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## **STUDY OF DECOMPOSITION OF LOW-RATED PHOSPHORITES OF THE AKZHAR DEPOSIT BY GREAT EXCESS OF PHOSPHORIC ACID**

### **Abstract**

The article describes about the phosphorite deposit and its reserves. At present, the revived production of phosphorus and complex fertilizers is based mainly on Karatau phosphorites. However, the reserves of rich phosphate ores in Karatau are being depleted, and in recent years there has been a need to involve low-grade natural phosphates in circulation. Therefore, the integrated use of mineral raw materials and ensuring the environmental safety of the technologies used are now becoming more and more relevant. The article provides about IR spectroscopy and XRF analysis of the Akzhar phosphorite deposit in the Karatau basin. And also, the results of the chemical composition of phosphorite and calculations of the kinetics of the decomposition of phosphorite of the Akzhar deposit of the Karatau basin with a 5-fold excess of phosphoric acid are presented.

**Keywords:** acid, apatite, mineral fertilizers, phosphorite decomposition.

### **Introduction**

Kazakhstan is one of the largest regions in the world with significant reserves of mineral raw materials and the prospects for expanding their use in chemical industry [1].

The production of mineral fertilizers is one of the priority areas of economic development, since the Republic of Kazakhstan has the largest deposit of phosphate raw materials in the Karatau basin, the reserves of which are more than 15 billion tons, and huge areas for grain, vegetables and industrial crops are in dire need of fertilizers to obtain sustainable and high yield [2].

At present, the revived production of phosphorus and complex fertilizers is based mainly on Karatau phosphorites. However, the reserves of rich phosphate ores in Karatau are being depleted, and in recent years there has been a need to involve low-grade natural phosphates in circulation.

At the same time, the integrated use of mineral raw materials and ensuring the environmental safety of the technologies used are now becoming more and more relevant, and much attention is paid to research related to the creation of technologies focused on obtaining not one, but several end products with the involvement of local raw materials in the production of local raw materials.

The Akzhar phosphorite ore deposit is currently one of the largest in terms of its reserves. Akzhar phosphorites belong to low-quality phosphate raw materials: washed concentrate contains 15-19% P<sub>2</sub>O<sub>5</sub> and a significant amount (up to 40%) of impurities, in particular SiO<sub>2</sub>. In addition, they are characterized by strong mutual intergrowth of their constituent minerals, which does not allow to enrich the raw material sufficiently efficiently and economically by flotation. In this regard, the question of finding new rational methods for the preparation and processing of such phosphorites, the search for ways of direct processing of low-grade phosphate raw materials into high-quality valuable products, arose sharply [3-5].

Known in the literature data on the kinetics of decomposition of Karatau phosphorites by a 4-8-fold excess of phosphoric acid refer to the study of low-grade phosphorites from the Zhanatas and Koxsu deposits. The lack of information on the kinetics of decomposition of poor phosphorites of the Akzhar deposit and the effect on the technological parameters of various process parameters (temperature, consumption rate of reagents, duration of interaction of reagents and concentration of phosphoric acid) necessitates additional studies in order to select optimal parameters [6-7].

### Materials and methods

To study the kinetics of decomposition, we used an ore sample with the following chemical composition, %: P<sub>2</sub>O<sub>5</sub>-22.5, CaO-31.9, MgO-4.5, Al<sub>2</sub>O<sub>3</sub>-0.53, Fe<sub>2</sub>O<sub>3</sub>-0.63, F-1.43, CO<sub>2</sub>-12.9, I.R.-24.8, Na-0.4, ΣLn<sub>2</sub>O<sub>3</sub>-0.43.

Mineralogical composition of phosphorite, calculated in accordance with its chemical composition, %: phosphorite-45.64, dolomite-9.66, calcite-9.37, glauconite-1.89; quartz-18.79; merwinites - 0.82; phosphosiderite - 4.23[1,2].

