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Study of the Process of Recycling Sodium-Phosphate Sludge

Khujamberdiev Sherzod, Mirzakulov Kholtura, Rakhmanov Begzod, Karshiev Nurali

Doctoral student, Department of Chemical Technology of Inorganic Substances, Tashkent Institute of Chemical Technology, Tashkent, Uzbekistan

Professor, Department of Chemical Technology of Inorganic Substances, Tashkent Institute of Chemical Technology, Tashkent, Uzbekistan

Teacher, General Chemistry and Chemical Technology Substances, Denau Institute of Entrepreneurship and Pedagogy, Denau, Uzbekistan

Teacher, General Chemistry and Chemical Technology Substances, Denau Institute of Entrepreneurship and Pedagogy, Denau, Uzbekistan

ABSTRACT: The research results on the recycling of sodium-phosphate sludge formed during the neutralization of extraction phosphoric acid with sodium carbonate in the presence of sodium hydroxide are shown. By neutralizing sodium-phosphate sludge with a sodium hydroxide solution to pH=11, it was demonstrated that 78-80% of the phosphorus oxide contained therein can be extracted, making the solution suitable for the production of sodium orthophosphate.

I. INTRODUCTION

One of the urgent issues in the world today is to fully meet the needs of industrial sectors for phosphate salts. Due to the decreasing rich raw material sources of phosphorus products on a global scale, it is important to develop efficient technologies for processing phosphorus sludges generated during production.

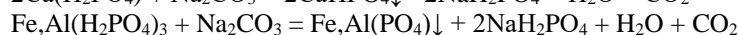
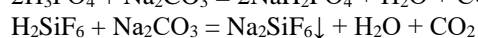
Phosphoric acid, obtained from various phosphorus-containing raw materials, is required to obtain phosphate salts. The production of phosphoric acid and phosphorus fertilizers is increasing every year. Specifically, in 2014, the production reached 42.7 million tons, and by 2018, this figure had increased to 46.6 million tons [1].

As the demand for phosphate salts increases, the demand for phosphorus raw materials is also growing, and consequently, their consumption is increasing accordingly. The decrease in high-phosphorus content ores and the increasing use of lower-grade phosphorites due to technological advancements, in turn, require solving a number of problems [2-6].

Sodium orthophosphates are widely used in various industries and public sectors, and in some cases, they cannot be replaced by other substances while maintaining technical and economic indicators [7, 8].

II. RESEARCH METHODS

Purified phosphoric acid is neutralized with calcined soda to a pH environment of 4.5. In this environment, the formation of a monosodium phosphate solution is observed. Additionally, calcium, magnesium, aluminum, iron, and fluorine compounds present in extraction phosphoric acid are observed to form precipitates. The neutralization process in the first stage was found to be based on the following physicochemical reactions:



After the neutralization process, the sodium-phosphate sludge filtered and separated consists of phosphate precipitates of sodium, calcium, aluminum, iron, and remaining monosodium phosphates. The composition of sodium-phosphate sludge

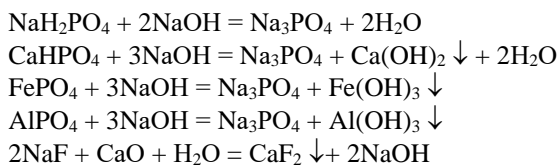
neutralized at pH=4.5 contains, by weight: Na₂O - 11.30%; P₂O₅ - 25.26%; SO₃ - 0.93%; CaO - 7.31%; MgO - 0.58%; Al₂O₃ - 2.65%; Fe₂O₃ - 2.42%; F - 1.21%.

The technology for obtaining sodium phosphates involves processing sodium-phosphate sludge with sodium hydroxide in an aqueous medium at a temperature of 80°C, filtering the resulting mixture, and converting the separated sodium phosphate solution into commercial trisodium phosphate. Sodium hydroxide solution was used to carry out the experiment. The mixture of sodium hydroxide solution at 80°C and the selected sodium-phosphate sludge as the research object was maintained in an environment with a pH of 11.0-11.5.

III. EXPERIMENTAL RESULTS

The content of phosphorus oxide in the sodium-phosphate sludge is approximately 30-40%. Due to the high content of sodium, iron, aluminum, and other elements in the sludge, it cannot be used as a mineral fertilizer or for other targeted purposes. It is known that currently, resource-saving, waste-free technologies in production are being implemented globally, including in our country, as part of various state programs. The processing of sodium-phosphate sludges is distinguished by the complexity of the processing process and the limited usability of the obtained products.

