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PROCESSING OF ZAGLIK ALUNITE ORE BY HEAP AND TANK LEACHING

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The present work is continuation of our investigations on heap leaching, suggested for the first time. Various variants of alkaline leaching of alunite from alunite ore without preliminary roasting have been studied. In present work the parameters of heap and tank alkaline leaching of alunite ore have been determined. The proposed methods can also be used to extract aluminum from poor alunite rocks. At alunite was washed 17 times with 3% NaOH solution, the degree of weight loss was 30.65%, while with 14 times washing with 10% alkali solution, the weight loss was 47.82 %. The results obtained are also typical for percolation leaching. An increase in alkali concentration and temperature intensifies the leaching process. At a ratio of solid and liquid phases of 1:5, a stirring speed of – 700 cycles/min, and an alkali concentration of 110.9 g/l, at 80°C within 60 minutes' alunite dissolves up to 96% and goes into solution with accompanying components.

Keywords: *alunite, heap and tank leaching, alkali, time, temperature, yield.*

doi

Introduction

Alunite ore, in the past, was used to obtain alumina. It is currently a promising source of natural raw materials for the production of alumina. There are many alunite deposits in the world. The largest of them is the Zaglik ore deposit, located near the Zaglik village of the Dashkesan region of Azerbaijan. According to estimates, its proven reserves amount to more than 170 million tons.

Despite a large number of alunite deposits, the results of technological research carried out in many countries have not led to the development of a real operating plant for processing alunite ore. The reason is that the cost of the proposed technological processes does not justify itself in comparison with the changing market price of the final product. Numerous studies on the extraction of valuable components from alunite ore have been proposed [1–10].

For the first time in the world, the Ganja Alumina Refinery began its activities in 1965 by the alkaline reduction method proposed by Labutin G.V., an employee of VAMI [10] and the alunite processing plant was shut down in 1997 due to several technological violations. Problems (decrease in rich ore resources, exces-

sive consumption of energy resources, loss of useful products, non-use of gases and dust emitted into the atmosphere etc.).

Numerous books, articles and patents have been published on the complex processing of the Zaglik alunite ores [8–10].

According to studies carried out all over the world, the first stage in the processing of alunite ore is grinding, incineration under reducing conditions, and then dissolving the fired material in alkaline and acidic solutions. Transportation of alunite ore with waste over long distances, heating the ore together with loose rocks to 600°C and burning them for several hours, consuming large amounts of fuel and capturing fumes, purchasing reagents and materials from outside (usually from abroad), and, finally, solving the problems which arise in solving environmental problems makes the cost of the process very high. The USA, Canada, Russia, Turkey, China, Iran and other countries have not been able to bring alunite processing to an industrial level due to the complexity and cost of processing.

Even before the emergence of an acute demand for the supply of local aluminum raw materials for the aluminum industry, research into the technology of processing alunite ores

have never been stopped and have constantly been improved.

The creative team, organized by scientists of M.Nagiyev Institute of Catalysis and Inorganic Chemistry and the Institute of Geology and Geophysics of ANAS, continues scientific research on developing a simple and effective alunite technology based on modern electrochemistry and membrane technologies as a result of numerous experimental researches.

One of the relatively simple and economical processing methods which has been developed in recent years is heap leaching, which is based on the dissolution of non-ferrous, rare and precious metals in alkaline, acidic and saline solutions [11–16]. This method can be considered cost-effective even if the concentration of the extracted metal in the raw material is less than 1%.

In the world practice [12, 13, 15–17] heap leaching is a cheap technology for the removal of gold, uranium, copper, nickel and rare earth metals from ores and their processing waste on an industrial scale. In accordance with new technology, all the components contained in the alunite ore are intended to be extracted directly at the deposit by heap leaching and leaching in tanks, and all the chemical reagents required for the ore processing process will be obtained from solutions using the original membrane electrodialysis [18].

The presented article discusses the research and results of the technology of primary processing of alunite, which is simple, inexpensive, efficient and eco-friendly, allowing the exploitation of both poor and rich alunite deposits.

The purpose of work is to determine the kinetic parameters of the dissolution of alunite mineral in alkali by a heap and tank leaching of the raw alunite ore without preliminary roasting, as well as the factors which accelerate the process.

