UDC 669.712
DOI: 10.31643/2021/6445.22

IRSTI 53.37.91

Modification of the phase composition of low-grade gibbsite-kaolinite bauxites

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Received: *25 March 2021*
Peer reviewed: *14 May 2021*
Accepted: *15 June 2021*

ABSTRACT

The known methods of processing low-quality bauxite have a number of disadvantages that make it difficult or impossible to use them in the conditions of production of JSC "Aluminum of Kazakhstan", therefore, it is necessary to effectively develop a technology for preliminary enrichment. Enrichment of low-quality bauxites is possible by separating the clayey - high-siliceous and coarse-crystalline fractions. Electron-microscopic analysis of the original bauxite showed that the coarse-crystalline fraction is tightly pressed by the fine one. The total silicon modulus of the sample is 2.49. The silicon modulus of the fine fraction is 2.19, and that of the coarse fraction is 3.89. When enriching bauxite, as a result of the separation of fines, it is possible to increase the silicon modulus. After the chemical activation of bauxite in a sodium bicarbonate solution, the finely dispersed fraction is effectively separated from the coarse one and the phase composition changes - the calcium silicate phase disappears and the calcite phase is formed. With an increase in the activation temperature, the content of kaolinite and siderite decreases, and the content of quartz and hematite increases. Studies have shown that at temperatures of chemical activation of 120 ° C, duration of more than 120 minutes and 200 ° C, duration of more than 40 minutes, a dawsonite phase is formed in bauxite, which densifies the mineral structure. When determining the optimal regime for carrying out chemical activation, it is necessary to take into account the negative possibility of dawsonite formation.

Keywords: Gibbsite-kaolinite bauxite, chemical activation, sodium bicarbonate, phase composition, conditioning, technology.

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Introduction

At present, at the Pavlodar aluminum plant of Aluminum of Kazakhstan JSC, the bauxites of the Krasnogorsk deposit are processed according to the sequential-combined method Bayer-sintering. Bauxites are characterized by a low silicon modulus and a high content of harmful components: siderite, chamosite, hematite, pyrite, organic and other impurities, and their quality continues to decline, which leads to a sharp deterioration in the composition of solutions, industrial products and a decrease in technical and economic indicators [1]. This is the basis for carrying out a set of theoretical and technological research for the development of an effective technology.

The known method of conditioning low-quality bauxite, combining washing of clay components and hydroalkaline decarbonization. The method makes it possible to simultaneously increase the silicon modulus and reduce the carbonate content in bauxite. The main disadvantages of this technology are: low degree of washing due to the use of alkaline wash water; increase in material flow [2].

Processing of low-quality bauxite is possible by the method of separate leaching of clay and stony bauxite fractions, however, the separation of bauxite fractions in an alkaline does not provide the required degree of separation of the clay part [3, 4].

Thus, the known methods of processing low-quality bauxite have a few disadvantages that make it difficult or impossible to use them. It is required to create new solutions to ensure efficient processing of low-quality gibbsite - kaolinite bauxite from the Krasnogorsk deposit to obtain high-quality alumina.

To involve in the processing of most bauxite deposits in Kazakhstan, we propose a technology for processing low-quality gibbsite - kaolinite bauxite with two-stage concentration and hydrochemical processing, including preliminary chemical activation [5, 6].

Experimental part and discussion of results

The purpose of the research was to develop a technology for conditioning gibbsite-kaolinite bauxites of the Krasnogorsk deposit before concentration and hydrochemical processing. To achieve the goal, the method of preliminary chemical activation in a solution of sodium bicarbonate was used.

The work was carried out on a representative sample of Krasnogorsk bauxite of composition, wt. %: Al_2O_3 42.0; SiO_2 11.5; Fe_2O_3 19.5; CaO 1.08; Na_2O 0.22; MgO 0.18; K_2O 0.03; TiO_2 2.05; SO_3 0.24; Cl^- 0.04; pp 23.16; µSi 3.65.

The work used: chemical, X-ray fluorescence, thermal, electron microscopic and IR analyzes.

