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STUDY OF THE DESULFURIZATION PROCESS OF BRINE FROM THE KARAUMBET AND BARSAKELMES LAKES

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Abstract. The research results on the desulfurization of brine from the Karaumbet and Barsakelmes lakes using distiller liquid, a by-product of soda production, are presented. It is shown that the optimal technological parameters are a distiller liquid dosage of 100-105%, a temperature of 20-30°C and a process duration of 20-30 minutes, under which the degree of desulfurization reaches 95%. Deep purification with sodium carbonate reduces the calcium chloride content by 0.66% and introducing barium chloride in 100% relative to the residual sulfate content reduces the sulfate concentration to 0.001%.

Keywords: *Magnesium, distiller liquid, sodium carbonate, desulfurization process, temperature.*

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1. Introduction

Understanding the desulphurization process of brine from Karaumbet and Barsakelmes lakes has a special relevance to environment conservation, sustainable utilization of resources and various industries. The other form of brine which is sulfate type is found in saline lakes and comes with environmental issues especially when natural ecosystems are fragile to formation of chemical equilibrium. The extant stenohaline and euryhaline subpopulations of the temporary lakes of Karaumbet and Barsakelmes foray in arid areas with significantly elevated levels of salinity and sulfate. Therefore, their desulfurization is both a scientific and industrial problem, as well as a potential for resource recovery (Wang *et al*., 2023; Abdullayev *et al*., 2023a; Xiao *et al*., 2022; Wang *et al*., 2023 Shuangchen *et al*., 2016).

Overally, desulfurization processes, are processes that seek to eliminate or minimize or sulfate, sulfide or sulfur dioxide in various environments or substances. Consequently, the present work seeks to establish the most suitable methods of sulphate removal from

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brine with regard to the chemical composition of the resulting brine with regards to the effects on water bodies in the surrounding environment and for the potential extraction of sulfates and other minerals (Zhao *et al*., 2024; Telesca *et al*., 2024).

These two lakes are absolutely original in the processes of their formation and have highly mineralized brine containing different salts and sulphates in particular. Awareness of the reactions involving these chemical compounds as well as various desulfurization methods' efficiency determines future efficient preservation of the environment and optimal use of these chemical compounds in industry (Song *et al*., 2022; Yang *et al*., 2023). Consequently, this work is aimed at comparing various methods for desulfurization, using the results of this research to determine the efficiencies, costs and environmental impacts of the different options available for practicing desulfurization of brine or other sulfur-containing compounds in various industries for purely local purposes or for world-wide applications. Second, the research brought into focus the conservation of natural resources as well as encouraging sustainable industrial development (Abdullayev *et al*., 2023b; Guan *et al*., 2021; Cherif *et al*., 2024).

In this paper we will therefore analyze the chemical content of brine from these lakes; look at all the methods used in desulfurization in similar cases and provide our findings on the most efficient process in this case.

2. Experimental part

For the research, brine from Lake Karaumbet with the following composition (wt. %) was used: Na^+ - 8.01; Mg^{2+} - 3.27; Cl^- - 17.70; SO_4^{2-} - 6.66 and brine from Lake Barsakelmes with the following composition (wt. %): Na^+ - 8.90; Mg^{2+} - 2.77; Cl^- - 16.90; SO_4^{2-} - 4.21. Chemical analysis of the main components in the liquid phases of the samples was carried out using well-known methods.

Brines from the Karaumbet and Barsakelmes deposits, located in the Kungrad region of the Republic of Karakalpakstan, 45 km and 80 km from Kungrad, respectively, were utilized for the experiments. Notably, the surface salt composition of the Barsakelmes deposit showed a more than 10% increase in sodium chloride content, along with an increased presence of insoluble residues and a 3-4 fold decrease in calcium sulfate. A similar shift was noted in the intercrystalline brine composition, where the sodium chloride concentration rose by approximately 3%, while the calcium magnesium bicarbonate content decreased.

