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**Metallurgy**

# Pre-activation of nepheline before the enrichment

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**ABSTRACT**

Due to limited reserves of bauxite, nepheline can be used in the industrial production of alumina in Kazakhstan. The most promising deposit is the nepheline syenites of the Kubasadyr deposit. Currently, there is no effective technology for processing nepheline ores. High energy intensity, capital intensity, and significant emissions into the atmosphere are the main drawbacks of the conventional technology of nepheline ore processing by the sintering method. Efficient hydrochemical processing of nepheline requires pre-enrichment with the separation of a part of silica. According to the existing technology of chemical enrichment in an alkaline solution at a temperature of 280°C, the silica extraction degree is no more than 36%. A pre-roasting at 500°C is used to increase the extraction rate of silica and this process permits an increase in the extraction rate to 65%. The paper presents the results of the chemical activation of nepheline syenites in a solution of sodium hydrogen carbonate. The optimum conditions of activation are determined. Activation at a temperature of 280 °C resulted in a change in the phase composition of the feedstock and increases the degree of silica extraction up to 65.5%. The results obtained showed the possibility in principle of using hydrochemical enrichment technology for the resulting high-quality nepheline concentrate and replacing the energy-intensive roasting process.

***Keywords:*** nepheline, enrichment, preliminary activation, silica, alumina.

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## Introduction

The global aluminium industry is growing dynamically and is one of the leaders in the production of construction materials [[1]], [[2]].

The traditional source of aluminium is bauxite. However, their quality reserves are limited and the currently known reserves will be exhausted within the next 20 years and the reserve base will be adequate for not more than 25 years [3]. This has led to a search for alternative sources. Aluminosilicate minerals such as kaolins or nepheline and others can be considered as such [[4]], [5], [6], [7], [8], [9], [[10]].

Nepheline is processed in the world only in Russia, Canada, and Norway [11].

Large accumulations of non-bauxite aluminium raw materials have been identified in Kazakhstan. These are high alumina minerals: nepheline of alkaline, ultrabasic and granitoid rocks. There are three main varieties of rocks rich in nepheline: nepheline rocks of alkaline or ultrabasic massifs, nepheline-leucite rocks of alkaline-basic massifs, and nepheline syenites of massifs of alkaline granitoids [12].

Several dozens of nepheline rock massifs are known in different regions of Kazakhstan:

- Kosistek in Western Kazakhstan;

- Yesil, Kentassk, Derzhavinsk, Karsakpai, and Shynsai in Central Kazakhstan;
- Irisu, Mashat, Badam, Kulandinsk in Southern Kazakhstan;
- Semeytausk in Eastern Kazakhstan.

The geological reserves of alumina raw materials at the above deposits are huge and counted in billions of tonnes of complex ores, by category C2 - over 500 million tonnes. At the present stage of exploration, the largest, well-studied is the Yesilsk massif of the Kubasadyr nepheline-leucite syenite deposit, which is represented by two sections: Ashchilisai and Taskuduk. The intrusive is located in a large deeply eroded caldera of an Early Devonian stratovolcano, confined to the intersection of two deep faults of northwest and northeast directions. The host rocks are metasomatically reworked nepheline syenites [12].

Reserves of alumina raw materials in category C2 total 239 million tons in two areas, but there is no production of their processing. To organize the production of nepheline processing in Kazakhstan, it is necessary to develop an effective technology that takes into account the peculiarities of the mineral composition of raw materials.

The information used to define industrial mineral quality relates to consumer specifications: minimum $SiO_2$, maximum $Fe_2O_3$, or whiteness, among others [13].

Sintering or hydrochemical methods can be used for nepheline processing. Sintering nepheline with limestone at 1200 $^oC$ decomposes it to form sodium and potassium aluminates and a two-calcium silicate. However, this process is very energy and capital-intensive.

Potentially, the processing of 1 million tons of ore could produce 150 thousand tons of alumina on existing technologies, by the way, to get 1,7 million tons of cement, 70 thousand tons of soda, and 100 thousand tons of sulphuric potash. The production requires 2.5M tonnes of limestone, with capital costs of about $1bn.

There have been studies of nepheline sintering at lower temperatures, e.g. sintering with an excess 5–10 % of sodium carbonate at 760–880 °C for 1.0–1.5 h. Such processes would provide high Al extraction, and the high energy consumption and $CO_2$ footprint that derive from the thermal treatment step are of great concern [14].

