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# Methods for purifying table salt from the Suzak deposit

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**ABSTRACT**

One of the pressing issues today is common salt purification from harmful impurities and production of salt for medical and household purposes. To obtain high-purity sodium chloride salt, it is necessary to develop more effective methods for purifying salt from impurities. The article discusses modern methods for purifying Suzak deposit common salt from harmful impurities. The main goal of the scientific work is to study the methods of purifying sodium chloride from impurities. The common salt raw material composition was studied. The content of impurities of $Ca^{2+}$, $Mg^{2+}$ and $SO4^{2-}$ ions and heavy metals Pb (II), Cu (II), Cd (II), As (V) was determined. The solubility in the systems NaCl-$Na_2SO_4$-H'$_2$O, NaCl-$CaCl_2$-$H_2O$ and NaCl-$MgCl_2$-$H_2O$ at a temperature of 100-110°C was studied. The effect of temperature and time on the common salt purification degree using active reagents was studied. It was found that the highest common salt purification degree from $Ca^{2+}$, $Mg^{2+}$ and $SO4^{2-}$ at 30 minutes and 90°C, respectively, is 99.8%, 99.9%, 99.93%. It was found that the use of a three-component mixture of $Mg(OH)_2$:$CaCO_3$:$CaSO_4$ in a ratio of 1:4-5:6-7 for 20 minutes during purification allows purifying the NaCl solution from trace impurities of Pb(II), Cu(II), Cd(II), As(V) by 92.0-97.7% and obtaining 99.4% NaCl. To obtain high-purity salt, effective purification methods of salt from impurities are recommended, allowing to achieve a purification level of up to 99%.

***Keywords:*** sodium chloride, table salt, salt purification methods.

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## Introduction

Kazakhstan has huge reserves of mineral raw materials – common salt, the reserves of which amount to more than 1.4 billion tons. In the chemical industry, NaCl is the main raw material for obtaining caustic ash and other inorganic sodium-containing salts. Depending on the purpose, NaCl is used in many industries [[1], [2], [3]].

The main consumer of high-purity sodium chloride is the pharmaceutical and food industries. Particular attention is paid to the methods of purifying common salt from harmful impurities of calcium, magnesium, sulfate ions and heavy metals (Pb, Cd, As, Cu). The common salt purity is considered one of the most important requirements in production. At the same time, the technogenic impact on the environment is growing, which makes a negative contribution to the deterioration of its condition [[4], [5], [6]].

The well-known lime and lime-soda methods do not always purify salts from impurities to the required quality due to the purification system complexity [[7], [8]]. To obtain high-purity salt, large expenses are required, which increase the product cost. Therefore, it is necessary to use a more effective method of purifying salt from impurities [[9], [10], [11]].

This article examines modern methods of purifying common salt [[12], [13], [14], [15], [16]]. Currently, the demand for various methods of producing and processing common salt is growing. The authors [17] found that when using the phosphate method of purifying a salt solution, the purification degree from calcium and magnesium ions increases to 95-97%.

Currently, Kazakhstan does not produce “extra” grade salt due to the lack of production and supplies it mainly from Russia and other countries.

The authors conducted research work on salt purification and the results obtained do not have full-scale application. This is due to the technology developed taking into account the physical and chemical properties of the deposits [[18], [19], [20]. [21], [22]].

For large-scale use in the pharmaceutical and food industries as an extra salt, the Suzak deposit requires chemical purification, since the salt contains many impurities.

The purpose of the scientific article is to study the physical and chemical properties of table salt and to propose modern methods of purification from harmful impurities and heavy metals using active reagents and precipitants.

To study the common salt composition, samples from Suzak deposit were used (Table 1).

