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CONCENTRATION OF WET PROCESS PHOSPHORIC ACID FROM WASHED THERMO CONCENTRATE OF PHOSPHORITES CENTRAL KYZYLKUM AND ITS RHEOLOGICAL PROPERTIES

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ABSTRACT

The process of purification and concentration of wet process phosphoric acid (WPA) creates the possibility of obtaining more concentrated phosphorus - containing products, such as ammonium dihydrogen phosphate and ammonium hydrogen phosphate, calcium dihydrogen phosphate and calcium dihydrogen phosphate, liquid complex fertilizers for drip irrigation. WPA from the washed thermo concentrate (WTC), although low concentrated (≥ 20 % P_2O_5), contains alone 0.3 - 0.5 % MgO and is suitable for concentration by evaporation. To obtain concentrated phosphoric acid, it is necessary to carry out the processes of desulfurization and evaporation of the Kyzylkums WPA.

Keywords: *wet process phosphoric acid (WPA), strontium carbonate, desulfurization, purification, evaporation, rheology, washed thermo concentrate (WTC), density, viscosity.*

INTRODUCTION

The main application of phosphoric acid remains the production of phosphate and complex fertilizers, and therefore about 90 % of phosphorus-containing ore is used for these purposes [1]. Other areas include livestock farming, poultry farming, fishing, food, woodworking, metallurgical and pharmaceutical industries, production of household chemicals and others.

In the world, the main producers of phosphoric acid and ore for its production are located directly in the quarries and mining areas, accounting for almost half of all mined raw materials. According to 2021 data, the production capacity of phosphate fertilizers is distributed among companies with the following proportion: OCP Group (Marocco), The Mosaic Company (USA) - more than 4 million tons per year; Potash Corporation of Saskatchewan (Canada), Yunnan Yuntianhua International Chemical Co (China) - more than 2 million tons per year; PhosAgro (Russia) - more than 1 million tons per year.

The purification and concentration of weak wet process phosphoric acid (WPA) hold significant importance as they allow for the creation of more concentrated phosphorus-based products [2, 3]. ADP (ammonium dihydrogen phosphate), AHP (ammonium hydrogen phosphate) and TSP (triple superphosphate) as highly concentrated, 100 % water - soluble fertilizers are necessary for growing plants with a short growing season and on poor soils with low sorption capacity, as well as for sale abroad. Manufacturing them, using thermal phosphoric acid (TPA) as a foundation is simple, yet it results in the increased expense of the final product [4]. It has the potential to be substituted with wet process phosphoric acid (WPA). But the technology with WPA is suitable only for the Khibiny apatite concentrate (39.4 % P_2O_5) and similar foreign phosphate raw materials Khuribga 80 - 82 TPL, BuKraa 80 TPL (Morocco), Nauru 84 BPL (Nauru Island).

The raw materials mentioned above can be utilized to create various types of phosphate fertilizers with satisfactory technical and economic measures. WPA obtained from such phosphate materials (with low contents of Mg, Fe, Al, Ca and other impurities) is easily evaporated to 40 - 55 % P_2O_5. For example, WPA obtained from Khibiny concentrate without distillation contains (%): P_2O_5 29 - 30; SO_3 1.8 - 2.5; F 1.5 - 1.8; R_2O_3 1.3 - 2.0; CaO 0.2 - 0.4; MgO - no, but with evaporation: P_2O_5 50 - 54; SO_3 3.6 - 5.0; F 0.5 - 0.8; R_2O_3 1.6 - 1.9; CaO 0.2 - 0.6; MgO - no.

WPA from Karatau phosphorites contains (%): P_2O_5 - 22.0; MgO - 3.0; SO_3 - 2.5; F - 1.6; R_2O_3 - 2.2; CaO - 0.3; Fe_2O_3 - 1.0; $A1_2O_3$ - 1.2. However, it cannot be evaporated due to its high magnesium content. The reason for this is the formation of a colloidal solution containing MgF_2; when it cools, the system thickens into a viscous, inactive mass (gel).

