

The Role of Medium Condition for Uranium Separation from Central Kyzylkum's Low-grade Phosphorite after Sulfuric Acid Treatment

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Abstract—This article presents the results of separating radioactive elements, particularly uranium, from low-grade phosphorites of the Central Kyzylkum by low concentrated sulfuric acid solution, which is practically feasible at the Kyzylkum phosphorite combine (KPC). In world practice, uranium was extracted from phosphorite ore in the countries of the Middle East (Syria, Morocco, Algeria, Egypt) at the final stages of obtaining phosphorus fertilizers at metallurgical and chemical plants. The extraction of uranium from phosphorite ore is not practiced in Uzbekistan. The proposed method is fundamentally different from the above and is recommended to be applied at the initial stage of enrichment phosphorite ores in the enrichment plants. Studies have shown that the uranium completely passed into solution by treating phosphorite ore with weak concentrated sulfuric acid solution. Analyses of solution and sediment composition showed that the useful component, P₂O₅, remained in the sediment, increasing from 8.69% to 10.3%, while uranium passed into the solution with alkaline salts alkaline-earth elements. To obtain a finished product, the resulting enriched solution was added to the richer phosphorite ore. When performing the above processes, the pH of the medium was kept at the neutral level, which is of great practical importance for the implementation of this method with minimal economic costs using conventional inventory, corrosion resistance, and environmentally friendly phosphorus fertilizer.

Keywords—Francolite; pH medium; filtration; sorption; extracting; sediments; fertilizers.

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I. INTRODUCTION

Phosphorites react with sulfuric acid to form phosphoric acid and phosphogypsum (hydrated calcium sulfate). Phosphoric acid (H₃PO₄) is one of the most produced acids in the world. The phosphoric acid produced contains several undesirable hazardous impurities, including small uranium and lanthanides. However, with the high global production of phosphate fertilizers at about 150 million tons per year, the small amounts of these lanthanides and uranium impurities are of economic interest. In addition, 90% of the phosphorus produced is used in the pharmaceutical, food production, feed, and fertilizer industries, and the presence of these impurities is a source of danger to human health and the environment [1]. There are various methods of extracting uranium from the wet process phosphoric acid during the production of phosphoric fertilizers in chemical plants at the final stage, in the purification of wet-process phosphoric acid (WPPA). In

this case, selective ion-exchange resins are added to the wet process phosphoric acid solution mixed with uranium and rare earth elements, on which these elements are sorbed. Before declining uranium prices, this method was used to extract from phosphorites in the Middle East (Syria, Morocco, Algeria, Egypt) in world practice. After the Fukushima nuclear power plant disaster, uranium extraction from phosphorites was suspended [2].

There are also known methods for extracting uranium in phosphoric fertilizers. The method was used to extract uranium at the final production stage after treatment with concentrated sulfuric acid, releasing the wet process phosphoric acid into the solution and harmful impurities. At the same time, difficulties appeared with the formation of viscosity, technologically hard-filtered pulp, and the presence of 4-6% phosphorus in the waste in the form of phosphogypsum [3], [4].

II. MATERIALS AND METHOD

In the process of obtaining phosphate fertilizers from the wet process phosphoric acid solution mixed with uranium and rare earth elements, their extraction is carried out using selective ion-exchange resins [5]. Another example by synergistic mixture solvent of D2EHPA – TOPO, Uranium extraction from Philippine phosphoric acid using the synergistic mixture solvent of D2EHPA – TOPO shows promising results with 91% recovery rate. However, the process temperature must be - 40 °C, which appears to be more complicated [6].

Extracting uranium employing sorption from aqueous solutions could address resource recovery and decontamination [7]. A crucial factor in the sorption process is selecting efficient sorbents. Until now, natural minerals, carbon materials [8], [9], natural organic materials [10], [11], synthesized nanomaterials [12], [13], solid waste-derived sorbents [14,15], microorganism [16], phosphate [17], and metal-organic frameworks [18], [19] have been studied extensively for this purpose. Among them, inexpensive and sustainable adsorbents have drawn attention. Thus, greater attention should be paid to adsorbents derived from byproducts and solid waste from the viewpoint of green chemistry and cleaner production processes.