Experimental part

Research methods

The characteristics of raw materials and products of its processing were investigated by the following methods:

- determination of phases was carried out by X-ray phase analysis on an AXS diffractometer (Brooker, Germany);

- the composition of elements in solutions was determined by the ICP-AES method on an ICP-AES 8300 spectrometer (Perkin Elmer, Germany).

- determination of aluminum is performed by complexometric method.

Calculation method

Analysis of Al_2O_3 :

$$[\text{Al}_2\text{O}_3] = \frac{(x - x_1) \cdot T \cdot 10^3 \cdot P}{V} \quad (1)$$

Here: x – the volume of Trilon-B, ml; x_1 – the volume of zinc acetate used for titration, ml; T – the titer of 0.05 M Trilon-B ($T_{\text{Trilon-B}} = 2.55 \cdot 10^{-3}$); P – degree of dilution of aluminate solution; V – the volume of the initially analyzed solution after dilution, ml.

The metals yield (in per cent) is calculated according to the formula (2):

$$\text{Yield, \%} = \frac{(m_{\text{initial}} - m_{\text{fin}})}{m_{\text{initial}}} \cdot 100\% \quad (2)$$

Here m_{initial} and m_{end} is the initial and final masses of metals.

Due to the fact that quartz in alunite rock is insoluble in alkali, and alunite mineral dissolves in alkali even at room temperature, we used heap or tank methods for ore processing.

Materials

The alunite ore used in the research was taken from the Ganja Clay-soil Plant, and some samples were taken directly from the bed according to the hardness and color. The material was crushed, sieved through standard sieves, and samples with different fractions were taken.

The mass fraction of alunite in the selected rock samples varies in a wide range from 22.51% to 51%.

The results of the chemical analysis of the alunite rocks taken are shown in Table 1.

X-ray phase analysis shows that the main minerals of alunite ore are sodium-potassium alunite, kaolinite (dickite), hematite and quartz (Figure 1, *a*).

Table 1. Results of chemical analysis of alunite rocks

Amount, %								
SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SO ₃	CaO	MgO	Na ₂ O+K ₂ O	H ₂ O	Pure alunite
41.4	21.7	5.05	20	0.2	0.12	5.1	6.5	50.2
48.12	23.58	4.21	14.55	0.44	0.15	3.43	5.51	37
31.09	24.09	4.14	27.11	0.13	0.11	6.88	5.4	51.0
52.80	22.47	4.34	10.93	2.56	0.34	2.15	4.04	28.9
50.12	28.99	2.52	10.83	2.96	0.66	2.60	1.31	28.7
50.08	26.78	3.66	8.84	0.46	0.30	2.62	7.27	22.51

Fig. 1. X-ray phase analysis of alunite ore (a) and red mud (b) obtained after alkaline leaching of alunite ore.

Construction of an alunite ore heap.

The heap of ore to be washed is placed on an inclined platform, usually selected in an area close to the bed. A waterproofing layer consisting of clay or a membrane is laid on the surface of the site, and then crushed ore is collected on it. The alkaline solution is sprayed onto the alunite heap, and the leached solution containing alunite components is collected from the drainage hole. The idea of washing alunite ore by heap leaching has been first proposed by Ch.M. Kashkai, A.A. Heydarov and others [17].

At the same time, geological studies of the alunite deposit show that there are favourable conditions for the formation of an alunite heap directly at the site of the deposit [17]. Thus, alunite ore is distributed in a layer on the surface of the earth to a maximum depth of 50 m, on the other hand, there is a layer of clay un-

der the layer of ore, and under it are waterproof volcanic sedimentary rocks. If the proposed technology is tested on an industrial scale, then a dense network of wells must be drilled directly in the alunite deposit, in which small explosions will be produced, as a result of which the structure of a heap with spraying of an alkaline solution can be created directly in the deposit itself.

To implement the proposed technology in practice, initial experiments were carried out in laboratory conditions on model heaps (8 kg of alunite ore) (Figure 2, a) and in percolation, kilns simulating heap leaching (Figure 2, b).

To intensify the leaching process, the washing alkaline solution is supplied in a countercurrent flow (Figure 2, b). In this case, all the alunite particles are completely soaked with the alkaline solution. The experiments were carried out at room temperature.

