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Studying the Process of Obtaining Purified Phosphoric Acid from Dycalcium Phosphate

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ABSTRACT: The article presents the results of the process of obtaining purified phosphoric acid from dicalcium phosphate. The influence of the acid norm and the concentration of the circulating solution on the technological parameters of the process is studied. The optimal parameters of temperature and the T:W ratio for obtaining purified phosphoric acid together with pure phosphogypsum are investigated.

KEYWORDS: dicalcium phosphate, acid, filtration, acid norm, circulating solution, extraction, sulfuric acid, purified phosphoric acid, roentgenorgamma.

I. INTRODUCTION

Phosphoric acid is produced by a wet or oven (thermal) method. When applying the wet process phosphoric acid is formed by the chemical reaction of phosphate rock with a mineral acid. This method is known to be the most economical way to obtain phosphoric acid [1]. The wet process can use three mineral acids: nitric, hydrochloric and sulfuric. Due to the presence of impurities in the raw materials, phosphoric acid obtained inevitably has many other chemical compounds. Some of these impurities are detrimental to the quality of the acid during its final use in the composition of fertilizers, some emulsifiers used in the food industry [2]. For this, the EPA must be completely purified from accompanying impurities. There are several physicochemical methods of purification from impurities based on precipitation, adsorption, ion exchange and extraction with selective extractants and solvents. Today, for the production of phosphoric acid at a new pace, it is experiencing some technological changes. Especially in recent years, both in Russia and abroad, there is a decline in production of thermal phosphoric acid (TFA) and increased production of purified phosphoric acid (FCS) [3]. The present work is devoted to the production of purified phosphoric acid from dicalcium phosphate, which in turn is obtained from low-grade phosphorites of the Central KyzylKum in nitric acid extraction. In the application of thermal or furnace process phosphoric acid produced from elemental phosphorus. This process is used in the production of phosphoric acid for purposes other than fertilizer production, such as processing metals, refractories, catalysts, and food and beverage applications. The advantages of the OFK are low cost, high technological effectiveness of the process and the possibility of obtaining phosphoric acid of any quality (technical, food, pharmaceutical, reactive). Along with the low cost, the wide development and spread of the production of OFK is facilitated by the low energy consumption of the process (up to 200 kWh per 1 ton of P2O5 versus 7500 kWh) [4-6]. The main world exporters of phosphoric acid continue to increase the production capacity of extraction phosphoric acid (EPA) and expand the range of products produced on its basis [6].

II. SIGNIFICANCE OF THE SYSTEM

The article presents the results of the process of obtaining purified phosphoric acid from dicalcium phosphate. The study of literature survey is presented in section III, methodology is explained in section IV, section V covers the experimental results of the study, and section VI discusses the future study and conclusion.

III. METHODOLOGY

The object of research is purified phosphoric acid. The subject of research was the technology of obtaining EPA from low-grade phosphorites of the Central KyzylKum and its subsequent purification to the stage of the RPC. The purpose of the work was: to study the process of obtaining purified extraction phosphoric acid. Research

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methodology: 1. Chemical analysis; 2. Experimental (photocolorimetric, titrimetric, potentiometric, mass spectrometric, X-ray phase thermogravimetric and IR spectroscopic) [7-8].

IV. EXPERIMENTAL RESULTS

In laboratory conditions, the process of obtaining purified extraction phosphoric acid from dicalcium phosphate was carried out at a temperature of 80 °C at different ratios of the application of the rate of sulfuric acid and a change in the concentration of EPA in the circulating solution. In the study of the process of obtaining OFA and pure phosphogypsum from dicalcium phosphate with a mixture of recycled TPA and/or circulating pulp, in laboratory conditions, they were carried out on a model setup using recycled solutions with thermal phosphoric acid (TPA), concentration 17-25% P_2O_5, dicalcium phosphate containing (wt%) P_2O_5 – 32,0; CaO 32,8; and others using sulfuric acid with a concentration of 96%. A certain amount of dicalcium phosphate was treated with 96% sulfuric acid, diluted with circulating TPA, and / or circulating pulp for 5 minutes at 75-80 °C. The rate of sulfuric acid is 100 - 125% of the stoichiometry for CaO. The S:W ratio was 1: 3.5, respectively. It was found that the filtration rates of solutions formed when the acid rate changes are different. The densities of the RPC and washes obtained as a result of the reactions are presented in Table 1.

