

METHODS OF PURIFYING EXTRACTION PHOSPHORIC ACID

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Abstract

Phosphoric acid is one of the widely used reagents in the world. Its global annual production capacity is 48.5 mln. P₂O₅ is tons. Phosphoric acid is used in the food industry, poultry, livestock, fishery, metallurgy and several other fields, phosphoric acid is mainly used to obtain mineral fertilizers. On its basis, double superphosphate, nutrient mono- and dicalcium phosphate, mono- and diammonium phosphate, nitroammophos, etc. are produced.

Introduction

The method of defluoridation of EPA during the concentration increase of extractive phosphoric acid (EPA) by evaporation is the main one on an industrial scale, in the first stage of evaporation, it is possible to reach the degree of fluorination up to 65-70% at a concentration of 50-54% P₂O₅. The second stage, when the acid is increased to 63-66% P₂O₅, the degree of defluoridation reaches 85-90%. Fluorine is released as silicon tetrafluoride and hydrogen fluoride. When 45-47% P₂O₅ EPA is evaporated, the concentration of fluorine in the acid decreases, and the rate of release of fluoride compounds slows down when the acid concentration increases. This is due to the presence of the rest of the fluorine in the form of complex compounds with aluminum and iron.

Phosphoric acid organic solvents extraction purification; ion exchange; recrystallization; It is cleaned using precipitation and electrochemical methods [1]:

Extractive purification in organic solvents. A number of works are based on purification of EPA from impurities. One of the main methods of purification of EPA is extraction purification with organic solvents [1]. This process is recognized as the most promising technology by foreign manufacturers due to its high kinetic properties, ease of separation, high selectivity, and the possibility of good solvent regeneration.

The table below shows some of the physico-chemical properties of OSs in the "ch" category used for deep purification of desulfurized EPAs with various organic solvents (OSs):



The name of the solvent	Chemical formula of solvents (molecular mass)	Temperature, °C			Lower limit of explosive mixtures, vol.%
		freezing	boiling	evaporation	
Propyl alcohol	C ₃ H ₇ OH (60,09)	-126,1	97,2	22	2,2±0,1
Isopropyl alcohol	C ₃ H ₈ O (60,09)	-89,5	82,4	11,7	2±1
Isobutyl alcohol	C ₄ H ₁₀ O (74,12)	-108	108,4	22	1,68±0,1
Ethyl alcohol	C ₂ H ₅ OH (46,07)	-114,3	78,4	13	3,3±0,1
Acetone (dimethyl ketone)	C ₃ H ₆ O (58,08)	-95,4	56,2	-20	2,55±0,1
Acetic acid	CH ₃ COOH (60,05)	16,75	118,1	103	4±0,1

Extraction experiments were carried out in a flask equipped with a sieve. H₃PO₄ of desulfurized EPA: OS= 1:3 ratio and mixed with OSs at a temperature of 25 °C. The resulting precipitate is passed through a layer of "white" filter tape paper and the precipitate is dried at 100-105 °C. The dried precipitate was analyzed for the above components. The results of the analysis are presented in Table 1 below.

1 table Chemical composition of precipitates formed during the purification of desulfurized extraction phosphoric acid with various organic solvents ("clean" type)

Name of solvents	Composition of components in dry mass. weight.%						
	P ₂ O ₅	CaO	MgO	Fe ₂ O ₃	Al ₂ O ₃	SO ₃	F
Propyl alcohol	42,71	8,27	8,84	2,36	3,57	5,19	7,12
Isopropyl alcohol	50,14	8,87	8,91	2,73	4,30	4,98	7,50
Isobutyl alcohol	48,25	8,92	9,12	2,52	3,62	5,02	7,32
Ethyl alcohol	23,45	12,38	10,95	3,67	6,14	6,18	11,02
(dimethyl ketone)	34,22	9,58	9,95	3,26	5,12	5,10	6,25
Acetic acid	43,09	9,79	5,17	5,17	7,12	3,24	5,45

It can be seen from the table that when various OS are used three times more for the purification of EPA, the composition of the precipitate is as follows: from 34.22 to 50.14% P₂O₅, from 8.27 to 9.58% CaO, from 8.84 to 9.95% MgO, 2.36 to 3.26% Fe₂O₃, 3.57 to 5.12% Al₂O₃, 4.98 to 5.19% SO₃, and 6.25 to 7.50% F found. Also, this sediment can be used as a phosphorus-magnesium fertilizer.

The sedimentation levels of the components in EPA were also calculated from the components in the acid (Table 2).

