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Investigation of the Characteristics of the Obtained Extraction Phosphoric Acid and Phosphogypsum from Unfired Phosphorites

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Abstract

In this article, the process of obtaining wet-process phosphoric acid (WPPA) from unburnt washed, dried phosphorite concentrates of Central Kyzylkum (UWDC PCK) is studied. The influence of the duration of the extraction process and the ratio of fat was studied: T for the output technological parameters of the process.

It was found that the decomposition of UWDC PCK in the presence of a circulating solution of 16.2% P_2O_5 showed that the decomposition process takes place completely, at 80-85 ^oC in 2 hours, quite easily and quickly without excess sulfuric acid. At the same time, there is no intensive foaming, which is characteristic of unfired phosphorite of the Central Kyzylkum.

Keywords:unfired phosphorite, filtration, acid rate, gypsum, circulating solution, extraction, density, viscosity, rheological properties.

Introduction. According to the International Fertilizer Industry Association (IFA), consumption of phosphate fertilizers reached a record 40 million tons in 2010. Production of the three main types of phosphate fertilizers - monoammonium phosphate (MAP), diammonium phosphate (DAP) and triple superphosphate (TSP) - increased 16% to 28.4 million tons. This led to an increase in the volume of production and consumption of raw materials for their production, including phosphoric acid. The main application of phosphoric acid remains the production of phosphate and complex fertilizers, with more than 90% of phosphorus-containing ore used for this purpose. Other areas include animal breeding, food, wood and pharmaceutical industries, production of household chemicals, construction materials, etc.

Along with the launch of new production facilities, some manufacturers in Spain, Belgium, Japan and the United States announced the closure of obsolete and unprofitable plants. Most of the new phosphoric acid production facilities are integrated into the phosphate fertilizer industrial complexes, but in Jordan and Tunisia the end (marketable) product will be phosphoric acid, not fertilizer.

Table 1
Phosphoric acid capacity in some countries

Country	2011 y.	2015 y.
China	16,3	18,2
Morocco	4,4	6,7
Brazil	1,5	2,2
Tunisia	2,0	2,1
SaudiArabia	–	1,5
Jordan	0,6	1,3

To date, in the world as well as in Uzbekistan, a lot of research workis conducted to increase the range and improve the quality of phosphate and organomineralfertilizers, acids and other salts to meet the needs of agriculture and other industries [1-4].

Currently in Uzbekistan, wet-process phosphoric acid (WPPA) is received from a thermal concentrate of phosphorites of Central Kyzylkum that creates some technological and economic problems: high cost of raw materials, difficulty to regulate the extraction mode temperature, low concentration of the received acid [5-6].

Object and research methodology. The object of the study is a phosphate cake with the following composition: 23.0% P_2O_5; 51.25% CaO; 2.59% SO_3; 0.94% F.

The cake is formed during the desliming process as an intermediate product, which is washed, dried and allocated. In the processes after concentrating the cake, phosphate flour and heat-fired concentrated phosphorite are obtained.

Chemical analyses were conducted according to GOST mineral fertilizers [7]. X-ray analysis was performed on an empirical panalytic model device using the powder X-ray mode of gypsum, which was formed during the production of WPPA [8].Microscopic studies were performed on a scanning electron microscope with an EDS analyzer [9].

Results and discussion. At the beginning of the serial experiments to obtain WPPA from UWDC PCK, search experiments were conducted to determine the concentration interval of sulfuric acid on the decomposition process.

As the studies show, when using sulfuric acid in different concentrations - 56, 66, 76, 86 and 96% - the consistency of the reaction pulp changes from a foamy pulp, to liquid-fluid, mobile, wet and semi-dry, respectively. When 86% sulfuric acid is used, a foamy product with a density of 0.86 g/cm^{3}is formed and a degree of carbonization was no more than 47.6%.This slurry is used in the second extraction stage, which causes abundant wet foaming and wherein requires a large reactor volume.Screw reactors are needed to implement this process, but the technological

indicators of the second stage are worsened, in particular the filtration rate and techno-analytical extraction parameters.In order to prevent foaming, UWDC PCK was treated with different concentrations of sulfuric acid during 15 min.During the experiments, the degree of foaming, the state of the reaction mass, the degree of decarbonization, and the density of the reaction mass were monitored (Table 2).

