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Carnallite Flotation of Khur Biabanak Potash Complex using Kimiaflot619 as a New Collector

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Abstract

Carnallite, with the chemical formula $KMgCl_3.6H_2O$, is a mineral that was first discovered in the Urals Mountains in Russia. The reverse flotation has been established for carnallite processing in the current decades, and the alkyl morpholine collector is used for the removal of NaCl from carnallite using the reverse flotation. The carnallite processing method involves reverse flotation with the dodecyl morpholine collector, and then centrifugation and cold crystallization. In this research work, kimiaflot 619, as a new collector, is synthesized, and the bench-scale flotation shows that kimiaflot 619 reveals a better selectivity and affinity for the NaCl crystals at an acidic pH with a less collector dosages–only 1/2 of the Armoflot 619 collector. The flotation results indicate that the NaCl grade in carnallite concentrated by Armoflot 619 (200 g/t) is 2.86%, while the NaCl grade in carnallite concentrated by kimiaflot 619 collector (100 g/t) is 2.75%. The frother's stability of the Armoflot 619 collector after flotation is very high and uncontrollable, while kimiaflot 619 has solved this problem, and it is completely controllable.

1. Introduction

Potassium is one of the three essential plant elements, and is not substituted in the agriculture to maintain and expand food production in the world. About 95% of the world's potassium production is used in the agriculture with other minor applications in the soap, plastics, and pharmaceutical industries [1]. The Khur Biabanak Potash Complex is a potash producer in the Islamic Republic of Iran, and one of the largest potash producers in the Middle East. This company with a mineral deposit of 2000 square kilometres is located in the Khur Biabanak city, Isfahan Province. This company has been put with the aim of an annual production of 50,000 tons of potassium chloride as a chemical fertilizer and 3,000,000 tons of sodium chloride for petrochemical, food and pharmaceutical uses and 30,000 tons of magnesium hydroxide. In the potash complex, the cold process method is used to separate salt from carnallite crystals, and flotation causes the separation of sodium chloride. The flotation effluent enters a thickener, and after concentrating, it enters the centrifuge to separate the carnallite from the suspension (Figure 1). In the Khur Biabanak Potash Complex, the flotation operation of the potash plant is an important part of the overall process, which involves the separation of NaCl from carnallite. In the reverse flotation system, the sodium chloride salt floats and is separated. In the carnallite flotation, the Armoflot 619 collector is used.

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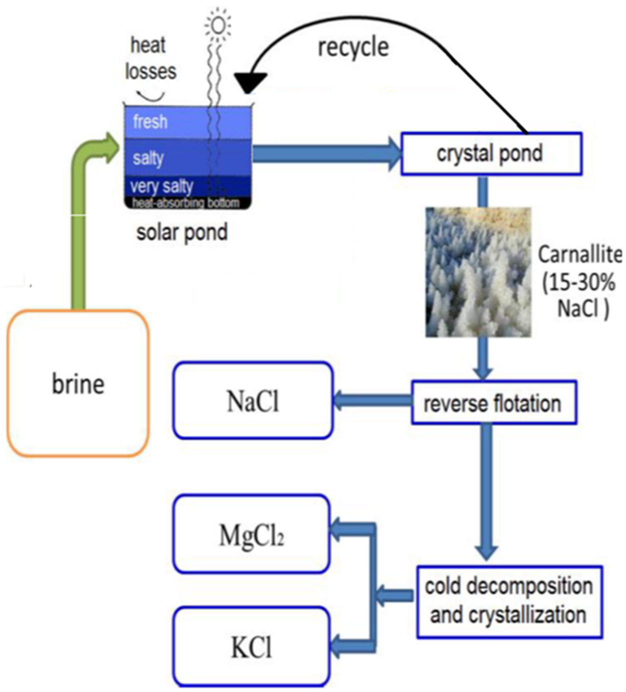

Figure 1. A flowsheet of reverse flotation of carnallite in Khur and Biabanak potash complex.