**Table 1** – Chemical composition of Suzak deposit common salt samples

| Salt composition of common salt, % | | | | | | |
|---|---|---|---|---|---|---|
| NaCl | KCl | $MgSO_4$ | $MgCl_2$ | $CaCl_2$ | $CaSO_4$ | Insoluble residue |
| 93.41 | 0.01 | 0.23 | 0.14 | 0.37 | 2.29 | 3.55 |
| 97.08 | 0.02 | 0.10 | 0.045 | 0 | 0.16 | 2.67 |
| 99.07 | 0.019 | 0.11 | 0.046 | 0 | 0.15 | 0.61 |
| 98.68 | 0.024 | 0.12 | 0.047 | 0 | 0.17 | 0.95 |

Table 1 shows that, according to the results of chemical analysis, natural common salt contains mainly halite mineral and impurities of clay-carbonate and sulfate materials – sulfate and calcium and magnesium, magnesium chloride, calcium chloride and potassium chloride. The salt is significantly contaminated with insoluble residues (silt, sand).

## Experimental part

***Object of research.*** The purpose of the work is to purify sodium chloride from harmful impurities and obtain high-purity salt. In accordance with the logic of scientific research, a research methodology was selected for conducting the experiment. It is a complex of theoretical and experimental methods, the combination of which makes it possible to most reliably study such a complex problem of complex purification of common salt from harmful impurities and heavy metals using active reagents and precipitants.

The following research methods were used in the work: chemical, mass spectrometry, X-ray phase, scanning electron microscopy and IR spectroscopy and differential thermal analysis. The experiments were conducted on a laboratory thermostatted unit.

***Experimental methodology.*** In the process of isotherm (Fig. 3, 4) of solubility of water-salt systems with the participation of NaCl at elevated temperatures, in the case of incomplete separation of Ca, Mg, $SO_4$ ions from saturated salt solutions, the remaining minor impurities contained in the liquid phase during hot filtration of solid sodium chloride pass into the filtrate.

The saturated NaCl solution separated from the sediment is evaporated at 100 - 110 °C to 1/2 of the original volume. The precipitated crystalline target product is separated from the hot solution. The mother liquor is again evaporated to 1/2 of the initial volume, the precipitated crystals are separated with NaCl and dried at 25-110 °C for 30 minutes.

## The discussion of the results

The average composition of Suzak deposit salt samples No.1 and No.2 was prepared. The elemental composition of table salt is as follows,%: O-1.05, Na-36.91, Mg-0.21, S-0.30, Cl-61.72, Ca-0.11. Microstructure of Suzak deposit natural table salt are shown in Figure 1.

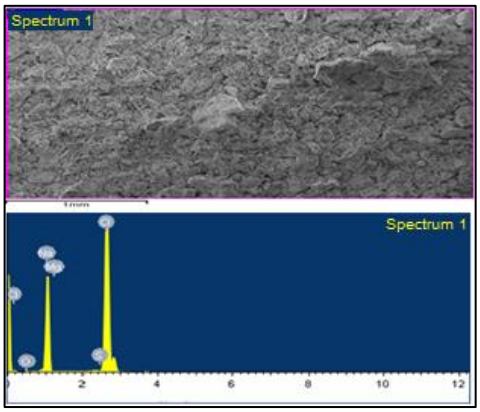


**Figure 1** – Energy dispersive analysis of Suzak deposit table salt

It is evident from Figure 1 that the studied sample contains sodium, magnesium, calcium, chlorine and sulfur bound in the form of compounds NaCl, KCl, $MgS0_4$, $MgS0_4$, $MgCl_2$, $CaCl_2$ and $CaSO_4$.

Figure 2 shows the IR spectrum of Suzak deposit common salt. The IR spectrum (Figure 2) of common salt has intense absorption bands with wavelengths of 609, 612, 1091 $cm^{-1}$, corresponding to the Na-CI bond, other absorption frequency intensities are not observed.

