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# NamMTI ILMIY-TEXNIKA JURNALI TAHRIR HAY'ATI A'ZOLARI

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# PRODUCTION OF MAGNESIUM CHLORIDE FROM MAGNESIUM-CONTAINING BRINES BY THE ISOTHERMAL METHOD AT 25 °C BASED ON THE SYSTEM $2Na^+, Mg^{2+} \parallel SO_4^{2-}, 2Cl^- – H_2O$

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**Abstract**: This study investigates the production of magnesium chloride from magnesium-containing brines by the isothermal method at 25 °C within the quaternary system $2Na^+, Mg^{2+} // SO_4^{2-}, 2Cl^- – H_2O$. Natural brines of the Alat and Kagan lakes (Bukhara region) were analyzed as potential raw materials. The solubility diagram of the reciprocal system was constructed, and the exchange reaction $2NaCl + MgSO_4 \rightleftarrows Na_2SO_4 + MgCl_2$ was confirmed. Crystallization fields of the main solid phases were determined, revealing the high solubility of magnesium chloride compared to other components. Based on phase equilibrium analysis, a technological approach for stepwise separation of sodium and magnesium salts is proposed, providing a scientific basis for organizing magnesium chloride production from local saline resources.

**Keywords:** magnesium chloride; isothermal method; quaternary system; phase equilibrium; saline brines; crystallization.

**Introduction.** Worldwide, significant attention is given to the production of magnesium compounds. Magnesium, being one of the most abundant elements, ranks eighth in terms of its content in the Earth's crust. The production of magnesium compounds increases annually, while their fields of application continue to expand. The main consumers are the refractory industry, construction sector, and metallurgical industry. In recent years, with the development of nanotechnology, special high-purity magnesium compounds have been used in the production of plastics.

**Main part.**The main sources of magnesia raw materials are magnesite ($MgCO_3$), brucite ($Mg(OH)_2$), dolomite ($CaCO_3 \cdot MgCO_3$), carnallite ($MgCl_2 \cdot KCl \cdot 6H_2O$), bischofite ($MgCl_2 \cdot 6H_2O$), seawater, and brines of inland lakes [81]. The most widely used type of industrial raw material is magnesite. The largest explored reserves of magnesite are found in China, Russia, Slovakia, the DPRK, Australia, and Turkey. These countries account for more than 90% of the total proven global reserves.

Seawater represents an almost inexhaustible raw material source for the production of magnesium compounds. Magnesium is present in even higher concentrations in the brines of salt lakes than in seawater.

Magnesium compounds and metallic magnesium were first produced from seawater by Dow Chemical at the Freeport plant (Texas, USA) in 1948. At present, the only remaining producer of metallic magnesium from seawater is Dead Sea Magnesium Ltd (Israel).

The largest producer of magnesium chloride in Israel is Dead Sea Works Ltd (DSW). Bischofite is extracted from the waters of the Dead Sea. Each liter of Dead Sea water contains approximately 170 g of magnesium chloride. Seawater is evaporated in solar evaporation ponds to a concentration of 33%. Magnesium chloride in the form of flakes or granules is obtained by further evaporation to a concentration of 47%. The production capacity exceeds 100,000 tons per year.

Most of the magnesium chloride produced in the United States is used for the manufacture of magnesium compounds. In the U.S., liquid magnesium chloride accounts for 90–93% of total production, while 7–10% is in flake form. The total domestic consumption of magnesium chloride is approximately 220 thousand tons per year. Magnesium chloride is obtained from the brines of salt lakes and from seawater.

Chinese manufacturers are primarily focused on the production of powdered and flake anhydrous magnesium chloride, as well as ingot products containing 98–99% $MgCl_2$, along with a smaller volume of liquid magnesium chloride (23–25% $MgCl_2$).

In Uzbekistan, more than 50,000 tons of magnesium chloride (bischofite) are imported annually from Russia, Turkmenistan, and China solely for the production of defoliants. In order to save foreign currency resources, it is necessary to organize the production of bischofite based on local raw material sources. In this regard, a key objective is the development of a technology for producing magnesium chloride from the saline lakes of the Kagan and Alat districts of the Bukhara region. It should be noted that the technologies currently known worldwide are not suitable for processing the brines of these lakes.

