphate pulps are shown in tables 3 and 4.

Table 3

Density of ammoniated phosphate pulps based on phosphoric acid decomposition of Khodzhakul phosphorite powder

Temperature, °C	Density (g/sm ³) at EPA:PP mass ratios					
	100:5	100:10	100:15	100:20	100:25	100:30
20	1.2106	1.250	1.3008	1.3445	1.3604	1.3765
30	1.2087	1.2479	1.2945	1.3406	1.3558	1.3707
40	1.206	1.2448	1.2893	1.3343	1.3502	1.365
50	1.2033	1.2417	1.2851	1.3279	1.3435	1.3593
60	1.2015	1.2386	1.28	1.3217	1.338	1.3537
70	1.1979	1.2346	1.2749	1.3155	1.3325	1.3481
80	1.1945	1.2307	1.2698	1.310	1.3271	1.3425

Table 4

Viscosity of ammoniated phosphate pulps based on phosphoric acid decomposition of Khodzhakul phosphorite powder

Temperature, °C	Viscosity (centipoise) at EPA:PP mass ratios					
	100:5	100:10	100:15	100:20	100:25	100:30
20	8.51	15.00	24.00	31.13	38.44	45.76
30	7.26	13.24	20.70	27.45	34.22	40.28
40	6.02	11.48	17.98	24.48	30.18	35.88
50	5.11	10.29	16.04	21.80	26.95	32.10
60	4.55	9.43	15.00	20.06	24.13	28.20
70	4.19	8.55	14.05	18.52	22.06	25.55
80	3.97	7.89	13.56	16.99	20.90	23.56

From the table. 3 it can be seen that with the ammonization of acid phosphate pulp and a temperature of 20°C with an increase in the mass fraction of phosphate

raw materials from 5 to 30 g, the density of the ammonium phosphate pulp increases from 1.2106 to 1.3765 g/cm³, at 40 °C from 1.206 to 1.365 g/cm³, at 60 °C from 1.2015

to 1.3537 g/cm³ and at 80°C from 1.1945 to 1.3425 g/cm³. The temperature of the process also has a tangible effect on the rheological properties of the ammonium phosphate pulp.

So, for the mass ratio of EPA:PP = 100:10 an increase in temperature from 20 to 80°C contributes to a decrease in the density of the ammonium phosphate pulp from 1.250 to 1.2307 g/cm³, for EPA:PP = 100:20 from 1.3445 to 1.310 g/cm³ and for EPA:PP = 100:30 from 1.3765 to 1.3425 g/cm³. A similar pattern is observed for the viscosity of the ammonium phosphate pulp, that is, the more phosphate raw materials are introduced into the system, the higher the viscosity values, and the higher the temperature, on the contrary, the lower the viscosity of the ammonium phosphate pulp.

Thus, at a temperature of 20°C with an increase in the mass fraction of phosphate raw materials from 5 to 30 g, the viscosity of the ammonium phosphate pulp increases from 8.51 to 45.76 centipoise, at 40°C from 6.02 to 35.88 centipoise, at 60°C from 4.55 to 28.19 centipoise and at 80°C from 3.97 to 23.56 centipoise. For EPA:PP = 100:10 an increase in temperature from 20 to 80°C leads to a decrease in pulp viscosity from 15.00 to 7.89 centipoise, for EPA:PP = 100:20 from 31.13 to 16.99 centipoise and for EPA:PP = 100:30 from 45.76 to 23.56 centipoise.

The highest density (1.3765 g/cm³) and viscosity (45.76 centipoise) values are found in the pulp obtained at an EPA:PP ratio of 100:30 and a temperature of 20°C. In any case, such pulp is easily pumped through pipelines to the BGS apparatus for granulation and drying.

Laboratory experiments have established that the most optimal mass ratios of EPA:PP appeared 100:20 and 100:25.

Under these conditions, the ammonium phosphate pulp at a temperature of 60°C has a density and viscosity of 1.32-1.34 g/cm³ and 20.06-24.13 centipoise, respectively.

Thus, the yellow phosphorite powder of the Khodzhaikul deposit is quite suitable for the production of ammonium phosphate fertilizer, where it can serve as a secondary phosphate raw material.

At the same time, the obtained acid phosphate and ammonium phosphate pulps can be pumped from one device to another in a liquid-flowing manner and without any difficulties.

Conclusion.

1. In the range of 20-90°C, the density and viscosity of acid phosphate pulps obtained from the decomposition of phosphorite powder Khodzhaikul with phosphoric acid in a wide range of initial components (EPA:PP = 100:5-30). It is shown that the pulps have high fluidity (no more than 1.37 g/cm³ and 7.08 centipoise).

2. The density and viscosity of phosphate pulps ammoniated to pH = 4.5 were measured. At optimal EPA:PP ratios = 100:20 and 100:25, ammonium phosphate pulp at 60°C has a density and viscosity of 1.32-1.34 g/cm³ and 20.06-24.13 centipoise. It is easily transported by pumping devices without any restrictions and is granulated and dried in a BGS apparatus.

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INVESTIGATION OF THE CHEMICAL-MINERALOGICAL COMPOSITION OF BENTONITE OF THE KHAUDAG DEPOSIT AND SYNTHESIS OF WINE FINING AGENTS BASED ON ITS

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Abstract. Fining is about removing unwanted material from wine while still in the cellar. It is part of the clarification and stabilisation process and involves adding a substance to the wine that will flush out

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