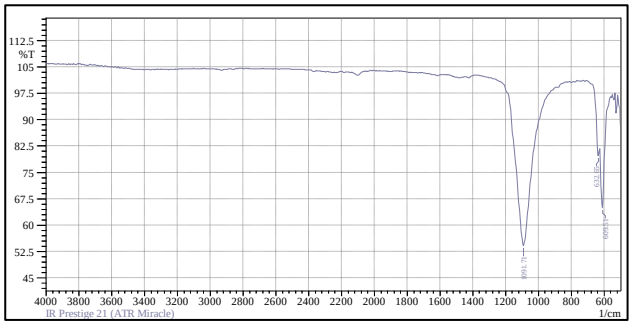


**Figure 2** – IR spectrum of Suzak deposit table salt

Suzak district has large reserves of rock table salt, which is mined in an open way. There are practically no overburden rocks. Suzak steppe is rich in minerals and provides the Republic of Kazakhstan with various types of raw materials. One of the leading places in reserves is occupied by common salt with the following composition of deposits: NaCl – 90.9-98.2%; Ca – 0.45-0.69%; Mg – 0.12-0.19%; $SO_4^{2-}$ – 1.98-2.7%; insoluble residue – 2.0-3.0%.

For deep purification and obtaining high-purity common salt, it is necessary to study the solubility diagrams of $NaCl-CaCl_2-H_2O$ and $NaCl-MgCl_2-H_2O$. To study the salt composition, salt samples were selected, the composition of which is given in Tables 2 and 3.

**Table 2** – Chemical composition of Suzak deposit table salt samples

| Salt composition of common salt, % | | | | | | |
|---|---|---|---|---|---|---|
| NaCl | KCl | $MgS0_4$ | $MgCl_2$ | $CaCl_2$ | $CaSO_4$ | Insoluble residue |
| 93.41 | 0.01 | 0.23 | 0.14 | 0.37 | 2.29 | 3.55 |
| 97.08 | 0.02 | 0.109 | 0.045 | 0 | 0.16 | 2.67 |

Table 2 shows that natural table salt contains mainly halite mineral and impurities of sulfate and calcium and magnesium, magnesium chloride, calcium chloride and potassium chloride.

To determine the content of other impurities and heavy metals, 2 samples of pre-prepared salt brine were analyzed using a Varian ICP-820MS inductively coupled plasma mass spectrometer. Table 3 shows the content of trace impurities (Pb, Cu, Cd, As).

**Table 3** – Content of heavy metals in salt

| Sample No. | Trace impurity content, μg/$dm^3$ | | | |
|---|---|---|---|---|
| | Pb | Cu | Cd | As |
| 1 | 18.21 | 67.53 | 9.00 | 14.01 |
| 2 | 16.01 | 60.12 | 7.3 | 12.43 |

The analysis results of common salt samples (Table 3) show that in addition to impurities such as calcium, magnesium, aluminum, iron, etc., the salt also contains heavy metals such as lead, copper, cadmium and arsenic, which requires purification to obtain “extra” common salt.

As can be seen, Suzak salt contains sodium, magnesium, calcium, chlorine and sulfur bound in the form of compounds NaCl, $CaCl_2$, $CaSO_4$, $MgS0_4$, $MgCl_2$, KCl and heavy metals. In this regard, it is of interest to study the solubility diagrams of saturated solutions of common salt in the presence of the above-mentioned impurities [[18], [19]].

For deep purification of salt from impurities, it is necessary to study solubility in the systems $NaCl-CaCl_2-H_2O$ and $NaC1-MgCl_2-H_2O$. The solubility isotherm in the system $NaCl-Na_2SO_4-H_2O$ was studied at the temperature of the saturated sodium chloride solution – 108.5°C. The time for establishing equilibrium was found by reaching equilibrium of sodium chloride in the solution [19].

The experimental data on the solubility in the above-mentioned systems are shown in Gibbs coordinates on Figures 3 and 4.

