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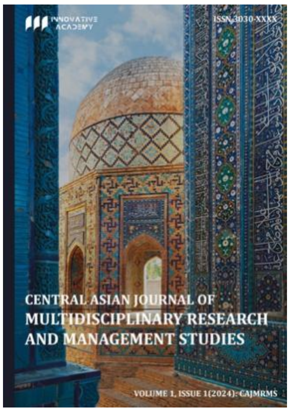

STUDY OF PHOSPHORIC ACID PURIFICATION DURING EXTRACTION AND ITS APPLICATION IN THE PRODUCTION OF HIGH-QUALITY NITROGEN–PHOSPHORUS FERTILIZERS STUDY OF PHOSPHORIC ACID PURIFICATION DURING EXTRACTION AND ITS APPLICATION IN THE PRODUCTION OF HIGH-QUALITY NITROGEN–PHOSPHORUS FERTILIZERS

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ABSTRACT

Objective: The purpose of this study is to investigate the purification of phosphoric acid derived from Central Kyzylkum phosphates using fluoride and sulfate additives during the extraction process. The work focuses on the removal of impurities such as iron, aluminum, calcium, magnesium, sulfate, and fluorine during the ammonium phosphate acid extraction stage, as well as the development of environmentally friendly, fully water-soluble ammonium phosphate fertilizers.

Methods: The research employed chemical and physicochemical techniques, including photocolorimetry, pH measurement, electron microscopy, along with statistical analysis of experimental results.

Results: Optimizing the phosphoric acid extraction process by simultaneous elimination of fluorine and sulfate impurities enabled effective removal of iron, aluminum, calcium, magnesium, fluorine, and other contaminants. The final product exhibited a high content of the main nutrient, P_2O_5.

Conclusion: The study demonstrates that co-treatment with calcium carbonate or washed calcined phosphate concentrate (WCPC) during extraction sludge processing allows simultaneous fluorine removal and desulfurization of wet-process phosphoric acid (WPPA) from Central Kyzylkum phosphates. Partial purification of phosphoric acid during extraction, followed by complete purification via ammoniation, produces high-quality, fully water-soluble ammonium phosphates. These nitrogen-

phosphorus fertilizers are suitable for drip irrigation systems and hydroponic cultivation

Introduction. Currently, the global demand for phosphate raw material is 190 million tonnes or 43 million tonnes of P2O5 per year. By 2030, demand for phosphate raw materials is projected to increase to 2 million tons. By 2050, the demand for raw materials will reach 220 million tonnes of phosphate, or 70 million tonnes of P2O5 [1].

As a result of large-scale concrete activities in the country, high results have been achieved in scientific research in the production of new types of phosphorus fertilizers based on local raw materials and in the provision of high-quality mineral fertilizers to agriculture.

In the third direction of the Strategy for Action of the Republic of Uzbekistan for 2017-2021 «... accelerated development of high-tech processing industries, primarily production of finished products with high value added based on deep processing of local raw materials. » Important tasks have been identified [2]. In this regard, it is important to create a technology for the production of high quality ammonium phosphates that meet the requirements of drip and hydroponic irrigation.

Therefore, the removal of fluorine and other additives from the product plays a special role in the process. Because fluorine compounds are very harmful to the environment. Research shows that fluorine not only affects plants but also causes many serious diseases in humans and other organisms.

The recovery and use of fluorine from phosphate feedstock solve two problems: increasing the production of fluorine compounds and preventing damage to the biosphere due to the release of fluorine into the environment. Some of the fluorine from the phosphate feedstock is extracted during extraction and the rest is removed by evaporation, granulation and drying. Phosphate extraction is also achieved by neutralizing phosphoric acid with non-fluoric containing components to reduce the relative fraction of fluorine in the product [3, 4, 5].