In Uzbekistan, at the chemical plant Almalyk-Maksam, which produces phosphorus fertilizer-amorphous, it is not practiced to extract uranium from solution [20]. Washed burnt phosphate concentrate from KPC with P₂O₅ content of 28% is used as a starting material [21]. This concentrate is treated with concentrated sulfuric acid, which produces the vicious, hard-filtered mixture, making it difficult to filter the solution, which contains harmful impurities in uranium, arsenic, and fluorine rare-earth elements [22]. All this mixture goes into the phosphate fertilizer and negatively affects the purity of amorphous [23]. In addition, due to the concentrated sulfuric acid interaction, phosphogypsum is formed in waste, which takes about 4-6% of P₂O₅.

In the production of phosphoric acid from phosphorites, thousands of tons of uranium are lost annually. Since 0.01-0.02% of uranium contained in phosphorite is not extracted from phosphorites [24] on an industrial scale, it is possible to extract simultaneously with the production of 30% technical phosphoric acid obtained by the wet method. In the late 1940s and early 1950s, various methods of uranium extraction from phosphorites - extraction, chemical, and ion exchange - were developed in the United States [25]. The main disadvantages of all known methods are the high consumption of sulfuric acid, the technological difficulties arising in the filtration of the resulting viscous solutions, which is formed after the addition of concentrated sulfuric acid, which entails an increase in energy costs and the purchase of special corrosion-resistant equipment [26].

The purpose of the research was to study the possibility of enriching low-grade phosphorite ores at the initial stage of production when obtaining phosphorite concentrates with the following extraction of harmful impurities in the form of radioactive elements and salts of alkaline and alkaline earth elements. To perform this experiment, we used a method to separate uranium from phosphate ore into solution using a low concentrated sulfuric acid solution to study the kinetics of uranium leaching into solution while retaining the useful component as P₂O₅ in the precipitate.

In connection with the above, laboratory studies were carried out at the Navoiy State Mining Institute to study the chemical composition of low-grade granular phosphorites of the Dzheroi-Sardarinsky deposit, which is presented in Table 1.

TABLE I
CHEMICAL COMPOSITION OF CENTRAL KYZYL KUM'S PHOSPHORITES

No	Comp.	Cont. of el. (%)	No	Comp.	Cont. of el. (%)
1	P ₂ O ₅	8.69	8	U	0.0032
2	Al ₂ O ₃	1.5-3.0	9	REE total	0.04-0.089
3	SiO ₂	6.0-8.0	10	Florium	1.8-3.2
4	CaO	42-48,1	11	SO ₃	2.5- 3.5
5	MgO	2.5-3.5	12	H ₂ O	10.0
6	Fe ₂ O ₃	0.6-0.8	13	Organic substance	2.8-3.0
7	CO ₂	8-15	14	Insoluble residue	8.0-8.2

The chemical composition of phosphorites is presented (Table 1) by the predominant amount of calcium oxide (CaO - 42-48.1%) presented in the form of CaCO₃ calcite mineral. Next is the aluminosilicates - a mixture of aluminum oxides (1.5-3.0%), silicon (6.0-8.0%) and magnesium oxide (2.5-3.5%).

The research work was carried out in the Chemical engineering and Mineral processing laboratories of Navoiy State Mining Institute and the Central Laboratory of the State Committee for Geological and Mineral Resources of the Republic of Uzbekistan, as shown in Fig. 1.

1) The amount of P₂O₅ in the feed ore sample was determined by the Spectrophotometer UV-1280 [27].

2) Samples were obtained in 100 g mass which taken from phosphorite ores by quartering method after ore blinding:

- each sample was put in the glass flasks
- pour the sulfuric acid solution (in different concentrations in different glass flasks) in this glass flask with the ratio L:S=10:1, with the 30 minutes stirring time;

The concentrations of sulfuric acid (0 – 40 g/l) were used as options to determine the effect of changes in sulfuric acid concentration on the solubility rate of uranium in the sample obtained and to determine the optimal concentrations that has high efficiency.

3) The resulting solution was filtered by filter paper and divided into filtrate and solid phase;

4) In the mass spectrometer ICP-Agilent 7500 SX, the elemental composition of the obtained filtrates and sediments was determined by the inductive plasma mass spectral method [28];

5) The total amount of P₂O₅ in the obtained filtrates and sediments (State Standard: 20851.2-75. Mineral fertilizers. Methods for the determination of phosphate) was determined by the Spectrophotometer UV-1280 [27];

6) Microscopic analyzes of sediments obtained were carried out on a binocular microscope of the NLCD-307B brand with digital modeling and with a monitor based on the Android OS "Ningbo optics" (China);

7) The IR spectra of the feed ore and sediments obtained samples were analyzed on a SHIMADZU device (laboratory of the Novosibirsk State Geological Institute, Department of Chemical Technology) brand IR-Fourier spectrometer IRTracer-100, by preparing pressed tablets in KBr. The absorption bands of infrared light correspond to antisymmetric wavelengths ranging from 400 to 4000 cm^{-1} .