Fig. 2. Device for heap (a) and percolation (b) leaching of alunite ore.

Different size fractions (1–2.5 cm) of alunite ore of known weight by tank washing method (tank leaching), some experiments were carried out by agitation leaching to speed up the dissolution process, by "Roll bottle" tests in polyethylene containers, with a mechanical mixer (Figure 3, a). During solution, the administration of a new alkaline solution should be renewed every 3–5 days. The results of an alkaline solution of 8 kg alunite ore ball at room temperature did not give effective results due to the fact that the process was carried out without mixing and the particles size was large.

Various fractions (1–2.5 cm) of alunite ore of a certain weight were processed by the

method of tank leaching, and to speed up the dissolution process, some experiments were carried out by mixing ("Roll Bottle" tests) in polyethylene containers with a mechanical mixer (Figure 3, a). In the process of dissolution, it is necessary to resume the introduction of a fresh alkaline solution every 3–5 days. Carrying out the leaching process at room temperature without stirring did not give effective results.

Alunite ore weighing (0.5–8.0 kg), crushed to a particle size of 0.75 mm $\geq$ 1–2.5 cm, was filled into the percolation column, which was irrigated in a drip mode by the alkali solution of various concentrations (3–10% NaOH) (Figure 2, a).

Fig. 3. Leaching of alunite ore by mixing with the alkaline solution ("Roll bottle" tests) (a) and dissolution on the magnetic stirrer at room temperature (b).

The solution was fed into the column in accordance with the parameters of countercurrent heap leaching with a flow speed of 0.4 l/h (Figure 2, *b*). In the course of the experiments, the factors influencing the quality of the obtained solution (concentration of the washing solution, time, pH) and the output of aluminum into the solution were monitored, and the weight of the alunite mass was measured after the end of the process.

During the period of work at the Ganja Aluminum Refinery, rocks containing more than 50% alunite were used as raw materials. An attempt to enrich poor alunite ore (containing less than 50% alunite) was unsuccessful [10].

As shown by the crystal-optical analysis of the samples, the reason was that the alunite mineral has a thin dissemination in the empty rock, and almost all alunite inclusions are interconnected with each other in the empty rock (in quartz) [9].

Results and discussion

Studies have shown that of the four minerals contained in alunite ores (alunite, dickite, quartz and hematite), alunite dissolves rather

well in alkaline solutions and dickite is worse:

So, the author [9] showed that the treatment of ore by a 10% NaOH solution at a temperature of 100°C after 30 min provides 100% decomposition of alunite, and by a 5% NaOH solution under the same conditions, 96.6% of alunite is leached within one hour. In contrast to the author [9], for the first time, we carried out experiments with unfired alunite at room temperature by heap and tank leaching.

Laboratory experiments were carried out with ore fractions 1–2.5 cm, 3 and 10% alkaline solution. The results are shown in Table 2.

At 17-fold leaching of the same mass of alunite ore (200 g) with the 3% alkaline solution, its mass decreases by 30.65%, and at 14-fold leaching with the 10% alkaline solution, the ore mass decreases by 47.82%. The latter shows that aluminum in alunite ore in the form of alunite is almost completely dissolved (Table 2).

Table 2. Results of the tank-leaching experiments (condition: weight of alunite 200 g, room temperature, ore size 1–2.5 cm, alkali concentration 3 and 10%)