Taking these problems into account, research was conducted on the processes of processing sodium-phosphate sludges with sodium hydroxide solution. The process of processing sodium-phosphate sludges with sodium hydroxide involves the following reactions:



The effect of sodium hydroxide norms, process temperatures, and durations on the chemical composition of liquid phases has been studied. The duration of the process ranged from 10 to 50 minutes, and sodium hydroxide norms for the formation of sodium phosphate were carried out at 60-80°C with a stoichiometric ratio of 50 to 75%. In the additional neutralization process of sodium phosphate solution, the separation of P₂O₅ in the sludge reaches a maximum value when the NaOH norm is 65-70%. Increasing the process temperature from 60 to 80°C almost does not affect the chemical composition of the liquid phase.

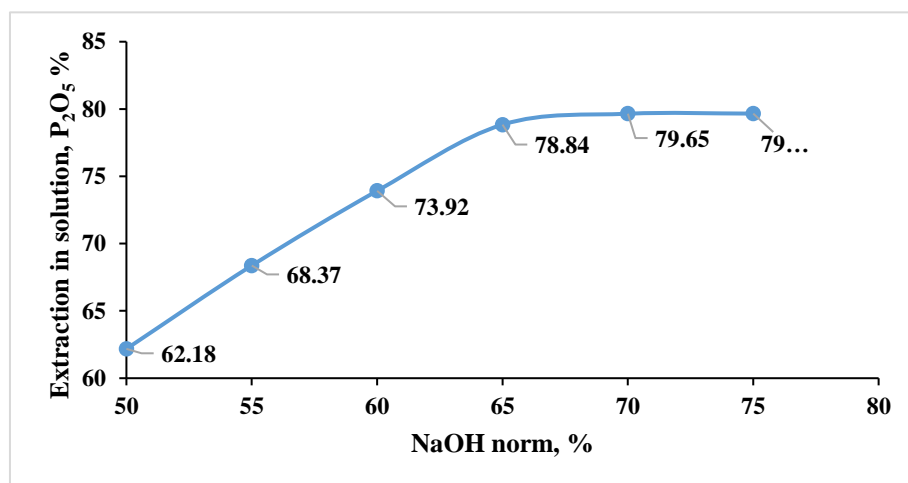


Fig. 1. The effect of NaOH norms on the separation of P₂O₅ in sodium-phosphate sludge.

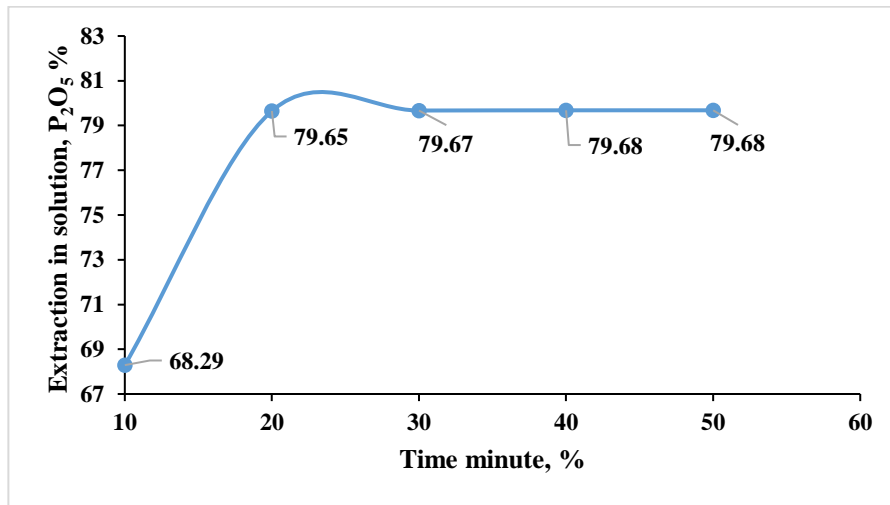


Fig. 2. The effect of time on the separation of P₂O₅ in sodium-phosphate sludge.

Figure 2 shows the results of processing sodium phosphate sludge with sodium hydroxide solution, specifically the effect of sodium hydroxide norms on the separation degree of P₂O₅ in the sludge during the neutralization process to trisodium phosphate. The duration of the process varied from 10 to 50 minutes, with a stoichiometric norm of sodium hydroxide being 70%.