Table 1
Density of the liquid phase of the extraction phosphoric acid and wash water, g/cm^3

№	EPC	First wash	Second wash	Third wash
1	1,195	1,141	1,080	1,021
2	1,212	1,147	1,086	1,017
3	1,248	1,148	1,059	1,013
4	1,192	1,137	1,062	1,015
5	1,217	1,138	1,061	1,015
6	1,247	1,131	1,051	1,010
7	1,260	1,158	1,056	1,012
8	1,286	1,161	1,052	1,012
9	1,272	1,160	1,055	1,012

In the table above, the densities of each of the PFCs obtained in the stage-by-stage washes are presented in order of priority by stage. In this case, the density of the acid was 1,195-1,248 g/cm^3. With an increase in the concentration of circulating TPA, a gradual change in the density of the obtained phosphoric acid towards an increase in concentration was noted. To determine the composition of the obtained products, we performed photometric, titrimetric and gravimetric analyzes. The results, which are shown in Table 2, are the general chemical analysis of OPA and pure phosphogypsum, obtained by decomposition of DKF at different concentrations of circulating TPA, at a temperature of 80 °C for 4 hours.

Table 2
Results of chemical analysis of RPC and pure phosphogypsum at various parameters of sulfuric acid norms

№	Reaction time, h	Rate of acid, g	C total solution, %	Purified phosphoric acid, %			Pure phosphogypsum. %		
				P_2O_5	CaO	SO_3	P_2O_5	CaO	SO_3
1	4	100	17	18,25	0,47	1,56	0,48	16,75	44,56
2	4	100	20	20,08	0,69	1,72	0,39	17,33	46,13
3	4	100	25	24,67	0,73	1,41	0,61	17,21	44,79
4	4	115	17	18,95	0,87	2,23	0,33	17,17	48,78
5	4	115	20	21,45	0,78	2,29	0,20	18,19	51,44
6	4	115	25	24,52	0,25	2,53	0,21	17,67	48,17
7	4	115	30	29,4	0,57	1,81	0,23	17,56	47,89
8	4	115	35	34,6	0,54	1,82	0,24	17,84	48,63
9	4	125	30	29,6	0,52	1,84	0,28	17,65	48,72

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As the results of chemical analysis show, with an increase in the rate of sulfuric acid from 100 to 115 g, the P_2O_5concentration increases from 37,3 to 37,9%, and when using recycled TPA with a concentration of 17%, and with 25% recycled TPA, the amount of P_2O_5 increases with48,21 to 49,04%, respectively.

Table 3

Influence of the consumption of the acid rate and the concentration of the circulating solution on the technological parameters in the process of obtaining purified phosphoric acid from dicalcium phosphate

name	Norm H_2SO_4	T:J	C Total solution	J	Phosphogypsum moisture	Filtration rate	density	m phosphogypsum
№1								
EPC	100	1:3,5	17	219	47	209,67	1,195	40
1-flush			12	169,5	52	133,03	1,141	
2-flush			6	137,7	54	123,90	1,080	
3-flush			0	138,3	49	115,76	1,021	
№ 2								
EPC	100	1:3,5	20	251,7	51	481,13	1,212	40,6
1-flush			12	142,5	50	118,27	1,147	
2-flush			6	136,1	46	114,03	1,086	
3-flush			0	139,9	48	110,77	1,017	
№ 3								
EPC	100	1:3,5	25	249,5	53	490,83	1,248	35,6
1-flush			12	149	52	97,10	1,148	
2-flush			6	140,2	53	116,58	1,059	
3-flush			0	136,4	46	108,86	1,013	
№ 4								
EPC	115	1:3,5	17	243,7	56	484,41	1,192	39,3
1-flush			12	135,1	56	166,49	1,137	
2-flush			6	162	57	121,64	1,062	
3-flush			0	140,2	54	117,56	1,015	
№ 5								
EPC	115	1:3,5	20	239,4	52	675,76	1,217	41
1-flush			12	140,9	53	199,65	1,138	
2-flush			6	138,8	52	192,77	1,061	
3-flush			0	137,1	46	194,35	1,015	
№ 6								
EPC	115	1:3,5	25	257,1	48	1283,21	1,247	35,8
1-flush			12	137,3	52	843,27	1,131	
2-flush			6	134,8	49	680,07	1,051	
3-flush			0	134,5	51	635,91	1,010	
№ 7								
EPC	115	1:3,5	30	237,2		1461,83	1,260	41,6
1-flush			12	153,7		609,45	1,158	
2-flush			6	137,6		637,97	1,056	
3-flush			0	139,8		653,11	1,012	
№ 8								
EPC	115	1:3,5	35	251,6		1406,14	1,286	42,6
1-flush			12	152,0		512,38	1,161	
2-flush			6	138,9		628,38	1,052	
3-flush			0	137,2		514,72	1,012	
№ 9								
EPC	125	1:3,5	30	236,6		1489,67	1,272	41,5
1-flush			12	145,3		579,96	1,160	