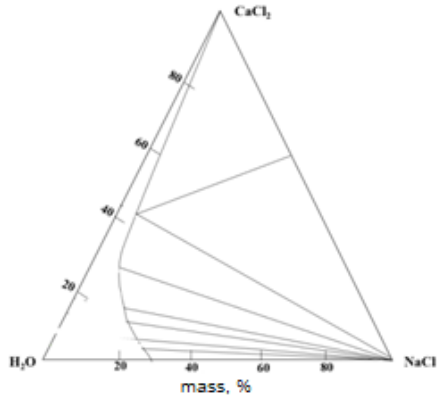


**Figure 3** – Solubility isotherm in the system

NaCl-$CaCl_2$-$H_2O$ at 100°C

From the data in Figure 3 it is evident that the solubility isotherm of the system NaCl-$CaCl_2$-$H_2O$ at 100°C consists of two branches: the NaCl crystallization branch in the region of 6.1÷28.3 mass % NaCl and the $CaCl_2$ crystallization branch in the region of 60.3÷41.5% mass % $CaCl_2$. The eutonic point has the following composition: liquid phase 1.9% NaCl; 41.5% $CaCl_2$; solid phase 39.5% NaCl; 55.1% $CaCl_2$.

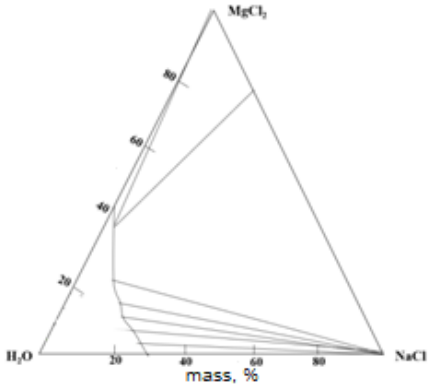


**Figure 4** – Solubility isotherm in the system NaCl-$MgCl_2$-$H_2O$ at 100°C

Similarly, from the data in Figure 4 it is evident that the solubility isotherm in the system NaC1-$MgCl_2$-$H_2O$ at 100°C consists of two branches: the NaCl crystallization branch in the region of 28.3÷0.9 mass % NaCl and the $MgCl_2$ crystallization branch in the region of 42.2÷35.0 mass % $MgCl_2$. The eutonic point has the following composition: liquid phase 0.9% NaCl; 35.0% $MgCl_2$; solid phase 5.5% NaCl: 38.5% $MgCl_2$. As shown by the solubility isotherms of aqueous salt systems with the participation of NaCl at elevated temperatures, in the case of incomplete separation of Ca, Mg, and $SO_4$ ions from saturated solutions of technical salt, the remaining minor impurities contained in the liquid phase during hot filtration of solid sodium chloride pass into the filtrate.

To purify the obtained sodium chloride from trace impurities (Pb, Cu, Cd, As), studies were conducted at various concentrations of sodium chloride solution 80-140 g/l NaCl, temperature 70-90°C, contact time of the residue with the solution of 20 minutes and molar ratio of $Mg(OH)_2$:$CaCO_3$:$CaSO_4$ in the residue 1:4-5:6-7. The three-component mixture was subsequently used as a precipitant for trace impurities.

A solution of magnesium and calcium chloride, soda and alkali in an amount of 0.1-0.2% of the total mass of the solution was added to the previously obtained solution. The resulting mixture was stirred for 20 minutes, the residue was allowed to settle and the residue was filtered from the sodium chloride solution. Quantitative determination of trace impurities of lead, copper, cadmium and arsenic was carried out by the atomic adsorption method on a Contra device. The experimental results are given in Table 4.

**Table 4** – Residual content of trace impurities after purification (μg/$dm^3$), and purification degree (%)

| Elements | Concentration of sodium chloride solution, g/l | | | | |
|---|---|---|---|---|---|
| | 80 | 100 | 120 | 130 | 140 |
| Pb | 0.65 | 0.65 | 0.82 | 1.01 | 1.25 |
| | 91.2 | 93.4 | 93.1 | 93.3 | 93.1 |
| Cu | 1.81 | 1.80 | 2.20 | 2.10 | 3.37 |
| | 93.4 | 95.1 | 95.2 | 95.1 | 95.0 |
| Cd | 0.37 | 0.36 | 0.46 | 0.56 | 0.70 |
| | 89.9 | 92.6 | 92.4 | 92.3 | 92.2 |
| As | 0.13 | 0.43 | 0.71 | 1.32 | 2.78 |
| | 97.7 | 94.3 | 93.8 | 88.4 | 80.1 |
| * numerator – content of trace impurities, μg/$dm^3$; denominator – purification degree, % | | | | | |