Many plants absorb large amounts of fluorine. For example, 1 kg of tea contains between 57 and 1,370 mg of fluorine and 4,500 mg [6, 7]. In this process, fluoride accumulates in the seeds of cotton, and most of it becomes cotton oil. Studies show that the content of fluoride in soil increases when fluorine is added to the soil, including mineral fertilizers [8]. In the presence of nitrogen phosphorus potassium fertilizers, the amount of fluorine absorbed by plants increases [9].

Phosphorus fertilizers are the main source of fluorine in soil. For example, apatite and phosphate contain, on average, 3.0% and 2.7% fluoride, respectively. The extracted phosphate acid (EPA) based on phosphate Central Kyzylkum contains about 1.2% fluoride. The method of deposition of acids from alkali metals in the form of silicon fluoride is not very effective as EPA contains almost very small amounts of acid-soluble silicon compounds [10].

When processing natural phosphates, the fluorine contained therein is distributed between the gas, liquid (EPA) and solid (phosphogypse) phases. According to the dihydrate scheme, 80-85% of the phosphoric acid in the raw material (apatite and phosphate) is converted into a hydrofluoric acid, and after processing into fertilizers.

Increasing the production of phosphorus, especially complex fertilizers, to fully meet the demand for agricultural fertilizers creates a risk of fluorine saturation of soils, plants and water bodies [11]. The negative effects of fluorine compounds entering the atmosphere and open water bodies and accumulating in living organisms and flora have been sufficiently studied [8, 9, 12].

Sulphates, chlorides, phosphates, carbonates and sodium and potassium hydroxides are used to precipitate EPA fluorides as compounds of silicon fluoride of insoluble alkali metals [13]. These methods are based on the chemical interaction of the above-mentioned salts with silica in EPA. Fluorine clearance is up to 90%.

The process of purifying EPAs based on Central Kyzylkum phosphate with alkali metal salts is well described in the literature. EPA is first purified from sulphates and then from fluorine. [14]

The authors have studied in detail the process of fluoridation of EPA based on phosphate Central Kyzylkum sulphate, dihydrophosphate, sodium metasilicate and have shown that the degree of fluoridation can be increased from 38-40% to 80-85% and EPA. Desorption technology developed [15, 16].

There are also materials for desulphurization of defluorinated EPAs by unprocessed raw materials of the Central Kyzylkum [17], carbonate and calcium oxide washed with enriched phosphate [18]. A patent has been obtained for obtaining nutrient sediments by purifying EPA of fluorine and sulphate compounds [19]. However, there are no materials for simultaneous removal of fluoride and sulphate from the extraction pulp when EPA is extracted from the phosphate of the Central Kyzylkum.

Methods. The simultaneous purification of EPA from fluorine and sulphate results in, first, an environmentally friendly product, second, a high contribution of useful substances to the product, third, some coverage of low-grade raw materials, fourth, fluorine - valuable raw materials, joins can be isolated in the process itself. Therefore, research on simultaneously purifying EPA from fluoride and sulphate is relevant.

In order to purify fluorine and other additives in the extracted phosphoric acid and to produce high-quality ammonium phosphates, purification processes for the extraction of phosphoric acid and the neutralization of purified acid with gaseous ammonia were studied.

In the composition for the study, in% by weight: P2O5 = 26,20; CO2 = 3,08; CaO = 57,64; MgO = 1,07; R2O3 = 0,79; SO3 = 2,18; F = 2,88; e.v. = 1.54 The washed phosphate concentrate of the Central Kyzylkum (WRPC) is used.

Deposition of fluorine and sulphate from Calcium Carbonate extraction sludge at 60-150% of the calcium oxide fluoride norm and 80-100% of the SO3 binding norm as calcium sulphate (limestone etc.)) and conducted by WRPC. The process of extraction of phosphoric acid with sulphuric acid was carried out in dihydrate mode, before filtration of the resultant extraction pulp fluorine and free sulphuric acid in it were deposited with calcium carbonate or WRPC.

Ammonia with gaseous ammonia up to pH = 3.8-5.5 to obtain relatively pure solutions of pre-purified acid from fluorine and sulphate.