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2-flush			6	138,6		769,15	1,055	
3-flush			0	137,1		795,68	1,012	

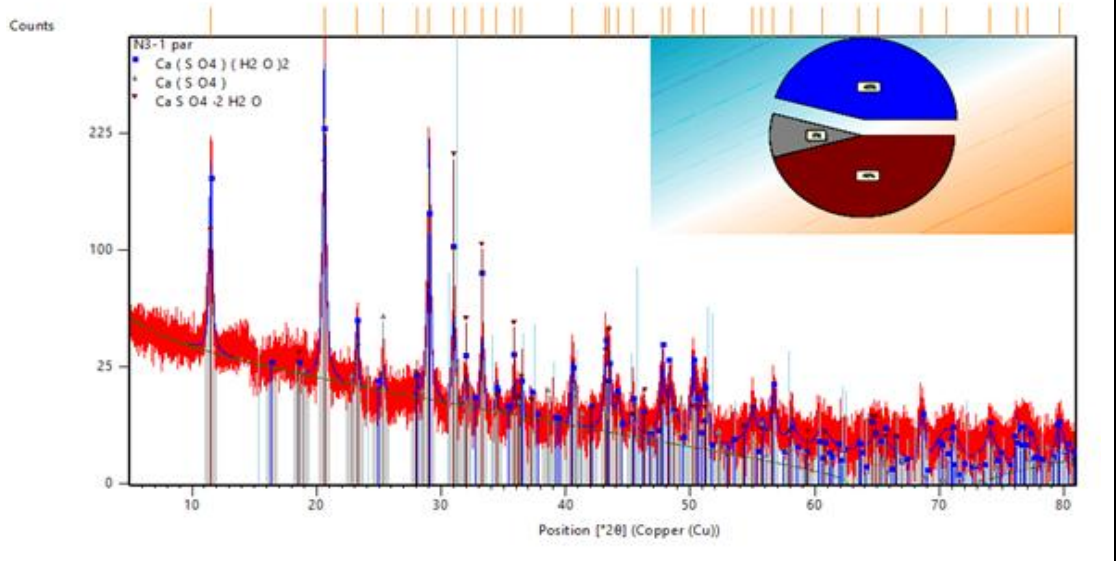
Table 3 above shows the results of the preparation of RPCs performed using thermal phosphoric acid as the circulating solution. The filtration rate is considered to be the main technological indicators.In connection with this, we studied the filtration rate of the phosphoric acid suspension (Table 4).

