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The effect of halite mineral impurities on the technological parameters of the sodium chloride production process

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ABSTRACT
This paper shows the findings of a detailed investigation of the natural halite from the Bakhyt-Tany deposit. The mineral's composition is sodium chloride with the addition of calcium sulfate, magnesium salts, and some other matters, including a residue of less than 2% insoluble residue. Elemental assaying indicates the occurrence of elements like Ca, Mg, Al, Si, Fe, and Pb, which points to the occurrence of clay and some sulfate impurities. To understand how impurities are distributed in different sizes, a sample was classified using a sieve with a mesh of 0.2 mm. It was discovered that less than twenty per cent of the salt mass is a fine fraction (d < 0.2 mm), where up to 3.4% of insoluble impurities are found, and in the coarse fraction (d > 0.2 mm), this value is less than 1.8%. A mathematical model developed showed that the fine fraction and the total amount of the residue insoluble are directly related, which supports its use for estimating contamination and evaluating the effectiveness of the processes of desalination. Moreover, the generated 3D model revealed that temperature and humidity, in addition to raising the concentration of insoluble impurities, also increase the concentration of such impurities in the fine fraction even more. The results obtained also support the need for the pre-purification of halite before its use in food and other technological applications, and support the statement of the fractionation and desalination based purification process for halite.

Keywords: halite, sodium chloride, mineral impurities, colloidal particles, recrystallization, purification.

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Introduction

Sodium chloride is produced on the basis of halite minerals extracted by mining; brines are obtained by in-situ leaching or processing of solid sediments of salt lakes, which is the most cost-effective method. The Republic of Kazakhstan is regarded as one of the most mineral-rich countries in Central Asia with regard to salt minerals. The latest data indicates that the number of salt lakes in Kazakhstan exceeds 2,500, with an annual production of sodium chloride that surpasses 1 million tons. Notably, more than 80% of this production is concentrated within the Kyzylorda region. It is estimated that approximately 40% of the global production of table salt is exported [[1], [2], [3], [4], [5]].

The rich reserves of salt deposits in the Sarysu region could be of industrial importance to the Zhambyl Oblast region. The reserves are estimated at 45 million tons, and only one of the nearby deposits, Majdekenkol, has about 10 million tons. There are 22 salt deposits in the Zhambyl district. They are located at a considerable distance from each other and spread over a vast territory from the foothill plain of the Karatau Range in the south to the valley of the Shu River in the north [[6], [7]].

Exploration works were carried out at four sites: Aydyn, Yunkikol, Tuzkol and Maidegenkol.

On the lake "Koibagar", salt deposits consist of two layers. The upper layer with seam thickness from 1 to 3.4 m contains mainly mineral halite (NaCl), the lower layer with ore thickness from 0.3 to 2 m contains the following minerals: halite (NaCl), tenardite, astrakhanite, glauberite, espomite and gypsum. Lakes Isteken and Kokalegel are sulphate lakes, varying in thickness from 0.2 to 1.4 m and containing the following minerals: halite (NaCl), tenardite (Na_2SO_4), myrobilite, astrachanite, glauberite and gypsum. In all explored deposits, the content of insoluble waste is concentrated in the range of 2-6% [[3], [8], [10]].

In terms of its technical characteristics, it is the opinion of experts that the percentage of water-insoluble substances, potassium ions, magnesium and sulphate, in both food products and technical salt, is below the permissible standards. The sodium chloride content of the composition is found to be 98.6%, which has been demonstrated to have a positive effect on quality [[8], [9]].

A significant presence of colloidal and mineral impurities has been identified in the composition of Halite ores. Such additives include sulfates, carbonates, silt and other substances of organic origin, which greatly interfere with the production of pure sodium chloride [[10], [11], [12]].

During the purification process of ore from impurities, colloidal particles function not only as mechanical impurities but also facilitate the destruction of the crystal structure through adsorption processes, accumulating on the crystal surface and thereby impeding the process [13]. The presence of colloidal particles has been demonstrated to have a number of effects on the process of crystallisation. These include the slowing down of crystal growth, the increase of the energy barrier at the start of crystallisation, and the formation of micropore effects [[14], [15]].