At present, measures are being taken to eliminate the consequences of rising water levels and salinization. As a result of decreasing water levels and increasing salinity, numerous salt layers are formed. An example of this is the reserve sources of mineral salts rich in various chemical elements located within the “Kogan” fishery in the Kagan district of the Bukhara region.

The study of salt lakes formed in the Alat district and the analysis of their composition can provide practical assistance in addressing the above-mentioned tasks.

The conducted investigations have established that mineral salts and their solutions contain a number of valuable chemical elements and their compounds.

It has been established that these saline waters consist mainly of sodium, magnesium chloride, and sulfates; however, their complete separation has not been studied. This leads to the formation of salt dust during strong winds, which adversely affects crop yields and human health. By processing these brines, it is possible to obtain magnesium compounds, meet the existing demand for them, and prevent environmental pollution.

This article presents the results of a scientific study aimed at investigating the process of magnesium chloride production from the existing lakes of the Bukhara region and at developing an appropriate technology.

In the study, brine from the Alat district lake with the following composition (wt. %) was used: $Na_2O$ — 11.53; CaO — 3.11; MgO — 10.55; $Cl^-$ — 16.90; $SO_4^{2-}$ — 8.35; $MgCl_2$ — 8.88; NaCl — 32.65; $MgSO_4$ — 14.31; insoluble residue — 0.33.

The composition of the saline water from the Kagan district lake is as follows (wt. %): $Na_2O$ — 12.33; $K_2O$ — 2.8; CaO — 4.10; MgO — 11.55; $Cl^-$ — 17.37; $SO_4^{2-}$ — 9.69; $MgCl_2$ — 9.2; KCl — 5.35; NaCl — 31.35; $MgSO_4$ — 15.5; insoluble residue — 0.43.

The pH of both liquid and solid salts ranges from 6.8 to 7.1, and the density is 1.340–1.420 $g/cm^3$.

The above data indicate that the saline water contains significant amounts of sodium and magnesium sulfates as well as chlorides, and that, with knowledge of their mutual equilibrium characteristics, their separation can be achieved. Therefore, the solubility diagram of the system $2Na^+, Mg^{2+} // 2Cl^-, SO_4^{2-} – H_2O$ was studied by the isothermal method at 25 °C.

The most common ions in natural waters that determine their properties are $Na^+$, $K^+$, $Ca^{2+}$, $Mg^{2+}$, $Cl^-$, $SO_4^{2-}$, $HCO_3^-$, and $CO_3^{2-}$; present in smaller amounts are $Fe^{2+}$, $Fe^{3+}$, $Mn^{2+}$, $F^-$, $Br^-$, $I^-$, $B_2O_3$, $HPO_4^{2-}$, $SO_3^{2-}$, $HS^-$, and $HSiO_3^-$. These minor components generally do not significantly affect the properties of the brine, but in certain cases they determine its technological characteristics.

The chemical composition of natural waters and their total dissolved salt content, referred to as salinity, vary widely.

The compositions of many natural waters have been plotted on combined salt projections of three- and four-component systems: sodium chloride – magnesium sulfate – water; magnesium chloride – calcium chloride – sodium chloride – water; as well as sodium chloride, sulfate, and bicarbonate systems with water.

As can be seen, the composition of natural waters, with certain limitations, can be described by a system containing at least eight components: $Ca^{2+}, Mg^{2+}, Na^+, K^+ // CO_3^{2-}, 2HCO_3^-, Cl^-, SO_4^{2-} – H_2O$. A clear graphical representation of such complex systems requires significant simplifications and largely loses its practical meaning. The analysis and investigation of the processes occurring within them are extremely complicated; therefore, a rational classification becomes necessary [1].

**Methods**. In halurgical practice, the process occurring in the system $2Na^+$, $2K^+$, $Mg^{2+}$ // $SO_4^{2-}$, $2Cl^-$ – $H_2O$ is of particular importance, as it approximately corresponds to the composition of seawater at 25 °C. This system adequately describes the processes of seawater concentration under both laboratory and natural conditions. Since natural evaporation takes place in regions with an arid (desert) climate, the average temperatures during the evaporation season range from 25 to 35 °C [2, 3].