Table 4

Influence of technological parameters on the filtration process when obtaining OFC from DKF

№	Production EPC, kg/m2·h		First wash, kg/m^2·h		Second wash, kg/m^2·h		Third wash, kg/m^2·h		Phosphogypsum moisture, %
	solid	liquid	solid	liquid	solid	liquid	solid	liquid	
1	209,67	516,51	133,03	256,53	123,90	196,33	115,76	186,38	51
2	481,13	1424,72	118,27	200,65	114,03	186,98	110,77	188,99	50
3	490,83	2017,53	97,10	242,35	116,58	278,45	108,86	257,36	37
4	484,41	1452,04	166,49	279,09	121,64	248,51	117,56	210,50	49
5	675,76	2084,77	199,65	362,52	192,77	344,59	194,35	343,38	47
6	1283,21	4850,96	843,27	1727,05	680,07	1387,31	635,91	1312,62	45
7	1461,83	4718,83	609,45	1309,93	637,97	1263,28	653,11	1353,24	42
8	1406,14	5003,64	512,38	1133,81	628,38	1308,77	514,72	1091,66	41
9	1489,67	4705,71	579,96	1156,11	769,15	1504,03	795,68	1583,74	44

From the data obtained, it follows that with an increase in the concentration of circulating TPA from 17 to 25% and the use of a rate of sulfuric acid from 100 to 125 g, the filtration rate increases in the liquid and solid phases, respectively. In the first variant of the experiment, the filtration rate was 209,67 kg/m^2·h in the solid phase, and with an increase in the rate of sulfuric acid to 115 g in the presence of 17% P2O5 recycled TPA, the filtration rate increased more than two times and amounted to 484,41 kg/m^2·h. X-ray photographs of phosphogypsum were obtained on a high-precision X-ray fluorescent energy-dispersed apparatus of the Shimadzu XRD-6100 model (Fig. 1).



a)

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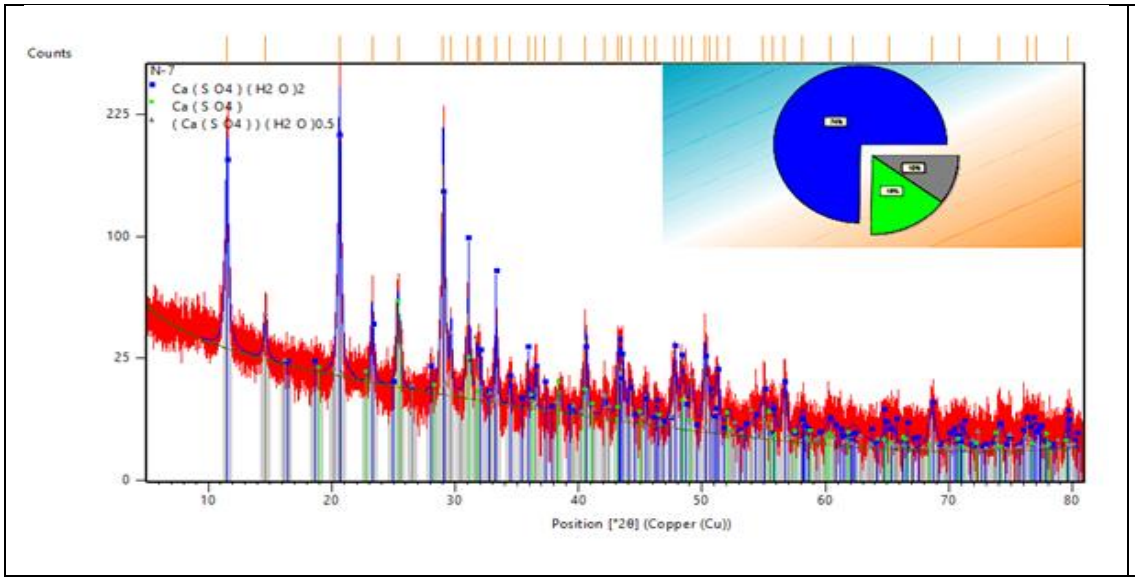

b)

Rice.1. Radiographic images: a) sample №3- phosphogypsum;b) sample №8–phosphogypsum

X-ray studies (Fig. 1) showed that with an increase in the concentration of the circulating solution and an increase in the rate of sulfuric acid from 17 to 25%, the yield of P_2O_5 increased from 18,25 to 24,67%, respectively. Mineralogical composition of samples