Considering the data obtained, concentrations from 56 to 96% were chosen as the optimal intervals for varying the concentration of sulfuric acid. In order to maintain the water balance of the extraction process, it is desirable to dilute the sulfuric acid with a concentration of 96% recycled WPPA. As studies have shown, when using recycled WPPA with 15% P_2O_5 to dilute sulfuric acid and bringing up its concentration to 56%. This means that in order to preserve the reactivity of sulfuric acid, it should be diluted as much as possible with less concentrated recycled WPPA. A mixture of sulfuric and phosphoric acids is formed.

Table 2

Influence of technological parameters at the first stage of the obtaining process WPPA

№	Concentration H_2SO_4, %	Acid mixture temperature, %	Stateofthepulp	Acid mixture density, g/sm^3
1	96	20	Semi-dry	1.835
2	86	72.5	Wet	1.745
3	76	91	Movable	1.689
4	66	98	Fluidity	1.612
5	56	103	Foamy	1.542

When studying the process of obtaining WPPA by pretreatment of UWDC PCK with the mixture of recycled WPPA and/or circulating pulp in the laboratory conditions, the laboratory studies were carried out on the model setup using recycled solutions of WPPA with 15-16.2% P_2O_5 concentration, UWDC PCKwith content, (wt.%) P_2O_5 – 23.0; MgO – 1.58; Al_2O_3 – 0.24; CaO – 51.25; F – 1.8; CO_2 – 14.50 and others, sulfuric acid with a concentration of 96% was used, which was diluted

with phosphoric acid and brought to 56%. A certain amount of UWDC PCK was treated with a 56% mixture of sulfuric acid diluted with a portion of recycled WPPA and/or circulating pulp during 10-15 min at 70-80°C. The rate of sulfuric acid is 100% of the stoichiometry on CaO [10].

It should be noted that when treated with 56% sulfuric acid, abundant foaming is observed, while after 10 min an abundant foam mass was formed. In the case of processing this mass in the second stage, there is almost the same abundant foaming as in the first stage.

Studies have shown that during the treatment of phosphorite with 56% sulfuric acid, the foaming of the pulp is practically not observed. Therefore, we further studied the extraction process with 56% sulfuric acid after decarbonization.

Therefore, in this work, a certain amount of UWDC PCKwas treated with a 56% mixture of sulfuric acid, obtained with diluted 96% sulfuric acid, recycled WPPA and / or circulating pulp for 10-15 minutes at 70-80 °C [8-9]. The rate of sulfuric acid is 100% of the stoichiometry on CaO [11-14].

We investigated the influence of the duration of the extraction process and the ratio L:S on the output technological parameters of the process (Tables 2, 3).

The data of chemical analysis (Table 3) of WPPA and gypsum obtained by decomposition of UWDC PCK in the presence of circulating solution containing 16% P_2O_5 showed that the decomposition process goes almost completely, at 80-85 ^oC for 2 hours quite easily and quickly without excessive sulfuric acid.In this case, there is no intense foaming characteristic of unburnt phosphorite CK.