The froth flotation is used to separate the valuable minerals from non-valuable minerals [2], which can be considered as a recovery process with time rate [3]. However, currently, there are a few ways to improve the separation of sylvite and halite [4, 5]. Flotation of KCl crystals from other salt crystals in their saturated salt water is the main metallurgical process for the recovery of potash with crystallization [6]. In the recent years, the reverse flotation carnallite ores have been used with the N-alkyl amides collector ($R-CONH_2$) [7]. N-alkyl morpholines ($R-N(CH_2CH_2)_2O$) have been widely used in the industrial production due to their selectivity and excellent collectability [8]. However, these factors have limited mineral collection properties due to their unique hydrophobic group [9]. The collector performance can be affected by temperature [10]. The flotation of NaCl and KCl from carnallite has been investigated by Titkov et al. [11] using alkyl morpholine as a collector. The results of flotation show that it can selectively float halite from a carnallite mixture with alkyl morpholine. The chain length difference of alkyl morpholine has also been investigated, and the results obtained show that the flotation activity of alkyl morpholine with a longer carbon chain is much higher than alkyl morpholine with a shorter carbon chain [11]. Also the addition of a frothing agent increases the absorption of alkyl morpholine at the surface of halite and decreases the consumption of morpholine. Zhang et al. have investigated the behavior of halite flotation using dodecyl morpholine (DDM) as a collector, explaining the mechanism of absorption but the conclusions are unclear. They made similar observations where the addition of frother increases the adsorption density [12]. Most research works focus on understanding the mechanism of uptake of long carbon chain amines at the sylvite mineral surface (KCl), and several theories have been proposed to explain the mechanism of adsorption of collectors/surfactants in salt flotation [13, 14], and have been comprehensively reviewed by Du et al. [15]. The ion exchange model explains that the selective adsorption of amines at the sylvite surface is due to the fact that the RNH^{3+} collector ions are located at the K^+ surface sites in the KCl network (Figure 2) but RNH^{3+} cannot be located in the NaCl network [16]. Most research works have focused on understanding the adsorption mechanism of long carbon chain amines in the sylvite mineral surface (KCl), and several theories have been proposed to explain the adsorption mechanism of collectors/surfactants in salt flotation [13-15]. The ion exchange model explains that the selective adsorption of amines at the sylvite surface is due to the fact that the RNH^{3+} collector ions are located at the K^+ points in the KCl lattice (Figure 2) but RNH^{3+} cannot be located in the NaCl network [16]. However, this theory cannot explain why long-chain morphine collectors can float halite and sylvite [17]. The interfacial structure model of water indicates that a salt may be classified as a constructor of water structures or water structures based on the analysis of salt hydration and the measurement of solution viscosity [17]. The salt that makes up the structure of water constantly reacts with water molecules, while the brittle salt in the structure of water tends to break down the structure of water at the surface of the salt. Therefore, the collector molecule can easily replace the water molecule on the surface of a brittle salt in the water structure. According to the interfacial water structure model [17], KCl is a brittle salt of water structure, while NaCl is a structural salt of water structure. Adsorption, therefore, occurs at the KCl surface. The interphase water structure theory cannot explicate the dodecyl morphine adsorption at the NaCl surface. According to the surface charge model [18], the mechanism governing flotation is the electrostatic interaction between the salt surface and the collector species [19]. The cationic amine collector is absorbed by the electrostatic adsorption force since the surface of the sylvite mineral has a negative charge. As mentioned earlier, the unbalanced electrophoretic mobility measurements [18] show that KCl and NaCl have opposite surface charges but both KCl and NaCl can be used as collectors using DDM. It seems that the surface charge model also cannot explain the adsorption behavior. Despite the fact that DDM is used in the industry successfully, in some cases, carnallite ($KCl.MgCl_2.6H_2O$) is concentrated by reverse flotation and used to produce sylvite (KCl) as a raw material by cold decomposition with the formation of $MgCl_2$ in solution and KCl as a solid phase. The octadecylamine hydrochloride (ODA) collector is used in carnallite flotation [20, 21]. The halite particles cannot be collected by ODA in the theory of interfacial water structure [22-24], while the amount of Na^+ concentrate in batch experiments is relatively high. As a result, based on the SEM-EDS analysis and the theory of interfacial water structure, it was judged that part of the halite particles could float for concentration, primarily due to the locking of the sylvite and halite, and causing the halite particles lock upwards with floating silhouette [25]. The amount of halite (NaCl, 15-30%), (KCl, 10-15%), sylvite, and water-insoluble clay usually contains the carnallite ore [26]. The carnallite ores should be concentrated because the cold decomposition process usually requires less than 4-6% halite [26]. Therefore, it is necessary to remove the halite from the carnallite ore before decomposition. In this work, a flotation method was developed using kimiaflot 619 as a collector for flotation of halite and sylvite from carnallite. A new collector was synthesized for carnallite flotation and compared with Armoflot 619. The 1-bromooctadecane ($C_{18}H_{45}Br$) and morpholine was selected as the raw materials for synthesis of the Kimiaflot 619 collector. The synthesis method was divided into two stages. First, 1-bromooctadecane and potassium hydroxide was mixed, and then morpholine was added to produce the Kimiaflot 619 collector.

