Removal of Fluorine during the Extraction of Phosphoric Acid

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Annotation: Task The article The defluorization and desulfurization of EPA in the process of its extraction by the introduction of sodium sulfate in an improved filtration mode in the presence of a chemical reagent sodium naphthenate, which has flocculation and biological active properties, which makes it possible to reduce fluorine emissions during the evaporation (concentration) of EPA, and to obtain defluorinated fertilizers have been studied.

Methods. One of the valuable results of these studies is that the introduction of a small amount of alkali metal petroleum (or oil refinery waste) into the extractor can improve the structure of calcium sulfate crystals (especially when desulfurizing the extraction pulp by introducing an additional amount of phosphate), leading to improved filtration of the extraction pulp in 1, 5 - 1.7 times and, accordingly, increase the productivity of the extraction shop.

Results In order to reduce the rate of the precipitant and increase the effect of defluorization at the same time, as well as improve the crystallization (i.e., the filterability of the pulp) of calcium sulfate (by reducing the silicofluoride ions in the solution) and the precipitation of silicofluorides on the "pad" of phosphogypsum during the decomposition of Karatauphosphorites, the extractor was introduced sodium naphthenate (first used for this purpose), which can affect crystallization as surface active agents. To improve the process of defluorination of EPA and crystallization of calcium sulfate, 10% sodium sulfate was replaced with sodium naphthenate (40% aqueous solution). Sodium naphthenate when interacting with hydrofluorosilicic acid forms large crystals of sodium silicofluoride and naphthenic acid. Therefore, the aim of these studies was to defluorinate EPA with a simultaneous improvement in the filterability of the extraction pulp.

Conclusion. The process of defluorination with simultaneous desulfurization using sodium sulfate and with partial replacement of it with sodium naphthenate during the extraction of phosphoric acid from phosphates has been studied and obtaining defluorinated fertilizers.

Keywords: Phosphorite, Karatau, sodium naphthenate, sodium silicofluoride, naphthenic acid, phosphogypsum, fluorine, defluorination of EPA, sodium sulfate, desulfurization, silicon hexafluoride, calcium sulfate, extraction pulp.

Introduction

Phosphate ores contain, in addition to phosphorus, a large amount of minerals in the form of impurities. Thus, Karatauphosphorites contain 24-26% P2O5, 0.5 to 5% MgO, 35-42% CaO, about 3% R2O3, 2 to 3% F, 3-10% insoluble residue. When receiving mineral fertilizers in the process of acid processing of phosphates, the main part of fluorine, remaining in fertilizers, is lost irretrievably. Magnesium, aluminum and iron complicate the processing of

phosphate raw materials and reduce the assimilation of phosphorus in fertilizers, binding it into fluorophosphate complexes.

From the literature it follows that it is necessary to develop scientific research on the development of effective technological schemes for processing poor phosphate raw materials, solving the problem of enriching phosphorite ores of various deposits, improving the instrumental and technological schemes for the production of phosphorus-containing fertilizers in the direction of maximum use of magnesium, aluminum, fluorine and ensuring minimum emissions of harmful gases into the atmosphere.

The main source of environmental pollution in the production of mineral fertilizers is fluoride compounds released at all stages of processing of phosphate raw materials. Fluorine is a biosphere pollutant and plant poisoning component. Its presence in ammophos leads to a deterioration in its agrochemical properties and an irreversible loss of deficient fluorine for the national economy. Waste fluoride gases formed during the processing of natural phosphates into fertilizers are the main source of fluorine for the production of its compounds. The large scale of production of phosphate fertilizers provides the possibility of the associated production of significant amounts of fluoride salts. Another source of fluorine is natural calcium fluoride - fluorspar or fluorite CaF2.

Currently, the scale of consumption of fluorine-containing ores by various sectors of the national economy is sharply increasing, which leads to a shortage of fluorine-inorganic products. Fluorine resources in the phosphate fertilizer industry, which can practically be utilized, in 1 year (calculated for 1975) reach 167 thousand tons (72 thousand tons in the phosphate fertilizer industry, 95 thousand tons from slags in the production of phosphorus).

Methods.

However, for the practical implementation of this possibility, the methods of utilization of fluorine and unorganized experimental-industrial and large-scale production of fluorine-containing products have not yet been sufficiently studied. Therefore, the purpose of the presented work is to study the possibility of fluorine extraction at some stages of the process of processing Karatauphosphorites for fertilizers, leading to an increase in the quality of the products obtained.