Table 3

The chemical composition of the wet-process phosphoric acid and gypsum from the unburnt cake of the washed phosphoconcentrate

№	Extraction time, h	S:L ratio	Contentofcomponents, wt.%							
			wet-process phosphoric acid				Gypsum			
			P_2O_5	CaO	SO_3	F	P_2O_5	CaO	SO_3	F

1	2	1:2.5	18.45	0.36	5.79	1.02	8.61	40.42	29.87	1.67
2	2	1:3.0	18.28	0.39	2.69	1.11	6.12	29.21	23.94	1.09
3	2	1:3.5	19.59	0.33	2.94	1.21	1.99	33.64	34.88	0.33
4	1	1:3.0	17.75	0.36	6.60	0.91	7.73	33.62	27.04	1.67
5	4	1:3.0	18.31	0.37	2.69	1.19	1.91	27.0	26.61	0.44

With increasing the S:L from 1:2.5 to 1:3.5 at the duration of 2 hours, the content of total P_2O_5 in gypsum decreases from 8.61 to 1.99%, and with increasing the duration of the process from 1 to 4 also at S:L = 1:3 the content of P_2O_5 in phosphogypsum decreases from 7.23 to 1.91%.

It should be noted that in experiments 3, 4 and 5, the temperature of the recycled solution was 40 °C lower than in other experiments.

On the basis of the above, it can be concluded that when using the recycled solution with 16% P_2O_5 at a temperature of 20-25 °C, can be obtained WPPA with a content not less than 19% at temperature and S:L process 80 °C and 1:3 respectively, the duration of 2-4 hours, stoichiometric rate - 90%.

For the selection of equipment for the extraction and filtration process, the rheological properties of the intermediate reaction products are necessary, and therefore we studied the rheological properties of the suspension depending on the S: L and temperature (Table 4).

Table 4
Rheological properties of the obtained WPPA and phosphogypsum

Ж:Т	Density,g/sm^3				Viscosity, cPs (sec)			
	20 °C	40 °C	60 °C	80 °C	20 °C	40 °C	60 °C	80 °C
19,6% P_2O_5								
2:1	1.398	1.330	1.322	1.335	9.821	8.985	7.427	7.285
3:1	1.316	1.306	1.296	1.286	5.522	5.210	4.974	4.814
4:1	1.307	1.301	1.292	1.280	5.412	5.165	4.923	4.783
5:1	1.296	1.290	1.286	1.275	5.331	5.142	4.894	4.699

17% P_2O_5								
2:1	1.378	1.369	1.366	1.362	7.645	7.318	6.977	6.752
3:1	1.332	1.322	1.315	1.302	6.336	6.045	5.400	5.288
4:1	1.288	1.280	1.277	1.266	5.774	5.291	4.961	4.778
5:1	1.280	1.276	1.266	1.262	5.694	5.244	4.921	4.682

It follows from the data that with an increase in the mass ratio of L:S from 2:1 to 5:1 at a temperature of 20 °C, the density decreases from 1.398 to 1.296 g/sm^3. As the temperature increases to 80 °C, the density of the suspense decreases from 1335 to 1.275 g/sm^3. The viscosity of the suspension also varies depending on the temperature and the ratio L:S of the suspension decreases from 9800 to 4699 cPs.

Electron microscopic (SEM) studies also showed (Fig. 1) that phosphogypsum sediments are mainly composed of Ca, S and P in the amount of 20.3; 17.5 and 1.2 %, respectively. As shown by microscopic analyzes, individual thin crystals of small thickness are found in the sediments.

