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Research of the Process of Obtaining Pure Solutions of Sodium and Magnesium Chlorides from Dry Mixed Salts of Lake Karaumbet

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ABSTRACT: The results of studies on obtaining solutions purified from accompanying impurities from dry mixed salts of Lake Karaumbet are presented. The conditions under which the maximum solubility of dry mixed salts is achieved, the chemical and mineralogical compositions of the resulting solutions and solutions after freezing sodium sulfate, purification from sulfates with a distiller liquid are established**.**

I. INTRODUCTION

Magnesium compounds are used in many industries. The chemical, metallurgy, glass, textile, energy, pharmaceutical and other industries need magnesium compounds. Magnesium chloride or bischofite is a raw material in the production of magnesium chloride defoliants, fungicides, and is used as an anti-icing preparation [1–3]. The need of the Republic only for bischofite for the production of defoliants exceeds 30 thousand tons per year. Due to the lack of own production of magnesium compounds, the need for them is fully met by imports.

II. LITERATURE SURVEY

Uzbekistan has vast reserves of raw materials for the production of magnesium and sodium salts. One of these types of raw materials are dry mixed salts of the Karaumbet Lake. Industrial reserves in the contour of the project open pit of the deposit amount to 612.55 thousand tons in terms of magnesium chloride. Dry mixed salts of Lake Karaumbet contain on average (mass %): Na_2SO_4 43-61; $MgCl_2$ 11-15; NaCl 13-19; undissolved precipitate 4-30. In addition to dry mixed salts, huge reserves of magnesium and sodium salts are also concentrated in the brine of lakes Karaumbet and Barsakelmes [4].

Despite the great need for magnesium compounds, the presence of a powerful raw material base, they are not produced in the Republic. This is due, first of all, to the lack of developed technologies for processing brine and dry mixed salts. Therefore, research aimed at developing a technology for processing dry mixed salts of Karaumbet Lake, with the production of magnesium compounds, is very relevant.

In order to involve dry mixed salts of Lake Karaumbet in industrial production, studies were carried out to study their solubility in water, to purify the resulting solutions from accompanying impurities, water-insoluble residues and subsequent processing into magnesium compounds.

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III. RESEARCH METHODS

Dry mixed salts of Karaumbet composition (mass %) were used for the experiments: Na_2SO_4 – 60.69; NaCl - 18.81; $MgCl_2$ - 15.30; $MgSO_4$ - 0.42; $CaCl_2$ - 0.31. Chemical analysis for the content of the main components of the still liquid, suspensions, mother liquors, and solid phases was carried out according to known methods [5, 6].

IV. EXEPERIMENTAL RESULTS

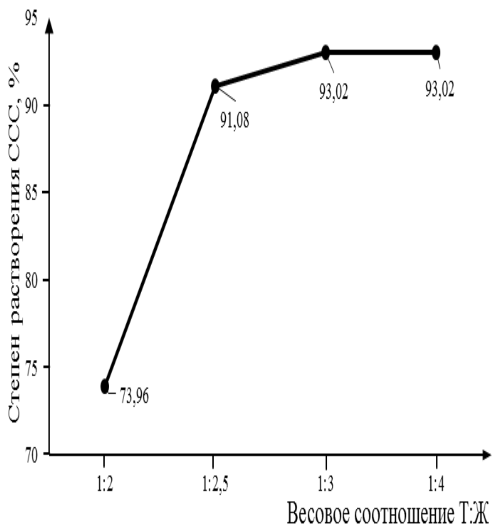
To establish the optimal technological parameters for obtaining purified solutions from DMS, their solubility in water was studied depending on the mass ratio S : L, the duration of the process, temperature, and the effect on the composition of the aqueous suspension. The studies were carried out at a temperature of 25°C, a constant stirring speed and a process duration of 30 minutes.

Figure 1-a shows the results of the degree of dissolution of DMS depending on the mass ratio T : L. It can be seen from the figure that with an increase in the liquid part of the suspension in relation to the solid, the solubility of DMS in water increases, reaching at S : L = 1 : 3 its the maximum value is 93.02%. A further increase in the mass fraction of water to S : L = 1 : 4 has practically no effect on the solubility of the DMS. Therefore, to obtain solutions with a sufficient degree of dissolution of DMS, we can consider the ratio S : L, equal to 1 : 3.

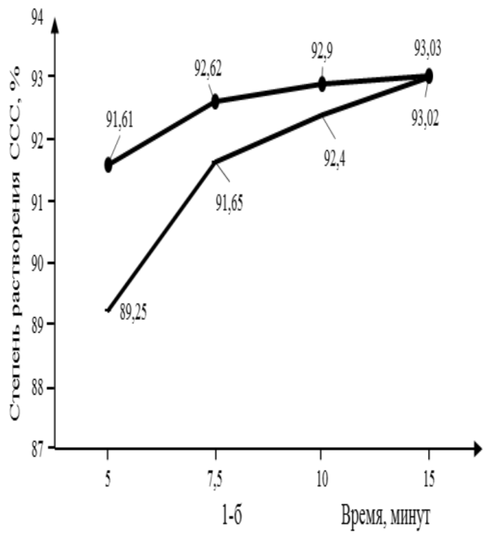
With an increase in the ratio S : L from 1 : 2 to 1 : 3, the amount of wet water-insoluble residue decreases from 22.63% to 4.69% relative to the total mass of the suspension.

