ISSN: 2350-0328
International Journal of AdvancedResearch in Science, Engineering and Technology
Vol. 11, Issue 11, November 2024

Production of Sodium Tripolyphosphate from Phosphate Sludge

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ABSTRACT: The results of research on the production of purified solutions and sodium phosphate salts from phosphate sludge formed during the neutralization of extractive phosphoric acid with ammonia are presented. The norms of an alternative technological regime are determined. It is shown that by neutralizing phosphate sludge with a sodium carbonate solution, 70-75% of the phosphorus oxide in its content can be separated and monodisodium phosphate salts can be formed, which are suitable for the production of sodium polyphosphates.

I. INTRODUCTION

Sodium tripolyphosphate are used in many industries due to its specific properties. However, the main consumer are manufactures of synthetic washing-up liquids where sodium tripolyphosphate is used for water softening [1-3]. Sodium tripolyphosphate is an active filler that provides the best characteristics and the minimum influence on environment. For manufacture of washing-up liquids application sodium tripolyphosphate with the required specific volume density, granulometric composition, crystal phase (form I and II) and moisture content [4-6].

A large part of the sodium phosphates used for the preparation of boiler water, and for cleaning boilers. At their addition to feeding water the scaling in coppers is prevented. [7, 8]. Them also enter into structure of lubricant-cooling liquids in the process of mechanical processing of metals [9-11].

Sodium tripolyphosphate is used for the preparation of boiler water and cleaning boilers. At its addition to feeding water the scaling in boilers is prevented [12, 13]. It also enter into structure of lubricant-cooling liquids in the process of mechanical processing of metals [14-16].

II. RESEARCH METHODS

The process of obtaining a monoammonium phosphate solution by ammonization of partially defluorinated and desulfated extraction phosphoric acid is based on the following chemical reactions:

$$H_3PO_4 + NH_3 \rightarrow NH_4H_2PO_4$$
$$Ca(H_2PO_4)_2 + NH_3 \rightarrow CaHPO_4\downarrow + NH_4H_2PO_4$$
$$3Al(H_2PO_4)_3 + NH_3 \rightarrow 3AlPO_4\downarrow + 6NH_4H_2PO_4$$
$$3Fe(H_2PO_4)_3 + NH_3 \rightarrow 3FePO_4\downarrow + 6NH_4H_2PO_4$$
$$Ca(H_2PO_4)_2 + 2NaF \rightarrow CaF_2\downarrow + 2NaH_2PO_4$$

After the ammonization process, the composition of the phosphate sludge separated by filtration consists of phosphate deposits of calcium, aluminum, iron, and unseparated monoammonium phosphates. The chemical composition of phosphate slurry is given in table 1.

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Table 1. Chemical composition of phosphate slurries

no	Chemical composition, mass %							
	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	N	SO_4^{2-}	F
1	23,84	9,36	3,17	1,39	1,41	3,28	0,26	1,13
2	25,47	8,55	3,23	1,42	1,46	3,54	0,29	1,17

This technology for the production of alkali metal phosphates involves the treatment of phosphate sludge with sodium carbonate or hydroxide in an aqueous medium at a temperature of 85-95°C, subsequent filtration of the resulting mixture and processing of the separated sodium phosphate solution into a commercial product of sodium tripolyphosphate. A 30% sodium carbonate solution was used to carry out the experiment. The mixture of sodium carbonate solution at a temperature of 85-90°C and phosphate sludge selected as the object of study was maintained at an ambient pH of 6.5-6.7.

The obtained sodium tripolyphosphate samples were analyzed using X-ray and IR spectrum methods.

The samples were identified based on diffraction patterns obtained on an XRD-6100 diffractometer (Shimadzu). CuKα radiation (β filter, Ni, 1.54178, tube current and voltage mode 30 mA, 30 kV) and a constant detector rotation speed of 4 deg/min with a step of 0.02 deg (ω/2θ coupling) were used, and the scanning angle varied from 4 to 80°.

The spectra of the samples were recorded using an IRTracer-100 FTIR spectrometer equipped with a single-pass ATR attachment with a diamond/ZnSe MIRacle 10 prism in the scanning range: 4600 – 600 cm^{-1}.

III. EXPERIMENTAL RESULTS

In the experimental research, studies on the effect of process time and temperature on the degree of separation of the phosphorus component in the slurry into the solution were studied.