Chemical analysis of the samples was carried out on an Optima 2000 DV inductively coupled plasma optical emission spectrometer (PerkinElmer, USA).

X-ray fluorescence analysis was performed on a Venus 200 PANalyical B.V. (PANalyical B.V., Holland).

X-ray experimental data were obtained on a BRUKERD8 ADVANCE apparatus using copper radiation at an accelerating voltage of 36 kW and a current of 25 mA.

IR analysis was obtained on an Avatar 370 FT-IR spectrometer in the spectral range of 4000-400 cm^{-1} from preparations prepared from 200 mg of KBr and 2 mg of a sample. Experiment attachment: Avatar Diffuse Reflectance.

Thermal analysis was performed using an STA 449 F3 Jupiter synchronous thermal analysis instrument.

The micrographs were taken on a JEOL JSM-6610LV thermal emission cathode (LaB6) scanning low-vacuum electron microscope.

The chemical activation of bauxite was carried out in a solution containing 120 g / dm^3 $NaHCO_3$ at a ratio L: T = 10.0 and temperatures of 90 - 200 ^{0}C using a thermostatted installation with 6 autoclaves rotating through the head, with a working volume of 250 cm^3. The required content of sodium bicarbonate in the solution of 120 g / dm^3 was selected taking into account its solubility limit.

The silicon modulus of the samples was determined from the ratio Al_2O_3/SiO_2.

The study of the material composition of the bauxite sample was carried out.

The X-ray phase composition of gibbsite - kaolinite bauxite is presented in Table 1.

Table 1 - Phase composition of gibbsite - kaolinite bauxite from Krasnogorsk deposit

Name	Formula	%
Gibbsit	$Al(OH)_3$	54.95
Kaolinite-1A	$Al_2Si_2O_5(OH)_4$	10.02
Siderite	$FeCO_3$	6.09
Hydroaluminosilicate	$Al_2Si_2O_5(OH)_4$	5.17
Quartz	SiO_2	5.10
Calcium silicate	Ca_5Si_3	5.07
Hematite	Fe_2O_3	4.89
Titanium oxide	TiO_2	4.88
Ferrotitanium oxide	Fe_2TiO_5	3.83

The infrared spectrum of a bauxite sample is shown in Figure 2.

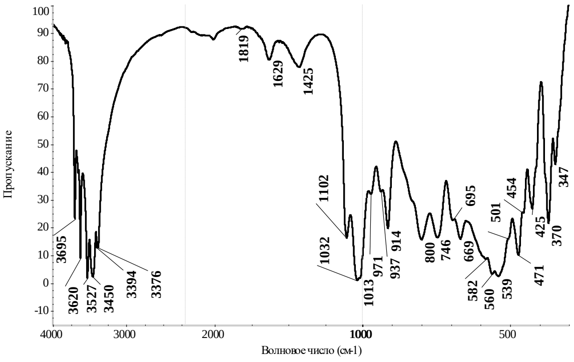


Figure 2 - Infrared spectrum of bauxite from Krasnogorsk deposit

Infrared spectroscopy determined the presence of the following phases: gibbsite γ-Al $(OH)_3$; kaolinite $Al_4[(OH)_8 \mid Si_4O_{10}]$; goethite α-FeOOH; siderite $FeCO_3$ [7]. Present: hematite Fe_2O_3 [8]; anatase TiO_2 [9]; diaspores $\alpha\text{-}AlOOH^{-1}$ [7].

The study of a bauxite sample by the thermal analysis method (Figure 3) showed: on the DTA curve there are intense endothermic effects with a maximum development at 334.5 ° C, 557 °C; on the dDTA curve there are additional exothermic effects with peaks at 897.8 °C and 989.9 °C; the DTG curve recorded minimums at 287.9 °C, 319.4 °C, 532.8 °C.