*	Leaching of 3% NaOH					Leaching of 10% NaOH				
	Weight change	AlOOH at 130°C	AlOOH total weight	Weight reduction	dissolution share	Weight change	AlOOH at 130°C	AlOOH total weight	Weight reduction	dissolution share
	G	g	g	%	%	g	g	g	%	%
0	200	–				199.8				
1	198.6	1.438	1.438	99.3	0.7	186.07	4.976		93.13	5.931
2	195.48	1.45	2.888	97.99	2.01	176.306	14.2	19.177	88.24	10.87
3	192.34	1.42	4.308	96.17	3.83	162.678	8.1	27.27	81.42	17.76
4	186.47	1.40	5.708	93.24	6.76	155.1	13.92	41.19	77.63	21.59
5	184.78	1.372	7.08	92.39	7.61	142.00	5.876	47.07	71.07	28.22
6	182.06	1.219	8.299	91.00	9.00	132.49	10.824	57.846	66.31	33.52
7	180.01	0.998	9.297	90	10	122.021	7.0	64.896	61.07	38.32
8	176.77	2.667	11.963	88.38	11.614	119.122	5.0	69.846	59.62	39.78
9	175.24	0.603	12.566	87.62	12.38	114.998	4.8	74.696	57.55	41.862
10	172.89	1.244	13.81	86.44	13.55	110.75	6.43	81.126	55.43	44.01
11	169.05	2.525	16.335	84.525	15.475	107.31	4.01	85.136	53.70	45.75
12	153.01	1.89	18.135	76.505	23.495	106.14	3.59	88.726	53.12	46.34
13	150.40	2.5	20.635	75.2	24.8	104.5	7	95.726	52.30	47.17
14	148.46	2.2	22.838	74.23	25.72	103.2	3.59	99.316	51.65	47.82
15	146.63	2.2	25.035	73.315	26.685					
16	144.39	2.1	27.135	72.19	27.805					
17	138.70	1.515	28.65	69.355	30.651					

* leaching multiplicity

The pH of the solutions obtained from each leaching was measured, aluminum hydroxide was precipitated, and its weight was determined after drying in an oven at 130°C. At 130°C, amorphous aluminum hydroxide converts to crystalline boehmite (AlOOH).

The mass of the precipitated AlOOH makes up 28.65 g, the weight loss – 30.65%. According to theoretical calculations, from 100 g of alunite 57.97 g of AlOOH is obtained. In our experiments 28.65 g was obtained. This mass corresponds to the yield of 49.42% Al from alunite ore (Table 2). As can be seen from the table, the weight of the insoluble residue (red mud) is reduced to 50%. X-ray phase analysis of the alkaline leaching residue of alunite confirms that it does not contain the mineral alunite (Figure 1, b).

To accelerate the washing process, experiments were carried out with "Roll bottle" tests by the mechanical stirring of Zaglik alunite ore with a size of more than 0.75 mm in a laboratory setup (Figure 3). Alunite ore weighing 100 g was leached with the solution of 10% alkali at room temperature for 32 hours. Al(OH)₃ was precipitated from the obtained solutions, the precipitate was filtered, and after drying at 130°C, the mass of AlOOH and the percentage of sulfur yield were determined. The results are shown in Table 3. From Table 3 it follows that the mass of AlOOH, passed into solution when mixing alunite with a 10% alkaline solution for 32 hours and precipitated at 200°C, is 14.69 g (practically). As a result of theoretical calculations, it was determined that 21.74 g of AlOOH can be obtained from 100 g of ore containing 50% of the mineral alunite. And this indicates to that the AlOOH yield is 67.57% ($\eta = \frac{14.69}{21.74} \cdot 100 = 67.57\%$). If the leaching time is extended and a new portion of alkali is used

for leaching, alunite can completely go from alunite ore into solution.

To increase the yield of aluminum from alunite ore into solution and accelerate the process, the experiments were carried out in a mixer equipped with a thermostat (Figure 3, b). The studies were carried out at various concentrations of alkali [$C_{\text{KOH}} = 52.96$ g/l (5%), 110.9 g/l (10%), 174.6 g/l (15%), 243.8 g/l (20%)]. A 100 ml beaker was placed in a thermostat with an alkaline solution of mentioned above concentration, and after the achieving temperature of the solution of the desired mode was reached, the time of the experiment was recorded. The results are shown in Figure 4.

Test experiments have shown that when the number of mixer cycles is 700 min⁻¹, the rate of transition of alunite into the solution does not depend on the stirring rate. Therefore, the experiments were carried out with the number of rotations of 700 min⁻¹.

It is seen from Figure 4 that an increase in the concentration of alkali accelerates the decomposition of the alunite mineral and the transition of aluminum into solution. To avoid high consumption of alkali, the experiments were carried out with 5% and 10% solutions. Another most important parameter that determines the leaching of alunite from the primary rock is the temperature (Figure 5). If at 90°C the solubility of alunite is 96% in 40 minutes, at 80°C this yield is 82%. By increasing the dissolution time to 90 minutes, a yield at 90°C and a 80°C, equal to (96%) can be obtained.