2 tables Sedimentation coefficient of components during purification of desulfurized extractive phosphonic acid with organic solvents

Name of solvents	Sinking coefficient. weight. %						
	P ₂ O ₅	CaO	MgO	Fe ₂ O ₃	Al ₂ O ₃	SO ₃	F
Propyl alcohol	23,43	87,68	96,66	76,67	77,82	87,09	64,90
Isopropyl alcohol	27,54	94,19	97,58	88,83	93,88	83,70	68,47
Isobutyl alcohol	1,98	7,08	7,47	6,13	5,91	6,31	4,99
Ethyl alcohol	7,97	81,67	67,95	79,56	89,09	74,49	61,12
Acetone (dimethyl ketone)	16,56	97,89	95,65	91,08	96,89	86,25	60,10
Acetic acid	10,12	69,84	58,23	63,22	63,49	60,29	58,23

The precipitation coefficient (%) of the components using different OSs was determined, depending on the amount of components in the precipitate in relation to the acid content when using isobutyl alcohol (7.08% CaO, 7.47% MgO, 6.13% Fe₂O₃, 5.91% Al₂O₃, 6.31% SO₃ and 4.99% F) compared to other organic solvents, it was found that the components of EPA are less precipitated. Isopropyl alcohol is considered to be the best precipitant for magnesium oxide and fluorine (97.58 and 68.47%), but a significant loss of phosphorus (27.54% P₂O₅) is observed with precipitation. When using propyl alcohol - 87.68% CaO, 96.66% MgO, 76.67% Fe₂O₃, 77.82% Al₂O₃, 87.09% SO₃ and 64.9% F, and the level of phosphorus loss - 23, 43% Forms P₂O₅. When ethyl alcohol was used to clean EFK from additives, the level of precipitation of components was respectively as follows: CaO; MgO; Fe₂O₃; Al₂O₃; SO₃ and F 81.67; 67.95; 79.56; 89.09; 74.49 and 61.12. When acetic acid is used, the degree of deposition of components is as follows: CaO; MgO; Fe₂O₃; Al₂O₃; SO₃ and F 69.84; 58.23; 63.22; 63.49; 60.29 and 58.23.

Acetone can be recommended as an effective precipitant for cleaning phosphoric acid, because when acetone is used, the precipitation coefficient of the components in the acid is as follows: CaO - 97.89%, MgO - 95.65%, Fe₂O₃ - 91.08%, Al₂O₃ - 96.89%, SO₃ - 86.25% and F 60.10%[2].

Ion exchange. One of the effective methods of cleaning EFK from fluorine, calcium, magnesium, iron and other additives is the ion exchange method. The choice of coal adsorbents depends on the state of impurities in phosphoric acid solutions. Activated carbons work by a mechanism close to ion exchange in cleaning EPA. Modification or activation of carbon adsorbents results in the production of "oxidized" carbons suitable for cation removal. To remove anions, sorbents are treated with alkaline solutions. Mechanical chemical activation of coal significantly improves the porosity of sorbents and the number of active sites.

Recrystallization. This method is one of the oldest methods of purifying phosphoric acid by crystallization when it is cooled to a certain low temperature, but the main disadvantage of this method is that it consumes a lot of energy, so this method is hardly used nowadays.



Drowning. Precipitation methods include defluoridation, desulfation, precipitation of heavy metal oxides. For the purification of EPA, low-soluble potassium or sodium silicate fluorides, calcium, barium or strontium sulfates, heavy metal oxides are based on precipitation in the form of phosphates and calcium fluorides. The advantage of the precipitation method of fluorine removal is the simplicity and low cost of the process, with a sufficiently high level of fluorine removal. As a reagent, sodium or potassium carbonate is used to defluorinate metasilicates. EPA desulfation processes are based on precipitation of sulfate ions in the form of poorly soluble sulfate compounds - calcium, barium, strontium. K_2CO_3 , NH_3 , NH_4OH , NH_4HCO_3 , $NaOH$, Na_2CO_3 , $NaHCO_3$ are used for partial neutralization in the process of cleaning EFK from heavy metal oxides. In this process, a solid precipitate is formed in the form of insoluble compounds of fluorine, aluminum and iron complex salt. For example, a precipitate is formed when neutralized with ammonia at a pH of 1 to 1.6. At pH = 2.2-2.5 $(Fe,Al)NH_4H_8(PO_4) \cdot 6H_2O$ salt $(FC,A1)NH_4(HPO_4) \cdot 0.5H_2O$ turns into an amorphous compound. At pH = 2.35-2.40, almost all iron and aluminum precipitates in the form of a complex compound with ammonium.

Electrochemical method. This method is mainly used for industrial enterprises. Electrochemical methods are considered new and developing directions. The essence of this method and technology is based on continuous current passing between the electrode plates.

Electrolysis is the use of electrolysis for the purification or recovery of heavy metals, this method can remove various methane ions at the same time, but this method consumes a lot of electricity and expensive steel, so this method is not used.

Among the mentioned methods, one of the most technologically optimal options in our conditions is deposition using organic solvents. Vinegar, formic acids, acetone, ethyl alcohol, tributyl phosphate, etc. can be used as organic reagents. The advantage of using these is that they are easily regenerated and can be reused several times in the process.

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