The data in Figure 1-b show that at S : L = 1 : 3 and a temperature of 25 ° C, after 5 minutes the degree of dissolution of the DMS is 89.25%, after 10 minutes - 92.4% and after 15 minutes - 93.02% , and at 50°C these figures are 91.61%, 92.90 and 93.03%, respectively, which indicate that 15 minutes is enough to dissolve the DMS.

Figure 1-c shows the data on the influence of temperature and duration of settling on the degree of clarification of the DMS suspension at S : L = 1 : 3. The choice of settling method is determined by the fact that water-insoluble particles settle at a high rate and, moreover, by the ease of use of the sump.



a - The degree of dissolution of DMS depending on S : L at 25°C for 30 minutes.



b – Degree of DMS dissolution depending on time and temperature (25 and 50°C) at S : L = 1 : 3.

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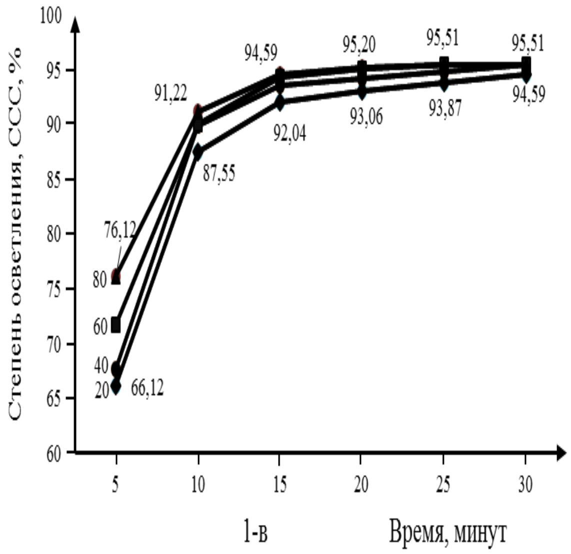
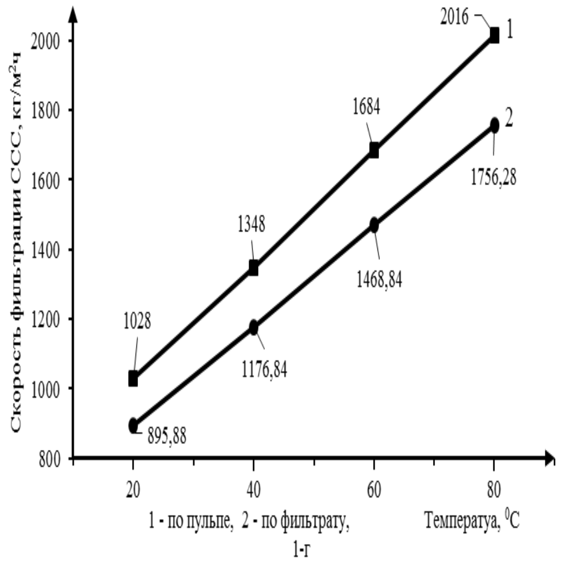


c – The degree of clarification of the DMS suspension depending on the settling time and temperature (20; 40; 60; 80°C) at S : L = 1 : 3.



d – Filtration rate of the thickened part of the DMS suspension over the pulp (1) and sediment (2) depending on the temperature.

Rice. 1. Influence of various technological parameters on obtaining a suspension of dry mixed salts (DMS).

Figure 1-c shows that the clarification of the suspension is most intense in the first 15 minutes, then slows down and after 30 minutes reaches 94.59% at 20°C and 95.51% at 40; 60 and 80°C. The optimal settling temperature and time are 40°C and 20 minutes, at which the degree of clarification of the DMS suspension is at least 95%.

To separate the solid phase from the liquid, studies were carried out on the filtration of the DMS suspension. The filtration rates of the thickened part of the DMS depending on the temperature are shown in Figure 1-d.

Table 1

Effects of temperature on the filtration rate of thickened sludge

№	Temperature, °C	Filtration rate, kg/m^2•h		
		by pulp	by solid phase	by filtrate
1	20	1028	132,64	895,88
2	40	1348	174,16	1176,84
3	60	1684	217,28	1468,84
4	80	2016	260,04	1756,28

The data shows an increase in filtration rate with increasing temperature. So, when the temperature rises from 20 °C to 80 °C, the filtration rate for the thickened pulp increases from 1028 kg/m^2•h to 2016 kg/m^2•h kg, and for the filtrate from 895.88 kg/m^2•h to 1756.28 kg /m^2•h. This produces from 132.64 kg to 260.04 kg of solids per square meter of filter per hour.