Residual sulphates and fluorine are deposited with calcium ions as CaSO4 va CaF2 and iron and aluminium ions are deposited as FePO4 and AlPO4. The sediment is separated from the solution by decantation.

Results. The territory of the republic consists of calcium and magnesium carbonates that meet technological requirements of industrial production: calcite, limestone, dolomite and other local minerals are found in large quantities [20-22]. In the process of extraction of phosphoric acid with sulphuric acid, the use of local carbonate raw material in fluoridation and desulphurization of extraction suspension is very efficient. The results of the research are presented in Tables 1, 2 and 3, as well as in Figures 1, 2 and 3.

Discussions. Table 1 shows that when EPA is produced without adding calcium carbonate, 5.45% of all fluorine in phosphate is converted into a gas phase, 40.5% into phosphogypse and 54.05% of fluorine remains in EPA. The following reactions may occur when adding calcium carbonate to the extraction suspension:

$CaMg(CO3)2 + H2SO4 = CaSO4 + MgSO4 + CO2 + H2O$

$CaMg(CO3)2 + H3PO4 = Ca(H2PO4)2 + Mg(H2PO4)2 + CO2 + H2O$

$2Ca(H2PO4)2 + H2SO4 + 2HF = CaSO4 + CaF2 + 4H3PO4$

$2Mg(H2PO4)2 + H2SO4 + 2HF = MgSO4 + MgF2 + 4H3PO4$

Magnesium fluoride is relatively soluble in acids than calcium fluoride, reacting with strong acids, including sulphuric acid:

$MgF2 + H2SO4 = MgSO4 + 2HF$

magnesium sulfate and hydrogen fluoride.

This process is complex because magnesium monophosphate and sulphate, which are well soluble in EPA, can react with calcium carbonate to form calcium sulphate monophosphate. However, magnesium carbonate also reacts with phosphoric acid to form magnesium monophosphate. This is confirmed by the fact that the amount of magnesium in the EPA remains constant at 0.80-0.82% (Table 1).

With the addition of calcium carbonate and the formation of calcium fluoride, the amount of fluorine released into the gas phase decreases from 5.45% to 4.11%, indicating that the bulk of it is released at the beginning of the process. When CaCO3 is added to the decomposition process at a rate of 100-150%, the total conversion of fluorine to the gas phase and phosphogypsum is 86.6-90.9%. The fluoride content of EPA is 0.25-0.32%, which is 4.1-5.3 times less than in the absence of calcium carbonate.

table.1.

Effects of calcium carbonate norm on EPA chemical composition, gas phase and phosphogypso permeability of fluorine, as well as technological parameters of production of fluorinated and desulphurized EPA

Name the indicators	Calcium carbonate content for binding free fluorine in stoichiometry,%						
	-	60	80	100	120	140	150
Standard for calcium carbonate binding free H2SO4 to stoichiometry,%	-	80	100	100	100	100	100
EPA chemical composition, heavy. %							
P2O5	20,15	20,08	19,97	19,85	20,24	19,94	20,02
CaO	0,41	0,39	0,58	0,94	1,46	2,14	2,61