The identification of the initial phosphorite by the X-ray diffraction method (Figure 1) confirmed the calculated mineralogical composition of the raw material. The X-ray phase analysis data are confirmed by the results of the IR-spectroscopic study of phosphorite. According to the absorption spectra of infrared radiation (Figure 6), the composition of phosphorite includes fluorapatite with partial replacement of the F<sup>-</sup> group by CO<sup>2-</sup> bands – 603 cm<sup>-1</sup>, 1045 cm<sup>-1</sup>, dolomite-bands – 1455 cm<sup>-1</sup> 877-884 cm<sup>-1</sup>, merwinite and forsterite bands – 460-466 cm<sup>-1</sup>, glauconite – 3550-3600 cm<sup>-1</sup>, quartz-absorption bands – 1085-1090 cm<sup>-1</sup>, phosphosiderite FePO<sub>4</sub> 2H<sub>2</sub>O-bands – 790-797 cm<sup>-1</sup>, 3500-3600 cm<sup>-1</sup>.

The decomposition was carried out in a thermostated vessel equipped with a reflux condenser and a paddle stirrer at a rotation speed of 300 rpm. Phosphorite was introduced in small portions into phosphoric acid heated to 363 ± 2K, and then the reagents were stirred at this temperature for a specified time. After the required decomposition time had elapsed, the reaction was stopped by adding a calculated amount of 0.1n NaOH solution to the suspension to neutralize the acid.

### Results and discussion

At the end of the experiment, the solid phase was separated from the liquid using a vacuum of 0.06 MPa. The filter cake was washed with water, dried, and its P<sub>2</sub>O<sub>5</sub><sup>total</sup> content was determined and P<sub>2</sub>O<sub>5</sub><sup>water</sup> by the standard method according to GOST 21560-82 [2]. According to the results of the analysis, an indicator of the completeness of the process was calculated, i.e. the degree of extraction of P<sub>2</sub>O<sub>5</sub> into solution or the decomposition coefficient according to the formula:

$$K_p = 1 \frac{m_p(P_{2O_5}^{total} - P_{2O_5}^{water})}{m_0 P_{2O_5}^{\phi_{total}}} \quad (1)$$

where  $m_0$  – dry weight of insoluble sediment, g;

$m_p$  – mass of phosphorite taken for decomposition, g;

P<sub>2</sub>O<sub>5</sub><sup>total</sup> и P<sub>2</sub>O<sub>5</sub><sup>water</sup> – content in dry sludge, respectively

P<sub>2</sub>O<sub>5</sub><sup>total</sup> и P<sub>2</sub>O<sub>5</sub><sup>water</sup>, %;

P<sub>2</sub>O<sub>5</sub><sup>φ<sub>total</sub></sup> – content in phosphorite P<sub>2</sub>O<sub>5</sub><sup>total</sup>, %;

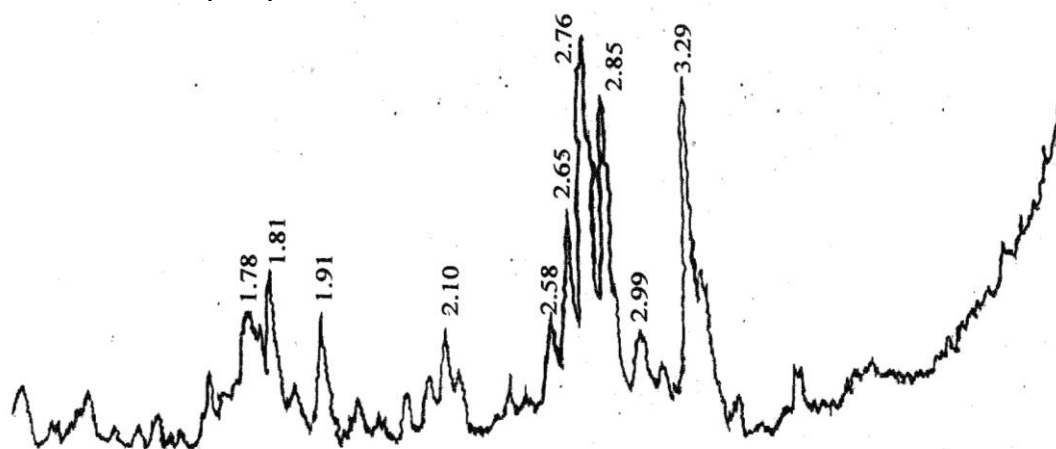


Figure 1 – X-ray diffraction pattern of phosphorite from the Akzhar deposit representative sample obtained from the Akzhar deposit