The constituent ternary systems have been sufficiently well studied by the solubility method [4–11].

In the study of the aqueous system $2Na^+$, $Mg^{2+}$ // $2Cl^-$, $SO_4^{2-}$ – $H_2O$ at 25 °C, sodium and magnesium sulfates and chlorides interact according to the reaction:

$$2Na^+, Mg^{2+} // 2Cl^-, SO_4^{2-} - H_2O$$

$$2NaCl + MgSO_4 \rightleftarrows Na_2SO_4 + MgCl_2$$

In our study of solubility in the mutual aqueous system of sodium and magnesium chlorides and sulfates, phase equilibrium was established within 3–4 days. The starting points for investigating the quaternary system $2Na^+$, $Mg^{2+}$ // $2Cl^-$, $SO_4^{2-}$ – $H_2O$ were the binary eutonic solutions of the boundary ternary systems.

Upon addition of the third component, the figurative points of the equilibrium liquid phase moved inward into the concentration quadrilateral along the boundary lines separating the fields of two solid phases, terminating at two isothermal invariant points corresponding to the coexistence of three different solid phases (Fig. 1, Table 1).

As shown in Figure 1, at a temperature of 25 °C the crystallization field is divided by the phase boundaries of NaCl, mirabilite — $Na_2SO_4 \cdot 10H_2O$, thenardite — $Na_2SO_4$, bischofite — $MgCl_2 \cdot 6H_2O$, epsomite — $MgSO_4 \cdot 7H_2O$, and the compound $Na_2SO_4 \cdot MgSO_4 \cdot 4H_2O$.

The field bounded by nodal points 8, 9, 15, 19, and 25 corresponds to the crystallization of $Na_2SO_4 \cdot MgSO_4 \cdot 4H_2O$ — astrakhanite. Point 19 is a triple point and represents the simultaneous crystallization of sodium chloride and magnesium sulfate together with sodium sulfate. The second field, located between points 6, 7, 15, and 25, occupies the smallest area and corresponds to the precipitation of thenardite. The third field belongs to mirabilite and is separated from the fields of other salts by points 2, 6, 8, and 25.

The solubility diagram shows that the major portion of the diagram is occupied by the crystallization fields of astrakhanite, epsomite, and sodium chloride, whereas the crystallization field of magnesium chloride occupies the smallest area. This indicates its high solubility compared to the other components of the system.

The solubility diagram indicates that astrakhanite dissolves in water at 40 °C at a specific component ratio; the resulting solution is then cooled, and at 0 °C mirabilite crystallizes. The eutectic solution, after separation of mirabilite, is subjected to evaporation. The concentrated solution obtained is cooled to 25 °C, resulting in the precipitation of epsomite. After separation of epsomite crystals, the mother liquor is processed by two alternative methods.

According to the first method, the mother liquor is diluted with water at a specified component ratio; the resulting solution is cooled, and the cycle is repeated. This method is carried out under batch conditions, and after each cycle the total mass of the system gradually decreases.

The second method consists in mixing the obtained solution at 40 °C with the initial astrakhanite; the component ratios vary depending on the position of the figurative points of the system. The resulting mixture is dissolved in water at 40 °C, then cooled, and mirabilite crystallizes at 25 °C. Thus, the cycle is repeated.

To separate sodium chloride and sodium sulfate, the resulting salt mixture is dissolved in water at 20 °C at a specified component ratio. The obtained suspension is cooled to 0 °C, and after separation of mirabilite, a solution is formed. This solution is then sent to the evaporation stage. The filtrate is directed to the repulpation stage of the initial precipitate.