Both the raw materials and the final product were analyzed for the concentrations of calcium, magnesium, sulfur, chlorine, carbonates, insoluble residues and moisture. Calcium and magnesium were quantified using the complexometric method, which is based on the colour change of indicators such as fluorexon (for calcium determination) and acid chrome dark blue (for magnesium determination) when interacting with Trilon B in the presence of these ions.

Sodium levels were measured by flame photometry and sulfate was determined gravimetrically, a method that involves the precipitation of sulfate ions with barium chloride in an acidic medium, followed by washing and weighing of the resulting precipitate. Chlorine content was analyzed using the volumetric argentometric method, which relies on the colour change of silver chloride precipitates formed by the interaction of silver ions with potassium dichromate. Moisture content in solid samples was determined by drying in a constant-temperature oven at 100-105°C.

3. Results and discussion

Previous studies on the two-stage evaporation of brine from the Barsakelmes and Karaumbet lakes, without preliminary purification, demonstrated the possibility of obtaining magnesium chloride solutions with a concentration of 19-19.5%. However, the resulting concentrate contains 6-7% sulfate ions, whose content is regulated for raw materials. To obtain purer crystals of magnesium chloride and sodium from the brines of the Karaumbet and Barsakelmes lakes, it is first necessary to remove the sulfate ions, which hinder the direct processing of the brine into bischofite and table salt.

In laboratory conditions, the desulfation process of the brine was studied using distiller liquid (DL) – a by-product of soda production at JSC "Kungrad Soda Plant" It should be noted that in the production of 1 ton of soda ash, 9-10 m^3 of DL is generated. Its average composition is as follows (wt. %): $CaCl_2$ - 10.82; NaCl - 5.54; $CaSO_4$ - 0.08; $Ca(OH)_2$ - 0.11; with a density of 1.134 g/cm^3. At JSC "Kungrad Soda Plant" with an annual production of 100 thousand tons of soda ash, approximately 1 million tons of DL is generated.

This creates the problem of land occupation by sludge storage areas, known as "White Seas" and the mineralization of natural water bodies. The technologies currently used for the utilization and processing of DL only partially solve the problem. As a result, most of the primary waste - DL - accumulates in sludge storage areas and/or is discharged into nearby water bodies. However, the discharge of DL into water bodies is strictly regulated, so the waste is mainly accumulated in sludge storage areas, which limits the production capacity.

For the experiments, brine from the Karaumbet and Barsakelmes lakes, as well as DL, were used, with their compositions presented in Table 1.

The experiments were conducted as follows: the initial DL was loaded into a glass reactor and placed in a water thermostat. The reactor was equipped with a screw stirrer to ensure proper mixing.

Table 1. Chemical composition of the brine from the Karaumbet and Barsakelmes lakes and distiller liquid

Name	Ionic composition, wt. %					Salt composition, wt. %			
	Na^+	Mg^{2+}	Ca^{2+}	Cl^-	SO_4^{2-}	$MgCl_2$	NaCl	$CaC1_2$	$MgSO_4$
Brine from Lake Karaumbet	8.01	3.25	0.02	17.53	6.66	6.28	20.37	0.05	8.33
Brine from Lake Karaumbet after summer settling	8.40	5.70	0.06	25.94	4.25	17.41	21.37	0.20	5.41
Brine from Lake Barsakelmes	9.06	1.64	0.01	17.07	2.34	4.13	23.05	0.03	2.54
Distiller liquid	2.18	0.007	3.03	8.74	0.03	0.03	5.54	8.41	0.04