Table 2 shows the composition of DMS solutions depending on the ratio S : L. The table shows that with an increase in the proportion of the liquid phase relative to the solid, the content of components in the DMS solution steadily decreases, although the degree of DMS dissolution increases.

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Table 2

Influence of S:L on the composition of an aqueous suspension of DMS at a temperature of 25°C and a dissolution time of 30 minutes

S:L	Ionic composition, wt. %					Chemical composition, wt. %			
	Na^+	Mg^{2+}	Ca^{2+}	Cl^-	SO_4^{2-}	Na_2SO_4	$MgCl_2$	NaCl	$CaSO_4$
1:2,0	9,41	1,40	0,024	3,97	14,35	21,15	5,48	6,55	0,08
1:2,5	7,83	1,16	0,024	3,30	11,93	17,57	4,55	5,44	0,08
1:3,0	6,82	1,01	0,024	2,88	10,40	15,31	3,97	4,74	0,08
1:4,0	5,45	0,81	0,024	2,30	8,32	12,23	3,17	3,79	0,08

Dry mixed salts contain more than 60% mirabilite. To reduce the content of sodium sulfate, the solutions obtained at S:L = 1:3 were subjected to cooling at temperatures of +5, 0 and -5 °C.

Chemical analysis of the compositions of solutions from dry mixed salts obtained at S:L=1:3 before and after mirabilite freezing is shown in Table 3.

Table 3

Effect of temperature on the chemical and salt compositions of solutions of dry mixed salts and the yield of mirabilite

T, ºC.	Chemical composition of the liquid phase, wt. %					Salt composition of the liquid phase, wt. %				Exit Na_2SO_4, %
	Na^+	Mg^2	Ca^{2+}	Cl^-	SO_4^{2-}	Na_2SO_4	$MgCl_2$	NaCl	$CaSO_4$	
25	7,92	1,01	0,022	5,83	10,41	15,31	3,97	4,74	0,08	-
5	3,51	1,41	0,032	8,14	1,96	2,82	5,54	6,61	0,11	81,58
0	3,44	1,43	0,032	8,22	1,75	2,51	5,52	6,68	0,11	83,61
-5	3,37	1,44	0,032	8,29	1,56	2,23	5,64	6,74	0,11	85,43

It follows from the data obtained that when cooled to a temperature of 0°C, the content of sodium sulfate in a solution of dry mixed salts decreases from 15.31% to 2.51%. The content of the remaining components of the solution increases. The content of magnesium chloride increases from 3.97% to 5.52%, sodium chloride from 4.74% to 6.68%. The content of calcium sulfate is 0.11%.

In view of the high residual content of sulfates in the solution (1.75%), after the precipitation of mirabilite, additional purification from sulfates was carried out with a distiller liquid - a waste from soda production of the composition (wt.%): Na^+ - 2.18; Mg^{2+} - 0.007; Ca^{2+} - 3.03; C1 - 8.74; SO_4^2 - 0.03.

Desulfation was carried out at a rate of calcium distillation liquid 95, 100, 102 and 105% on SO_4^{-2} solution, at a temperature of 25°C and a process duration of 30 minutes (Table 4).

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Table 4

Influence of the rate of distilling liquid on the chemical composition and degree of desulfurization of solutions of dry mixed salts

Norma DL., %	Chemical composition of the liquid phase, wt. %					Salt composition of the liquid phase, wt. %				The degree of desulfurization,%
	Na^+	Mg^{2+}	Ca^{2+}	Cl^-	SO_4^{2-}	Na_2SO_4	$MgCl_2$	NaCl	$CaSO_4$	
95	3,72	0,90	0,11	8,08	0,61	0,54	3,52	8,99	0,33	65,14
100	3,46	0,88	0,11	7,94	0,24	-	3,46	8,84	0,34	86,29
102	3,45	0,87	0,14	7,90	0,23	-	3,43	8,80	0,32	86,86
105	3,44	0,86	0,19	7,83	0,22	-	3,39	8,74	0,31	87,43

At the same time, the content of SO_4^{-2} is reduced from 1.75% to 0.22-0.24%. Sodium sulfate reacts with calcium chloride in the still liquid to form sodium chloride and calcium sulfate. The content of sodium chloride rises to 8.74-8.84%, and magnesium chloride to 3.39-3.46%. At a distillation liquid rate of 100-105%, there is no sodium sulfate in the solution and a small amount of calcium chloride appears. The content of calcium sulfate is 0.31-0.34%, which corresponds to its solubility in aqueous solutions.

The compositions of the solutions obtained at a rate of 100-105%, in terms of the content of impurities, are close to the compositions of purified solutions obtained from the brine of the Karaumbet and Barsakelmes lakes, which are processed with good technical and economic indicators into magnesium chloride and magnesium hydroxide [7, 8].

V. CONCLUSION

Thus, the conducted studies have shown the fundamental possibility of obtaining solutions of sodium and magnesium chlorides purified from impurities from DMS, suitable for further processing into magnesium chloride and hydroxide, magnesium oxide.

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