From the above data of Table 6 it is evident that deep purification of the sodium chloride solution from trace impurities of Pb, Cu, and Cd is within 89.9-95.1% and from As by 80.1-97.7%. The concentration of mixtures in these solutions is more than 3 mmol/l, while the molar ratio of the mixture $Mg(OH)_2$:$CaCO_3$:$CaSO_4$ should be 1:4-5:6-7, and the contact time of the residue is not less than 20 minutes. The three-component mixture $Mg(OH)_2$:$CaCO_3$:$CaSO_4$ obtained in the process for purification of the sodium chloride solution from trace impurities of heavy metals is more effective than the known single-component coagulants based on $Fe(OH)_3$, $Mg(OH)_2$, $CaCO_3$, $CaSO_4$.

Based on the obtained data, 5 $cm^3$ of 0.1 M $Ca(OH)_2$ and $BaCl_2$ solutions are added to 1 $dm^3$ of the sodium chloride solution (100 g/$dm^3$) for purification. Magnesium hydroxide is precipitated, then 2 $cm^3$ of 1 M sodium carbonate solution and 3 M ammonia solution are added to pH 12-13.

The resulting purified sodium chloride solution has the following composition, mass %: NaCl – 13.9; $Na_2SO_4$ – 0.0002; $CaSO_4$ – 0.02; $MgSO_4$ – 0.001; $H_2O$ – 86.06 and trace impurities, μg/$dm^3$: Pb 0.65-1.01; Cd 0.37-0.56; Cu 1.81-2.10; As 0.13-1.32. After cooling the solution to 25°C, sodium chloride

crystals containing 99.4% NaCl precipitate. Figure 5 shows the energy dispersion analysis of the obtained sodium chloride. The elemental composition of table salt is as follows,%: O-0.95, Na-37.37, Mg-0.001, S-0.000, Cl-61.46, Ca-0.002. Figure 5 shows the energy dispersion analysis of the obtained sodium chloride.

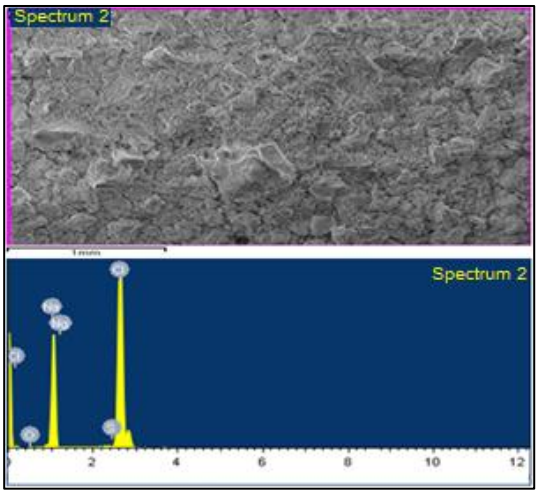


**Figure 5** – Energy dispersion analysis of the purified salt

From Figure 5 it can be seen that the impurity content is Ca-0.002%, Mg-0.001%, and there is no sulfate ions.

Figure 6 shows derivatograms of purification of sodium chloride obtained using a Q-1500 Derivatograph.

**Figure 6** – DTA of the residue obtained during the purification of sodium chloride

On the derivatograms of the resdiues (Figure 6) obtained during the purification of sodium chloride, the endothermic effect of dehydration of $CaSO_4 \cdot 2H_2O$ (140-160 and 120-180ºC ) is visible, the endothermic effect of dehydration of $CaSO_4 \cdot 0{,}5H_2O$ (160-180$^{O}$C) is clearly expressed, i.e. dehydration of hemihydrate to anhydrite occurs.

X-ray phase analysis of the studied table salts was carried out on a Panalytical Empyrean X-ray diffractometer and results are shown in Figure 7.