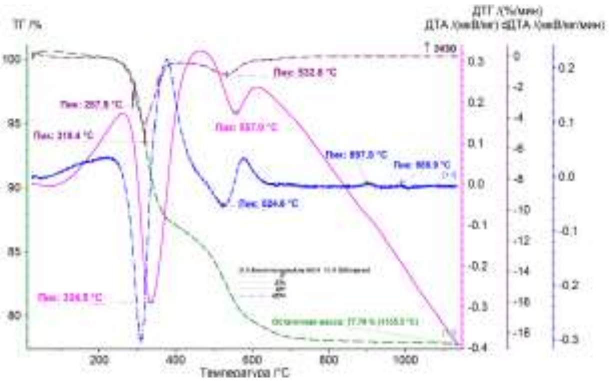
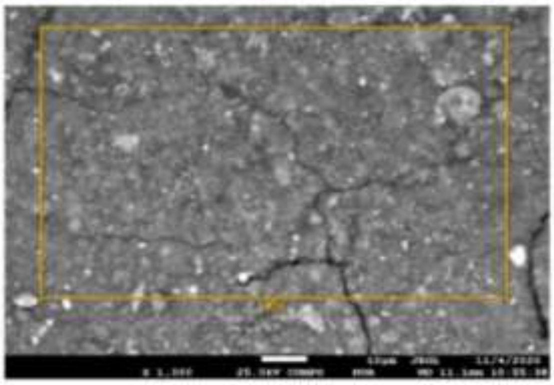
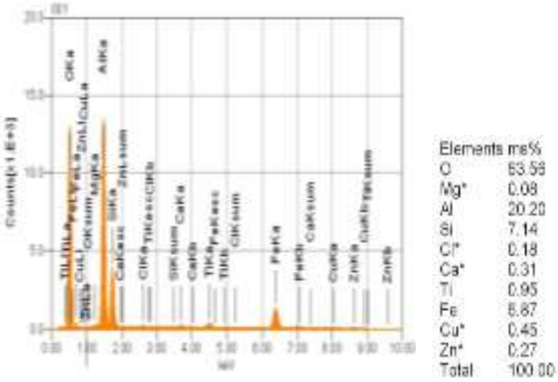


Figure 3 - Thermogram of the initial sample of gibbsite - kaolinite bauxite from Krasnogorsk deposit

A deep endothermic effect with a maximum development at 334.5 ° C, in the development area of which two minima appeared on the DTG curve (287.9 ° C, 319.4 ° C), reflects the dehydration of gibbsite and iron hydroxide. The combination of the endothermic effect with an extremum at 557 ° C on the DTA curve and a weak exothermic effect with a peak at 989.9 ° C on the dDTA curve mb. manifestation of kaolinite. In addition, the combination of the same endothermic effect with an exothermic effect with a peak at 897.8 ° C on the dDTA curve may reflect the presence of coarse-crystalline siderite.

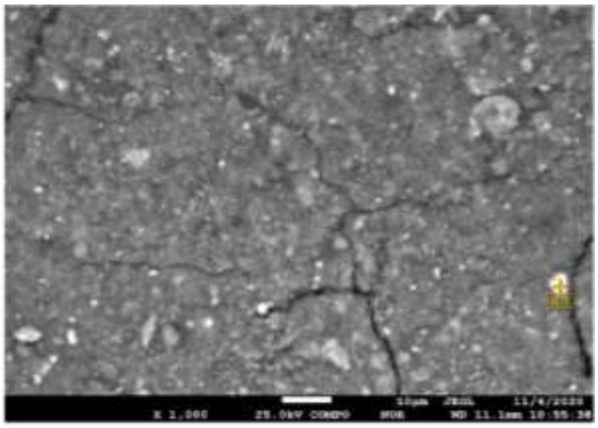
Micrographs and electron microscopic analysis of bauxite are shown in Figure 1 (a, b, c).