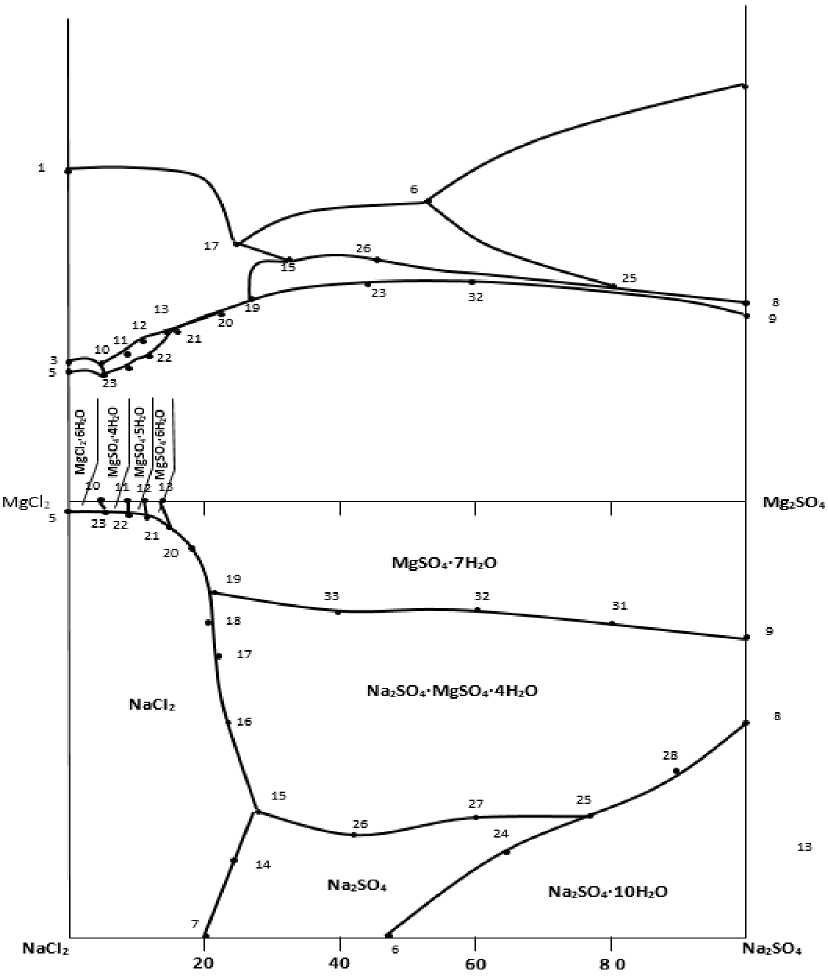

**Figure 1.** Solubility diagram of the reciprocal system $2Na^+$, $Mg^{2+}$ // $2Cl^-$, $SO_4^{2-}$ – $H_2O$ at 25 °C

Their ratio is determined by the position of the figurative points of the system, while their quantity depends on the ratio of water to the constituent components of the system. The resulting pulp contains a salt mixture consisting of NaCl, $Na_2SO_4$, and $Na_2SO_4 \cdot 10H_2O$.

To dissolve NaCl and recrystallize thenardite ($Na_2SO_4$) into mirabilite ($Na_2SO_4 \cdot 10H_2O$), additional water is introduced. The required amount of water is determined from the solubility diagram. Thus, the cycle is closed, and as a result of evaporating the glaserite solution, a mirabilite precipitate is formed, which is returned to the stage of glaserite production and finished edible sodium chloride After this, magnesium chloride is obtained as the final product.

