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Satbayev University

ХАБАРЛАРЫ

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ
НАУК РЕСПУБЛИКИ
КАЗАХСТАН
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KAOLINITE RAW MATERIALS OF KAZAKHSTAN AND THE METHOD OF THEIR BENEFICIATION

Abstract. The only enterprise in the Republic of Kazakhstan that produces alumina is the Pavlodar aluminum plant, where low-quality bauxites of the Krasnogorsky mine of the Krasnooktyabrsky deposit serve as raw materials. Bauxites have a low flint modulus and high concentrations of iron and carbonates impurities, which complicates the processing technology.

Kaolinite clays can serve as an additional source of alumina in Kazakhstan, both as an additive to improve the sintering process of red sludge by reducing the iron content, and for self-processing into alumina. The most promising are kaolinite clays of the Alekseevsky deposit, however, when using standard techniques, satisfactory enrichment results were not achieved.

Kaolinite clays are subjected to gravity enrichment before processing into alumina, to increase the efficiency of which preliminary hydrothermal treatment is used.

This study presents the results of gravitational beneficiation of kaolinite clays of Alekseevskoe deposit, which includes hydrothermal pre-treatment in a solution of sodium hydrogen carbonate. It is determined that after hydrothermal treatment the yield of kaolinite fraction increases with increasing temperature, duration and concentration of sodium hydrogen carbonate solution and does not depend on L:S ratio.

After hydrothermal pre-treatment of clay the yield of kaolinite fraction increased from 41.4 % to 59.3 % and dispersion of material.

The maximum yield of kaolinite fraction was obtained at temperature 150 ^{o}C, ratio L:S=10.0, duration of 300 minutes and concentration of sodium hydrogen carbonate solution 120 g/dm^3.

Key words: kaolinite, quartzite, fraction, silica module, hydrothermal treatment, product yield.

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ҚАЗАҚСТАННЫҢ КАОЛИНИТ ШИКІЗАТЫ ЖӘНЕ ОНЫ БАЙЫТУ ӘДІСІ

Аннотация. Қазақстан Республикасында глинозем өндіретін жалғыз кәсіпорын Павлодар алюминий зауыты, мұнда шикізат ретінде Краснооктябрь кен орны Красногор кенішінің төмен сапалы бокситтері. Бокситтердің құрамы төмен кремний модулі және темір мен карбонат қоспалары жоғары болғандықтан бұл өңдеу технологиясын қиындатады.

Қазақстанда глиноземнің қосымша көзі ретінде каолинитті саздар темірдің құрамын төмендету арқылы қызыл шламды жентектеу процесін жақсарту үшін, сондай-ақ глиноземде өз бетінше өңдеу үшін қоспа ретінде қолдануға болады. Алексеев кен орнының каолинитті саздары ең перспективалы болып табылады, бірақ стандартты әдістерді қолдану кезінде байытудың қанағаттанарлық нәтижелеріне қол жеткізілген жоқ.

Каолинитті балшықтар глиноземге өңдеуден бұрын гравитациялық байытуға ұшырайды, оның тиімділігін арттыру үшін алдын-ала гидротермиялық өңдеу әдісі қолданылады. Бұл жұмыста натрий гидрокарбонат ерітіндісінде алдын-ала гидротермиялық өңдеуді қамтитын каолинит саздарының гравитациялық байыту нәтижелері көрсетілген. Гидротермиялық өңдеуден кейін каолинт фракциясының шығуы температураның, уақыттың және концентрациясының жоғарылауымен жоғарылайды және С:Қ арақатынасына байланысты болмайды.

Балшықты алдын ала гидротермиялық өңдеуден кейін, каолинт фракциясының шығуы 41,4% -дан 59,3% -ға өсті, материалдың дисперсиясы артты. Каолинт фракциясының максималды шығуы 150 ^{o}C температурада, С:Қ = 10,0 қатынасында, уақыты 300 минут және натрий гидрокарбонаты ерітіндісінің концентрациясы 120 г / дм^3 алынды

Түйін сөздер: каолинит, кварцит, фракция, кремний модулі, гидротермалды өңдеу, өнім шығару.