The mineral additives most frequently employed include kaolinite and montmorillonite, which are characterised by their silty composition, in addition to calcium and magnesium sulfates and carbonates, and heavy metals [16].

The movement and distribution of colloidal particles within a sodium chloride solution flow are contingent on environmental conditions, thereby exerting a substantial influence on the optimal parameters of solution purification. In the presence of salt gradients, the rate and direction of colloid migration are contingent on temperature and contact time, thereby affecting the efficiency of impurity separation [17]. For instance, at elevated temperatures, diffusiophoretic processes are enhanced, thus facilitating faster migration of particles to the interface. Concurrently, the optimal residence time is such that the maximum concentration of particles is achieved in the target zone. Consequently, the selection of temperature and duration in NaCl purification systems utilising colloids must consider the kinetics of their movement, thereby achieving a balance between the purification speed and the degree of contaminants removed [[18], [19]].

A recent study published on Phys.org demonstrates that salt gradients can effectively control the direction of motion of colloidal particles in microfluidic systems through a combination of diffusiophoresis and diffusioosmosis [20]. Researchers at Yale University have discovered that even a minor variation in salt concentration can induce focusing and redirection of colloids, obviating the necessity for external fields. This finding offers a promising outlook for the development of passive and energy-efficient systems for liquid purification and targeted delivery of substances in medicine and the environment [[19], [20]].

There are also studies on the purification of solutions from colloidal particles using membranes and electrocoagulation. Bharti et al. [21] presented a comprehensive review of electrocoagulation as a universal method for purifying wastewater and natural water from colloidal ions, organic substances, and turbidity. The authors showed that electrocoagulation provides a high degree of removal of dispersed particles due to the formation of hydroxide flocs, which makes it promising for the preliminary purification of mineral raw materials. Aouni et al. and Moneer et al. focused on the combination of electrocoagulation with membrane processes such as ultrafiltration and reverse osmosis. It has been shown that hybrid schemes can significantly reduce membrane fouling and ensure effective removal of colloids, increasing the stability of seawater desalination processes. The authors showed that such hybrid systems provide improved selectivity and efficiency in the removal of impurities, including finely dispersed colloids, which is particularly important in the preparation of complex brines and wastewater for further processing [[22], [23]].

While modern methods of sodium chloride purification by evaporation, recrystallization, filtration, and even sedimentation work wonders, they all have one thing in common: dealing with colloidal and mineral impurities remains a challenge. As an example, evaporation can be energy intensive, especially when heating to overcome colloids stabilizing the solution.

Recrystallization poses a challenge when the solution has a lot of clay particles. These clay particles adsorb onto the crystals, disrupting the shape, disturb, and purity of the crystals [[23],[24]].

The filtration method does not work well with stable colloidal systems, especially if the particles in the colloidal solution are not likely to aggregate into larger structures.

Time and, at the same time, very sensitive to external influence such as pH, ionic strength, and, if the solution has a lot of contaminants, they may not be as effective [[20], [25]].

In this particular case, studying the behavior of colloidal particles in saturated sodium chloride solutions and developing effective colloidal particle removal strategies are very relevant. One example is the removal of colloidal impurities before crystallization, a process known as desliming. Desliming (deslammation) is a technological process for removing finely dispersed particles (particles typically < 10–20 µm in size) from mineral raw materials or ore pulp.

Improving the desliming process directly translates into purification. This desliming process improves the final purity while reducing energy consumption and preventing excessive waste. The purpose of the work is to evaluate the influence of the fractional composition and external conditions on the distribution of impurities in halite to optimize the purification scheme.

Experimental part

For this article, a sample of natural halite was used, which was taken from the Bakhyt-Tany deposit. The initial material was first dried at room temperature for 24 hours, after which it was mechanically crushed to obtain fractions of different particle size distribution. To determine the distribution of impurities into fractions, the sample was separated by dry sieving through a sieve with a mesh diameter of 0.2 mm. Thus, two fractions were separated: coarse (f > 0.2 mm) and fine (d < 0.2 mm). To accurately separate and sort the material by size, a vertically oscillating laboratory sieve shaker Analysette 3 PRO (FRITSCH) was used. Samples of about 50 g were subjected to wet sieving for approximately 45 minutes, with the vibration amplitude set to 1–2 mm.