It should be noted that the production of phosphorus - containing fertilizers in the Republic of Uzbekistan is limited by the quality of the available phosphorite at the Central Kyzylkum deposit. This is a raw material with a low phosphorus content and contains many impurities. A large amount of acid is not consumed in the decomposition of fluorapatite, but a large amount of waste is generated. To produce top - notch phosphorus fertilizer from these raw materials, the initial step involves enhancing its quality through enrichment. Therefore, at the Kyzylkum Phosphorite Plant (KPP), multi - stage enrichment was carried out: crushing to produce ordinary phosphate rock, high enrichment, and chlorine washing. This plant provides phosphate raw materials from the Kyzylkum Phosphorite Plant (KPP). KPP annually produces three types of phosphate rock products: 400.000 tons of washed calcined phosphorus concentrate (WCPC - 27 % P_2O_5), 200.000 tons of washed dried concentrate (WDC - 18 % P_2O_5) and 200.000 tons of ordinary phosphorite powder (OPP - 17 % P_2O_5) [5].

Wet process phosphoric acid from washed thermo concentrate of phosphorite Kyzylkum containing 26 % P_2O_5 (Washed Thermo Concentrate - 26), having a low concentration (> 20 % P_2O_5), contains 0.3 - 0.5 % MgO, which is suitable for concentration by evaporation. It should be noted that phosphoric acid obtained by sulfuric acid extraction of natural phosphates contains a total of up to 15 mass % impurities, the main ones of which are the following:

cations: Ca^{2+}, Mg^{2+}, K^+, Na^+, Fe^{3+}, Al^{3+}, Pb^{2+} and etc;

anions: SO_4^{2-}, F^-, SiF_6^{2-}, $C1^-$, HSO_4^-, AsO_4^{3-}.

In the series of anions, SO_4^{2-} are sulfates of Ca, Mg and others, and HSO_4- is free sulfuric acid [6, 7].

Traditional technologies for purification and concentration of WPA include the following stages: desulfation, clarification, stripping with hot gaseous coolants (defluorination), precipitation of impurity components with various inorganic and organic substances [8].

Acid desulfation is the first and determining stage in the process of WPA purification, since the content of sulfate ions in it exceeds the concentration of other impurities, reaching 2.5 - 3.0 % (mass). Currently, the most common way to reduce free sulfuric acid or its soluble salts in WPA is to treat the latter with natural phosphates and calcium carbonates [9, 10]. The main disadvantage of the method is that calcium or phosphate minerals do not completely precipitate sulfate ions from the WPA composition (up to 85 % SO_4^{2-}).

EXPERIMENTAL

The process of desulfation of WPA with strontium carbonate was carried out. Since, unlike barium compounds, strontium salts are low-toxic substances [11], they reduce the residual SO_4^{2-} content in the WPA solution by almost 30 - 40 times than that of phosphates and calcium carbonate. It is known that at 18°C the solubility of $SrSO_4$ in water is 0.011 %, while for $CaSO_4$ this value is 0.5 - 0.6 % [12].

Concentrated solutions were prepared from desulfated acid by evaporation. The content of P_2O_5 in the samples was determined by photocolorimetric, SO_3 - by gravimetric, F - by potentiometric methods. Calcium and magnesium were determined by the complexometric method. The method is based on changing the color of the indicator (fluorexone for the determination of calcium and dark blue acid chromium for the determination of magnesium) with Trilon B. The sulfate content was determined by gravimetric method. The method involves causing sulfate precipitation using barium chloride in an acidic setting, and then determining the weight of the sediment formed. The complexometric method was employed to ascertain the levels of iron and aluminum oxides content. The technique relies on titrating 0.0125 M iron with Trilon B solution, facilitated by sulfosalicylic acid, followed by back titration of excess Trilon B using a 0.0125 M zinc sulfate solution. This allows for the determination of aluminium in the presence of xylene orange indicator. The density of solutions was determined by pycnometric methods, and kinematic viscosity by viscometric methods.