The rotational speed of the electric motor (250 rpm) was regulated using a rheostat. Then, the calculated amount of salt brine was gradually dosed into the reactor. The dosage rate (DL) was varied from 75 to 110% of the stoichiometric amount required to bind SO_4^{2-} into $CaSO_4$ and the process temperature was adjusted between 20 and 60°C. During the mixing of these components, a reaction occurs between calcium chloride and sodium magnesium & sulfate:

$$CaCl_2+Na_2SO_4(MgSO_4)+nH_2O=CaSO_4\cdot 2H_2O+2NaCl(MgCl_2\cdot 6H_2O)$$

Initially, the effect of the desulfation process duration (from 5 to 180 minutes) was studied using the brine from Lake Karaumbet with a 100% DL. It was found that increasing the time from 5 to 20 minutes led to an intense desulfation process, which then significantly slowed down. For example, at 20°C, the degree of desulfation of the Karaumbet brine was 58.37% after 5 minutes, 88.59% after 20 minutes, 89.8% after 30 minutes and 90.33% after 60 minutes. After 180 minutes, the increase was only 0.48% (Table 2).

Table 2. The effect of duration and temperature on the composition and degree of desulfation of Karaumbet brine at a 100% dosage rate of distiller liquid

Time, min.	Ionic composition of the liquid phase, wt. %					Salt composition of the liquid phase, wt. %			
	Na^+	Mg^{2+}	Ca^{2+}	Cl^-	SO_4^{2-}	$MgCl_2$	NaCl	$CaC1_2$	$MgSO_4$
Desulfation temperature 20°C									
5	5,67	2,33	0,40	15,24	0,96	8,27	14,43	1,12	1,20
10	5,72	2,35	0,22	15,36	0,52	8,79	14,55	0,60	0,65
15	5,74	2,36	0,14	15,41	0,34	8,99	14,60	0,40	0,42
20	5,75	2,36	0,11	15,43	0,27	9,08	14,62	0,32	0,33
30	5,75	2,36	0,10	15,44	0,24	9,11	14,62	0,28	0,30
60	5,75	2,36	0,09	15,44	0,23	9,13	14,63	0,27	0,28
180	5,75	2,36	0,09	15,45	0,22	9,14	14,63	0,26	0,27
Desulfation temperature 40°C									
5	5,67	2,33	0,44	15,22	1,04	8,18	14,41	1,21	1,30
10	5,71	2,35	0,25	15,34	0,60	8,69	14,53	0,71	0,76
15	5,73	2,35	0,19	15,38	0,46	8,85	14,56	0,54	0,58
20	5,73	2,35	0,17	15,40	0,40	8,93	14,58	0,46	0,49
30	5,74	2,36	0,15	15,41	0,36	8,97	14,59	0,43	0,45
60	5,74	2,36	0,14	15,41	0,34	8,99	14,60	0,40	0,43
180	5,74	2,36	0,14	15,42	0,33	9,01	14,60	0,38	0,41
Desulfation temperature 60°C									
5	5,66	2,32	0,47	15,19	1,13	8,08	14,39	1,32	1,42
10	5,68	2,33	0,37	15,26	0,87	8,38	14,46	1,02	1,09
15	5,70	2,34	0,29	15,32	0,69	8,59	14,50	0,80	0,86
20	5,71	2,35	0,25	15,34	0,60	8,70	14,53	0,69	0,74
30	5,72	2,35	0,21	15,37	0,51	8,80	14,55	0,59	0,63
60	5,72	2,35	0,20	15,37	0,49	8,83	14,56	0,57	0,61
180	5,72	2,35	0,20	15,38	0,47	8,84	14,56	0,55	0,59

The remaining portion of calcium sulfate dihydrate stays in the brine due to its partial solubility (at 20°C, the solubility of $CaSO_4\cdot 2H_2O$ is 0.206 g/100 g of H_2O). An increase in temperature leads to a reduction in the degree of desulfurization of the saline brine. Thus, at temperatures of 20, 40 and 60°C, the degree of desulfurization of the brine

is: after 5 minutes - 58.37, 54.93 and 50.88%; after 30 minutes - 89.80, 84.50 and 78.27% and after 60 minutes - 90.33, 85.40 and 79.21%, respectively.

This can be explained by the fact that with an increase in temperature, the solubility of $CaSO_4 \cdot 2H_2O$ increases and accordingly, its content in the liquid phase increases. With the increase in the duration of desulfation, the content of Ca^{2+} in the liquid phase decreases. At 20°C, after 5, 30 and 60 minutes, the calcium ion content is 0.40%, 0.10% and 0.09%, respectively and at 60°C, it is 0.47%, 0.21% and 0.20%, respectively.