The analysis of raw materials and products was conducted using a combination of spectral microscopy, X-ray analysis, and differential thermal studies. Elemental analysis (elemental composition) was performed using X-ray fluorescence spectroscopy on an INCA Energy 450 energy dispersive microanalysis system mounted on a JSM 6610 LV scanning electron microscope, JEOL, Japan. A scanning electron microscope JSM 6610 LV, JEOL, Japan was used to study the microstructure of the samples. Differential thermal analysis (DTA) was carried out on a Q-1500D derivatograph at a heating rate of 10 °C/min in an air atmosphere.

To determine the moisture content, the sample was pre-weighed in analytical scales and dried at 100 ^{0}C in a desiccator. Drying time 6 hours, interval 30 minutes. Chemical analysis was performed according to GOST R 51574-2000 [26].

The discussion of the results

The halite sample came from the Bakhyt Tany deposit (Fig.1). The salt exhibits the typical cubic crystal habit. However, it is distinguished by a grayish-beige color with significant dark inclusions. This is a raw natural substance with contaminants, likely clay and organic matter. Some salt will crumble into a fine powder when rubbed, suggesting the presence of colloidal particles. These characteristics explain the reasons for a preliminary treatment and the refined steps that might be required in the further treatment of the sample. The sample's chemical composition is shown in Table 1 (Fig.2) below.

Figure 1 - The halite sample from the Bakhyt Tany deposit

According to the elemental analysis of the mineral, it can be said that the salt composition is dominated by sodium and chlorine; there are also such elements as silicon, calcium, magnesium and aluminium, which confirm the presence of clay colloidal impurities. Such impurities are usually in a dispersed form and negatively affect the process of crystallization, and so they are very poorly filtered. This, in turn, harms the purification parameters

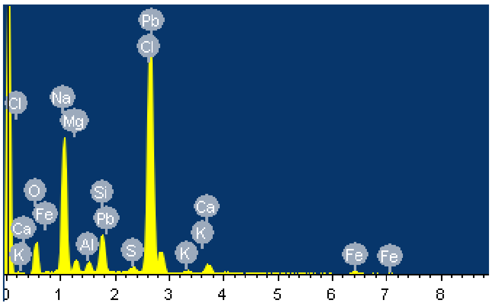

Figure 2 - Elemental composition of halite mineral of the Bakyt-Tany deposit

Table 1 - Elemental composition of halite mineral of Bakyt-Tany deposit, %

Element	Mass %
O	19.61
Na	25.40
Mg	2.36
Al	1.57
Si	4.97
S	0.54
Cl	39.46
K	0.64
Ca	2.21
Fe	1.12
Pb	2.11
Total	100.00

The results of thermal analysis of the natural salt mineral of the Bakhyt-Tany deposit are shown in Figure 3.

Figure 3 - The following report presents the DTA results of the natural sodium salt of the Bakhyt Tany deposit

The DTA of the salt sample from the natural mineral of the Bakhyt-Tany deposit is characterised by three endo effects and three exo effects. During the preliminary heating stage, two non-intensive endothermic effects have been identified at temperatures of 330°C and 560°C. These effects are attributed to the removal of moisture from the crystal structure of sodium and calcium chloride minerals. The intensive endothermic effect in the region of 820-830°C is indicative of the melting of sodium chloride. Endo effects in the region of 470°C and 685°C are associated with the burnout of a small amount of organic sulfur-containing magnesium compounds.

The data obtained has revealed that the natural sodium salt of the Bakhyt Tany deposit contains a high concentration of sodium chloride, with only a minor presence of impurities. It is reasonable to hypothesise that the natural sodium salt obtained at the Bakhyt Tany deposit has the potential to be utilised as a raw material in the production of table salt and soda ash. The calcium sulfate and other silicate compounds contained within insoluble precipitates have the potential to be utilised in the production of construction materials.