The effect of the process time on the degree of P_2O_5 release into the solution during the treatment of phosphate sludge with sodium carbonate solution was studied in the range from 45 to 110 minutes. It was shown that 60.81% of P_2O_5 was released into the solution at 45 minutes, 72.95% at 60 minutes, 74.49% at 75 minutes, 75.12% at 75 minutes, and 75.36% at 110 minutes. The data show that at 60-75 minutes, the release of P_2O_5 reaches 73-75%. Later, with increasing time, it changed very little.

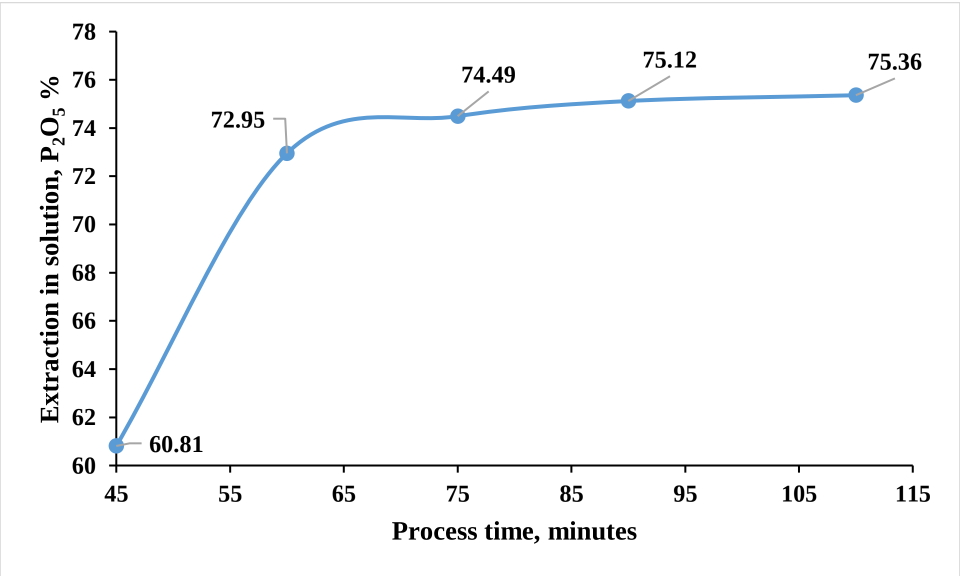

Fig. 1. Effect of process time on the extraction rate of P_2O_5 in phosphate slurry

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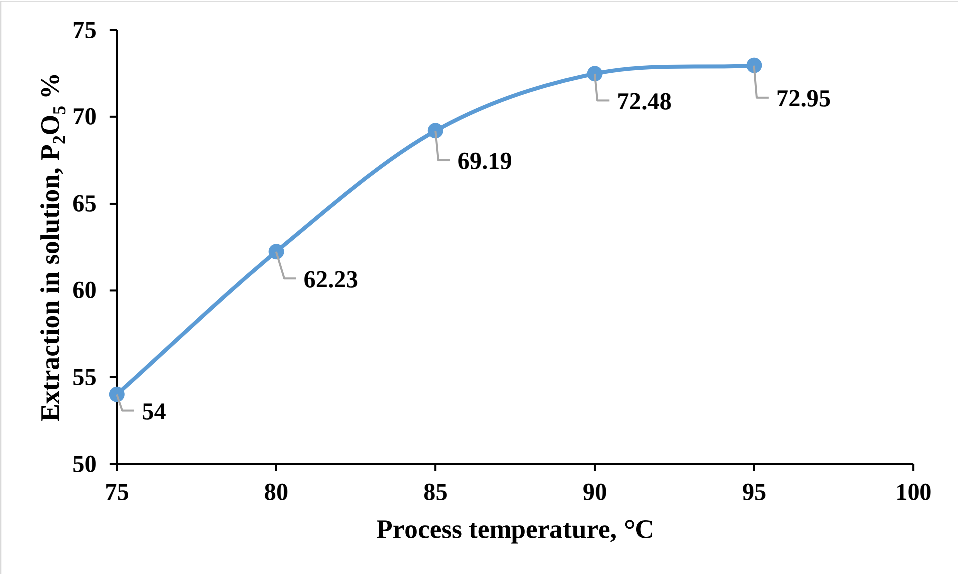

Fig. 2. Effect of process temperature on the extraction rate of P_2O_5 in phosphate slurry

The effect of process temperature on the degree of P_2O_5 release into the solution during the treatment of phosphate sludge with sodium carbonate solution was studied in the range from 75°C to 110°C. At 75°C, 54.0%, at 80°C - 62.23%, at 85°C - 69.19%, at 90°C - 72.48% and at 95°C - 72.95% of P_2O_5 was released into the solution. The data show that the release of P_2O_5 increases in the temperature range from 75 to 95°C.