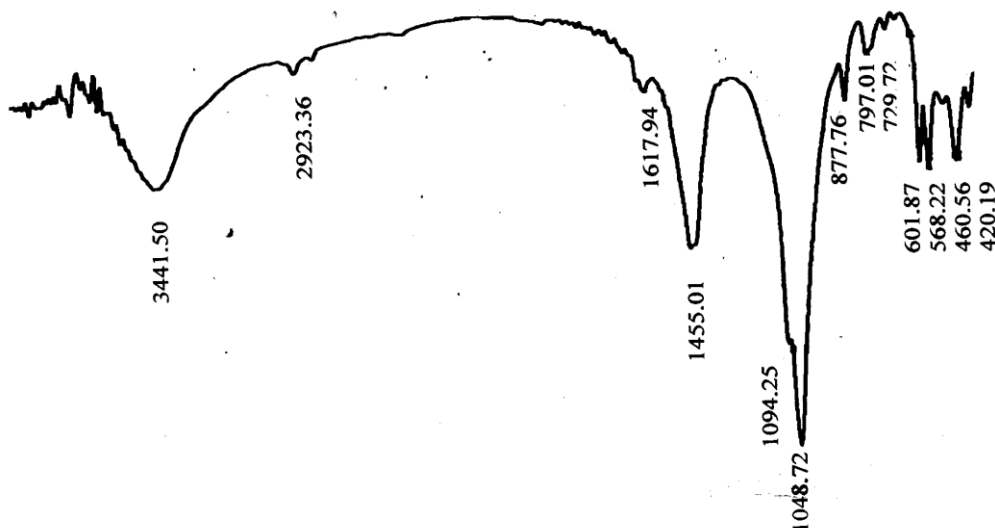


Figure 2 – IR absorption spectrum of phosphorite from the Akzhar deposit

The concentration of phosphoric acid 36.2%  $P_2O_5$  and its norm – 5.0-fold excess were selected on the basis of graphic calculations according to the  $CaO-P_2O_5-H_2O$  solubility diagram to obtain a  $CaO$ -unsaturated solution, from which monocalcium phosphate crystalline hydrate could be isolated during cooling.

The precipitate washing factor was determined by the formula:

$$K_{OTM} = 1 - \frac{P_{2O_5\text{water}} \cdot \frac{m_t}{m_{ph}}}{P_{2O_5\text{water}} \cdot K_{ph}} \quad (2)$$

The kinetics of decomposition of phosphorite at a known rate and concentration of phosphoric acid was studied at the following parameters: temperature – 348-368K, duration of interaction of reagents – 1-50 min.

From the results of the studies given in Table 1 and Figures 3,4 and it follows that with an increase in the duration of the interaction of the reagents in the range from 1 to 50 minutes, the decomposition coefficient increases, and the bulk of the phosphorite decomposes in the first 10 minutes. With a further increase in time, additional decomposition of the raw material occurs, and by 40 minutes, almost complete decomposition of phosphorites is achieved ( $K_p = 0.98$ )[1].

The effect of temperature on the degree of decomposition of phosphorites is insignificant. An increase in temperature from 348 to 368K causes an increase in the decomposition coefficient by only 0.05-0.07%. Thus, the maximum attainable degree of decomposition is 0.98 for 348 K and 0.92 for 368K with a process duration of 30 minutes and is the same – 0.98 with a process duration of 40 minutes.

Almost complete opening of phosphorite is ensured by the use of a 5-fold excess of phosphoric acid. In this case, a solution of monocalcium phosphate is formed, unsaturated with respect to  $CaO$ , the formation of a new solid phase - production monocalcium phosphate, does not occur, the process is significantly intensified due to the exclusion of sludge formation of grains by the newly formed solid phase. This explains the given nature of the dependence shown in Figure 3.