According to the results of the study, it was found that the mass fraction of moisture in halite samples ranges from 0.6% to 1.0%. These values are typical for natural materials stored under standard conditions and not subjected to additional drying. Based on the results of the chemical analysis, the approximate salt composition of the studied mineral was calculated. The base is sodium chloride (NaCl), the proportion of which reaches 88.4% of the total mass. In addition, the sample contains impurities in the form of calcium sulfate ($CaSO_4$) — about 2.5%, magnesium sulfate ($MgSO_4$) — 0.18%, and magnesium chloride ($MgCl_2$) — 0.37%. The presence of such impurities significantly affects the quality of salt and its suitability for various types of processing. In particular, the mineral contains up to 2% of the mass fraction of an insoluble residue, which makes it impossible to use it directly in the food industry without a preliminary stage of desalination — the removal of fine and colloidal impurities. Such a level of contamination can not only worsen the organoleptic properties of salt but also affect the technological parameters of crystallisation and filtration during processing. The analysis showed that the insoluble residue has a complex nature and consists of two main phases. The first is a clay mass, represented by finely dispersed insoluble components of natural origin. The second is clearly distinguishable transparent crystals of calcium sulfate, resistant to dissolution in water and prone to precipitation. For a more detailed study of the composition of the insoluble residue, an elemental analysis using X-ray fluorescence spectroscopy was performed. Micrographs, as well as data on the elemental composition of the insoluble residue obtained during the study of halite from the Bakhyt-Tany deposit, are shown in Tables 2 and 3, and also shown in Figures 4 and 5.

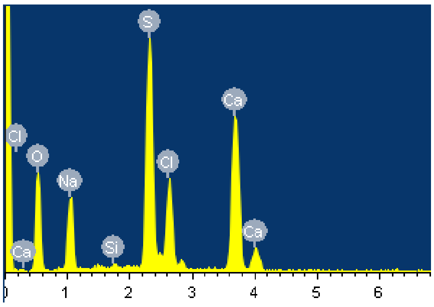

Figure 4 - Elemental composition of crystalline phase of insoluble precipitate of sodium salt mineral of Bakhyt-Tany deposit

The main peaks correspond to Na and Cl, which confirm the presence of halite (NaCl). Additional peaks of S and Ca indicate the occurrence of $CaSO_4$ phases. Minor Si and O peaks are attributed to traces of clay impurities. In Fig.5, besides the dominant Na and Cl peaks, significant signals of Si, Al, Mg, K, and Fe were observed, which are typical of clay minerals such as kaolinite and montmorillonite. The presence of Ca and Ti also supports the association with mineral impurities of terrigenous origin.

Table 2 - Elemental composition of the crystalline phase of the insoluble residue of the mineral of the Bakhyt-Tany deposit, %

Element	Mass %
O	41.34
Na	10.87
Si	0.28
S	18.49
Cl	8.85
Ca	20.16
Total	100.00

The presence of calcium sulfate in halite is of particular importance. On the one hand, $caso_4$ is an undesirable impurity: in the production of soda, it increases the formation of scale in evaporators and reduces the efficiency of the process, and in edible salt, it affects the purity requirements. On the other hand, calcium sulfate is a valuable raw material. Recent research [[27], [28]] highlights its widespread use in building materials, ceramics, soil-improving agents, and asphalt composites. Thus, the removal of $caso_4$ from halite not only improves the quality of salt but also opens up opportunities for processing and reuse of this byproduct in other industries.

The data obtained allowed not only to identify the composition of impurities, but also to understand their distribution inside the material. In order to study the distribution of insoluble impurities by size fractions, the salt sample was pre-crushed and fractionated using a laboratory sieve with a diameter of 0.2 mm. According to the results of sieving, it was found that up to 20% of the total mass of salt is a fine fraction with a particle size of less than 0.2 mm. It contains up to 3.4% of the mass fraction of the insoluble residue, the main part of which is the clay component.