From the data, it can be concluded that the optimal temperature for desulfation is 20-30°C and the optimal duration is 20-30 minutes.

Tables 3-5 present the ionic and salt compositions, as well as the degree of desulfation of the brines from the Karaumbet and Barsakelmes lakes, depending on the norm of DL (desulfurizing agent) and the temperature of the process over 30 minutes.

Table 3. The composition and degree of desulfation of the brines from the Karaumbet and Barsakelmes lakes depend on the norm of distiller liquid at 20°C

Norm, wt. %	Ionic composition of the liquid phase, wt. %					Salt composition of the liquid phase, wt. %			
	Na^+	Mg^{2+}	Ca^{2+}	Cl^-	SO_4^{2-}	$MgCl_2$	NaCl	$CaC1_2$	$MgSO_4$
Brine of Karaumbet Lake									
75	5.80	1.99	0.15	14.36	1.37	6.51	14.75	0.40	1.71
90	5.65	1.80	0.13	14.21	1.24	6.69	14.20	0.36	1.22
100	5.43	1.77	0.12	13.86	0.29	6.72	13.82	0.34	0.36
105	5.36	1.73	0.15	13.75	0.18	6.66	13.63	0.43	0.23
110	5.29	1.69	0.21	13.64	0.14	6.54	13.44	0.58	0.18
Brine of Barsakelmes Lake									
75	7.55	1.37	0.26	19.68	1.25	4.13	19.20	0.73	1.56
90	7.31	1.32	0.06	20.11	0.41	4.77	18.59	0.18	0.51
100	7.15	1.29	0.10	20.37	0.24	4.82	18.19	0.28	0.30
105	7.08	1.28	0.13	20.50	0.19	4.83	18.01	0.36	0.24
110	7.00	1.27	0.17	20.63	0.17	4.81	17.80	0.46	0.21
125	6.79	1.23	0.28	20.99	0.13	4.68	17.29	0.78	0.17
150	6.47	1.17	0.46	21.55	0.09	4.51	16.46	1.27	0.11

The data in Table 3 show that at 20°C, with an increase in the distiller liquid (DL) norm from 75% to 110%, the degree of desulfation of the Karaumbet brine increases from 67.1% to 96.0%, the Karaumbet brine after its summer settling from 51.06% to 92.69% and the Barsakelmes brine from 50.03% to 92.5%.

An increase in the DL norm above 100%, although it ensures the maximum removal of sulfate ions, on the other hand, leads to a noticeable increase in the calcium ion content in the purified brine, i.e., the appearance of a residual amount of calcium chloride. A similar pattern in the change in the degree of desulfation and the content of Ca^{2+} ions depending on the DL norm is maintained for other temperatures (Tables 4 and 5). The higher the temperature, the lower the degree of desulfation, which is explained by the increased solubility of calcium sulfate.

Table 4. The composition and degree of desulfation of the brines from the Karaumbet and Barsakelmes lakes depend on the norm of distiller liquid at 40°C

Norm, wt. %	Ionic composition of the liquid phase, wt. %					Salt composition of the liquid phase, wt. %			
	Na^+	Mg^{2+}	Ca^{2+}	Cl^-	SO_4^{2-}	$MgCl_2$	NaCl	$CaC1_2$	$MgSO_4$
Brine of Karaumbet Lake									
75	5.77	1.98	0.25	14.29	1.60	6.24	14.69	0.68	2.01
90	5.55	1.81	0.24	14.00	0.97	6.35	14.12	0.66	0.94
100	5.41	1.76	0.23	13.80	0.55	6.42	13.75	0.65	0.68
105	5.34	1.72	0.25	13.70	0.42	6.39	13.57	0.71	0.53
110	5.26	1.68	0.31	13.58	0.38	6.27	13.39	0.86	0.48
Brine of Barsakelmes Lake									
75	7.41	1.36	0.43	19.58	1.6	3.53	19.58	1.16	2.27
90	7.27	1.31	0.10	20.01	0.52	4.56	18.96	0.20	0.73
100	7.11	1.28	0.16	20.27	0.24	4.80	18.92	0.44	0.43
105	7.04	1.27	0.21	20.40	0.21	4.71	18.37	0.57	0.34
110	6.99	1.26	0.28	20.53	0.16	4.70	18.15	0.73	0.30