From literary [1-12]sources it is known that the average content of fluorine in the earth's crust is about 10 billion tons, in the soil the content of fluorine is about 0.02%, in the waters of rivers - 0.00002%, in the waters of the oceans - 0.0001%... Fluorine is most widespread in phosphorites and apatites. The fluorine content in apatites ranges from 1.3 to 3.9%. This means that already now it is possible to obtain 700 thousand tons of fluorine with 50% of its extraction from apatite. In other minerals, the fluorine content is no more than 1%. The main types of raw materials for the industrial production of fluorine and its compounds are fluorine-containing gases, which are wastes from the production of phosphorus fertilizers, and fluorspar CaF2.

When receiving phosphoric fertilizers, fluorine, depending on the technology used and the raw materials used, is distributed between phosphogypsum, extraction phosphoric acid and the gas phase in different ways. The distribution of fluorine in the production of EPA by the dihydrate method is influenced by the parameters of the technological regime and the content of alkali metals in the feedstock. Silicon tetrafluoride, depending on the conditions, either evolves in a gaseous state or reacts with water to form H2SiF6, part of H2SiF6, forms magnesium, potassium and sodium silicofluorides.

In the production of EPA by the hemihydrate method, 50% of the fluorine is removed into the

gas phase. In the production of defluorinated phosphates, about 97% of fluorine is contained in the exhaust gases in the form of HF; during the electrothermal sublimation of phosphorus, about 80% of the fluorine contained in the feed passes into slag, which is a waste product. The main sources of fluoride pollution in the environment are factories producing phosphate fertilizers and the fertilizers themselves.

Currently, there are no standards for the content of fluoride in fertilizers. 1 ton of superphosphate carries on the field 9-10 Kgfluorine; 1 ton double superphosphate 15-17 kgfluorine; 1 ton of ammophos 40-50 Kgfluorine, i.e. from 50 to 80% of fluorine from its content in phosphate raw materials is emitted to the fields, and this amount of fluorine in fertilizers is in a sufficiently soluble form, absorbed by plants through vegetables, fruits, grain enters the human body and animals. It is washed out by waters, getting into ponds, rivers, lakes, and hence into drinking water.

In order to reduce the rate of the precipitant and thereby increase the effect of defluorization, as well as improve the crystallization (i.e., the filterability of the pulp) of calcium sulfate (by reducing the silicofluoride ions in the solution) and the precipitation of silicofluorides on the "pad" of phosphogypsum in the process of decomposition of Karatauphosphorites, the extractor was introduced sodium naphthenate (first time used for this purpose), which can affect crystallization as surface active agents[13-20]...

Discussion.

In experiments on defluorination of EPA during its extraction, Karatauphosphorites of the following composition were used, in mass. %: P_2O_5 = 25.5, CaO = 38.0; MgO = 1.6; R_2O_3 = 2.69; Fe_2O_3 = 0.93; Al_2O_3 = 1.76; F = 3.71; CO_2 = 8.26; n.d. = 21.05 and H_2O = 0.21.

It has been found that the optimal rate of the precipitant is 120% to bind all the fluorine contained in the feed. At the same time, the degree of defluorination of EPA is 76.6%. Production EPA contains 0.4% fluorine. This process has a certain effect on the technological parameters of the extraction of EPA from Karatauphosphorites. The coefficients of extraction and yield of production acid are reduced by 0.4-0.1%. It is advisable to maintain the content of free sulfuric acid in the liquid phase at 3.0-3.5 g / 100 ml in terms of SO_3, which will improve the filterability of the suspension.

The decrease in the degree of extraction of phosphoric acid can be explained by the sloughing of phosphorite particles by fine-crystalline silicofluorides of alkali metals. In order to reduce the ballast in the production EPA for defluorization of its optimal rate of the precipitant, 120% amount of sodium sulfate was taken to bind fluorine in the form of silicon hexofluoride.

To improve the process of defluorination of EPA and crystallization of calcium sulfate, 10% sodium sulfate was replaced with sodium naphthenate (40% aqueous solution).

Sodium naphthenate interacting with hydrofluorosilicic acid forms large crystals of sodium silicofluoride and naphthenic acid. The conditions of the experiment are similar to the above, but with a certain distinctive character of the variants: precipitation of fluorides by the introduction of sodium sulfate (120% of stoichiometry) without desulfurization of the extraction pulp and with desulfurization, i.e. the introduction at the end of the process (in the second reactor) of phosphorite to bind free sulfate ions to calcium sulfate; precipitation of fluorides by introducing sodium sulfate (110% of stoichiometry) and sodium naphthenate (10% of stoichiometry) was carried out in the same way in the above two variants. But in the process of desulfurization, the additional crystals formed do not have time to grow, and lead to an increase in the density of the phosphogypsum layer (cake) on the filter and to clogging of the filter cloth.