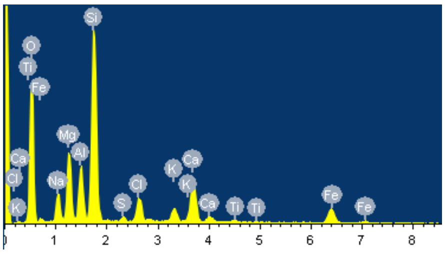

Figure 5 - Elemental composition of the clay phase of the insoluble residue of the sodium salt mineral of Bakhyt-Tany deposit

Table 3 - Elemental composition of the clay phase of the insoluble residue of the mineral of the Bakhyt-Tany deposit, %

Element	Mass %
O	44.64
Na	4.86
Mg	7.79
Al	5.58
Si	19.36
S	0.53
Cl	3.15
K	1.94
Ca	6.27
Ti	0.37
Fe	5.52
Total	100.00

This confirms the tendency of fine impurities to pass into small fractions during grinding and enrichment. The remaining 80% of the mass is accounted for by a large fraction (particles d > 0.2 mm), where the insoluble residue content is much lower — about 1.8%. In this case, calcium sulfate crystals predominate, forming in the salt structure in the form of dense transparent inclusions. Thus, the data obtained allow us to conclude that a comprehensive purification scheme for this halite is necessary, including not only mechanical fractionation and filtration, but also methods aimed at the effective removal of clay and sulfate impurities. Accounting for the distribution of salt by fractions can be useful in designing the technological process of salt processing for food or technical purposes. However, it was found that simple dry sieving is not sufficient to remove the mucous fraction. This may be because colloidal particles are bound to calcium sulphate crystals, forming dense inclusions in the salt matrix. Therefore, the authors conducted additional experiments on mucus removal using a saturated NaCl solution [29]. The results of the experiments showed that the optimal ratio of solid to liquid substances is 1:3, which ensures the most effective removal of clay and colloidal impurities with minimal NaCl losses.

Figure 6 shows the model dependence of the insoluble residue content in the fine salt fraction on temperature and humidity. The graph clearly demonstrates that with an increase in both parameters, both temperature and humidity, an increase in the amount of insoluble impurities is observed.

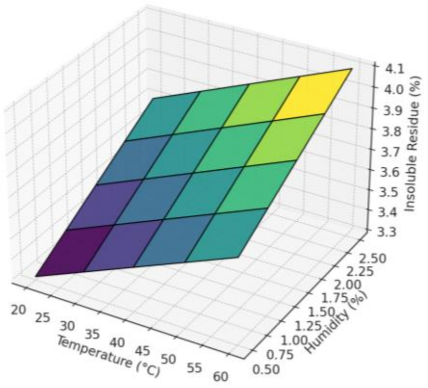

Figure 6 - Simulated 3D Dependence of Insoluble Residue (%) on Temperature(^{0}C) and Humidity (%)

This is because at higher temperatures, degradation and desorption processes are activated, contributing to the release of impurities fixed on crystals, and increased humidity increases the migration of clay and colloidal particles into solution, where they are more difficult to remove. This trend is especially relevant for the storage and processing of natural halite containing up to 2-3% insoluble impurities. The growth of these indicators in conditions of high humidity and temperature requires an adjustment of the cleaning process, for example, an increase in the settling time, the dose of coagulant, or the choice of another method of pre—desalination. Thus, the obtained model makes it possible to evaluate the behavior of impurities under changing external conditions and can be used in designing or optimizing the salt preparation scheme for processing.

Conclusions

The results of the study showed that up to 20% of salt mass goes to the fine fraction, and up to 80% of salt mass remains on the sieve in the coarse fraction. The fine fraction, d<0.2 mm, contains up to 3.4 wt.% of insoluble residue, and most of the clay residue passes into this fraction. The coarse fraction, d>0.2 mm, contains up to 1.8 wt. % of insoluble residue, and the main mass of insoluble residue consists of calcium sulfate crystals.

The compiled mathematical model confirmed a direct correlation between the increase in the fraction of fine fraction and the increase in the total content of insoluble impurities. Based on the fractional composition, it is possible to evaluate the products, which allows optimising the desliming technology.

On the other hand, a model of the effect of temperature and humidity as external factors on the behaviour of impurities was studied. It was demonstrated on a 3D plot that as the mentioned factors increase, the fraction of insoluble residue, especially its fine fraction, increases towards the insoluble residue. This needs to be taken into account in long-term and short-term salt operations to minimise impurities or improve performance.

It was also shown that simple dry sieving is not sufficient for the removal of fine clay and colloidal impurities, since colloidal particles are often associated with calcium sulfate crystals. Effective desliming was achieved only when using a saturated NaCl solution, where the optimal solid-to-liquid ratio was determined experimentally as 1:3.