Given the negative attitudes in the world towards the sintering process due to high carbonate emissions into the atmosphere, hydrochemical technologies are preferable.

In the hydrochemical process, the nepheline is decomposed by an alkaline solution in the presence of lime in autoclaves at a temperature of 280$^o$C. Under these conditions, sodium and potassium aluminates pass into the solution and silica remains in precipitation in the form of sodium-calcium hydro silicate. High aluminium recovery is achieved at $Na_2O$ content in the solution of 400-500 g/dm$^3$.

The hydrochemical method is characterized by a high alkali turnover, which predetermines a considerable heat consumption for solvent evaporation. However, in contrast to the sintering process specific material losses of solutions and heat consumption per tonne of $Al_2O_3$ do not depend much on the solid phase ($SiO_2$ + CaO), i.e. on the silicon ratio of raw materials ($SiO_2$: $Al_2O_3$). During sintering, the specific fuel consumption per tonne of $Al_2O_3$ is almost proportional to the silicon ratio. This implies that the hydrochemical process is of interest for processing alkaline silica-rich aluminosilicate rocks and the profitability of production should increase as the quality of aluminosilicate raw materials deteriorates.

Nepheline syenite is also processed by acid treatment [15].

There are also technologies of combining the hydrochemical method with chemical enrichment, roasting, and others [[16], [17]].

Chemical enrichment of nepheline is used to reduce its silica content, leading to a reduction in the flow of raw materials for further processing in alumina production.

Chemical enrichment is carried out in an alkaline solution containing 240 g/dm$^3$ $Na_2O$ at a temperature of 280$^o$C and a duration of 40 minutes [16]. Under these conditions, the degree of silica extraction into the solution of 38.6% was obtained. To increase of efficiency of chemical enrichment offered to carry out preliminary activation of nepheline by roasting with the steam-air mixture at a temperature of 500$^o$C [17]. As a result, the degree of silica extraction is increased to 60.0-65.0%. However, roasting is an energy-intensive process.

In the conducted research to increase the efficiency of the enrichment process, the possibility of pre-chemical activation of nepheline syenites of the Kubasadyr deposit in a solution of sodium hydrogen carbonate has been studied.

Preliminary chemical activation of raw materials in a solution of sodium hydrogen carbonate leads to a change in the phase composition, which will allow for obtaining higher values for further processing [[18]], [19], [20].

Preliminary chemical activation was tested in the processing of low-quality gibbsite-kaolinite [18], waste products [19], and semi-products of ferrochrome production [20], and ash and showed high results.

## Experimental part

X-ray fluorescence analysis of the chemical composition was carried out on a Venus 200-wave dispersion spectrometer (PANalytical B.V., Holland).

Chemical analysis was performed on an optical emission spectrometer with inductively coupled plasma Optima 2000 DV (USA, Perkin Elmer).

Semi-quantitative X-ray phase analysis was carried out on the D8 Advance diffractometer (BRUKER) using Cu-$K_\alpha$ radiation at an accelerating voltage of 36 kV, current 25 mA.

Chemical activation of nepheline syenites was carried out in a solution containing 120 g/dm$^3$ $NaHCO_3$ at an L: S ratio of 3 and a temperature range of 120 - 280 $^0$ using a thermostatically controlled unit with 6 autoclaves rotating through the head, with a working volume of 250 cm$^3$ and activation time 90 minutes. The maximum sodium hydrogen carbonate content in the solution was 120 g/dm$^3$, taking into account its solubility limit.

Chemical enrichment was carried out in an alkaline solution containing 240 g/dm$^3$ $Na_2O$ at a temperature of 280$^0$C and a duration of 40 minutes.

## Results and discussion

Physical and chemical studies of the material composition of nepheline syenites of the Kubasadyr deposit were carried out.

Chemical composition of the initial sample, mass %: $Al_2O_3$ – 17,77; $SiO_2$ – 48,37; $Fe_2O_3$ – 3,18; CaO – 2,89; $Na_2O$ – 4,24; $K_2O$ – 5,34; MgO – 0,34; Cl – 0,3.

The phase composition of the initial sample is shown in figure 1 and table 1.