According to the laws of physical chemistry, at a solid dissolves, the concentration of chemical reaction products increases in solution, and in a stable closed system, the intensity of solubility decreases as the equilibrium is reached.

Table 3. Roll bottle test results ($m = 100$ g, $t = 20^\circ\text{C}$, $V_{\text{mixing}} = 20$ period/min)

Time, h	Insoluble residue, g	Weight of AlOOH at 160°C, g	The mass of sulfur in the solution, g	Sulfur content in the solution, %
1	90.653	5.49	1.239	16.03
2	89.909	7.74	1.36	17.59
3	88.514	11.44	1.82	23.55
7	84.89	13.05	2.07	26.78
24	79.08	15.02	2.33	30.15
32	78.23	14.67	2.808	36.33

Fig. 4. Dependence of the yield of alunite mineral from alunite ore on the concentration of alkali (temperature = 60°C, S:L= 1:5).

Fig. 5. Dependence of the leaching of the alunite mineral from the alunite ore on temperature ($C_{\text{KOH}} = 110.9 \text{ g/l}$, S:L = 1: 5).

To prevent this, many researchers try to increase the temperature and concentration of the alkali. However, if ions (AlO_2^- and SO_4^{2-}), which pass into solution during the reaction, are to be immediately removed from the system, then the reaction parameters will not change but will always remain in the intense zone. In this case, increasing the temperature does not make much sense, and the concentration of alkali can be reduced [19].

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ZƏYLİK ALUNIT FİLİZİNİN TOPA VƏ ÇƏN HƏLLOLMA YOLU İLƏ EMALI

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Hazırkı iş topa və çən həllolma üzrə apardığımız tədqiqatların davamıdır. Alunitin alunit filizindən əvvəlcədən yandırılmadan qələvi həllolmasının müxtəlif variantları öyrənilmişdir. İlk dəfə olaraq, topa və çən həllolma alunit filizinin qələvidə həllolmasının topa və çən həllolma parametrləri araşdırılmışdır. Təklif olunan üsullarla alüminiumu kəsib alunit filizindən də yuyub çıxarmaq mümkündür. 3%-li NaOH məhlulu ilə 17 dəfə çən yuma üsulu ilə alunitin çəki azalma faizi 30.65% edirsə, 10%-li NaOH-la 14 dəfə yumaqla bu azalma 47.82% edir. Alınan nəticələr perkolyasiya yolu ilə yumada da effektiv olur. Məhluldakı qələvinin qatılığının və temperaturun artması həllolma prosesini intensivləşdirir. Bərk-maye fazalar nisbəti 1:5, qarışma sürəti 700 dövr/dəq, qələvinin qatılığı 110.9 q/l olduqda 80°C temperaturda 60 dəq müddətində 96%-ə kimi alunit mineralı həll olub komponentləri ilə birlikdə məhlula keçir.

Açar sözlər: alunit; topa və çən həllolma, qələvi, vaxt, temperatur, çıxım faizi

ПЕРЕРАБОТКА ЗАГЛИКСКОЙ АЛУНИТОВОЙ РУДЫ КУЧНЫМ И ЧАНОВЫМ ВЫЩЕЛАЧИВАНИЕМ

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Работа является продолжением наших исследований по кучному выщелачиванию, предложенному впервые. Исследовались различные варианты щелочного выщелачивания алунита из алунитовой руды без предварительного обжига. В настоящей работе определены параметры кучного и чанового щелочного выщелачивания алунитовой руды. Предлагаемыми методами также можно извлекать алюминий из бедноалунитовых пород. При 17-кратном промывании алунита 3%-ным раствором NaOH степень снижения веса составила 30.65%, а при 14-кратном промывании 10%-ным раствором щелочи снижение веса составило 47.82%. Повышение концентрации щелочи и температуры интенсифицируют процесс выщелачивания. При соотношении твердой и жидкой фаз 1:5, скорости перемешивания -700 цикл/мин и концентрации щелочи – 110.9 г/л, при 80°C в течение 60 мин алунит растворяется до 96% и переходит в раствор с сопутствующими компонентами. Полученные результаты характерны также для перколяционного выщелачивания.

Ключевые слова: алунит, кучное и чановое выщелачивание, щелочь, время, температура, выход.