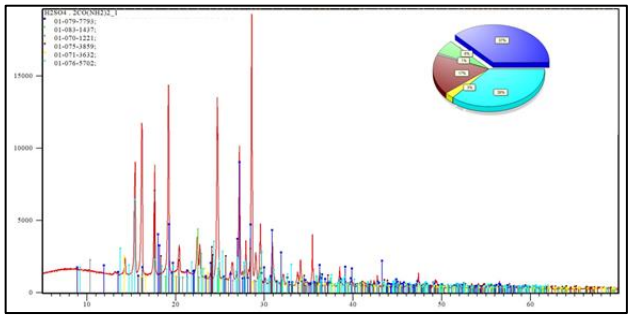

**Figure 7** – Diffractogram of the purified sodium chloride

X-ray phase analysis data (Fig. 7). compounds of table salt, it can be noted that all reflections in diffraction patterns, as a rule, are characterized by their own reflection angles, a set of interplanar distances and intensities of diffraction lines. This indicates the individuality of the crystal lattices of the resulting pure compounds of table salt

Thus, the use of a three-component mixture consisting of $Mg(OH)_2$:$CaCO_3$:$CaSO_4$ in a ratio of 1:4-5:6-7 and contact of the residue with it for 20 minutes allows to purify the NaCl solution from trace impurities of Pb(II), Cu(II), Cd(II), As(V) by 92.0-97.7% and obtain 99.4% NaCl that meets the requirements of GOST P51574-2018.

Based on the work carried out, it was determined that the proposed method for purifying table salt from harmful impurities is effective, since the degree of purification is up to 99.0%. This method is aimed at the Suzak deposits, taking into account the physical and chemical properties of table salt.

A distinctive feature of this method from other methods is the study of the physicochemical properties of table salt, taking into account the composition of harmful impurities and heavy metals using active reagents and precipitants.

## Conclusions

As a result of the research work, Suzak deposit common salt's raw material composition was studied and the content of impurities of $Ca^{2+}$, $Mg^{2+}$ и $SO_4^{2}$ ions and the content of heavy metals Pb (II), Cu (II), Cd (II), As (V) were determined. The solubility in the systems NaCl-$Na_2SO_4$-$H_2O$, NaCl-$CaCl_2$-$H_2O$ and NaCl-$MgCl_2$-$H_2O$ was studied and the isotherm of salt solubility at a temperature of 100-110°C was constructed.

The effect of temperature and time on the purification degree of common salt using barium chloride and sodium carbonate was studied, it was found that the highest purification degree of common salt from $Ca^{2+}$, $Mg^{2+}$ и $SO_4^{2}$ ions at 30 minutes and 90°C, respectively, is 99.8%, 99.9%, 99.93%. It was found that the use of a three-component mixture of $Mg(OH)_2$:$CaCO_3$:$CaSO_4$ in a ratio of 1:4-5:6-7 for 20 minutes allows purifying the NaCl solution from trace impurities of Pb(II), Cu(II), Cd(II), As(V) by 92.0-97.7% and obtaining 99.4% NaCl.

Based on the data obtained, effective methods for purifying salt from impurities were recommended, and the combined method can achieve salt purification above 99.0%.

***CRediT author statement:*** **A. Anarbayev:** Conceptualization, formal analysis, investigation, data writing, original draft preparation, writing–review and editing; **B. Kabylbekova:** Data curation, writing draft preparation, methodology; **B. Smailov:** Resources, supervision, software, investigation; **G. Ormanova:** visualization, validation.