In the process of evaporation of desulfated acid, samples of concentrated WPA containing 45.62 were obtained; 50.01; 55.25 and 60.34 % P_2O_5. After settling concentrated acids for 24 hours, samples of the clarified part and their sediments were obtained. The acid precipitates were separated and washed with acetone until neutrality, then X - ray analysis of the dried sediments was carried out. X - ray diffraction analysis was carried out on an XRD - 6100 diffractometer (Shimadzu, Japan). CuK_α radiation was used (β - filter, Ni, tube current and voltage mode 30 mA, 30 kV) and a constant detector rotation speed of 4 deg min^{-1}, and the scanning angle varied from 4 to 80°. When removing samples, a rotating chamber was used, where the rotation speed was 30 rpm. The obtained results of X - ray diffraction patterns were analysed by comparison with the American ASTM map and tables of radiometric indicators for the determination of Mikhaev's minerals [14]. Some information was determined from X - ray structural analysis of Kitaygorodsky [15]. In addition, the analysis of the acquired X - ray patterns were conducted utilizing Mirkin and Giller's X - ray mineral identification method [13, 16].

RESULTS AND DISCUSSION

In laboratory, WPA produced by Ammophos - Maxam JSC was subjected to desulfation and evaporation with the following composition (mass %): P_2O_5 - 18.95; CaO - 0.27; MgO - 0.26; Fe_2O_3 - 0.41; Al_2O_3 - 0.56; F - 0.45; $SO_{3total.}$ - 2.96; $SO_{3wat.}$ - 0.15 and strontium carbonate grade "chemical pure" (not less than 98 % $SrCO_3$). The rate of strontium carbonate was taken from 95 to 105 % of the stoichiometry for the formation of $SrSO_4$ according to the reaction below:

$SrCO_3 + H_2SO_4 \rightarrow SrSO_4\downarrow + CO_2\uparrow + H_2O$

The initial WPA was placed inside a reactor equipped with a rotating screw and warmed using a water thermostat up to a temperature of 75 degrees Celsius. Then the calculated amount of strontium carbonate powder was gradually dosed into it. The duration of the mixing process was 30 min. After a given period, the contents of the reactor settled for 60 min at 60 - 65°C. Afterward, the desulfated WPA was filtered to separate it from the sediment, dried, and subsequently measured in terms of weight. After which the total content of SO_3 and P_2O_5 in the clarified part of the desulfated WPA and its sediment was determined according to the methods [17 - 19]. Based on the data obtained, the degree of desulfation of WPA was calculated. The P_2O_5 content in the dried sediment results in the loss of phosphorus from WPA. The results are shown in Table 1.

It shows that the optimal rate of $SrCO_3$ can be considered 100 % of the stoichiometry, at which an acceptable degree of WPA desulfation is achieved (97.34 %). Continuing to raise the dosage does not result in a significant rise in this measure. Additionally, it causes the WPA mixture to become contaminated with strontium salts.

The phosphoric acid thus obtained, purified from sulfate ion, and contains 20.53 % P_2O_5, 0.28 % CaO, 0.27 % MgO, 0.45 % Fe_2O_3, 0.62 % Al_2O_3, 0.10 % SO_3. Next, it was evaporated to various concentrations of P_2O_5. Evaporation of the acid was carried out in a tubular quartz reactor equipped with a quartz stirrer

Table 1. The degree of desulfation of wet process phosphoric acid depending on the rate of strontium carbonate.

Norm $SrCO_3$, %	$WPA_{desulfated}$, %		P_2O_5 content in dry sediment, %	Desulphurization degree, %
	P_2O_5	SO_3		
95	20.86	0.15	0.71	96.17
97.5	20.86	0.13	0.73	96.48
100	20.53	0.10	0.79	97.34
102.5	21.04	0.10	0.82	97.36
105	21.39	0.10	0.91	97.37

Table 2. The content of impurity components in desulfated evaporated WPA depending on the concentration of phosphorus pentoxide.

Concentration P_2O_5 in WPA, %	Contents of components, %						
	CaO	MgO	Fe_2O_3	Al_2O_3	SO_3	F	Phase degree by mass
45.62	0.63	0.60	1.01	1.39	0.21	0.42	100
50.01	0.69	0.63	1.08	1.54	0.24	0.40	100
55.25	0.75	0.73	1.23	1.68	0.28	0.38	100
60.34	0.82	0.78	1.34	1.83	0.30	0.32	100

under atmospheric pressure at temperatures of 150; 160; 165 and 180°C (up to boiling), respectively, for a concentration of 45.62; 50.01; 55.25 and 60.34 % P_2O_5. The composition of evaporated WPA is given in Table 2. It was visually observed that when WPA is evaporated to a concentration of 60 % P_2O_5, it does not thicken and is in a fluid state.