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КАОЛИНИТОВОЕ СЫРЬЕ КАЗАХСТАНА И СПОСОБ ЕГО ОБОГАЩЕНИЯ

Аннотация. Единственным предприятием в Республике Казахстан, которое производит глинозем является Павлодарский алюминиевый завод, где в качестве сырья служат низкокачественные бокситы Красногорского рудника Краснооктябрьского месторождения. Бокситы имеют низкий кремневый модуль и повышенные содержания примесей железа и карбонатов, что усложняет технологию переработки.

Дополнительным источником глинозема в Казахстане могут служить каолинитовые глины как в виде добавки для улучшения процесса спекания красного шлама путем снижения содержания железа, так и для самостоятельной переработки на глинозем. Наиболее перспективными являются каолинитовые глины Алексеевского месторождения, однако при использовании стандартных приемов не было достигнуто удовлетворительных результатов обогащения.

Каолинитовые глины перед переработкой на глинозем подвергают гравитационному обогащению для повышения эффективности которой применяют предварительную гидротермальную обработку.

В настоящей работе представлены результаты гравитационного обогащения каолинитовых глин Алексеевского месторождения, включающего предварительную гидротермальную обработку в растворе гидрокарбоната натрия. Установлено, что после гидротермальной обработки выход каолинитовой фракции возрастает с увеличением температуры, продолжительности и концентрации раствора гидрокарбоната натрия и не зависит от отношения Ж:Т.

После предварительной гидротермальной обработки глины увеличился выход каолинитовой фракции с 41,4% до 59,3% и диспергация материала.

Максимальный выход каолинитовой фракции получен при температуре 150°С, отношении Ж:Т=10,0, продолжительности 300 минут и концентрации раствора гидрокарбоната натрия 120 г/дм^3.

Ключевые слова: каолинит, кварцит, фракция, кремниевый модуль, гидротермальная обработка, выход продуктов.

Introduction. The analysis of global alumina production shows that the rate of development of alumina production capacity is lagging behind that of primary aluminium production (*http://www.aluminalimited.com/uploads/ASX-announcement-2017-27-Half-Year-Results-presentation*. pdf). The lack of capacity has become one of the major factors behind the rising price of alumina and the main reason for increasing raw material shortages at aluminium smelters. One of the reasons holding back the development of alumina production capacity is the deterioration of raw material quality and lack of effective technologies for processing alternative raw material sources, among which kaolinite clays occupy one of the leading places (*https://minerals.usgs.gov/minerals/pubs/commodity/bauxite/*; Zhang et all., 2018; Abdulvaliyev et all, 2019; https://www.marketing-magazin.ru/ imgs/goods/826/ru_kaolin.pdf; https://docplayer.ru/116077422-Rossiyskiy-i-mirovoy-rynokgliny-i-kaolina-syrevaya-baza-v-2018-godu-russian-andworld-market-of-clay-and-kaolin-raw-materials-base-in-2018).

The world reserves of kaolinite are estimated at 16 billion tonnes. The modern mining and processing of kaolinite raw materials has an established infrastructure, is provided with raw material resources and can be adapted to the production of alumina and by-products in the future. Currently, kaolin is mined in the USA, UK, China, Ukraine, Russia, Kazakhstan, Arab Republic of Egypt, etc. (Abdulvaliyev et all, 2018; Youssef et all, 1994; Brichkin et all, 2019; Bazhin et all, 2017; Arsentiev et all, 2017). The use of standard techniques of gravity concentration of kaolinite raw materials has not achieved satisfactory results (Kovzalenko et all, 2018).

In order to increase beneficiation efficiency, hydrothermal treatment of kaolinite materials at elevated temperature (350^{o}C) in water was studied (Sysa, 2008). Hydrothermal treatment does not change their crystal structure but leads to formation of less strong coagulation structures with increased plasticity, consisting of particles with average grain size increased by 10 – 20%. As a result, the intensity of interaction between particles is reduced, which increases the efficiency of beneficiation and hydrochemical processing. Kaolinite was found to be the most resistant mineral to hydrothermal treatment.

In the studies carried out, a solution of sodium hydrogen carbonate was used to provide more severe conditions in the hydrothermal treatment of kaolinite clay. The choice of sodium hydrogen carbonate is due to the fact that this reagent has proven itself for the selective separation of components of raw materials (Patent RK 335383, 2019; Patent RK 32333, 2017). The kaolinite concentrate obtained as a result of clay beneficiation is intended for alumina production and therefore part of the unwashed sodium bicarbonate after treatment will not interfere in the further alkaline technology.