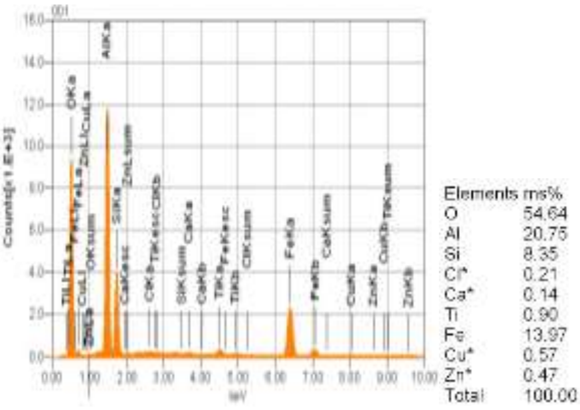




Elements	ms%
O	63.56
Mg*	0.08
Al	20.20
Si	7.14
Cl*	0.18
Ca*	0.31
Ti	0.95
Fe	6.87
Cu*	0.45
Zn*	0.27
Total	100.00

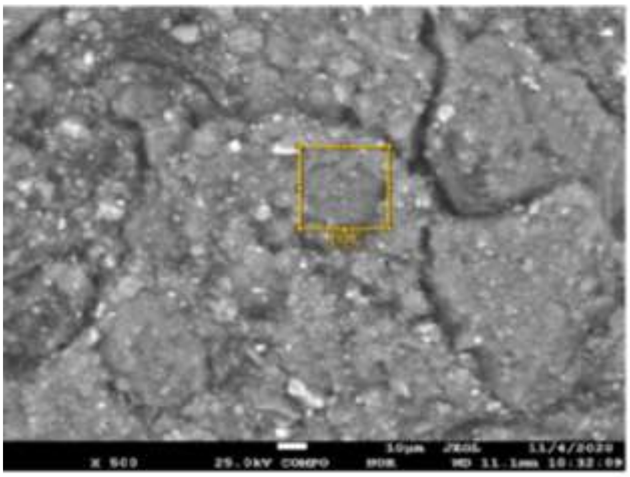
A

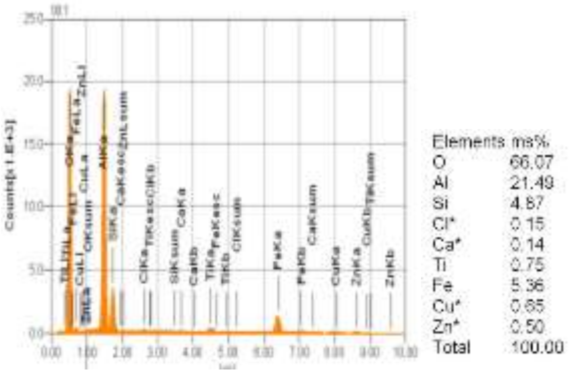




Elements	ms%
O	54.64
Al	20.75
Si	8.35
Cl*	0.21
Ca*	0.14
Ti	0.90
Fe	13.97
Cu*	0.57
Zn*	0.47
Total	100.00

B





Elements	ms%
O	66.07
Al	21.49
Si	4.87
Cl*	0.15
Ca*	0.14
Ti	0.75
Fe	5.36
Cu*	0.65
Zn*	0.50
Total	100.00

C

a - general composition; b - large fraction; c - fine fraction

Figure 1 - Micrographs and electron microscopic analyzes of gibbsite - kaolinite bauxite from Krasnogorsk deposit

The micrographs show that the coarse-crystalline fraction of bauxite is tightly pressed with a fine fraction. The total silicon modulus of the sample area (Figure 1 - a) is 3.45. The silicon modulus of the coarse fraction (Figure 1-b) is 4.41, of the fine fraction (Figure 1-c) - 2.19.

In order to increase the silicon modulus of bauxite by separating the fine fraction, the initial sample was subjected to chemical activation in a sodium bicarbonate solution.

To determine the optimal conditions for the chemical activation of bauxite, studies of the effect of temperature and process duration were carried out.

Studies of the effect of the temperature of chemical activation on the change in the material composition of bauxite were carried out at temperatures of 90-200 ° C and duration of 60 minutes.

The chemical composition of bauxite depending on the activation temperature is presented in Table 2.