**Table 1.** Solubility data for the system $2Na^+$, $Mg^{2+}$ // $2Cl^-$, $SO_4^{2-}$ – $H_2O$ at 25 °C

| Composition point number | Composition of the liquid phase,% | | | | | Composition of the liquid phase, mol/100 t of solution | | | | Indices | | | Solid phase |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | NaCl | $Na_2SO_4$ | $MgCl_2$ | $MgSO_4$ | $H_2O$ | NaCl | $Na_2SO_4$ | $MgCl_2$ | $MgSO_4$ | $Mg^{2+}$ | $SO_4^{2-}$ | $H_2O$ Mole/ Mole $\sum$ salts | |
| 1. | 26,41 | - | - | - | 73,59 | 0,2257 | 0 | 0 | 0 | 0 | 0 | 18,114 | NaCl |
| 2. | - | 21,8 | - | - | 78,2 | 0 | 0,1535 | 0 | 0 | 0 | 1 | 28,302 | $Na_2SO_4$*$10H_2O$ |
| 3. | - | - | 35,6 | - | 64,4 | 0 | 0 | 0,3738 | 0 | 1 | 0 | 9,571 | $MgCl_2$*$6H_2O$ |
| 4. | - | - | - | 26,7 | 73,3 | 0 | 0 | 0 | 0,2218 | 1 | 1 | 18,36 | $MgSO_4$*$7H_2O$ |
| 5. | 0,3 | - | 35,5 | - | 64,2 | 0,0026 | 0 | 0,3728 | 0 | 0,9931 | 0 | 9,501 | NaCl+$MgCl_2$*$6H_2O$ |
| 6. | 13,67 | 14,82 | - | - | 71,51 | 0,1168 | 0,1044 | 0 | 0 | 0 | 0,472 | 17,96 | $Na_2SO_4$*$10H_2O$+$Na_2SO_4$ |
| 7. | 23,10 | 6,89 | - | - | 70,01 | 0,1974 | 0,0485 | 0 | 0 | 0 | 0,1972 | 15,817 | NaCl+$Na_2SO_4$ |
| 8. | - | 18,80 | - | 15,34 | 65,86 | 0 | 0,1324 | 0 | 0,1274 | 0,4904 | 1 | 14,084 | $Na_2SO_4$*$10H_2O$+$Na_2SO_4$*$MgSO_4$*$4H_2O$ |
| 9. | - | 13,25 | - | 21,82 | 64,93 | 0 | 0,0933 | 0 | 0,1812 | 0,6601 | 1 | 13,141 | $Na_2SO_4$*$MgSO_4$*$4H_2O$+$MgSO_4$*$7H_2O$ |
| 10. | - | - | 34,3 | 2,25 | 63,45 | 0 | 0 | 0,3602 | 0,0187 | 1 | 0,0494 | 9,303 | $MgCl_2$*$6H_2O$+$MgSO_4$*$4H_2O$ |
| 11. | - | - | 31,5 | 3,25 | 65,25 | 0 | 0 | 0,3308 | 0,027 | 1 | 0,0755 | 10,131 | $MgSO_4$*$4H_2O$+$MgSO_4$*$5H_2O$ |
| 12. | - | - | 29,8 | 3,95 | 66,25 | 0 | 0 | 0,313 | 0,0328 | 1 | 0,0949 | 10,644 | $MgSO_4$*$6H_2O$+$MgSO_4$*$5H_2O$ |
| 13. | - | - | 26,3 | 4,8 | 68,9 | 0 | 0 | 0,2762 | 0,0399 | 1 | 0,1262 | 12,109 | $MgSO_4$*$7H_2O$+$MgSO_4$*$6H_2O$ |
| 14. | 19,4 | 8,2 | 2,4 | - | 70 | 0,1658 | 0,0577 | 0,0252 | 0 | 0,1013 | 0,232 | 15,637 | NaCl+$Na_2SO_4$ |
| 15. | 14,0 | 10,6 | 5,9 | - | 69,5 | 0,1197 | 0,0746 | 0,062 | 0 | 0,2419 | 0,2911 | 15,065 | NaCl+$Na_2SO_4$+Ac |
| 16. | 10,35 | 9,15 | 9,7 | | 70,8 | 0,0885 | 0,0644 | 0,1019 | 0 | 0,3999 | 0,2527 | 15,437 | NaCl+Ac |
| 17. | 5,15 | 8,6 | 14,9 | - | 71,35 | 0,044 | 0,0606 | 0,1565 | 0 | 0.5994 | 0,2321 | 15,182 | NaCl+Ac |
| 18. | - | 7,9 | 20,5 | 0,9 | 70,7 | 0 | 0,0556 | 0,2153 | 0,0075 | 0,7733 | 0,2266 | 14,108 | NaCl+Ac |