Table 5. The composition and degree of desulfation of the brines from the Karaumbet and Barsakelmes lakes depend on the norm of distiller liquid at 60°C

Norm, wt. %	Ionic composition of the liquid phase, wt. %					Salt composition of the liquid phase, wt. %			
	Na^+	Mg^{2+}	Ca^{2+}	Cl^-	SO_4^{2-}	$MgCl_2$	NaCl	$CaC1_2$	$MgSO_4$
Brine of Karaumbet Lake									
75	5.75	1.97	0.35	14.23	1.86	5.95	14.62	0.98	2.32
90	5.38	1.75	0.36	13.72	0.85	6.09	13.68	0.99	1.06
100	5.31	1.71	0.37	13.63	0.70	6.08	13.50	1.03	0.88
105	5.24	1.67	0.41	13.52	0.64	5.99	13.32	1.15	0.80
110	5.75	1.97	0.35	14.23	1.86	5.95	14.62	0.98	2.32
Brine of Barsakelmes Lake									
75	5.65	2.30	0.78	15.11	1.33	7.38	14.67	2.04	2.06
90	5.62	2.31	0.61	15.18	1.10	7.82	14.74	1.14	1.56
100	5.60	2.32	0.48	15.24	0.80	8.11	14.80	1.24	1.24
105	5.61	2.33	0.41	15.26	0.70	8.32	14.82	1.07	1.03
110	5.62	2.33	0.35	15.29	0.67	8.44	14.84	0.91	0.88

The influence of the DL norm and temperature on the degree of desulfation and the relative calcium content in the liquid phase is clearly seen in Figures 1 and 2.

Figure 1 clearly shows the effect of DL, where the degree of desulfation of the brine increases monotonically as the dosage rate rises, while the calcium content in the liquid phase increases with a breakpoint at the 100% dosage rate (Figure 2).

In any case, part of the calcium dihydrate remains dissolved in the brine, which is why the degree of desulfation does not exceed 90% within 30 to 60 minutes. For the

desulfation process of brine from both Karaumbet and Barsakelmes lakes, the optimal interaction time for the components is 20-30 minutes at a temperature of 20-30°C.

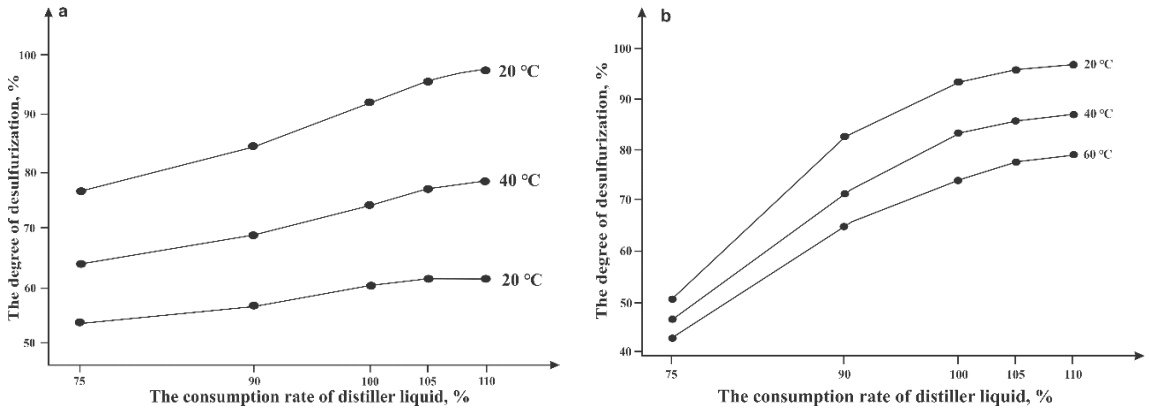

Figure 1. The effect of the consumption rate and temperature of distiller liquid on the degree of desulfurization of brine from the Karaumbet (a) and Barsakelmes (b) lakes