The highest decomposition rate is achieved in the first 5 minutes (4-5 g of phosphorite/min). A sharp decrease in the rate of decomposition of ores after 5 minutes is due, apparently, to a decrease in the activity of hydrogen ions.

Table 1 – Kinetics of the decomposition of phosphorite from the Akzhar deposit with a 5-fold excess of phosphoric acid in the liquid phase due to the neutralization of the first hydrogen ion

№	Temperature, K	Decomposition time, min.	Dry sediment weight, g	Content in i.r., %		Decomposition coefficient	Sludge washing factor	Filtration performance, kg/m <sup>2</sup> h	Decomposition rate of phosphorite, g/min
				P <sub>2</sub> O <sub>5</sub> total	P <sub>2</sub> O <sub>5</sub> water				
1	348	1	46,42	10,38	1,02	0,52			3,58
		2	42,80	9,5	2,61	0,62			3,6
		3	38,32	7,2	1,98	0,75			3,89
		5	28,60	4,7	1,12	0,70			4,28
		10	27,2	2,25	0,59	0,85			2,28
		20	26,75	1,78	0,52	0,89			1,16
		30	26,68	1,25	0,51	0,93			0,77
		40	25,91	0,62	0,36	0,98	0,98	409	0,57
		50	25,42	0,44	0,35	0,99	0,99	410	0,49
2	368	1	46,32	10,25	2,59	0,39			3,68
		2	40,6	10,16	2,07	0,52			2,86
		3	33,8	4,56	1,98	0,75			5,4
		5	28,22	3,25	1,63	0,86			4,36
		10	27,2	2,42	0,92	0,87			2,28
		20	26,20	1,38	0,54	0,921			1,19
		30	24,84	1,26	0,37	0,98			0,83
		40	24,64	0,50	0,30	0,98	0,98	411	0,63
		50	24,38	0,30	0,22	0,99	0,99	415	0,51

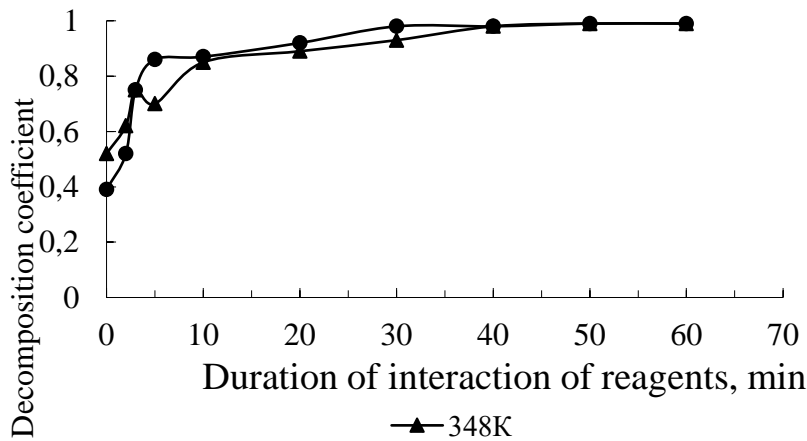


Figure 3 – Kinetics of phosphorite decomposition by 5.0-fold excess of phosphoric acid