The filtration rate decreases and phosphogypsum is poorly washed.

Therefore, the aim of these studies was to defluorinate EPA with a simultaneous improvement in the filterability of the extraction pulp. Our results (Table 1) show that the filtration rate τ_{cm}^{ϕ} is equal on average to 25 seconds, and $\tau_{cm}^{H_2O}$ is equal to 11 sec. (without desulfurization of the pulp) when sodium sulfate is introduced into the reactor. but during defluorization in parallel with desulfurization, the filtration rate deteriorates, as it becomes on average τ_{cm}^{ϕ} 35 sec., And $\tau_{cm}^{H_2O}$ is equal to 15 sec. (acidviscosityincreases). The introduction of a small amount of sodium naphthenate leads to the elimination of the above undesirable properties of the pulp. Homogeneous crystals of calcium sulfate dihydrate with a size of 320 x 40 microns and 400 x 60 (small enough) are formed and the filterability of the pulp with and without desulfurization (Table 2) has almost the same value τ_{cm}^{ϕ} on average is 15 seconds, and $\tau_{cm}^{H_2O}$ is equal to 7.5 seconds, that is, it accelerates by 1.5 - 1.7 times (the viscosity of the acid decreases). This, in turn, leads to an increase in the productivity of EPA production units by 1.5 - 1.7 times and the purification of EPA from fluorine and sulfuric acid by the ammonization of the obtained phosphoric acids (at a temperature of 900C to a pH of about five and drying the ammophos pulp at 1050C to constant weight, received ammophos (Table 3) with different content of fluorine (0.95–1.0%), sulfate ion (2%) and ammonium naphthenate (0.8–1.0%), depending on the method of obtaining EPA ...

Results of a baseline defluorination experiment using only sodium sulfate (stoichiometric ratio for fluorine 120%)

Experience number								Pulp filterability, sec		Phosphogypsum composition, etc.						
	P2O5,%	SO3, g / 100 ml	d, g / cm 3	η, cps at 800 C	CaO, %	MgO,%	F, %	τ_{stf}	τ_{stH2O}	H2O %	P2O5 total %	P2O5v.r%	Kizv, %	Kotm,%	Kvykh,%	F, %
Without desulfurization																
one	24.9	3.36	1.317	1.87	0.50	1.37	0.64	29.03	10.6	48.3	0.99	0.22	96.7	99.0	95.7	1.38
2	22.4	2.32	1.308	1.76	0.48	1.44	0.35	21.8	12.6	26.5	0.80	0.24	97.0	98.8	95.8	
3	23.9	1.8	1,301	1.66	0.67	1.34	0.52	27.4	11.33	39.0	0.98	0.33	97.1	98.5	95.7	1.58
4	23,7	2.36	1.295	1.91	0.55	1.32	0.37	26.8	10.9	33.3	0.82	0.32	97.7	98.6	98.6	
5	21.0	2.0	1.281	1.77	0.40	1.29	0.41	25.8	12.4	36.6	0.98	0.44	98.1	99.0	97.1	
With desulfurization																
6	24.98	1.6	1,318	2.23	0.62	1.39	0.51	45.5	16.9	39.8	0.79	0.27	97.7	98.8	96.6	1.95
7	24.51	1.45	1,313	2.47	0.77	1.62	0.48	31.2	15.7	41.2	1,2	0.48	96.8	97.8	94.8	2.04

8	24.07	1.48	1,318	2.13	0.38	1.38	0.44	30.2	14.5	42.2	1.49	0.53	95.8	97.6	93.6	
nine	24.8	1.23	1.32	2.43	0.34	1.24	0.43	32.6	13.8	37.1	2.01	0.40	93.0	98.3	91.4	
ten	22.03	1.20	1.27	1.75	0.43	1.24	0.44	36.1	16.5	26.7	1.17	0.62	97.65	97.3	95.0	

Experimental results using a mixture of sodium sulfate and sodium naphthenate 120% of the fluorine stoichiometry

(10% Na as sodium naphthenate without and with desulfurization).