As can be seen from Figure 2, the processing process is almost complete within 20 minutes, and the separation degree of P₂O₅ in sodium-phosphate sludge reaches a maximum of 79.65%. The quantitative ratio of sodium oxide and phosphorus oxide in the solution corresponds to trisodium phosphate.

The insoluble residue separated from the solution was analyzed using X-ray diffraction and scanning electron microscopy methods (Figures 3 and 4).

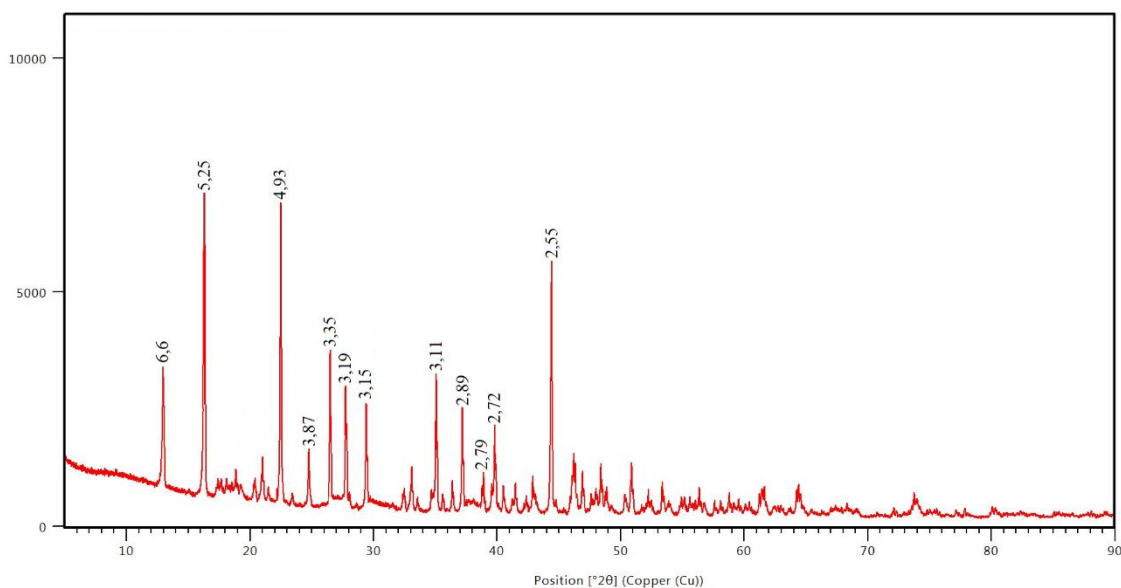
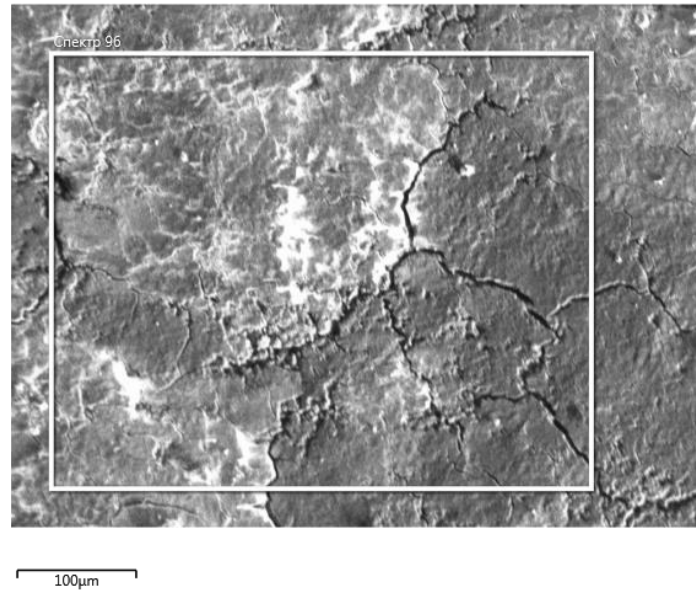


Fig. 3. X-ray analysis of the solid phase obtained from processing the sludge

The analysis of the results of the X-ray studies conducted was examined. The main peak indicators obtained were found at 4.93; 6.6; 2.89 Å - $\text{Ca}_3(\text{PO}_4)_2$, 3.11 Å - $\text{Ca}(\text{OH})_2$, 5.25 Å - $\text{Fe}(\text{OH})_3$, 2.55 Å - $\text{Al}(\text{OH})_3$, 3.1579 Å - CaF_2 , 2.72 Å - $\text{Mg}(\text{OH})_2$, 3.3514; 3.1988 Å - CaHPO_4 , 3.87; 2.79 Å - CaSO_4 .