The sodium phosphate suspension was kept at a temperature of 90-95°C for 90 minutes. Insoluble impurities in the suspension were separated by filtration. The solution was evaporated and thermally treated at a temperature of 360°C. The composition of the obtained sodium tripolyphosphate samples is presented in table 2.

Table 2. Chemical composition of sodium tripolyphosphate

№	Chemical composition, mass %						pH of 1% solution	Extraction of P_2O_5, %
	P_2O_5	Na_2O	MgO	Al_2O_3	Fe_2O_3	F		
1	56,5	41,43	0,0071	0,0079	0,0084	0,0021	9,6	71,51
2	56,7	41,40	0,0075	0,0077	0,0076	0,0019	9,5	72,95

In this case, sodium tripolyphosphate containing 56.5-56.7% P_2O_5 is obtained. The extraction of P_2O_5 from phosphate sludge is 71.51-72.95%, while the mass fraction of sodium tripolyphosphate is 96.8%, and the pH of a 1% solution of sodium tripolyphosphate is 9.5-9.6.

Thus, mass-spectral elemental analysis of sodium tripolyphosphate obtained and dried at a temperature of 360 °C was carried out. It was shown that the product additionally contains: Ca - 117; Mg - 115; Al - 57; Fe - 61; As - 14; Pb - 0.299 g/t.

Figure 3 shows an X-ray diffraction pattern of a sodium tripolyphosphate sample calcined at 360°C. During dehydration at a temperature of 360°C, only phase II is formed, which is characterized by intense peaks at 4.5993, 4.4621, 3.0631, 2.8143, 2.7390, 2.6928 Å.

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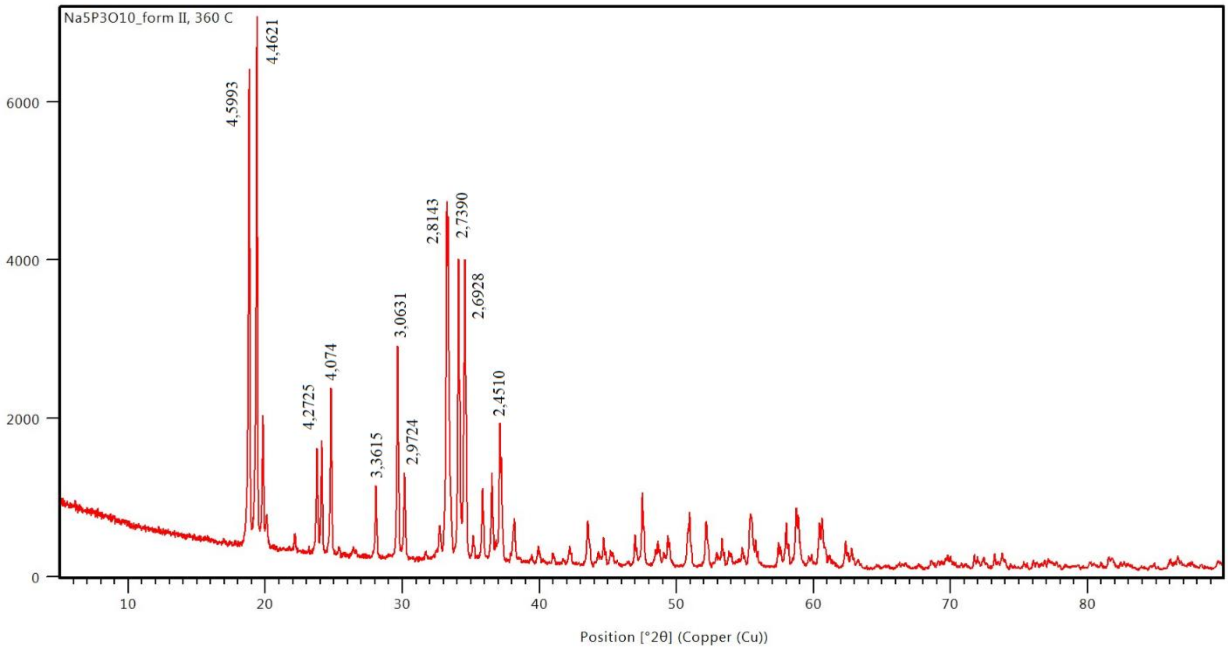

Figure 3. X-ray diffraction of sodium tripolyphosphate

The IR spectrum of the sample calcined at 360°C is shown in figure 4.