In general, the work showed that from this deposit, it is necessary to pre-treat the finished halite with the use of fractionation, desliming, and further with the introduction of thermal or chemical after-treatment.

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Натрий хлоридін алу процесінің технологиялық параметрлеріне галиттің минералды қоспаларының әсері

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ТҮЙІНДЕМЕ

Мақалада Бақыт-Таны кен орнынан алынған табиғи галитті кешенді зерттеу нәтижелері келтірілген. Құрамы бойынша бұл минерал – кальций сульфаты, магний тұздары және кейбір басқа заттар қосылған натрий хлоридінен, оның ішінде 2%-дан аз ерімейтін қалдықтардан тұрады. Элементтік талдау нәтижесінде Ca, Mg, Al, Si, Fe және Pb сияқты элементтердің болатыны анықталды, бұл саз балшық пен кейбір сульфатты қоспалардың бар екенін көрсетеді. Әр түрлі өлшемдегі қоспалардың қалай таралатынын анықтау үшін үлгі 0,2 мм ұяшықты елеуіш арқылы фракцияларға бөлінді. Зерттеу нәтижесінде тұз массасының 20 %-дан азы d < 0,2 мм болатын ұсақ фракцияны құрайтыны анықталды, ал бұл фракцияда ерімейтін қалдықтың мөлшері 3,4 %-ға дейін жетеді. Ал ірі фракцияда (d > 0,2 мм) бұл көрсеткіш 1,8 %-дан аспайды. Құрастырылған математикалық модель ұсақ фракцияның мөлшері мен ерімейтін қалдықтың жалпы мөлшері арасында тікелей байланыс бар екенін көрсетті. Бұл модель тұздың ластану деңгейін бағалау және тазалау процесінің тиімділігін болжау үшін пайдаланылуы мүмкін. Сонымен қатар, жасалған үш өлшемді модель температура мен ылғалдылықтың артуы тек ерімейтін қоспалардың концентрациясын жоғарылатып қана қоймай, олардың ұсақ фракцияда жинақталуын да күшейтетінін көрсетті. Зерттеу нәтижелері галитті тағам өндірісінде және басқа да технологиялық мақсаттарда қолданбас бұрын алдын ала тазалау қажет болатынын көрсетті және тазалау процесінің фракциялау мен тұзсыздандыруға негізделген тиімді концепциясын қолдайды.

Түйін сөздер: галит, тазалығы жоғары натрий хлориді, минералды қоспалар, коллоидтық бөлшектер, қайта кристалдану, тазарту.

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Влияние минеральных примесей галита на технологические параметры процесса получения хлорида натрия

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АННОТАЦИЯ

В данной статье представлены результаты детального исследования природного галита с месторождения Бахыт-Таны. По составу минерал представляет собой хлорид натрия с добавлением сульфата кальция, солей магния и некоторых других веществ, в том числе менее 2% нерастворимого остатка. Элементный анализ показывает наличие таких элементов, как Ca, Mg, Al, Si, Fe и Pb, что указывает на наличие примесей глины и некоторых сульфатов. Чтобы понять, как распределяются примеси разного размера, образец был классифицирован с помощью сита с ячейкой 0,2 мм. Было обнаружено, что менее двадцати процентов массы соли составляет мелкая фракция (d < 0,2 мм), в которой содержится до 3,4% нерастворимых примесей, а в крупной фракции (d > 0,2 мм) это значение составляет менее 1,8%. Разработанная математическая модель показала, что мелкодисперсная фракция и общее количество нерастворимого осадка находятся в прямой зависимости, что позволяет использовать ее для оценки загрязнения и эффективности процессов опреснения. Более того, созданная 3D-модель показала, что температура и влажность не только повышают концентрацию нерастворимых примесей, но и еще больше увеличивают концентрацию таких примесей в мелкой фракции. Полученные результаты также подтверждают необходимость предварительной очистки галита перед его использованием в пищевой промышленности и других технологических применениях и подтверждают концепцию процесса очистки галита, основанного на фракционировании и опреснении.

Ключевые слова: галит, хлорид натрия высокой чистоты, минеральные примеси, коллоидные частицы, перекристаллизация, очистка.

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