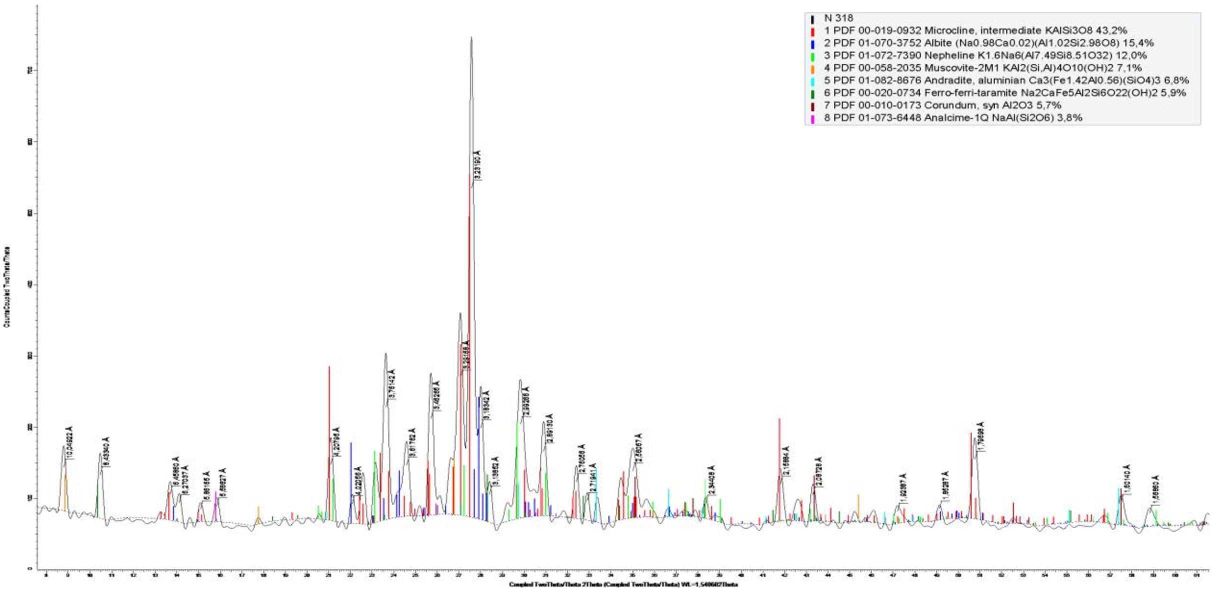

**Figure 1** - X-ray diffraction pattern of nepheline

**Table 1 –** Phase composition of nepheline

| Composition | | |
|---|---|---|
| Components | Components | % |
| Microcline | $KAlSi_3O_8$ | 43.2 |
| Albite | $(Na_{0.98}Ca_{0.02})(Al_{1.0}2Si_{2.98}O_8)$ | 15.4 |
| Nepheline | $K_{1.6}Na_6(Al_{7.49}Si_{8.51}O_{32})$ | 12.0 |
| Muscovite- | $KAl_2(Si,Al)_4O_{10}(OH)_2$ | 7.1 |
| Andradite, | $Ca_3(Fe_{1.42}Al0_{.56})(SiO_4)_3$ | 6.8 |
| Ferro-ferri-taramite | $Na_2CaFe_5Al_2Si_6O_{22}(OH)_2$ | 5.9 |
| Corundum, | $Al_2O_3$ | 5.7 |
| Analcime-1Q | $NaAl(Si_2O_6)$ | 3.8 |