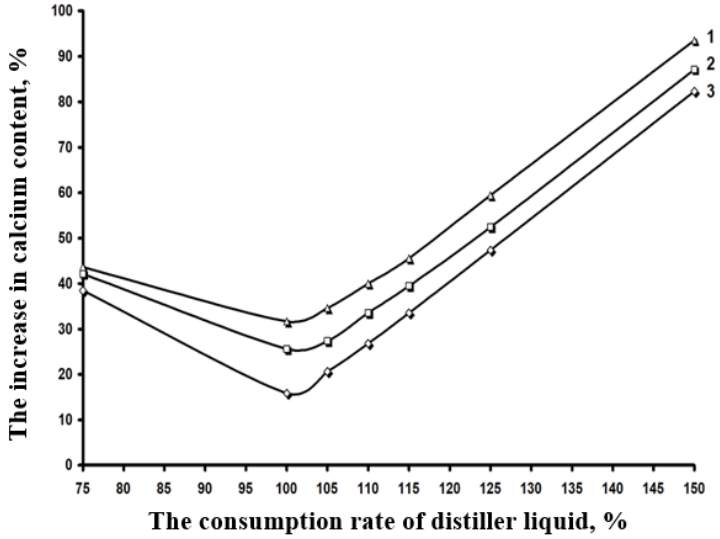

Figure 2. The effect of the consumption rate of distiller liquid and temperature on the calcium content in brine at the following temperatures: 1-20°C, 2-40°C, 3-60°C

Thus, the fundamental feasibility of using distiller liquid from "Kungrad Soda Plant" JSC for the desulfation of brine from the Karaumbet and Barsakelmes lakes-raw materials for the production of bisulfite has been demonstrated. The optimal conditions for the desulfation process are as follows: temperature 20-30°C, DL, 100-105% of the stoichiometric requirement and mixing duration 30 minutes. Under these conditions, the sulfate ion content in the brine is reduced from an initial 2.34-6.66% to 0.29-0.85% in the desulfated solution, while the Ca^{2+} content reaches 0.12-0.15%. To obtain purer solutions of sodium and magnesium chlorides, the desulfated brine must undergo deep purification, during which more effective chemical reagents should be used.

Sodium carbonate was used as a precipitant for calcium ions. The amount of Na_2CO_3 added relative to 1000 kg of brine varied between 7.52 and 12.53 kg, corresponding to 75-125% of the stoichiometric requirement. The process temperature

was room temperature 25°C and the interaction time was 15-20 minutes.

As can be seen from the experimental data (Table 6), with an increase in the Na_2CO_3 dosage from 75 to 105%, the calcium ion content in the form of calcium chloride significantly decreases from 0.86% to 0.66%. Further increasing the dosage to 125% does not result in a substantial reduction in the amount of calcium chloride in the liquid phase. The contents of the other components-magnesium chloride, sodium chloride and magnesium sulfate-also change insignificantly and are within the following ranges (mass %): 9.106-9.151, 15.223-15.665 and 0.227-0.228, respectively.