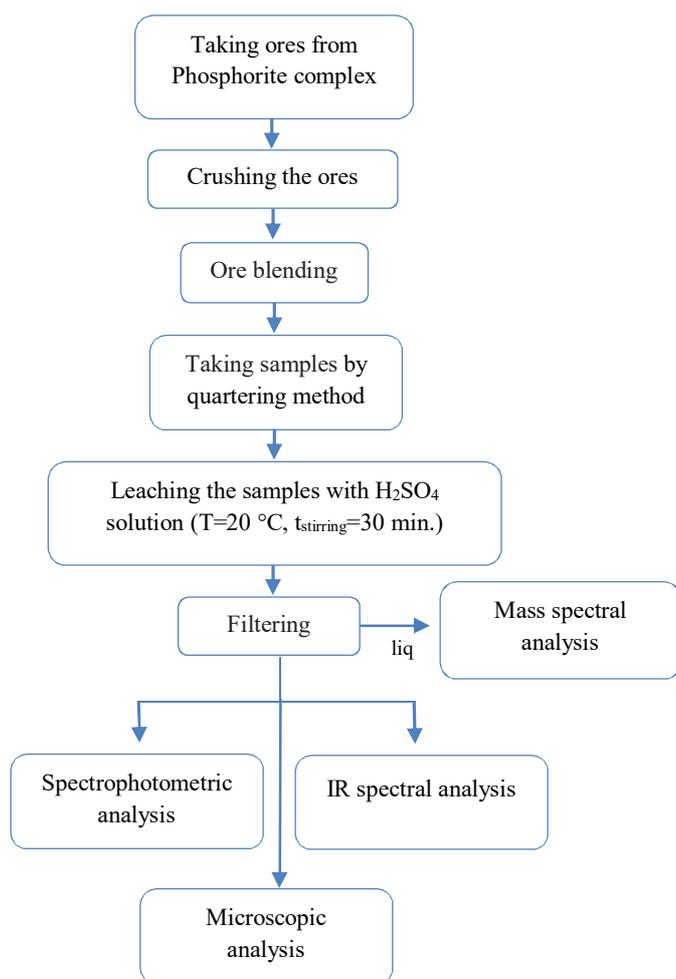


Fig. 1 The stages of conducting research.

The results of the analysis were obtained and collected. The microscopic analysis results were compared to the original state of francolite, gypsum, and quartz minerals. The results of mass-spectral, spectrophotometric, and IR analysis were compared with the facts studied by learning related articles and books. This final result was excellent, and it led us to conclude that this research method was well performed

The solution to the assigned task was achieved by sulfuric acid processing low-grade phosphorites that are not used at KPC for subsequent enrichment and their involvement in production. Considering the role of sulfuric acid as a strong oxidizing agent, it was assumed that interfering impurities in the form of radioactive elements with a certain amount of salts of alkaline and alkaline-earth elements could be extracted into

the solution, as well as the parallel enrichment of phosphorite with its subsequent involvement in the production at KPC. In mixing reactors, phosphorites were treated with low concentrated sulfuric acid (2, 4, 6, and 8 g/l) solutions in the ratio S: L = 1: 10. After stirring for 30 minutes, the whole mass was loaded into thickeners, in which the precipitate was easily separated from the solution. In the future, the resulting precipitate can be enriched at KPC, and the concentrate can be obtained. Also, uranium can be extracted from the separated solution using selective ion-exchange resins. After softening with milk of lime and disposal of chlorides, process water can also be reused as recycled water.

III. RESULTS AND DISCUSSION

Chemical analysis of the feed low-grade phosphorite ore showed the content of the useful component - P_2O_5 in the amount of 8.69%, uranium U 0.0032%. The gradual addition of low concentrated sulfuric acid solution (2-40 g/l) to phosphorites showed that there were no obvious deviations in the pH of the medium towards an increase in acidity, which indicates the release of alkali and alkaline earth metals' salts into the solution, which led to the formation of complex compounds with sulfuric acid, did not allow to decrease the pH of the solution. Analysis of the pH medium of the solutions with high concentrations - 20, 30 and 40 g / l, showed an insignificant decrease in pH and amounted to about 7.66, 6.49 and 6.70, respectively (Table 2).