Next, the density and viscosity of solutions of evaporated WPA without the clarification stage were studied depending on the P_2O_5 content (45.62 - 60.34 %) and temperatures (30 - 100 °C). Density was determined by the pycnometric method [20], and the coefficient of kinematic viscosity was determined in a TLV (transparent liquid viscometer) viscometer [21].

The table shows that as the P_2O_5 concentration increases from 45.62 to 60.34 %, the content of impurity components except F increases (mass %): CaO from 0.63 to 0.82; MgO from 0.60 to 0.78; Fe_2O_3 from 1.01 to 1.34; Al_2O_3 from 1.39 to 1.83; SO_3 from 0.21 to 0.30. In this instance, the F content decreases from 0.42 to 0.32 %.

And it is shown that the density and viscosity of these solutions depending on their concentration and temperature. It has been established that both the density and viscosity of WPA are directly related to its concentration and temperature. Thus, at the studied temperatures (30 - 100°C), the density of the initial desulfated but not evaporated (20.53 %) WPA is in the range of 1.1630 - 1.2010 g cm^{-3}, for WPA with a concentration of 45.62 % P_2O_5 from 1.575 to 1.5221 g cm^{-3} and for WPA with a concentration of 55.25 % P_2O_5 from 1.7891 to 1.7310 g cm^{-3}.

With an increase in the P_2O_5 content from 45.62 to 60.34 %, the density of evaporated acids at 70°C increases from the initial (unevaporated) 1.180 g cm^{-3} from 1.5444 to 1.8782 g cm^{-3}. As the temperature rises from 30 to 100°C, the density consistently decreases in all samples. For example, for the initial desulfated but not evaporated acid (20.53 % P_2O_5) this figure decreases from 1.1630 to 1.2010 g cm^{-3}, for WPA with a concentration of 45.62 % P_2O_5 from 1.575 to 1.5221 g cm^{-3} and for WPA with a concentration of 55.25 % P_2O_5 from 1.7891 to 1.7310 g cm^{-3}. Thus, an increase in temperature contributes to a noticeable decrease in the density of acids (Fig. 1).

The viscosity of evaporated phosphoric acids shows a comparable trend. If for the original desulfated WPA (20.53 % P_2O_5) at temperatures of 30 - 100°C the viscosity is in the range of 0.78 - 1.80 cP (centipoise), then for 45.62 % acid this figure is 6.41 - 34.46 cP,

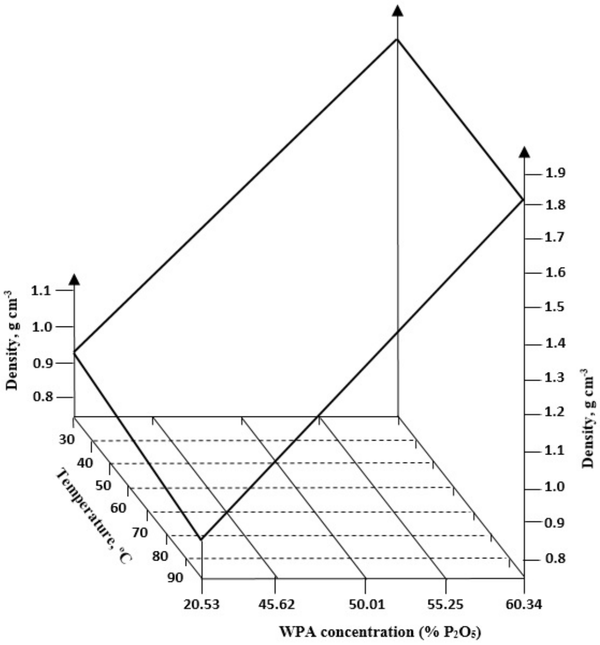

Fig. 1. Density of samples of evaporated phosphoric acids depending on temperature and concentration.

for 50.01 % acid 8.32 - 51.69 cP, for 55.25 % acid 13.51 - 89.3 cP. At a relatively increased concentration (60.34 % P_2O_5) and temperatures (30 - 50°C), the viscosity of WPA becomes significantly increased (140.55 - 354.49 cP), although in this case it is in a fluid state. This sets up ideal technological circumstances for transporting it via pipelines and converting it into completed mineral fertilizers (Fig. 2).