MgO	0,82	0,81	0,81	0,80	0,82	0,81	0,81
SO3	2,22	0,86	0,51	0,48	0,52	0,47	0,44
R2O3	0,61	0,61	0,60	0,60	0,59	0,60	0,59
F	1,32	0,69	0,51	0,32	0,27	0,25	0,25
particulate matter	0,25	0,32	0,28	0,19	0,24	0,17	0,16
Fluorine conversion rate, %							
Phosphogypsum	40,5	68,4	74,1	82,5	85,7	86,6	86,8
gaseous phase	5,4	5,3	5,1	4,1	4,2	4,0	4,1
All	45,9	73,7	79,2	86,6	89,9	90,6	90,9
Technological parameters of production of fluorinated and sulfated EPA							
Kp.. %	98,7	98,7	98,7	98,6	98,5	98,5	98,4
Ksep., %	95,8	95,7	95,5	95,5	95,6	95,5	95,5
Kwash., %	99,2	99,3	99,3	99,4	99,4	99,4	99,3
Kprod, %	95,0	95,0	94,8	94,9	95,0	94,9	94,8
The density of the porridge (ρ), g/sm3, 25°C	1,26	1,26	1,26	1,26	1,26	1,27	1,27
The stickiness of the porridge (η), spz, 25°C	3,31	3,48	3,58	3,61	3,66	3,70	3,72
Extraction porridge filtration rate, kg/m2·s	810,11	809,07	808,20	807,41	806,85	806,60	806,46
Size of phosphogypsum crystals, µm	100x24, 120x20, 220x20, more 560x80, 400x80, 360x28 and partially 100x16, 80x60, 60x20						

When the CaCO3 content in CaO is 100-120%, the EPA degree of fluorination is 75.4-80.5%, and the sulphating rate is 78.7-79.6% (figures 1 and 2). Figures show that adding more than 120% calcium carbonate to the amount of fluorine and sulphate in acid changes very little with the loss of fluoride and sulphate. The amount of fluorine remaining in EPA is 9.1-13.4% of the total phosphate. When the level of calcium carbonate in phosphate changes from 60 to 100%, the additional conversion of fluorine to phosphogypse is 27.9-42.0%. Increasing the level of calcium carbonate by 120-150% increases the conversion of fluorine to the solid phase by only 3.2-4.3%. Excess calcium carbonate from EPA is used in the deposition of fluorine to form calcium sulphate due to the presence of excess sulphuric acid and monocalcium phosphate due to its interaction with phosphoric acid. The data in Table 2 show that the SO3 content decreased from 2.22% to 0.44-0.52% and the calcium oxide content increased from 0.41% to 2.61%. The decomposition, separation, leaching and yield coefficients range from 60 to 150 per cent for fluorine binding, from 80 to 100 per cent for neutralizing the excess sulphuric acid for normal calcium carbonate 98.4 to 98.7 per cent and 95.5 to 95.8 per cent respectively. 99.2-99.4 per cent and 94.8-95.0 per cent.

table 2

Technological parameters of production of fluorinated and sulphated EPA with WRPC

Name the indicators	WRPC standard for binding free fluorine relative to stoichiometry,%						
	-	60	80	100	120	140	150
MOFK standard for binding free H2SO4 to stoichiometry,%	-	80	100	100	100	100	100
Chemical composition of EFK, heavy. %							
P2O5	20,15	20,87	21,28	21,13	21,71	21,81	21,89
CaO	0,41	1,04	1,17	1,26	1,55	1,94	2,15
MgO	0,82	0,85	0,86	0,85	0,88	0,89	0,88
SO3	2,22	0,90	0,61	0,47	0,48	0,48	0,48
R2O3	0,61	0,63	0,64	0,63	0,65	0,66	0,66
F	1,32	0,74	0,56	0,36	0,30	0,31	0,31
particulate matter	0,25	0,34	0,31	0,25	0,27	0,28	0,19

The filtration rate of phosphogypsum is relatively high, ranging from 806.46 to 809.07 kg/m2·s for dry matter.

Table 2 and figures 1 and 2 show the results of the fluorination and desulphurization of EPA at the Central Kyzylkum WRPC.

Unlike the use of calcium carbonate in acid cleaning, the use of UCFC increases the amount of P2O5 in EPA from 19.85-20.15% to 20.15-21.89%, the formation of calcium fluoride is 120-150% and the formation of calcium sulphate. The amount of CaO in the acid is 1.26-2.15%, the amount of MgO and SO3 is practically unchanged and the amount of fluorine in the acid is reduced to 0.31%.

Figures 1 and 2 present comparative data on fluoridation and desulphurization levels of calcium carbonate and WRPC. The figures show that with a normal calcium carbonate of 100-120% the fluorination level is 2.2-2.4% higher than that of WRPC and the sulphate level is 1.7-2.3% lower.