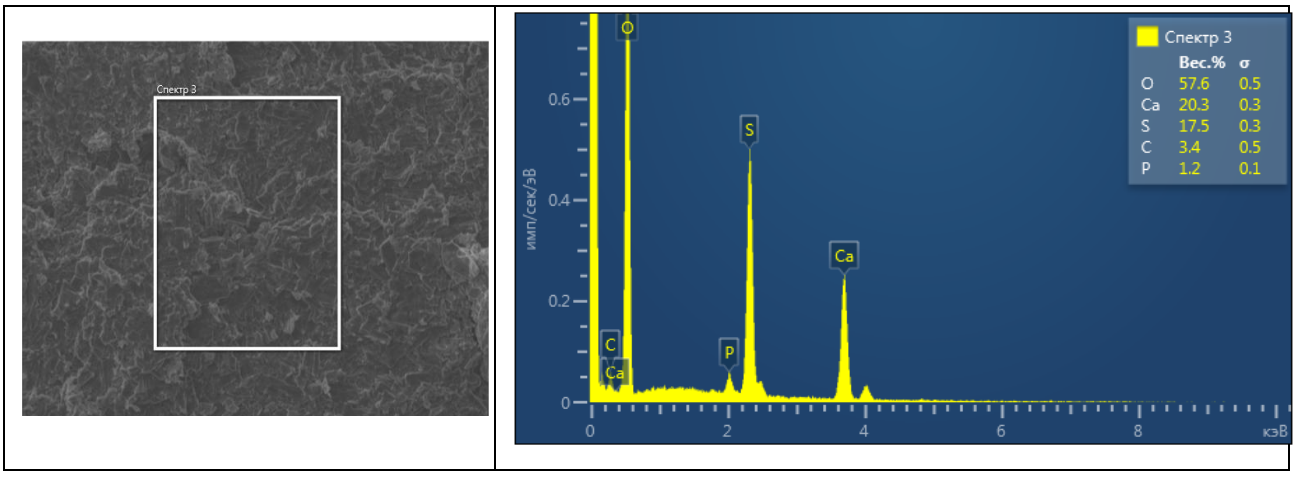


Figure 1. Energy dispersive spectrum of gypsum on a scanning electron microscope

From X-ray phase analysis (Fig. 2) it follows that the precipitate mainly consists of calcium sulfate dihydrate $CaSO_4 \cdot 2H_2O$ - 96%, and a small amount of anhydrite and undecomposed phosphorite.