Figure 4 shows the results of the scanning electron microscopic analysis (SEM) of the solid phase obtained from processing sodium phosphate sludge.



Element	Wt. %	Sigma Wt. %
C	0.08	0.03
O	45.32	0.81
F	5.56	0.73
Na	0.11	0.02
Mg	1.54	0.04
Cl	0.11	0.02
Al	6.67	0.15
Si	0.09	0.02
P	6.11	0.33
S	0.31	0.03
Ca	26.15	0.94
Fe	7.95	0.27
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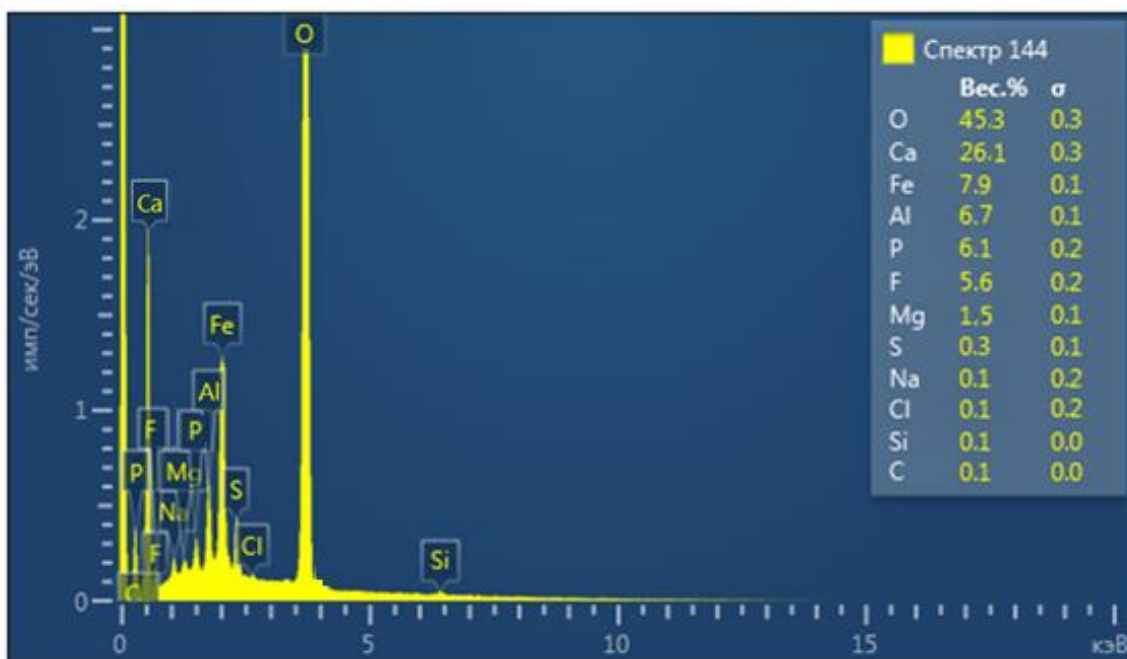


Fig. 4. Scanning electron microscopy analysis of the solid phase obtained from processing sodium phosphate sludge

The elemental composition of the solid phase: O - 45.32%, F - 5.56%; Na - 0.11%; Mg - 1.54%; Cl - 0.11%; Al - 6.67%; Si - 0.09%; P - 6.11%; Ca - 26.15%; S - 0.31%; Fe - 7.95%.

Figure 5 shows the principal technological scheme for processing sodium-phosphate sludges with sodium hydroxide.