As an alternative source of raw materials for alumina production in the Republic of Kazakhstan the most promising is Alexeyevskoe kaolinite deposit located 22 km north-west of Kokshetau city and processed by "AraiPro" LLP. Balance reserves of kaolin of the deposit by category B - 13,665 mln. tons, by category C_1 - 67,228 mln. tons, category C_2 – 174,261mln. tons. The average stripping ratio is not more than 1 m^3/t.

Currently, "AraiPro" produces about 300 thousand tonnes of raw kaolin, which is exported to the Russian Federation.

Statement of the problem. The aim of the study was to increase the beneficiation efficiency of kaolinite clays through hydrothermal pretreatment in a solution of sodium hydrogen carbonate.

Methods and methodology of the study. X-ray fluorescence, chemical, X-ray phase and thermal analyses were used in the studies.

X-ray fluorescent analysis was carried out on Venus 200 spectrometer with wave dispersion (PA Nalyical B.V., Holland).

Chemical analysis of samples was performed on optical emission spectrometer with inductively coupled plasma Optima 2000 DV (USA, PerkinElmer).

Semi-quantitative X-ray phase analysis was carried out on D8 Advance diffractometer (BRUKER) on copper Cu-Kα radiation at accelerating voltage 36 kV, current 25 mA.

The pre-thermal treatment of kaolinite clays was carried out in a solution containing 40 – 120 g/dm^3 $NaHCO_3$with L:S=2 – 10,0, at 90 - 230^{o}C and for 30 – 300 minutes using a thermostatically controlled plant with 6 autoclaves rotating through the head and having a working volume of 250 cm^3 (Figure 1). The maximum sodium hydrogen carbonate content of 120 g/dm^3 in the solution was taken to its solubility limit.

Figure 1 – Thermostatically controlled unit with autoclaves

The kaolinite fraction of the clay was separated by washing with running water at room temperature.

The silica modulus, μ_{si}, was calculated from $Al_2O_3 \div SiO_2$ ratio.

Study results. The initial product for the study was a representative sample of kaolinite clay Alexeyevskiy deposit, provided by "Araipro" LLP, corresponding to the standard of the organization ST LLP 101240014515-01-2019.

The appearance of kaolinite clay sample is loose sand of whitish colour, density of 2.06 g/cm3, bulk density of 1.36 kg/cm3, the average grain size of 2 mm.

Chemical composition of kaolinite clay sample from Alexeyevskiy deposit wt.%: Al_2O_3 – 26.9; SiO_2 – 56.6; Fe_2O_3 - 0.537; Na_2O – 0.07; SO_3 – 0.028; K_2O – 1.31; p.p.s. 14.555, μ_{si} – 0.47.

The dispersion and chemical composition of fractions and distribution of elements by size classes of kaolinite clay sample from Alexeyevskiy deposit are presented in Table 1.

Table 1 - Dispersion and chemical composition of fractions in a representative sample of kaolinite clay from the Alexeyevskiy deposit

Granularity class, mm	Yield, %	Contents, %				μ_{si}	Distribution, %		
		Al_2O_3	SiO_2	Fe_2O_3	p.p.		Al_2O_3	SiO_2	Fe_2O_3
+0.05	58.6	14.18	60.85	0.69	24.28	0.233	30.9	58.46	55.48
-0.05+0.02	38.06	30.89	49.8	0.597	18.71	0.62	65.08	38.53	41.27
-20 + 10 μm	3.05	32.07	48.09	0.631	19.209	0.67	3.6	2.7	2.9
-10 μm	0.29	30.35	46.59	0.689	22.37	0.651	0.42	0.31	0.35
Total	100						100	100	100

From the results it follows that kaolinite fraction of clay is mainly included in the composition of size classes equal to – 0.05.

Chemical composition of kaolinite fraction of clay, wt.%: Al_2O_3 - 31.2; SiO_2 – 51.6; Fe_2O_3 – 0.53; CaO 0.43; Na_2O – 0.095; MgO 0.2; SO_3 0.02; K_2O – 1.5; TiO_2 1.05; Cl^-0.02; p.p. 13.355; μ_{si} – 0.6.