Therefore, the findings from conducted research indicate that low WPA with less than 20 % P_2O_5, derived from WTC - 26 (washed thermo concentrate - 26), can be efficiently desulfated using strontium carbonate (with a purity of at least 97 %) and concentrated through evaporation to achieve a 60 % P_2O_5 content. Simultaneously, the concentrated phosphoric acid produced is highly appropriate for manufacturing top-notch ADP and AHP, as well as calcium phosphates for animal feed. It can also be involved in the production of liquid complex fertilizers such as ammonium polyphosphates of grades N : P_2O_5 = 10 : 34 and 11 : 37.

Further should be noted to study XRD investigation of the precipitates formed during the settling process of the desulfated and evaporated acids. The resulting XRDs are shown in Figs. 3-7.

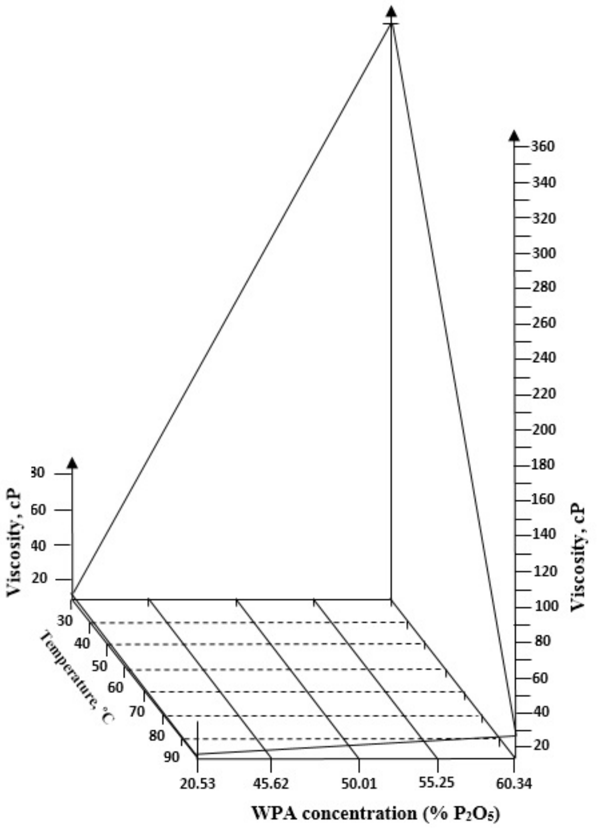

Fig. 2. Viscosity of evaporated phosphoric acid samples depending on temperature and concentration.