TABLE II
CHARACTERIZATION OF PH OF THE MEDIUM, MASS OF DRY SEDIMENT AND MASS SPECTROMETRIC ANALYSIS OF THE MAIN ELEMENTS' CONTENT IN PHOSPHORITE ORE

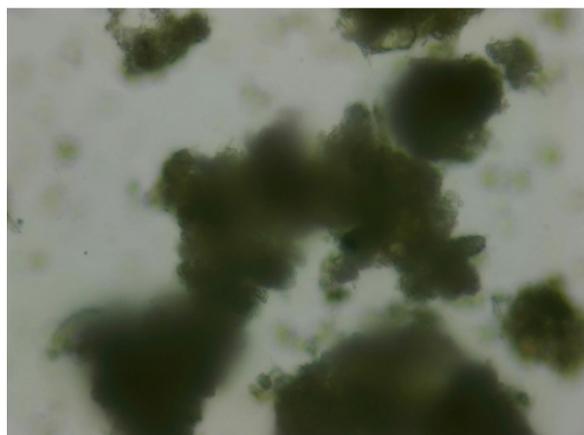
No	1	2	3	4	5
H_2SO_4 concentration (g/l)	0	2	4	6	8
pH of solutions	7,99	8,19	8,25	8,27	8,14
Sediment mass (g)	98,0	98,1	99,1	100,9	101,9
Contents of P_2O_5 in feed ore (%)					8,69
Content of P_2O_5 in solutions (%)	0,15	0,15	0,11	0,09	0,09
Contents of P_2O_5 in sediments (%)	8,8	9,2	9,5	9,7	10,3
Content of U in feed ore (g/t)					32,0
Content of U in solution (g/t)	8,8	16	32	32	32
No	6	7	8	9	10
H_2SO_4 concentration (g/l)	10	15	20	30	40
pH of solutions	6,58	6,49	7,66	6,49	6,70
Sediment mass (g)	102,8	105,9	110,2	117,3	123,9
Contents of P_2O_5 in feed ore (%)					8,69
Content of P_2O_5 in solutions (%)	0,12	0,09	0,11	0,15	0,08
Contents of P_2O_5 in sediments (%)	9,1	7,92	7,53	7,15	6,94
Content of U in feed ore (g/t)					32,0
Content of U in solution (g/t)	28	17	32	6,3	7,3

In addition to the below, the transformation of the calcite mineral CaCO_3 into Ca(OH)_2 was the main reason for keeping the pH within neutral levels. Determination of the content of the useful component - P_2O_5 and U in the composition of the solution showed that low concentrated sulfuric acid also did not cause to dissolve P_2O_5 due to the binding of sulfate ions by chemically more active alkali and alkaline earth metals. A study of the sediment mass obtained showed that the mass begins to increase from treating with 8 g/l and reaches the

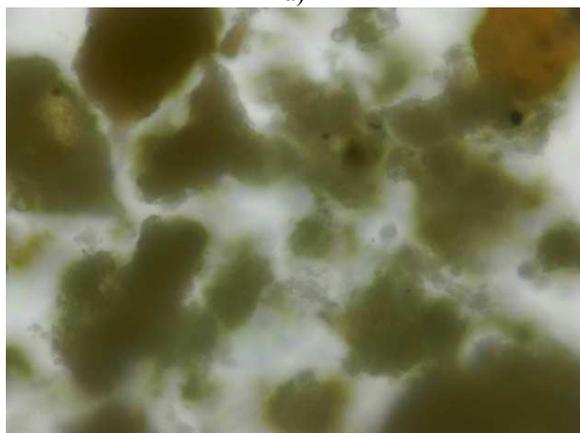
maximum level with the 40 g/l sulfuric acid solutions, reaching 123.9 g, instead of the initial 100 g. This fact indicates the beginning of the gypsum and phosphogypsum formation in the resulting sediment. In this process, phosphoric acid and gypsum were produced when sufficient amounts of sulfuric acid were used. The general reaction of apatite dissolution by sulfuric acid is written below (1) [29]:



However, the opposite case occurred with uranium, when this element began to actively dissolve by treating with low concentrated sulfuric acid solution and especially intensively at concentrations of 4, 6, and 8 g/l H_2SO_4 . Sulfuric acid mainly dissolves 6 valence uranium. This means that the phosphorites contained mainly 6 valence uranium. The oxidation of 4 valence uranium can most likely explain the reason for the 6 valence uranium appearance not only during the mining of phosphorite ore by the open method, in which, under the influence of atmospheric oxygen, it is oxidized to the 6 valence form but also by the fact that the layer of phosphorites is located on the near-surface areas, at a depth of 18-25 m. Thus, an important fact to separate uranium from P_2O_5 in phosphorites is noted.



a)



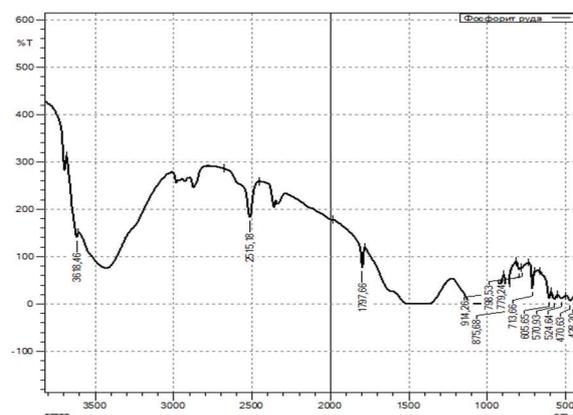
b)

Fig. 2 Structure of francolite and other mineral components. a) the feed low-grade phosphorite ore (magnification 10x40). b) low-grade phosphorite ore after treatment with sulfuric acid 8 g/l (magnification 10x40).

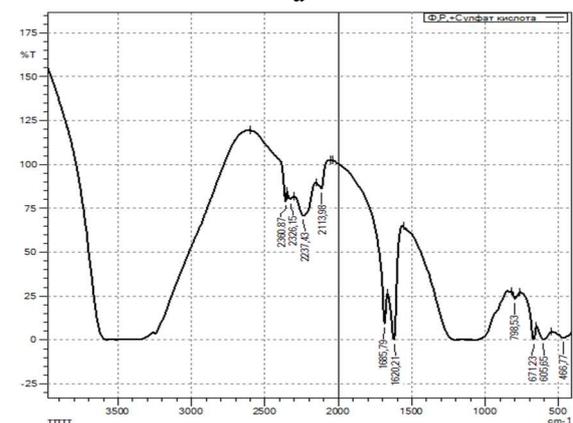
Microscopic analysis of the minerals' morphological structure of the feed phosphorites showed the dominance of francolite grains in all the studied fields of view, where they

had a dark brown color (Fig. 2. a). Grain size varied but most often ranged from 500 to 1.500 microns. The inclusions of calcite imparted a darker shade to the color of the francolite. In addition, the francolite mineral itself had a denser, dark brown, saturated hue. The edges of francolite grains were clearly outlined.

After treatment with a sulfuric acid solution (8 g/l) (Fig. 2. b), the color of francolite grains became lighter, and the grains themselves became friable. The edges of the grains became more friable and sparser, which indicates the gradual diffusion of sulfuric acid molecules into the structure of the mineral, thereby causing its loosening and gradual decomposition. Grain size after sulfuric acid treatment decreased slightly in size and ranged from 200 to 500 microns. Additionally, calcite crystals were separated from francolite, which gave the francolite grains a light gray color. Calcite under sulfuric acid separated from francolite acquired a white hue and resembled a powdered over francolite grains surface. When the sulfuric acid solution concentration was increased to 30, and 40 g/l, the translucent, elongated, and multifaceted shape of gypsum crystals began to stand out clearly in the microscopic images.



a



b

Fig. 3 IR spectrum. a) low-grade phosphate ore. b) phosphorite ore treated with sulfuric acid.

Study of IR spectra of low-grade phosphorites before (Fig. 3) and after (Fig. 3. a) treatment with a sulfuric acid solution (8g/l) showed a wide range of wavelengths for the distribution of functional groups in low-grade phosphorites where marked peaks in the spectra of francolite from 995 to 1600 cm^{-1} . The degree of density and infrared permeability of the francolite

mineral was very high and ranged from 0 to 8 %. Calcium compounds in the form of the calcite mineral were not only in the francolite but were also distributed as free calcite (CaCO_3) and CaO at wavelengths from 800 to 3800 cm^{-1} . Thereby, the IR spectra were able to determine the location of the main mineral of phosphorite - francolite in the composition of phosphate ore, located within the wavelengths from 800 to 1800 cm^{-1} .