Table 2 - Chemical composition of bauxite depending on the activation temperature

Name	Activation temperature, ° C					
	Initial	90	120	150	180	200
	Content, %					
Al_2O_3	42.0	42.4	42.0	42.2	42.6	42.8
SiO_2	11.5	11.4	11.3	11.3	11.4	11.5
Fe_2O_3	19.5	19.7	19.9	19.7	19.9	19.9
CaO	1.08	1.1	1.1	1.2	1.1	1.2
Na_2O	0.22	0.7	1.06	1.18	1.5	6.4
MgO	0.18	0.18	0.19	0.17	0.19	0.18
SO_3	0.24	0.17	0.18	0.18	0.19	0.16
K_2O	0.03	0.03	0.02	0.03	0.05	0.05
TiO_2	2.05	2.25	2.3	2.2	2.1	2.2
Cl^-	0.04	0.02	0.02	0.016	0.017	0.03
LOI	23.16	22.05	21.93	21.824	20.953	15.58

As a result of chemical activation obtained, that the content of the elements in the samples, except sodium oxide, remained at the same level. At an activation temperature of 200 ° C, a thick slurry was obtained.

The phase composition of bauxite depending on the temperature of chemical activation is presented in Table 3.

Table 3 - Phase composition of bauxite depending on the temperature of chemical activation

Name	Activation temperature, ^{0}C					
	Initial	90	120	150	180	200
	Content, %					
Gibbsite $Al(OH)_3$	54.95	55.59	56.45	55.7	52.38	55.34
Kaolinite-1A $AL_2Si_2O_5(OH)_4$	15.19	12.11	12.43	12.44	10.79	10.86
Siderite $FeCO_3$	6.09	11.52	9.77	7.69	6.82	7.29
Quartz SiO_2	5.11	4.8	4.91	6.37	7.63	8.04
Calcium silicate Ca_5Si_3	5.07	-	-	-	-	-
Hematite Fe_2O_3	4.89	5.09	5.89	6.23	6.99	7.22
Titanium oxide Ti_6O_{11}	4.88	4.69	4.59	4.57	4.51	4.64
Ferrotitanium oxide Fe_2TiO_5	3.82	3.67	3.70	3.80	3.76	3.41
Calcite $CaCO_3$	-	2.53	2.26	3.20	3.21	3.20

It follows from Table 3 that with an increase in the temperature of chemical activation in the phase composition of bauxite, the content of kaolinite and siderite decreases, the content of quartz and hematite increases, the calcium silicate phase disappears and the calcite phase is formed.

The influence of the duration of chemical activation on the change in the material composition of bauxite at temperatures of 120 and 200 ° C has been investigated.

The chemical composition of bauxite, depending on the duration of activation at a temperature of 120 ° C, is presented in Table 4.

Table 4 - Chemical composition of bauxite depending on the duration of activation at a temperature of 120 ° C

Name	Duration, min.						
	20	40	60	90	120	180	240
	Content, %						

Al_2O_3	42.2	42.25	42.24	42.4	42.4	38.8	36.3
SiO_2	11.2	11.1	11.3	11.3	11.1	10.9	10.6
Fe_2O_3	20.3	20.3	19.8	19.6	20.0	18.6	17.1
CaO	1.3	1.14	1.1	1.1	1.17	1.06	0.9
Na_2O	0.413	0.45	1.16	1.3	1.43	4.35	6.8
MgO	0.13	0.18	0.19	0.16	0.18	0.2	0.16
SO_3	0.19	0.16	0.18	0.17	0.17	0.04	0.19
K_2O	0.07	0.06	0.02	0.04	0.03	0.04	0.06
TiO_2	2.5	2.4	2.6	2.2	2.5	2.2	2.06
Cl^-	0.02	0.02	0.02	0.02	0.02	0.02	0.04
LOI	21.677	21.94	21.39	21.71	21.0	23.79	25.79

With an increase in the duration of activation to 180 minutes at a temperature of 120 ° C, the content of Na_2O in bauxite increases, and the content of the remaining elements remains the same. With an activation duration of 180 minutes or more, a thick pulp was obtained. the mass of which is greater than the initial mass of bauxite, therefore, the percentage of elements decreases.