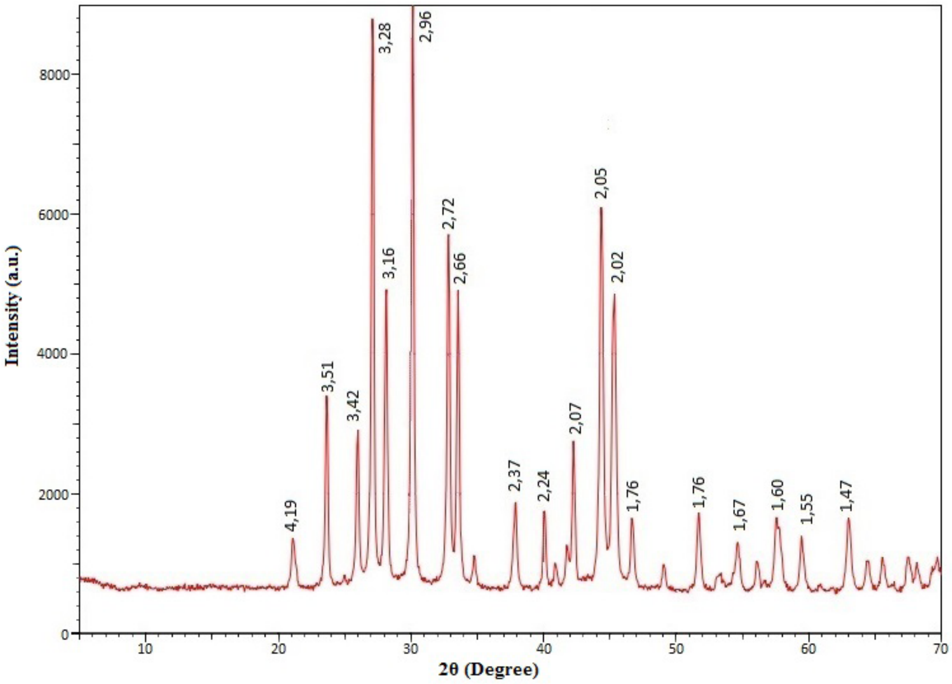

Fig. 3. XRD pattern of the original desulfated WPA.

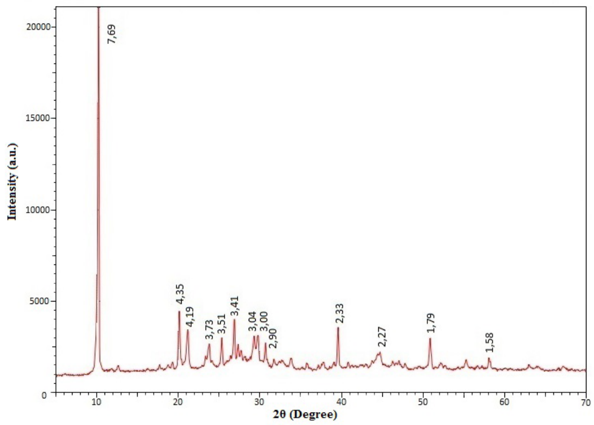

Fig. 4. XRD pattern of the sediment released from evaporated wet process phosphoric acid containing 45.62 % P_2O_5.

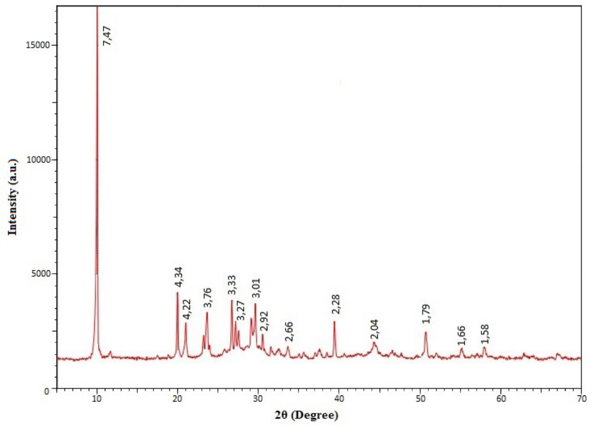

Fig. 5. XRD pattern of the sediment released from evaporated wet process phosphoric acid containing 50.01 % P_2O_5.

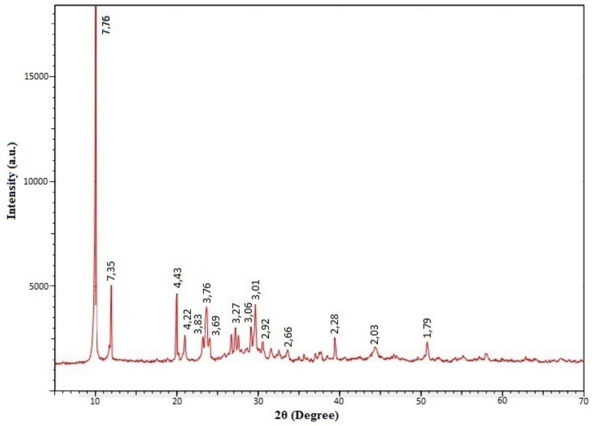

Fig. 6. XRD pattern of the sediment released from evaporated wet process phosphoric acid containing 55.25 % P_2O_5.