Table 6. The effect of sodium carbonate dosage on the composition of desulfurized brine

Dosage rate, Na_2CO_3, wt. %	Composition of the liquid phase, wt. %				Mass, kg	
	$MgCl_2$	NaCl	$CaC1_2$	$MgSO_4$	solution	chalk
75	9.151	15.223	0.86	0.228	994.31	7.33
90	9.138	15.356	0.73	0.228	994.85	8.50
95	9.133	15.400	0.70	0.228	994.89	8.78
100	9.129	15.445	0.68	0.228	995.02	9.00
105	9.124	15.489	0.66	0.228	995.29	9.14
110	9.119	15.533	0.66	0.227	995.70	9.20
115	9.115	15.577	0.65	0.227	996.14	9.23
120	9.110	15.621	0.65	0.227	996.62	9.24
125	9.106	15.665	0.65	0.227	997.11	9.25

Based on the data in Table 6, the degree of brine decalcification was calculated (Figure 3). The figure indicates that to reduce the brine contamination by calcium ions, it is sufficient to maintain the sodium carbonate dosage at 100-105%, at which the degree of calcium ion removal is 59.4-60.5%. The resulting solution contains more than 9% $MgCl_2$ and 15.5% NaCl.

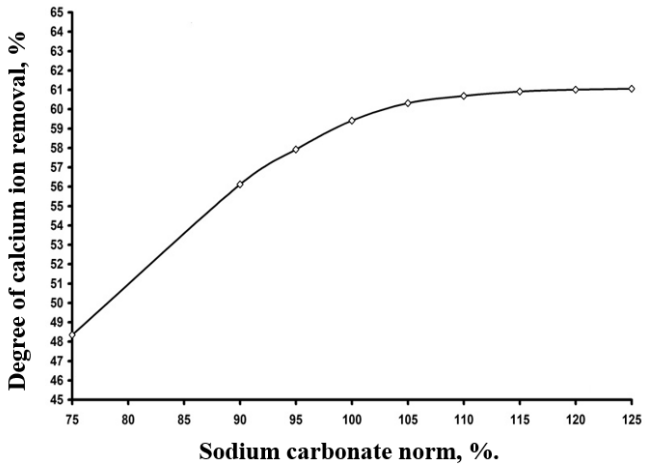

Figure 3. The effect of sodium carbonate dosage on the degree of calcium removal from desulfurized brine

For the deep purification of brine from residual sulfates, brine that had been pre-desulfurized and purified from calcium ions was used, with the following composition (wt. %): NaCl - 15.47; $MgCl_2$ - 9.13; $CaCl_2$ - 0.32; $MgSO_2$ - 0.39. The precipitation of SO_4^{2-} ions was carried out using a 10% barium chloride solution at a dosage of 70-105% of the stoichiometric amount required for the formation of $CaSO_4$, at a temperature of 25°C and with a mixing duration of 30 minutes. The obtained results are presented in Table 7.

From the table, it is evident that when the barium chloride dosage reaches 100%, the desulfurization process is almost complete. At this point, the degree of desulfurization reaches 99.5% and the SO_4^{2-} ion concentration in the purified brine does not exceed 0.001%.

Table 7. The effect of barium chloride dosage on the composition of brine and the degree of sulfate precipitation at 25°C with a process duration of 30 minutes

$BaCl_2$ dosage, wt. %	Composition of the liquid phase, wt. %				Degree of SO_4^{2-} precipitation, wt. %.
	NaCl	$MgCl_2$	$CaCl_2$	$MgSO_4$	
70	14.40	8.63	0.36	0.0550	75.65
80	14.26	8.56	0.36	0.0440	84.60
90	14.09	8.50	0.35	0.0160	93.04
95	14.02	8.46	0.35	0.0089	96.25
98	13.98	8.44	0.34	0.0010	98.63
100	13.95	8.43	0.34	0.0009	99.50
102	13.88	8.40	0.34	0.0008	99.65
105	13.82	8.37	0.60	0.0008	99.70

4. Conclusion

Research has shown the feasibility of removing sulfates from the brines of Karaumbet and Barsakelmes lakes using barium chloride. In this process, barium hydroxide reacts with sulfates to form insoluble barium sulfate, which can then be filtered out. Following this, the solution undergoes additional purification steps involving sodium carbonate and barium chloride, which help in further removing residual impurities. The resulting purified solution is suitable for the production of magnesium chloride. The purity of this magnesium chloride is 97.3%, which might be suitable for various industrial applications.

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