**Table 2** - Phase composition of nepheline syenite samples depending on the temperature of chemical activation

| Activation temperature, °C | Composition | | |
|---|---|---|---|
| | Components | Formula | % |
| 120 | Microcline, intermediate | $KAlSi_3O_8$ | 40.1 |
| | Albite | $(Na_{0.98}Ca_{0.02})(Al_{1.02}Si_{2.98}O_8)$ | 17.5 |
| | Nepheline | $K_{1.6}Na_6(Al_{7.49}Si_{8.51}O_{32})$ | 13.1 |
| | Corundum, syn | $Al_2O_3$ | 7.7 |
| | Andradite, aluminian | $Ca_3(Fe_{1.42}Al0_{.56})(SiO_4)_3$ | 3.8 |
| | Ferro-ferri-taramite | $Na_2CaFe_5Al_2Si_6O_{22}(OH)_2$ | 6.9 |
| | Muscovite-2M1 | $KAl_2(Si,Al)_4O_{10}(OH)_2$ | 6.6 |
| | Analcime-1Q | $NaAl(Si_2O_6)$ | 4.4 |
| 150 | Microcline, intermediate | $KAlSi_3O_8$ | 34.6 |
| | Albite | $(Na_{0.98}Ca_{0.02})(Al_{1.02}Si_{2.98}O_8)$ | 18.8 |
| | Nepheline | $K_{1.6}Na_6(Al_{7.49}Si_{8.51}O_{32})$ | 16.3 |
| | Ferro-ferri-taramite | $Na_2CaFe_5Al_2Si_6O_{22}(OH)_2$ | 7.0 |
| | Corundum, syn | $Al_2O_3$ | 6.9 |
| | Muscovite-2M1 | $KAl_2(Si,Al)_4O_{10}(OH)_2$ | 6.4 |
| | Analcime-1Q | $NaAl(Si_2O_6)$ | 6.3 |
| | Andradite, aluminian | $Ca_3(Fe_{1.42}Al_{0.56})(SiO_4)_3$ | 3.8 |
| 200 | Microcline, intermediate | $KAlSi_3O_8$ | 33.4 |
| | Albite | $(Na_{0.98}Ca_{0.02})(Al_{1.02}Si2.98O_8)$ | 18.2 |
| | Nepheline | $K_{1.6}Na_6(Al_{7.49}Si8_{.51}O_{32})$ | 15.7 |
| | Andradite, aluminian | $Ca_3(Fe_{1.42}Al_{0.56})(SiO_4)3$ | 4.2 |
| | Ferro-ferri-taramite | $Na_2CaFe_5Al_2Si_6O_{22}(OH)_2$ | 6.8 |
| | Corundum, syn | $Al_2O_3$ | 6.7 |
| | Muscovite-2M1 | $KAl_2(Si,Al)_4O_{10}(OH)_2$ | 6.1 |
| | Analcime-1Q | $NaAl(Si_2O_6)$ | 6.1 |
| 280 | Albite | $Na_{0.986}Al_{1.005}Si_{2.995}O_8$ | 31.5 |
| | Microcline, intermediate | $KAlSi_3O_8$ | 15.3 |
| | Nepheline | $NaAlSiO_4$ | 14.1 |
| | Calcium iron(II) Aluminum oxide | $Ca_4Fe_{1.5}Al_{17.67}O_{32}$ | 13.1 |
| | hydrosodalite, hexasodium | $Na_6(AlSiO_4)_6(H_2O)_8$ | 9.8 |
| | Calcium Iron Oxide | $Ca_2Fe_2O_{5.12}$ | 7.3 |
| | Ferro-tschermakite | $Ca_2Fe_3Al_2(Si_6Al_2)O_{22}(OH)_2$ | 6.6 |
| | Analcime-1Q | $NaAl(Si_2O_6)$ | 2.3 |

The phase composition of nepheline syenite samples depending on the temperature of chemical activation is presented in table 2.

During chemical activation depending on the temperature, there were changes in the phase composition of nepheline syenites: almost three times less microcline content; two times more albite content, hydro sodalite, calcium-iron oxide and analcite phases were formed, and the andradite and corundum phases disappeared (figure 2).