The phase composition of bauxite depending on the duration of chemical activation at a temperature of 120 ° C is presented in Table 5.

Table 5 - Phase composition of bauxite depending on the duration of chemical activation at a temperature of 120 ° C

Name	Activation temperature, ^{0}C						
	20	40	60	90	120	180	240
	Content, %						
Gibbsite $Al(OH)_3$	51.43	51.42	51.45	51.91	50.69	45.62	43.76
Kaolinite-1A $AL_2Si_2O_5(OH)_4$	11.67	11.98	10.94	10.22	10.37	9.06	9.3
Siderite $FeCO_3$	4.89	4.58	4.77	5.00	6.56	6.02	-
Quartz SiO_2	5.28	5.18	5.61	5.94	6.02	6.55	6.05
Hematite Fe_2O_3	10.02	10.25	10.23	10.1	9.81	9.35	8.09
Titanium oxide Ti_6O_{11}	6.99	7.0	7.09	6.94	6.99	7.11	7.1
Ferrotitanium oxide Fe_2TiO_5	6.32	6.2	6.25	6.6	6.54	6.23	6.71
Calcite $CaCO_3$	3.4	3.39	3.66	3.29	3.02	4.4	4.5
Dawsonite $NaAlCO_3(OH)_2$	-	-	-	-	-	5.66	14.49

As a result of chemical activation, the content of the gibbsite phase remains at the same level for a duration of 120 minutes, further, its amount decreases and a new aluminum-containing phase appears - dawsonite.

The chemical composition of bauxite, depending on the duration of activation at a temperature of 200 ° C, is presented in Table 6.

With an activation duration of 60 minutes, a thick pulp was obtained. A solid phase was obtained at a duration of 90 minutes, which filled the entire volume of the autoclave, there was no solution (Figure 4).

Table 6 - Chemical composition of bauxite depending on the duration of activation at a temperature of 200 ° C

Name	Duration, min.			
	20	40	60	90
	Content, %			
Al_2O_3	44.1	44.38	44.83	44.8
SiO_2	11.0	11.1	10.9	10.7
Fe_2O_3	20.1	19.4	19.9	11.08
CaO	1.14	1.1	1.2	0.6
Na_2O	0.2	0.9	6.4	23.1
MgO	0.18	0.18	0.18	0.12
SO_3	0.17	0.17	0.16	0.2
K_2O	0.04	0.05	0.05	0.01
TiO_2	2.6	2.53	2.5	1.64
Cl^-	0.02	0.02	0.02	0.01
п.п.	20.45	20.17	13.86	7.74

Figure 4 - Solid phase of bauxite activation. obtained at 200 ° C and duration 90 minutes

An increase in the final weight of bauxite after activation at a temperature of 200 ° C and a duration of 60 minutes or more led to a decrease in the percentage of elements, at the same time, the sodium oxide content increased sharply, which is associated with the formation of a new phase - dawsonite (table 7).

Table 7 - Phase composition of bauxite depending on the duration of chemical activation at a temperature of 200 ° C

Name	Temperature, ^{0}C			
	20	40	60	90
	Content, %			

Gibbsite $Al(OH)_3$	54.59	54.38	50.34	15.3
Kaolinite-1A $AL_2Si_2O_5(OH)_4$	9.58	8.85	9.8	6.34
Siderite $FeCO_3$	8.8	8.86	-	-
Quartz SiO_2	4.05	4.36	4.8	3.2
Hematite Fe_2O_3	5.45	6.28	6.22	5.45
Titanium oxide Ti_6O_{11}	2.69	2.83	2.4	1.2
Ferrotitanium oxide Fe_2TiO_5	8.49	8.19	8.41	2.8
Calcite $CaCO_3$	6.35	6.25	3.26	2.82
Dawsonite $NaAlCO_3(OH)_2$	-	-	14.77	62.89

Micrographs and electron microscopic analysis of bauxite after chemical activation are shown in Figure 5.