Chemical composition of quartzite fraction, wt.%: Al_2O_3, 7.65; SiO_2, 65.5; Fe_2O_3, 0.68; CaO 0.29; Na_2O, 0.031; MgO 0.08; SO_3 0.05; K_2O, 0.39; TiO_2 0.9; Cl-0.016; p.p. 24.413; μsi – 0.12.

X-ray patterns of kaolinite and quartzite fractions of clay from Alexeyevskiy deposit are shown in Figures 2, 3.

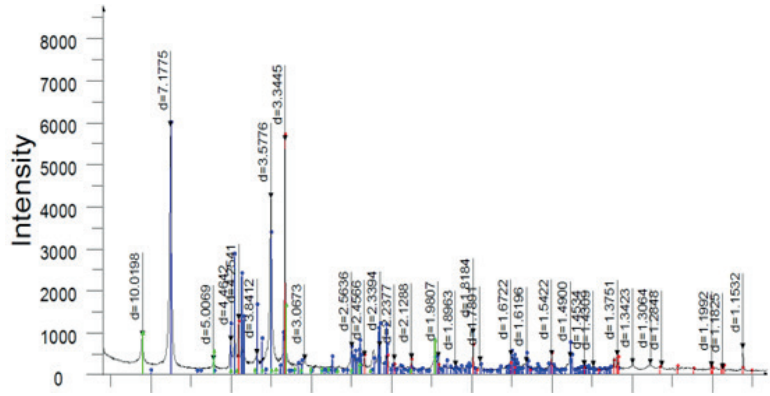

Figure 2 - X-ray diagram of the kaolinite clay fraction of the Alexeyevskiy deposit

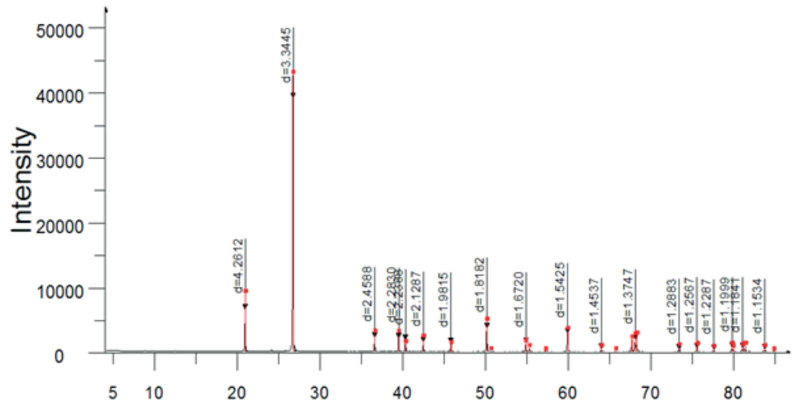

Figure 3 - X-ray diagram of the quartzite fraction of the Alexeyevskiy clay deposit

The yield of the kaolinite fraction in clay beneficiation was investigated as a function of temperature, duration, L:S ratio and concentration of sodium hydrogen carbonate solution during hydrothermal pre-treatment (Table 2).

Table 2 – Dependence of the kaolinite yield of the beneficiation fraction on the hydrothermal pretreatment conditions

Treatment conditions				Beneficiation results, %	
Temperature, °C	L:S	Time duration, min	Concentration $NaHCO_3$	Yield of kaolinite fraction	Yield of Al_2O_3 to kaolinite fraction
Without processing				41.4	72.6
Processing in water, 230°C	10	180	-	47.5	76.8
90	10	60	120	41.6	74.6

120	10	60	120	47.6	76.9
150	10	60	120	47.6	79.39
180	10	60	120	42.4	79.0
230	10	60	120	42.4	76.93
150	10	30	120	47.4	76.0
150	10	90	120	49.8	83.3
150	10	120	120	58.0	88.67
150	10	300	120	59.3	89.8
150	2	120	120	48.0	86.9
150	4	120	120	48.1	87.0
150	6	120	120	48.3	87.9
150	8	120	120	48.6	87.6
150	10	120	40	48.9	80.3
150	10	120	60	49.3	82.7
150	10	120	80	49.35	83.2
150	10	120	100	49.6	85.6

As a result of studies it was found that the yield of kaolinite fraction increases with increasing temperature, duration and concentration of sodium hydrogen carbonate solution after hydrothermal pretreatment and practically does not depend on the ratio L:S.