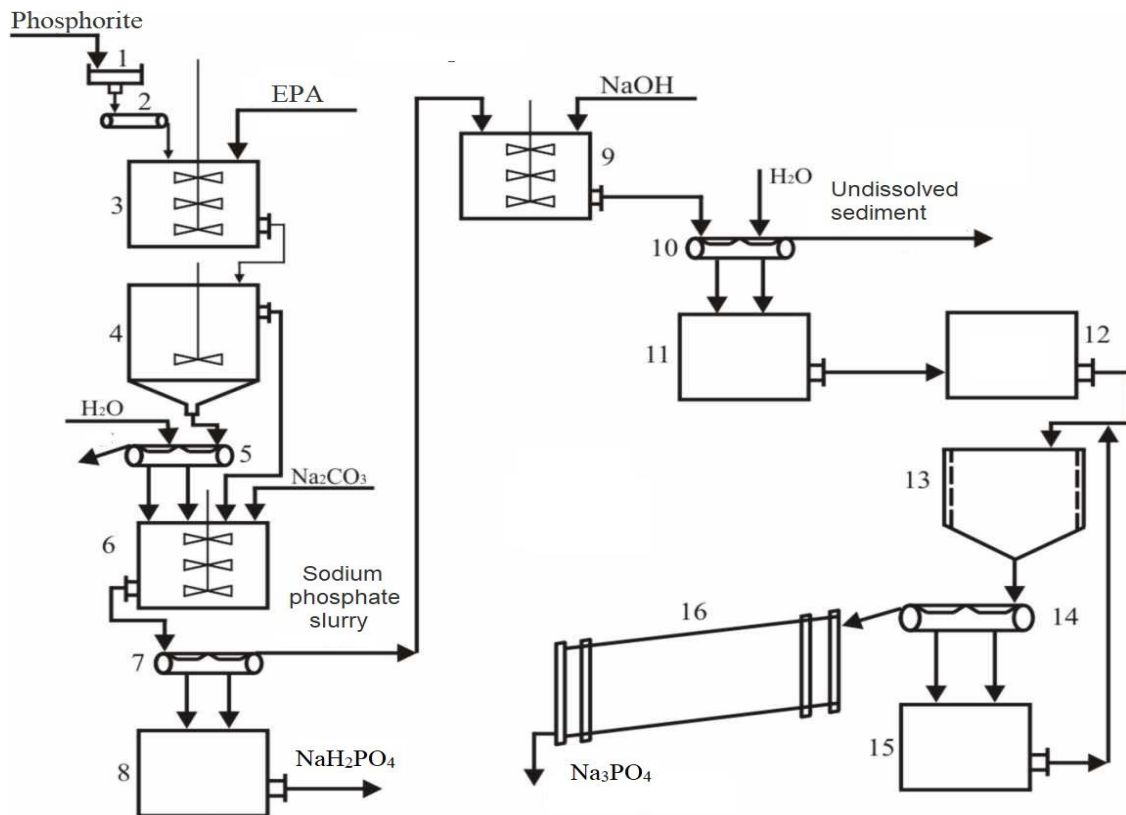


Fig. 5. Flexible principal technological scheme for obtaining sodium orthophosphates: 1 - bunker, 2 - belt feeder, 3, 6, 9 - reactors, 4 - settler, 5, 7, 10, 14 - filters, 8, 11, 15 - collectors, 12 - vacuum evaporation unit, 13 - crystallizer, 16 - drying drum.

Defluorinated extract phosphoric acid (EFPA) enters the reactor (position 3), where it is introduced from the receiving bunker (position 1) through the feeder (position 2). The resulting suspension is placed in the settler (position 4), and the thickened part is fed to the filter (position 5). The calcium sulfate precipitate is sent to the storage warehouse, while the filtrate and the solution separated from the settler are neutralized with sodium carbonate in the reactor (position 6). The resulting suspension is filtered in the filter (position 7), and the monosodium phosphate solution is sent to the container (position 8) for further processing into mono-, di-, trisodium phosphate, and polyphosphates. The sodium phosphate sludges obtained are then sent to the reactor (position 9) for further processing with sodium hydroxide solution. The sludge, consisting of hydrolyzed calcium, aluminum, iron hydroxides, calcium fluoride, and other impurities in the presence of sodium hydroxide, is separated in the filter (position 10). The resulting sodium orthophosphate solution is collected in the intermediate container (position 11) and evaporated in the vacuum evaporation unit (position 12). The sodium orthophosphate solution is transferred to the crystallizer (position 13) for crystallization. The sodium orthophosphate crystals are separated in the filter (position 14) and dried in the drying drum (position 16), and the circulating solution is returned to the crystallizer (position 13).

IV. CONCLUSION

In conclusion, it can be said that it is possible to obtain sodium orthophosphate by processing sodium-phosphate sludges formed during the neutralization of EPA obtained from phosphorites with sodium carbonate using sodium hydroxide solution.



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