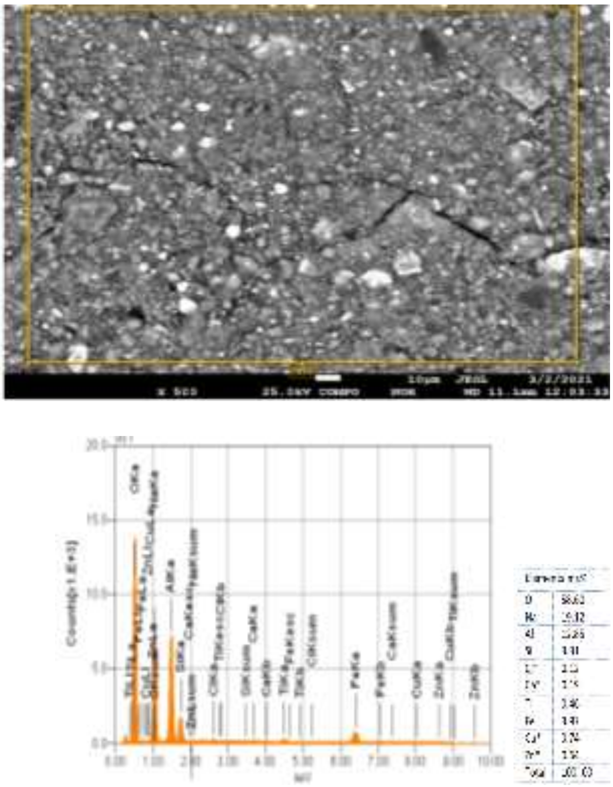


Figure 5 - Micrograph and electron microscopic analysis of gibbsite - kaolinite bauxite of the Krasnogorsk deposit after chemical activation at a temperature of 200 ° C and a duration of 40 minutes

The photomicrograph shows, that after chemical activation of bauxite there was a separation of the fine fraction from the coarse one, which will improve the efficiency of gravity and chemical processing, low-quality gibbsite - kaolinite bauxite from Krasnogorsk deposit for further processing to produce alumina.

Conclusions

The phase composition of gibbsite - kaolinite bauxite of the Krasnogorsk deposit is represented by gibbsite, kaolinite, siderite, hydroaluminosilicate, quartz, calcium silicate, hematite, titanium oxide and ferrotitanium oxide.

Electron-microscopic analysis of the original bauxite showed that the coarse-crystalline fraction is tightly pressed by the fine one. The total silicon modulus of the sample is 3.45. The fines silicon module is 2.19, large fraction - 4.41.

After the chemical activation of bauxite in a sodium bicarbonate solution, the finely dispersed fraction is effectively separated from the coarse one and the phase composition changes - the calcium silicate phase disappears and the calcite phase is formed.

With an increase in the activation temperature, the content of kaolinite and siderite decreases, and the content of quartz and hematite increases.

At temperatures of chemical activation of 120 ° C, duration of more than 120 minutes and 200 ° C, duration of more than 40 minutes, the dawsonite phase is formed in bauxite, which densifies the mineral structure and makes it impossible to carry out further enrichment by gravity methods. therefore, when choosing the activation conditions, it is necessary to exclude the formation of this phase.

After chemical activation of bauxite, the separation of the finely dispersed fraction from the coarse one took place, which will increase the efficiency of gravity and chemical enrichment of low-quality gibbsite - kaolinite bauxite from the Krasnogorsk deposit for further processing to produce alumina.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

Acknowledgement. *This study was funded by the Science Committee of the Ministry of Education and Science of the Republic of Kazakhstan (Grant No. AR08856046).*

Cite this article as: Abdulvaliyev R.A., Dyussenova S.B., Manapova A.I., Akcil A., Beisenbiyeva U.Zh. Modification of the phase composition of low-grade gibbsite-kaolinite bauxites. *Kompleksnoe Ispol’zovanie Mineral’nogo Syr’a. = Complex Use of Mineral Resources = Mineraldik Shikisattardy Keshendi Paidalanu.* - 2021. № 2 (317). pp. 94-102. https://doi.org/10.31643/2021/6445.22