After treatment with a weak concentrated sulfuric acid solution (Fig. 3. b), in the francolite, which consists mainly of two large, broad peaks, only one peak, corresponding to a wavelength of 1500 to 1900 cm^{-1} , was split. Another peak at wavelengths from 100 to 1400 cm^{-1} remained intact.

Thus, the experiments conducted with a sulfuric acid solution on phosphorites allowed to form a new technological scheme (Fig. 4) to separate uranium from low-grade phosphorites.

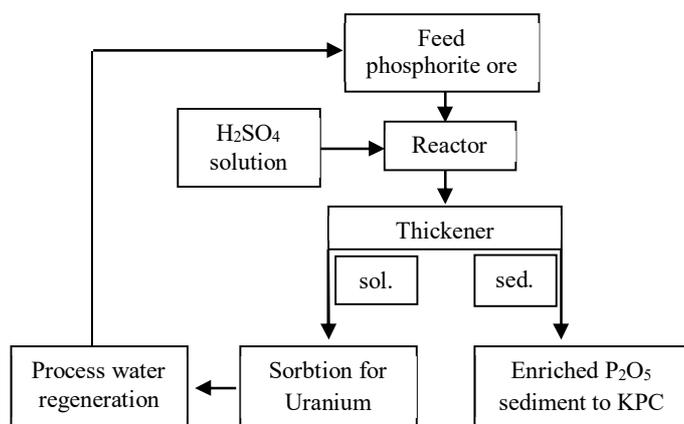


Fig. 4 Technological scheme of uranium separation from low-grade phosphate ore.

According to this scheme, initially prepare the weak concentrated sulfuric acid aqueous solution, in which phosphate ore is added in a ratio of S:L = 1:10, then stirred in the reactors for 30 minutes. The resulting mixture is transported to special thickeners, where the solution is separated from the sediment. In the process, uranium and some salts of alkaline and alkaline earth elements are separated into the solution. The uranium extraction from the solution can be carried out by adding selective uranium ion-exchange resins to the solution. After the sorption of uranium, the process solution is purified from impurities with milk of lime, and then this purified process water can be reused in the process.

The conducted research is new in its technological nature because uranium was extracted from low-grade phosphate ore of Central Kyzylkum at the initial stage of processing. Such low-grade ore is practically not used at KPC, and its waste has accumulated in the amount of more than 11 million tons, which is constantly growing, and due to being carried away by the wind in the form of fine dust, they cause environmental pollution. After processing low-grade ore and increasing its useful component in the form of P_2O_5 , it is recommended to involve it in the process by proportionally adding it to the main mass of phosphate ore used in the KPC.

The application of the presented methods of enrichment of low-grade phosphorites in the enrichment factory is practically possible with the original, averaged ore processed

at the plant because the chemical and mineralogical composition of phosphorites is the same except for the useful component P_2O_5 , whose content is much higher (more than 19 %) than in the low-grade fractions. It is also proposed to include step-by-step treatment, first with sulfuric acid when using, which the release of interfering impurities can increase even more.

IV. CONCLUSION

Thus, we have developed a fundamentally new method of separating uranium from low-grade phosphorite ores of the Central Kyzylkum Republic of Uzbekistan and the technological scheme, which can be used to separate harmful and interfering impurities from phosphorites. The process is practically feasible at the initial stage of ore processing. For this purpose, it was proposed to add sulfuric acid diluted in water to the phosphorite ore with an optimum concentration of 4 to 8 g/l. This variation in concentrations is due to the amount of uranium and the useful component, P_2O_5 , in the composition of the ore. After stirring for 30 minutes in the sulfuric acid solution, penetration of sulfuric acid into the mineral grains of francolite, which gradually begins to loosen and shrink in size, was noted. The resulting solution quickly settles and is easily separated from the sediment by simply pouring the liquid into another container. After the sorption extraction of uranium from the solution onto an ion-exchange resin, it is proposed to soften the solution and reuse it in the process.

The resulting phosphorus-enriched sediment, free from uranium and partially from salts of alkali and alkaline earth elements, is proposed to be added to the richer ore to obtain the finished product at the KPC. The technology is practically feasible for continuous applications.

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