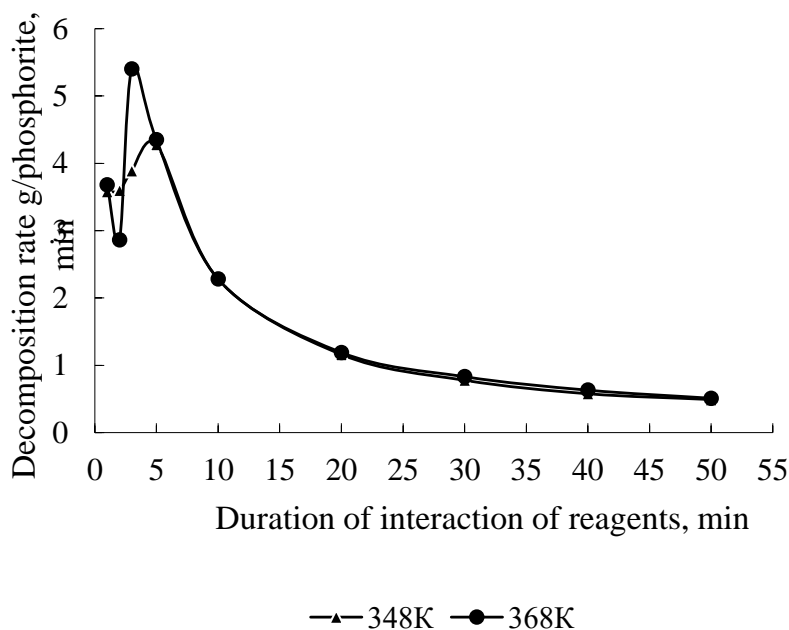


Figure 4 – Change in the rate of decomposition of phosphorite over time

At the same time, the temperature dependence of the degree and rate of decomposition is completely absent in the time interval from 10 to 50 minutes (Figure 4). The high speed of the process is ensured by the selected theoretically substantiated conditions – carrying out the decomposition with phosphoric acid with content of 36.2%  $P_2O_5$ .

## CONCLUSION

Thus, we have established that almost complete decomposition of phosphorites is achieved at a reaction time of 40 minutes and a process temperature of 368K.

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## **АҚЖАР КЕН ОРНЫНЫҢ ТӨМЕН БАҒАЛАНҒАН ФОСФОРИТТЕРІНІҢ ФОСФОР ҚЫШҚЫЛЫНЫҢ АРТЫҚ МӨЛШЕРІМЕН ЫДЫРАУЫН ЗЕРТТЕУ**

### **Түйін**

Мақалада фосфорит кен орны және оның қорлары туралы айтылады. Қазіргі уақытта фосфор мен күрделі тыңайтқыштардың жанданған өндірісі негізінен Қаратау фосфориттеріне негізделген. Алайда, Қаратауда фосфатқа бай кендердің қоры таусылып жатыр, соңғы жылдары айналымға төмен сұрыпты табиғи фосфаттарды тарту қажеттілігі туындап отыр. Сондықтан минералды шикізатты кешенді пайдалану және қолданылатын технологиялардың экологиялық қауіпсіздігін қамтамасыз ету қазіргі кезде өзекті бола түсуде. Мақалада Қаратау бассейніндегі Ақжар фосфорит кен орнының ИҚ спектроскопиясы және XRF талдауы туралы айтылады. Сондай-ақ, Фосфориттің химиялық құрамының нәтижелері және Фосфор қышқылының 5 есе артық мөлшерімен Қаратау бассейнінің Ақжар кен орнындағы фосфориттің ыдырау кинетикасының есептеулері келтірілген.

**Кілттік сөздер:** қышқыл, апатит, минералды тыңайтқыштар, фосфориттің ыдырауы.

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## **ИССЛЕДОВАНИЕ РАЗЛОЖЕНИЯ НИЗКОКАЧЕСТВЕННЫХ ФОСФОРИТОВ МЕСТОРОЖДЕНИЯ АКЖАР ПРИ БОЛЬШОМ ИЗБЫТКЕ ФОСФОРНОЙ КИСЛОТЫ**

### **Аннотация**

В статье рассказывается о месторождении фосфоритов и его запасах. В настоящее время возрожденное производство фосфора и комплексных удобрений основано в основном на Каратауских фосфоритах. Однако запасы богатых фосфатами руд в Каратау истощаются, в последние годы возникает необходимость привлечения в оборот низкосортных природных фосфатов. Поэтому комплексное использование минерального сырья и обеспечение экологической безопасности применяемых технологий в настоящее время становится все более актуальным. В статье рассказывается о ИК-спектроскопии и анализе XRF месторождения фосфоритов Акжар в Каратауском бассейне. Также приведены результаты химического состава фосфорита и расчеты кинетики разложения фосфорита на Акжарском месторождении Каратауского бассейна с 5-кратным избытком фосфорной кислоты.

**Ключевые слова:** кислота, апатит, минеральные удобрения, разложение фосфорита.