Based on the research, a material balance of purification of fluorine and sulphate in the production of extractive phosphate acid from washed phosphate concentrate of Central Kyzylkum was developed (Figure 3). Production of high-quality concentrated phosphorus fertilizers based on fluoride and non-sulphate EPA is ensured.

The technology of EPA production from fluorine and sulphates has been tested in the industrial production of OAO «Ammophos-Maxam» and a prototype of high-quality mineral fertilizer of ammophos is made from this acid. The difference between the cost of Class I and Class I is 94,000 sums per ton. The production of 100 thousand tons of fertilizer will produce an economic effect of 9.4 billion sum.

Figure 3. Material balance of production of EPA purified from fluorine and sulphate using carbonate feedstock

Adding calcium carbonate to the extraction solution solves three problems: partial discoloration and sulphate of extracted acid and calcium ion enrichment.

Ammonia with gaseous ammonia up to pH = 3.8-5.5 to obtain relatively pure solutions of pre-purified acid from fluorine and sulphate. Residual sulphates and fluorine are deposited with calcium ions as CaSO4 and CaF2, and iron and aluminium ions are deposited as FePO4 and AlPO4. The sediment is separated from the solution by decantation.

Table 3 shows the influence of ammonium level (pH) on the chemical composition of water-soluble ammonium phosphates.

table 3

Effects of ammonia level on the chemical composition of ammonium phosphates

pH	Chemical composition of ammonium phosphates, w. %				
	P2O5	N	F	SO3	H2O
3,8	56,91	11,54	0,11	0,24	1,43
4,5	58,63	12,83	0,03	0,08	2,54
5,2	59,95	13,71	0,01	0,04	2,18
5,3	58,81	14,03	0,01	0,03	2,27
5,5	57,48	14,21	0,01	0,02	1,34

When pH increased from 3.8% to 5.5, fluorine content decreased from 0.11% to 0.01% and sulphate from 0.24% to 0.02%, while P2O5 and nitrogen were 56.91% and 11.54% to 59.95% and 14.21%, respectively.

High-quality water-soluble ammonium phosphates can be obtained by partially purifying phosphoric acid during extraction and complete purification by extracting phosphoric acid with ammonia. The result is a simultaneous reduction of fluorine content from 0.11% to 0.01% and sulphates from 0.24% to 0.02% containing 56.91-59.95% P2O5 and 14.21-11.54%. A product with N was produced.

Conclusion. Studies have shown that simultaneous fluorination and desulphurization of EPA based on Central Kyzylkum phosphate can be achieved by adding calcium carbonate or WRPC to the extraction sludge. The optimal calcium carbonate and UFC levels are 100-120% for calcium fluoride and 100% for calcium sulphate. The sulphate content decreases from 2.22% to 0.44-0.48%, the fluorine content decreases from 1.32% to 0.25-0.30%, the rate of conversion of fluorine to the gas phase by extraction from 5.4% to 4%, and the rate of transition to phosphogypse increases from 40.5% to 82.5% to 85.7%. After purification from fluorine and sulphate, ammonium contains not more than 0.8-1.0% fluorine and more than 52.0% P2O5. The net profit from the conversion of refined EPA to ammonium is not less than 52.0% of P2O5 in the amount of 100,000 tonnes. Tonnes is 9.4 billion Sum.

High-quality water-soluble ammonium phosphates can be obtained from Central Kyzylkum phosphates by partial purification by phosphoric acid extraction and complete purification by the extraction of phosphoric acid with ammonia. The result is a simultaneous reduction of fluorine from 0.11% to 0.01% and sulfates from 0.24% to 0.02%, containing 56.91-59.95% P2O5 and 14.21-11.54%. A product with N was obtained. The products are integrated water-soluble nitrogen-phosphorus fertilizers that are used to grow crops by drip irrigation and hydroponics.

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