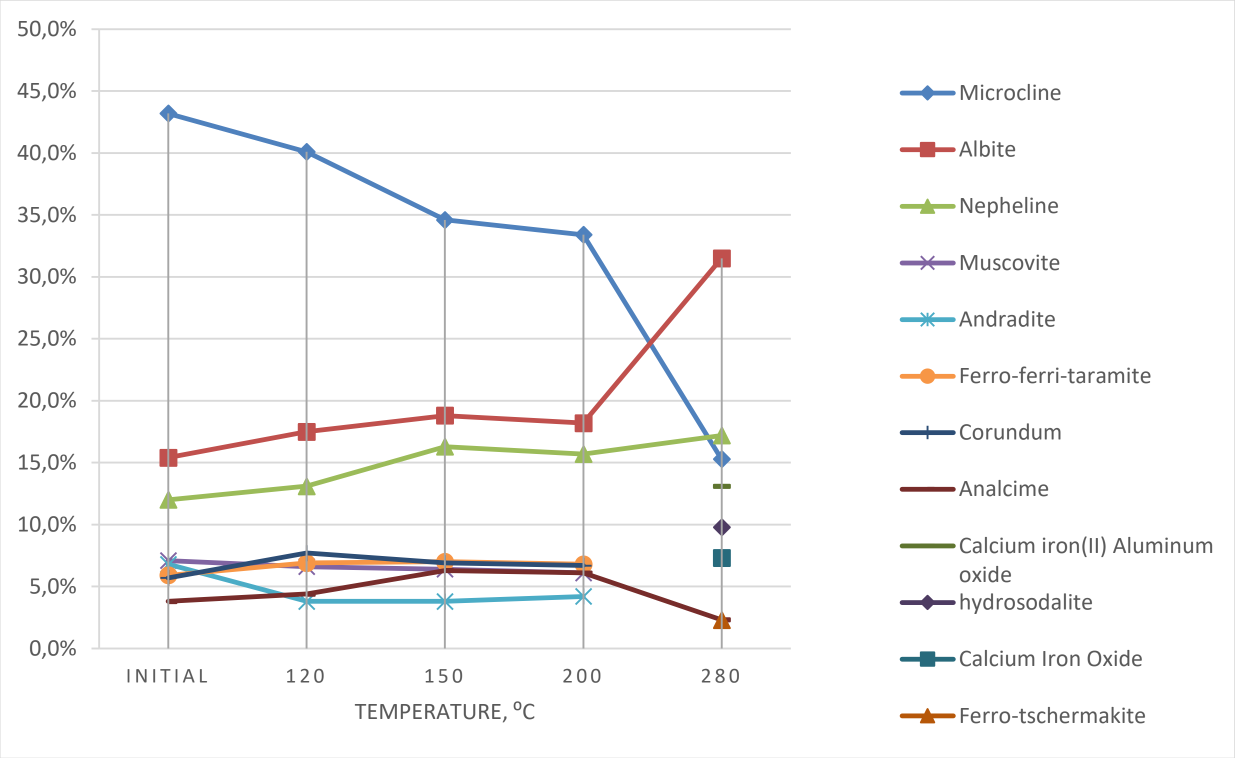

**Figure 2 -** Dependence of the change in the phase composition of nepheline syenites on the temperature of chemical activation

The influence of temperature of preliminary chemical activation of nepheline syenites on the degree of $SiO_2$ extraction at enrichment is investigated. It was shown that with increasing activation temperature the degree of extraction of silica in the solution increases (figure 3).

At an activation temperature of 280°C, the $SiO_2$ extraction rate during enrichment was 65.5 %. As a result of enrichment, the nepheline concentrate is received, composition wt.%: $Al_2O_3$ – 28.6; $SiO_2$ – 50.293; $Fe_2O_3$ – 5.08; CaO – 2.89; $Na_2O$ – 6.79; $K_2O$ – 4.74; MgO – 0.54.

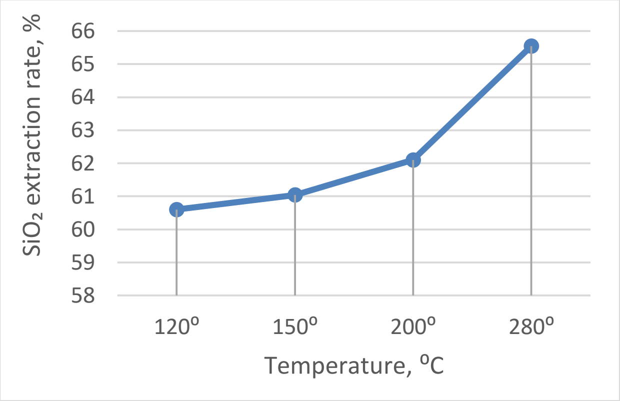

**Figure 3** – $SiO_2$ extraction rate

## Conclusions

Studies on the chemical activation of nepheline syenites of the Kubasadyr mine were conducted.

The obtained degree of enrichment of nepheline after preliminary chemical activation in a solution of sodium hydrogen carbonate is comparable with the degree of enrichment obtained after activation by pre-firing with the steam mixture at 500°C.

These results show the possibility in principle of obtaining high-quality nepheline concentrate, but require further research and refinement involving other techniques.