Visible	Ref.Code	Score	Compound Name	Displ.[2θ]	Scale Fac.	Chem. Formula
*	01-074-1905	51	Calcium Sulfate Hydrate	-0.120	0.558	$Ca(SO_4)\ (H_2O)_2$
*	01-075-5972	6	Calcium Sulfate	-0.142	0.090	Ca(SO4)
*	00-021-0816	34	Calcium Sulfate Hydrate	-0.120	0.496	$CaSO_42H_2O$
*	01-076-8724	38	Calcium Sulfate Hydrate	-0.103	0.608	$Ca(SO4)\ (H_2O)_2$
*	01-085-6122	14	Calcium Sulfate	0.019	0.126	$Ca(SO_4)$
*	01-076-6905	9	Calcium Sulfate Hydrate	-0.125	0.078	$(Ca(SO_4))\ (H_2O\ )0.5$

Derivatograms of the samples were recorded on a LabSysEvothermogravimetricderivatograph (Setaram, France) up to 1400 °C, the heating rate of the samples was 10 deg/min.

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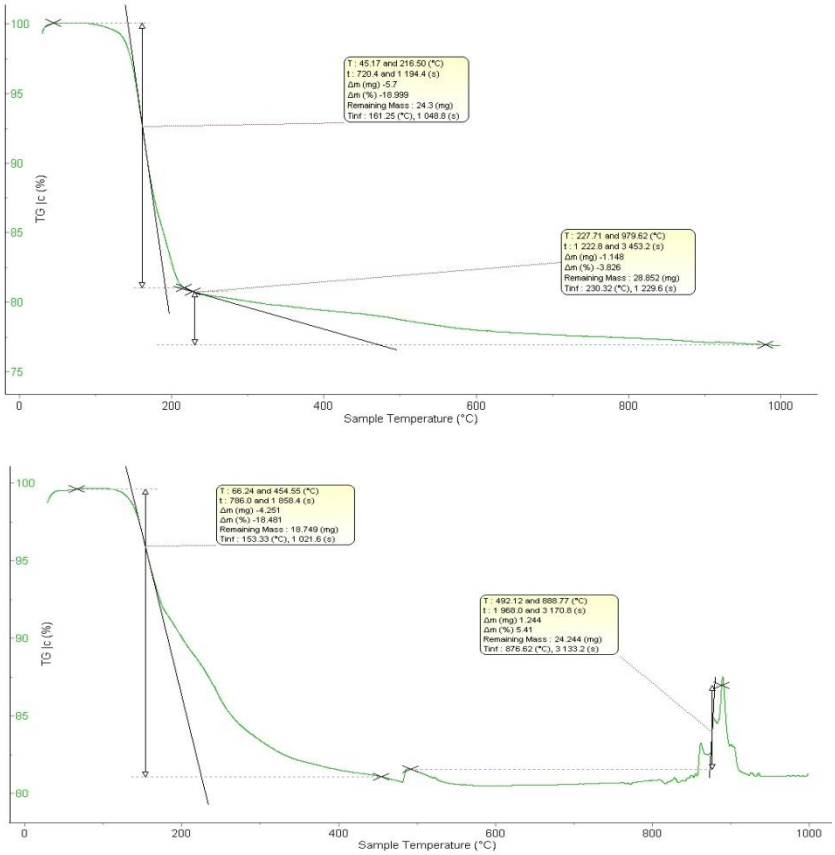

a

b

Rice.2. Thermogravimetry of phosphogypsum (according to tab. №2) a) phosphogypsum№ 3;b) phosphogypsum№. 6

The results obtained showed that in the variant at intervals of 90-980 °C, a deep endo-effect of a stepwise nature was noted with a total weight loss of 22,9%, characteristic of phosphate dihydrate, while the weight loss in quantitative terms was 6,6 mg.In the second variant, the weight loss was 18,25%, at a temperature of 90-560 °C

V. CONCLUSION AND FUTURE WORK

In this experimental work, the processes of obtaining purified extraction phosphoric acid by the dihydrate method were investigated.Thermal phosphoric acid and dicalcium phosphate were used as reagents.Based on the studies performed, the following conclusions can be drawn: 1. It was found that when processing dicalcium phosphate, the filtration rate increased, respectively, with the rate of sulfuric acid used and the concentration of the circulating solution.2. It was revealed that the output of P_2O_5 in the 8th variant of the experiment is the highest state

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