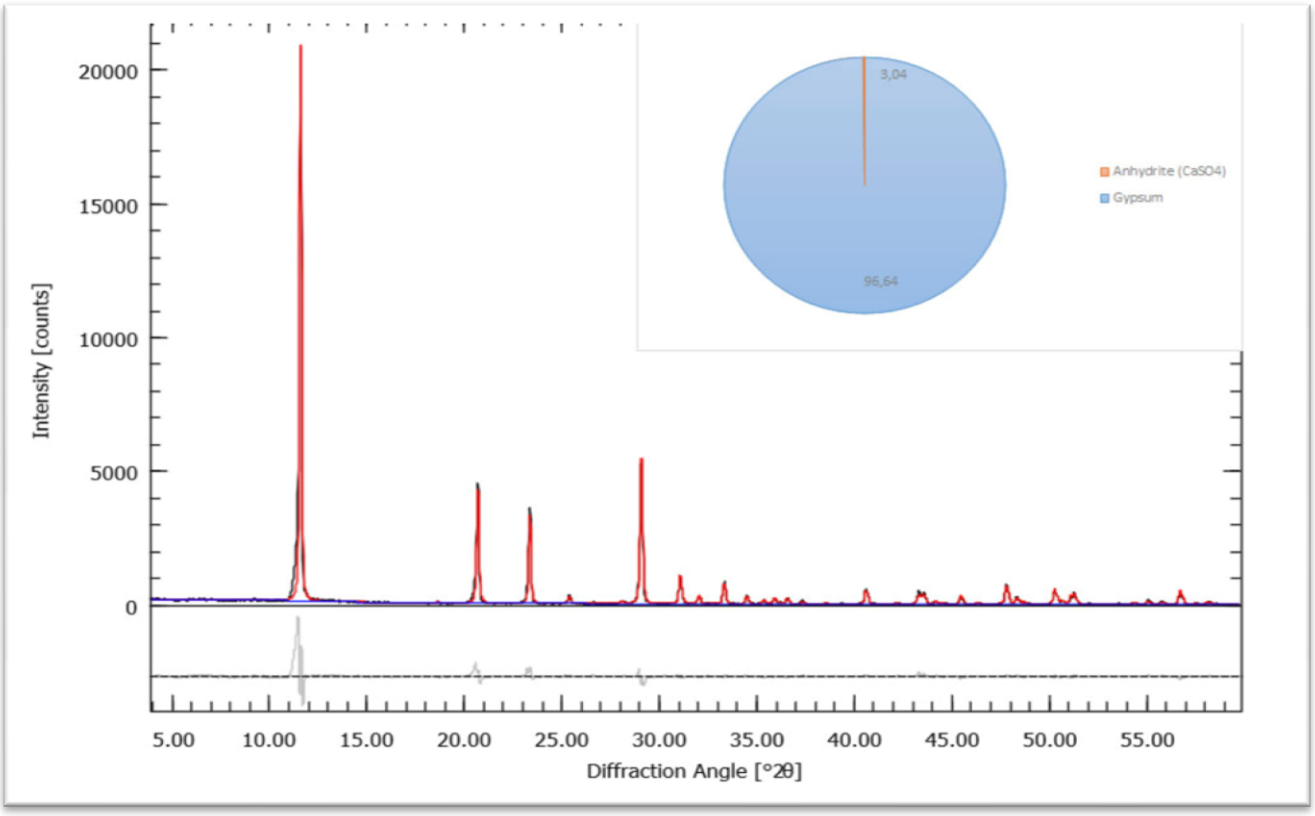
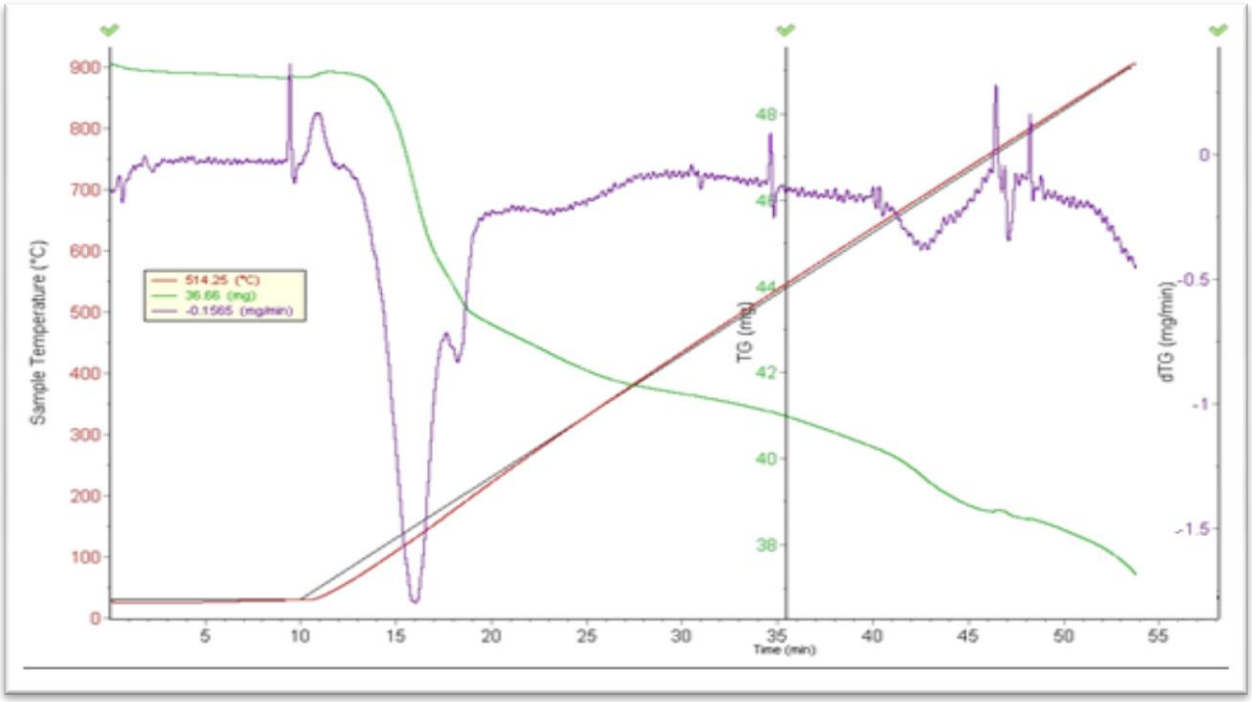


Figure 2. Radiographs of gypsum.