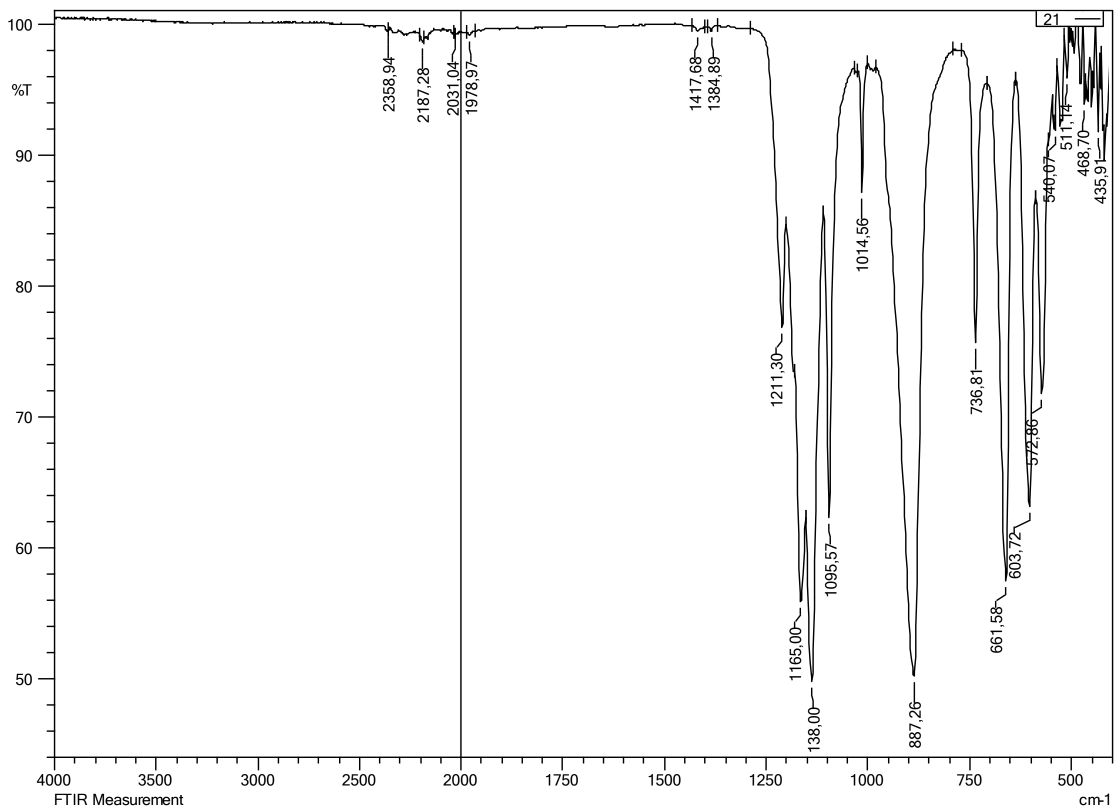

Figure 4. IR spectra of sodium tripolyphosphate

ISSN: 2350-0328

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The vibrational absorption spectra of the compounds characterize the features inherent in the IR spectra of polyphosphates. In the IR spectrum of $Na_5P_3O_{10}$, three groups of absorption bands can be distinguished in the ranges of 572.86-736.81, 887.26-1014.56, 1095.57-1211.30 cm^{-1}, indicating the long-chain structure of the compound anion. In the region of asymmetric vibrational absorption spectra of the compounds characterize the features inherent in the IR spectra of polyphosphates. The shift in the position of the absorption band maxima of the PO_2 and POP groups to the long-wave region, as well as the change in the intensity of the absorption bands reflect the influence of the nature of cations and the degree of polymerization of the anion.

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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