At chemical enrichment of nepheline syenites, the degree of extraction of silica is low (36%). As a result of pre-activation, the degree of extraction of silica in the solution reaches 65%.

During chemical activation depending on the temperature, there were changes in the phase composition of nepheline syenites: almost three times less microcline content; two times more albite content, hydro sodalite, calcium-iron oxide, and analcite phases were formed, and the andradite and corundum phases disappeared.

The results show the basic possibility ofobtaining high-quality nepheline concentrate, but require further research and refinement involving other techniques.

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**Conflict of interest.** The corresponding author declares that there is no conflict of interest.

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# Нефелинді байытуға дейінгі алдын ала белсендіру

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**ТҮЙІНДЕМЕ**

Қазақстанда алюминий тотығын өнеркәсіптік өндіру саласындағы Байер бокситтерінің шектеулі қорларының салдарынан боксит емес шикізат-нефелиндерді пайдалануға болады. Неғұрлым перспективалы кен орны Кубасадыр кен орнының нефелинді сиениттері болып табылады. Қазіргі уақытта нефелин кендерін өңдеудің тиімді технологиясы жоқ. Жоғары энергия сыйымдылығы, капитал сыйымдылығы және атмосфераға елеулі шығарындылар нефелин кендерін пісіру тәсілімен өңдеудің жалпы қабылданған технологиясының негізгі кемшіліктері болып табылады. Нефелиндерді тиімді гидрохимиялық өңдеу үшін кремнеземнің бір бөлігін бөліп алу арқылы алдын ала байыту қажет. Қолданыстағы сілтілі ерітіндіде 280 $^{o}$C температурада химиялық байыту технологиясына сәйкес кремнеземді алу дәрежесі 36 %-дан аспайды. Кремний тотығын алу дәрежесін арттыру үшін 500 $^{o}$C температурада алдын ала күйдіруге қолданылады, және бұл экстракция дәрежесін 65 %-ға дейін арттыруға мүмкіндік береді. Мақалада натрий гидрокарбонатының ерітіндісіндегі нефелинді сиениттердің алдын ала химиялық белсенділігінің нәтижелері берілген. Белсендірудің оңтайлы шарттары анықталды. 280 $^{o}$C температурасы кезіндегі белсенділік бастапқы шикізаттың фазалық құрамының өзгеруіне алып келді және 65,5 % байыту кезінде кремнезем алу дәрежесінде алды. Алынған нәтижелер алынған жоғары сапалы нефелинді концентраты үшін гидрохимиялық байыту технологиясын қолданудың және энергияны көп қажет ететін күйдіру процесін ауыстырудың түбегейлі мүмкіндігін көрсетті.

***Түйін сөздер:*** нефелин, байыту, алдын ала белсендіру, кремний тотығы, алюминий тотығы

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# Предварительная активация нефелинов перед обогащением

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**АННОТАЦИЯ**

Вследствие ограниченных запасов байеровских бокситов в сферу промышленного производства глинозема в Казахстане возможно использование небокситового сырья – нефелинов. Наиболее перспективным месторождением является нефелиновые сиениты Кубасадырского месторождения. В настоящее время, не существует эффективной технологии переработки нефелиновых руд. Высокая энергоемкость, капиталоемкость и значительные выбросы в атмосферу являются основными недостатками общепринятой технологии переработки нефелиновых руд способом спекания. Для эффективной гидрохимической переработки нефелинов требуется предварительное обогащение с отделением части кремнезема. По существующей технологии химического обогащения в щелочном растворе при температуре 280$^{o}$С степень извлечения кремнезема составляет не более 36%. Для повышения степени извлечения кремнезема используют предварительный обжиг при 500$^{o}$С, который позволяет повысить степень извлечения до 65%. В статье представлены результаты предварительной химической активации нефелиновых сиенитов в растворе гидрокарбоната натрия. Определены оптимальные условия активации. Активация при температуре 280 $^{o}$С привела к изменению фазового состава исходного сырья и получить степень извлечения кремнезема при обогащении 65,5%. Полученные результаты показали принципиальную возможность применения гидрохимической технологии обогащения для полученного качественного нефелинового концентрата и заменить энергоемкий процесс обжига.

***Ключевые слова:*** нефелин, обогащение, предварительная активация, кремнезем, глинозем.

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