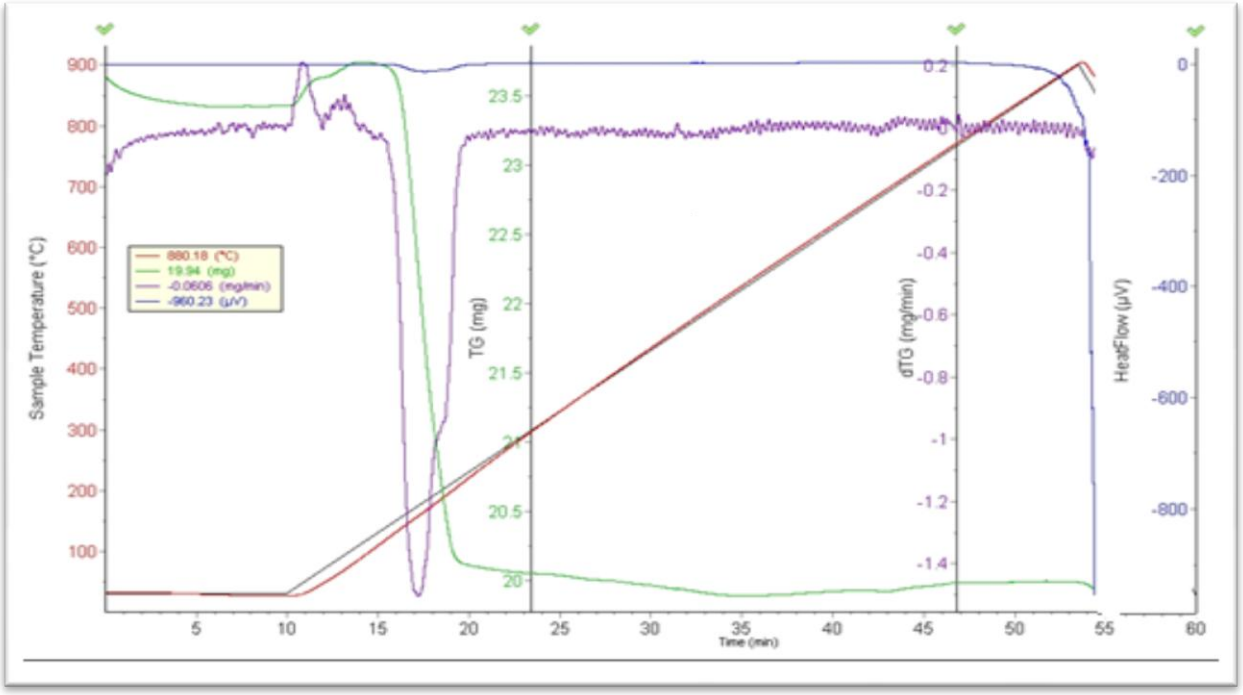


Fig. 3 it can be seen that in the intervals of 200-800 ° C there is a deep endo-effect of a stepwise nature with a total weight loss of ~ 15%, which is characteristic of calcium sulfate dihydrate and hemihydrate.

Conclusion. The results of the studies show that when maintaining the above-mentioned conditions, a decrease in foam formation during extraction of WPPA from unburnt phosphorites, as well as an increase in the amount of P_2O_5 in WPPA up to 19.59% is observed.

With an increase in T: W from 1: 2.5 to 1: 3.5 at the process duration of 2 hours, the content of P_2O_5 total. in phosphogypsum decreases from 8.61 to 1.99%, and with an increase in the duration of the process from 1 to 4 hours and at S:L = 1: 3, the content of P_2O_5 in phosphogypsum decreases from 7.23 to 1.91%.

It was found that phosphogypsum consists of 20.3; 17.5; 1.2% % Ca, S and P, respectively.

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