The maximum yield of kaolinite fraction of 59.3% was obtained after hydrothermal pre-treatment of clay at 150°C, ratio L:S = 10.0, time of 300 minutes and concentration of sodium bicarbonate solution of 120 g/dm^3. The yield of Al_2O_3 in kaolinite fraction was 89.8%.

The phase composition of kaolinite fraction of clay, obtained in these conditions is presented, %: kaolinite – 57.0; quartz 25.4; muscovite 8.9; aluminosilicate sodium 4.9 and magnetite 3.7 (Figure 4).

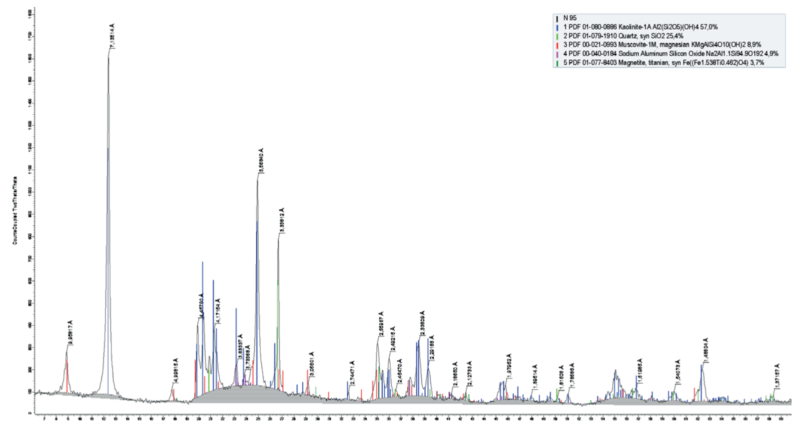

Figure 4 - X-ray of the kaolinite clay fraction after hydrothermal pre-treatment

The chemical composition of the kaolinite fractions after hydrothermal pre-treatment is shown in Table 3.

Table 3 - Dispersion, chemical composition of kaolinite clay fractions after hydrothermal pre-treatment

Granularity class, mm	Yield, %	Contents, %				μ_{si}	Distribution, %		
		Al_2O_3	SiO_2	Fe_2O_3	p.p.		Al_2O_3	SiO_2	Fe_2O_3
+0.05	40.7	5.83	77.7	0.98	15.49	0.075	10.2	62.09	61.75
-0.05+0.02	54.89	30.03	48.58	0.61	20.78	0.62	75.37	29.3	29.01
-0.02 + 0.01 µm	4.0	29.76	46.4	0.63	23.21	0.642	14.12	8.49	9.09
-0.01 + 0 µm	0.41	29.73	45.14	0.69	24.44	0.659	0.31	0.13	0.15
Total	100.0						100	100	100

Analysis of obtained results of dispersion composition shows that after hydrothermal treatment of clay in sodium hydrogen carbonate solution in kaolinite fraction the content of – (0.05 + 0.02) mm class increased, i.e. average particle size increased by 19.9 %. The obtained result corresponds to the conclusions of theoretical investigations of the mechanism of behaviour of kaolin during hydrothermal treatment (Kenzhaliyev et all, 2021).

According to the known method of hydrothermal pre-treatment in water, the yield of kaolinite fraction was 47.5% with an Al_2O_3 yield of 76.8%, i.e. the use of sodium hydrogen carbonate solution increased the efficiency of beneficiation.

Conclusions. The conditions of hydrothermal pre-treatment of kaolinite clay from Alexeyevskiy deposit of the Republic of Kazakhstan in solution of sodium bicarbonate for kaolinite fraction extraction at enrichment were investigated. The yield of kaolinite fraction due to treatment increased from 41.4% to 59.3%. Extraction of Al_2O_3 in kaolinite fraction was 89.8%.

As a result of hydrothermal treatment, the average particle size of the kaolinite fraction increased, which ensured better selectivity of the concentration operation.

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