**Conflicts of Interest.** On behalf of all authors, the correspondent author declares no conflict of interest.

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# Созақ кен орнының ас тұзын қоспалардан тазарту әдістері

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**ТҮЙІНДЕМЕ**

Бүгінгі күннің өзекті мәселелерінің бірі – ас тұзын зиянды қоспалардан тазарту және медициналық және тұрмыстық ас тұзын өндіру. Белгілі болғандай, «экстра» ас тұзына сұраныс жыл сайын артып келеді. Жоғары тазалықтағы натрий хлоридін алу үшін тұзды қоспа заттардан тазалаудың тиімді әдісін жасау қажет. Мақалада Созақ кен орнының ас тұздарын қоспалардан тазалаудың қазіргі жетілдірілген әдістері қарастырылған. Ғылыми жұмыстың негізгі мақсаты натрий хлоридін қоспа заттардан тазалау әдісін зерттеу. Ас тұзының бастапқы шикізатының құрамы зерттеліп, ол $Ca^{2+}$, $Mg^{2+}$ және $SO_4^{2-}$ иондарынан және Pb(II), Cu(II), Cd(II), As(V) ауыр металдарынан тұратыны анықталды. 100-110$^{O}$C температура аралығында NaCl - $CaCl_2$ - $H_2O$ және NaCl-$MgCl_2$-$H_2O$ жүйелеріндегі тұздардың ерігіштігі зерттелді. Белсенді заттарды қолданып ас тұзын тазалау дәрежесіне температура мен уақыттың әсері зерттеліп, ас тұзын $Ca^{2+}$, $Mg^{2+}$ және $SO_4^{2}$ иондарынан жоғары дәрежеде тазалау 30 минутта және 90$^{O}$C температурада болатыны және сәйкесінше тазалау дәрежесі 99,8%, 99,9%, 99,93% құрайтыны анықталды. $Mg(OH)_2$:$CaCO_3$:$CaSO_4$ үш компонентті қоспасын 1:4-5:6-7 қатынасында қолданып 20 минут ішінде NaCl ерітіндісін Pb(II), Cu(II), Cd(II), As(V) микроқоспаларынан 92,0-97,7% дейін тазартуға болады және тазалығы 99,4% NaCl алынатыны анықталды. Жоғары тазалықта тұзды алу үшін, 99% дейін тазарту деңгейін қамтамасыз ететін, қоспалардан тұзды тазартудың тиімді әдістері ұсынылады.

***Түйін сөздер:*** натрий хлориді, ас тұзы, тұзды тазалау әдістері.

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# Способы очистки поваренной соли Сузакского месторождения от примесей

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**АННОТАЦИЯ**

Одним из актуальных вопросов на сегодняшний день является очистка поваренной соли от вредных примесей и получение соли медицинского и бытового назначения. Как известно, ежегодно на потребность пищевой соли «экстра» растет. Для получения высокочистой соли хлорида натрия необходимо разработка более эффективных методов очистки соли от примесей. В статье рассмотрены современные методы очистки поваренной соли Сузакского месторождения от вредных примесей. Основная цель научной работы исследование методов очистки хлорида натрия от примесей. Исследован состав исходного сырья поваренной соли и определены содержание примесей $Ca^{2+}$, $Mg^{2+}$ и $SO_4^{2-}$ионов и тяжелых металлов Pb(II), Cu(II), Cd(II), As(V). Изучен растворимость в системе NaCl-$Na_2SO_4$-$H_2O$, NaCl - $CaCl_2$ - $H_2O$ и NaCl-$MgCl_2$-$H_2O$ при температуре 100-110$^OC$. Исследован влияние температуры и времени на степень очистки поваренной соли с использованием активных реагентов, установлен что наивышее степень очистки поваренной соли от $Ca^{2+}$, $Mg^{2+}$ и $SO_4^{2}$ при 30 мин. и 90$^OC$ соответственно составляет 99,8%, 99,9%, 99,93%. Установлена, что использование при очистке трехкомпонентной смеси $Mg(OH)_2$:$CaCO_3$:$CaSO_4$ в соотношении 1:4-5:6-7 в течение 20 минут позволяет очистить раствор NaCl от микропримесей Pb(II), Cu(II), Cd(II), As(V) на 92,0-97,7% и получить 99,4% NaCl. Для получения соли высокой степени чистоты рекомендованы эффективные методы очистки соли от примесей позволяющие достичь степень очистки до 99%.

***Ключевые слова:*** хлорид натрия, поваренная соль, методы очистки соли.

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