No. experience								Pulpfilterability, sec		Phosphogypsumcomposition, etc.						
	P2O5,%	SO3, g / 100 ml	d, g / cm3	η, cps at 800 C	CaO, %	MgO, %	F, %	τ_{stf}	τ_{stH2O}	H2O %	P2O5 total %	P2O5 wp %	Kizv, %	Kotm,%	Kvykh,%	F, %
Withoutdesulfurization																
one	22.08	3.50	1.303	1.60	0.53	1.23	0.54	18.50	8.01	21.0	0.97	0.56	98.22	97.6	96.26	1.66
2	21.40	2.76	1,290	1.62	0.57	1.52	0.50	18.50	8.36	27.1	0.87	0.60	98.78	96.50	95.37	1.64
3	22.01	3.46	1.291	1.64	0.38	1.29	0.54	16.13	7.70	41.6	0.91	0.31	97.32	98.60	96.02	1.65
4	20.51	3.70	1.285	1.54	0.45	1.31	0.56	14.45	6.70	35,7	0.69	0.44	98.91	98.07	97.0	1.66
5	21.14	3.80	1,290	1.58	0.39	1.29	0.46	17.07	7.59	33.3	1.27	1.10	-	-	-	1.83
6	21.54	4.12	1.289	1.57	0.39	1.21	-	14.30	6.90	30.3	1.76	1.01	96.73	99.55	96.29	1.79
7	22.35	2.90	1.286	1.59	0.58	1.35	0.50	15.36	6.20	32.1	0.93	0.57	98.43	97.48	95.94	1.80
Withdesulfurization																
8	23.70	1.54	1.288	1.67	0.64	1.47	0.50	15.89	7.50	44.4	1.84	0.58	94.62	97.33	92.02	1.82
nine	22.91	1.80	1,284	1.56	0.71	1.41	0.56	19.05	7.75	50.0	1.75	0.69	65.38	96.85	92.37	2.11
ten	22.72	1.55	1.287	1.83	0.51	1.33	0.54	19.90	7.33	41.4	1.07	0.74	98.57	96.73	95.73	2.06

Homogeneous crystals 320x40, 400x60 microns, few small.

Components	PhosphoritesofKaratau	EPA content,%		
		Regulardihydrate (acid -1)	with the introduction of 120% Na2SO4 (acid-2)	with the introduction of 110% Na2SO4 and R-COONa with desulfurization (acid-3)
P2O5	25.5	22.87	21,7	24.2
SO3	-	2.93	1.86	0.84
F	3.71	1.76	0.43	0.44
CaO	38.0	0.19	0.25	0.46
MgO	1.6	1.22	1.44	1.46
R2O3	2.69	1.67	1.68	1.44
Fe2O3	0.93	1.03	1.06	1.07
Al2O3	1.76	0.64	0.62	0.34
d, g / cm3	-	1.292	1.299	1.295
η, cpsat 80oC	-	1.51	1.67	1.60
CO2	8.26	-	-	-
but.	21.05	-	-	-
H2O	0.21	-	-	-
τ_{cm}^{ϕ}, sec.	-	18.9	29.03	14.5
$\tau_{cm}^{H_2O}$, sec.	-	9,7	12.6	6.2
R-COOH	-	-	-	0,4

However, precipitated fluorine is lost irretrievably with phosphogypsum.

One of the valuable results of these studies is that the introduction of a small amount of alkali metal petroleum (or oil refinery waste) into the extractor can improve the structure of calcium sulfate crystals (especially when desulfurizing the extraction pulp by introducing an additional amount of phosphate), leading to improved filtration of the extraction pulp in 1, 5 - 1.7 times and, accordingly, increase the productivity of the extraction shop.

Of interest is the partial release of fluorine into the gas phase during the decomposition of phosphates and due to the heat of mixing sulfuric and circulating phosphoric acids with simultaneous defluorization and cooling of the system by suction of fluorine gases (vapor-gas-air mixtures).

In extraction systems, where the decomposition of phosphate is carried out by the internal circulation of the slurry, there are no circulation centrifugal pumps, which simplifies the design of the pipelines. At the same time, there is no need for a vacuum evaporator and the cost of pumping large amounts of circulating slurry to barometric altitude (more10 m). In this case, the cooling of the pulp can be carried out by blowing the surface of the pulp with air (through special tubes in the extractor lid) supplied by a fan that simultaneously sucks fluoride gases. The Swenson Reactor is also an internal circulation extractor, and it is directly connected to the vacuum system and combines the functions of an extractor and an evaporative cooler. It is believed that with this scheme, costs are reduced by 10-12%. The

advantages of the reactor are the reduction of the residence time of the reagents in the reaction zone, optimization of the temperature regime with a deviation of no more than 0.30C, the release of gypsum crystals, uniform in size and shape, which facilitates and accelerates their filtration.

Conclusion. Thus, the process of defluorination with simultaneous desulfurization using sodium sulfate and with partial replacement of it with sodium naphthenate during the extraction of phosphoric acid from phosphates has been studied.and obtaining defluorinated fertilizers.

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