| 19. | - | 6,75 | 20,9 | 2,0 | 70,35 | 0 | 0,0475 | 0,2195 | 0,0166 | 0,8325 | 0,2260 | 13,781 | NaCl+Ac+$MgSO_4$*$7H_2O$ |
| 20. | - | 4,2 | 23,2 | 2,8 | 69,8 | 0 | 0,0296 | 0,2436 | 0,0233 | 0,9002 | 0,1784 | 13,0786 | NaCl+$MgSO_4$*$7H_2O$ |
| 21. | - | 2,3 | 25,95 | 3,3 | 68,45 | 0 | 0,0162 | 0,2725 | 0,0274 | 0,9488 | 0,1379 | 12,0304 | NaCl+$MgSO_4$*$7H_2O$+$MgSO_4$*$6H_2O$ |
| 22. | - | 0,8 | 29,8 | 3,3 | 66,1 | 0 | 0,0056 | 0,3129 | 0,0274 | 0,9838 | 0,0954 | 10,6164 | NaCl+$MgSO_4$*$6H_2O$+$MgSO_4$*$5H_2O$ |
| 23. | - | 0,4 | 34,4 | 1,9 | 63,3 | 0 | 0,0028 | 0,3612 | 0,0158 | 0,9926 | 0,0490 | 9,2593 | NaCl+$MgSO_4$*$6H_2O$+$MgSO_4$*$4H_2O$ |
| 24. | 4,55 | 22,3 | 4,7 | - | 68,45 | 0,0389 | 0,157 | 0,0494 | 0 | 0,2014 | 0,64 | 15,5026 | $Na_2SO_4$+$Na_2SO_4$*$10H_2O$ |
| 25. | - | 25,05 | 6.2 | 1,35 | 67,6 | 0 | 0,1764 | 0,063 | 0,0112 | 0,2961 | 0,7486 | 14,9864 | $Na_2SO_4$+$Na_2SO_4$*$10H_2O$+Ac |
| 26. | 10,3 | 14,2 | 5,6 | - | 69,9 | 0,088 | 0,1 | 0,0588 | 0 | 0,2382 | 0,4052 | 15,7346 | $NaSO_4$+Ac |
| 27. | 3,75 | 21,2 | 6,4 | - | 68,65 | 0,0321 | 0,1493 | 0,0672 | 0 | 0,2703 | 0,6006 | 15,3415 | $NaSO_4$+Ac |
| 28. | - | 22,0 | 2,8 | 8,8 | 66,4 | 0 | 0,1549 | 0,0294 | 0,0731 | 0,3982 | 0,8858 | 14,3314 | $Na_2SO_4$*$10H_2O$+Ac |
| 29. | - | 17,1 | 10,65 | 5,7 | 66,55 | 0 | 0,1204 | 0,1118 | 0,0473 | 0,5692 | 0,6000 | 13,2279 | $Na_2SO_4$+$MgSO_4$*$7H_2O$ |
| 30. | - | 18,1 | 5,4 | 11,95 | 64,55 | 0 | 0,1275 | 0,0567 | 0,0993 | 0,5503 | 0,8000 | 12,6494 | $Na_2SO_4$+$MgSO_4$*$7H_2O$ |
| 31. | - | 11,65 | 4,9 | 14,95 | 68,5 | 0 | 0,082 | 0,0515 | 0,1242 | 0,6818 | 0,8002 | 14,7676 | $Na_2SO_4$+$MgSO_4$*$7H_2O$ |
| 32. | - | 10,7 | 9,7 | 9,3 | 70,3 | 0 | 0,0754 | 0,1019 | 0,0773 | 0,7038 | 0,5998 | 15,3401 | $Na_2SO_4$+$MgSO_4$*$7H_2O$ |
| 33. | - | 9,45 | 15,0 | 4,55 | 71 | 0 | 0,0665 | 0,1575 | 0,0378 | 0,7450 | 0,3984 | 15,0665 | $Na_2SO_4$+$MgSO_4$*$7H_2O$ |

**Conclusion**. Thus, the constructed three-dimensional representation of the four-component system $Na^+$, $Mg^{2+}$ // $2SO_4^{2-}$, $Cl^-$ – $H_2O$ makes it possible to clearly determine the boundaries of adjacent salt crystallization fields and to calculate their volumetric fractions. This provides a basis for guiding technological calculations for the processing of marine-type saline deposits and for understanding the conditions of their formation.

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