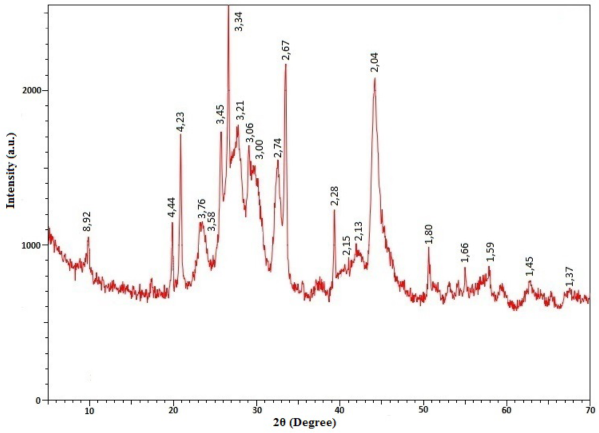

Fig. 7. XRD pattern of the sediment released from evaporated wet process phosphoric acid containing 60.34 % P_2O_5.

As it is seen that precipitates obtained from original desulfated WPA has diffraction peaks strontium sulfate with maximal 3.28, 2.96 Å and minimal 2.07, 2.02 and 2.05 Å. Whereas, diffraction lines with 3.51, 3.42, 3.16, 2.72 Å belong to gypsum (Fig. 3 and Table 3).

Diffraction peak with 7.86, 7.69, 7.47 Å and 2.28, 3.06, 3.01 Å characterizes phase gypsum dehydrate and gypsum hemihydrate which were precipitated when evaporation of the acid to 45.62; 50.01; 55.25 and 60.34 % P_2O_5. Remaining diffraction lines with traces are gangues such as iron and aluminium complex compounds revealed mainly amorphous state (Figs. 4-7).

Table 3. Results of XRD analysis of a sample of the original desulfated WPA.

№	dA	Corresponding substances
1	3.51	$CaSO_4$
2	3.42	$CaSO_4 \cdot 0{,}5H_2O$
3	3.28	$SrSO_4$
4	3.16	$CaSO_4$
5	2.96	$SrSO_4$
6	2.72	$CaSO_4 \cdot 2H_2O$
7	2.66	$(Al, Fe)PO_4 \cdot 2H_2O$
8	2.07	$SrSO_4$
9	2.05	$SrSO_4$
10	2.02	$SrSO_4$

CONCLUSIONS

Thus, the opportunity of desulfation of diluted WPA (18.95 % P_2O_5 and 2.96 % SO_3), from Kyzylkum phosphate, with strontium carbonate was created. As a result, SO_3 content in the acid was reduced from 2.96 to 0.10 % achieving a high level of desulfation of the phosphoric acid to be 97.34 %. Following the desulfated acid containing 20.53 % P_2O_5 was evaporated in order to obtain concentrated samples of the solutions ranged in 45.62; 50.01; 55.25 and 60.34 % P_2O_5.

The density and viscosity of desulfated and evaporated WPA were studied depending on their concentration and temperature. It has been established that at relatively low temperatures (30 - 50°C) and high concentrations (55 - 60 % P_2O_5), although WPA has an increased density and viscosity but it is in a fluid state.

The phase composition of the precipitates generated as a result settling concentrated samples of the acids were studied by XRD analyses. It was found that precipitate formed from diluted and desulfated WPA has diffraction lines belonging to strontium sulfate. At that time, the precipitates formed during concentrated WPA have mainly gypsum hemihydrate with maximal diffraction. Investigation carried out desulfation and evaporation industrial WPA can be reliable for production feed phosphate.

Authors' contributions: *The manuscript was written through contributions of all authors. All authorshave given approval to the final version of the manuscript. I.E.Kh. conducts a thorough purification of wet-process phosphoric acid derived from phosphorites of the Central Kyzylkum and contributes to the calculation of rheological properties. Sh.I.T. took part in research and analysis of scientific and technical literature. A.R.S. substantiated the idea of conducting research on the development of technology for purifying WPA and its practical application, and also took part in writing the article. Sh.S.N. created the conditions for conducting all laboratory experiments, the formulation of the goals and objectives of the research.*

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