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Мақала келді: *25 наурыз 2021*
Сараптамадан өтті: *14 мамыр 2021*
Қабылданды: *15 маусым 2021*

ТҮЙІНДЕМЕ

Төмен сапалы бокситті өңдеудің белгілі әдістерінің бірқатар кемшіліктері бар. Оларды «Қазақстан алюминийі» АҚ өндіріс жағдайында қолдану қиын немесе мүмкін емес, сондықтан алдын ала байыту технологиясын әзірлеу қажет. Төмен сапалы бокситті байыту - сазды жоғары кремнийлі және ірі кристалды фракцияларға бөлу арқылы мүмкін болады. Бастапқы бокситтің электронды-микроскопиялық талдауы ірі кристалды фракцияның майда бөлшектермен тығыз басылып қалатындығын көрсетті. Үлгінің жалпы кремний модулі 3,45 құрайды. Ұсақ фракцияның кремний модулі 2,19, ал ірі фракцияның 3,89-ды құрайды. Бокситтегі ұсақ бөлшектерді бөлу арқылы кремний модулін көбейтуге болады. Натрий гидрокарбонатының ерітіндісінде бокситті химиялық активтендіргеннен кейін ұсақ дисперсті фракция ірі бөліктен тиімді бөлінеді және фазалық құрамы өзгереді - кальций силикатының фазасы жойылып, кальцит фазасы түзіледі. Белсендіру температурасы жоғарылағанда каолинит пен сидериттің мөлшері азаяды, ал кварц пен гематиттің мөлшері артады. Зерттеулер көрсеткендей, химиялық активтендірудің температурасы 120° С, ұзақтығы 120 минут және 200° С, ұзақтығы 40 минуттан артық болғанда бокситте даусонит фазасы түзіліп, минералдық құрылымды тығыздайды. Химиялық активтендірудің оңтайлы режимін анықтағанда, даусониттің жағымсыз түзілу мүмкіндігін ескеру қажет.

Түйін сөздер: Гиббсит-каолинитті боксит, химиялық активация, натрий гидрокарбонаты, фазалық құрамы, кондициялау, технология.

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Модифицирование фазового состава низкокачественных гиббсит-каолинитовых бокситов

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Поступила: *25 марта 2021*
Рецензирование: *14 мая 2021*
Принята в печать: *15 июня 2021*

АННОТАЦИЯ

Известные методы переработки низкокачественных бокситов имеют ряд недостатков, делающих затруднительным или невозможным их применение в условиях производства АО «Алюминий Казахстана», поэтому необходима эффективная разработка технологии предварительного обогащения. Обогащение низкокачественных бокситов возможно осуществить путем разделения глинистой – высококремнистой и крупнокристаллической фракций. Электронно – микроскопический анализ исходного боксита показал, что крупно – кристаллическая фракция плотно запрессована мелкой. Общий кремниевый модуль пробы составляет 3,45. Кремниевый модуль мелкой фракции равен 2,19, крупной– 4,41. В результате отделения мелкой фракции боксита возможно повысить его кремниевый модуль. При химической активации боксита в растворе гидрокарбоната натрия происходит эффективное отделение мелкодисперсной фракции от крупной и изменение фазового состава – исчезает фаза силиката кальция и образуется фаза кальцита. С увеличением температуры активации уменьшается содержание каолинита и сидерита, увеличивается содержание кварца и гематита. Исследования показали, что при температурах химической активации 120^0 С, продолжительности более 120 минут и 200^0 С, продолжительности более 40 минут в боксите образуется фаза даусонита, которая уплотняет минеральную структуру. При определении оптимального режима проведения химической активации необходимо учитывать негативную возможность образования даусонита.

Ключевые слова: Гиббсит-каолинитовый боксит, химическая активация, гидрокарбонат натрия, фазовый состав, кондиционирование, технология.

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