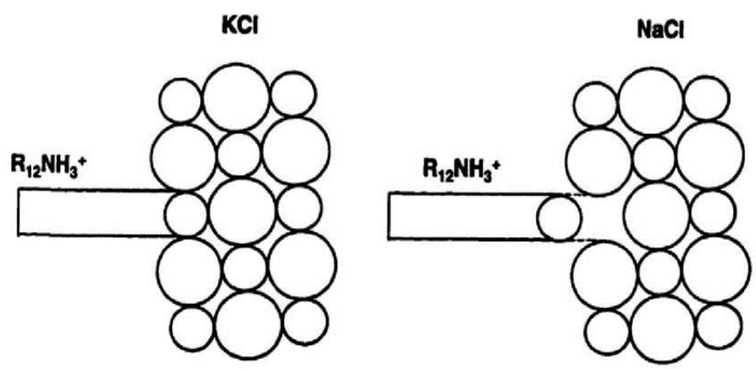

Figure 2. Mechanism of collector adsorption based on ion exchange model. Long chain amine exchange at KCl and NaCl surfaces [16].

2. Materials and methods

2.1. Feed Preparation

For the flotation separation tests, the carnallite mineral was obtained from the Khur Biabanak Potash Complex (Khur Biabanak city, Isfahan Province). The carnallite ores were analyzed as 19.11% sylvite (KCl), 17.13% halite (NaCl), 29.45% $MgCl_2$, 0.32% $CaCl_2$, and 33.65% H_2O. The carnalite samples were grinded to less than 1 mm using laboratory mills, and the crushed salts were sieved to separate the salts below 1 mm for use in the flotation process.

2.2. Solution Preparation

In order to prepare a circulating solution, certain proportions of carnallite and water were mixed together. A circulating solution (saturated solution of carnallite) with a density of more than 1.280 g/cm^3 was prepared for flotation.

2.3. Mashing

Since a high concentration of $CaCl_2$ disrupts the flotation, the mashing process is used to remove $CaCl_2$ from the solid phase. Garrett has reported the effect of $CaCl_2$ on flotation that increasing $CaCl_2$ concentration in solutions reduces the NaCl recovery [27]. First, the flotation feed and the circulating solution in the ventilation tank are mixed with $CaCl_2$, which is much more soluble than the other salts present in the feed dissolved in solution.

2.4. Synthesis of alkyl morpholine

The scientists have suggested a method for the synthesis of DDM using bromododecane ($C_{12}H_{25}Br$), which, in practice, has disadvantages for the synthesis of DDM, which include:

1. The formation of a white solid that is difficult to separate;
2. The white solid adheres to the reactor wall, and affects the reactor temperature;
3. Release of large amounts of CO_2 during the synthesis;
4. About 12 h is the synthesizing time;
5. About 84% is the efficiency of this method [28].

In 2001, an effective method was developed to produce DDM to be used in the flotation plants [28]. This method basically divides the above reaction into two stages. First, hydroxide and dodecyl-bromodecane are added before mixing, and then morpholine is added to produce DDM. Kimiaflot 619 was synthesized by two stages. First, bromo-octadecane and potassium hydroxide were mixed, and then morpholine was added. 1-bromoctadecane (1; 0.1 mol) was mixed with KOH (0.1 mol) in the reactor at 150 °C for 20 min.Then aqueous morpholine solution (2; 0.12 mol) was slowly injected. The reaction time was 8 h. Then N-(noctadecyl) morpholine was produced (3; 0.095 mol, 95% yield).

1 2 3

The result of the NMR spectrum analysis is shown in Figure 3. 400 MHz ^{1}H-NMR ($CDCl_3$, ppm): δ0.88 ($2CH_3$), 1.2-1.4 ($16CH_2$), 2.5(CH_2N^+), 2.4-2.6 ($CH_2N^+CH_2$), 3.5–4 (CH_2OCH_2).

2.5. Flotation

660 g of carnallite and 1200 cc of the circulating solution were added to the laboratory flotation cell with a volume of 1.6 L for each flotation test. The flotation cell and the flotation operation were performed with a stirrer speed of 910 rmp and a conditioning time of 2 min. After adding the collector, the air valve was opened for frothing, and the first concentrate was collected for 1.5 min. The second concentrate was collected for 4.5 min. Both concentrates of 1.5 min and 4.5 min were filtered, and the flotation tailings were filtered by a filtration machine in order to collect the filtered salts for the analysis of sodium, potassium, magnesium, and calcium. The first 1.5 min was considered as the first concentrate, the second 4.5 min as the second concentrate, and the flotation tailings as the precipitate.

3. Results and analysis

The flotation tests were designed as shown in Table 1. Figure 4 shows the NaCl grade in the carnallite flotation with kimiaflot 619 and Armoflot 619. The bench-scale flotation shows that kimiaflot 619 reveals a better selection for NaCl crystals from carnallite at a normal pH, resulting in lower collecting doses-only 1/2 molecule of Armoflot 619. The flotation result shows that the NaCl grade in carnallite concentrate by Armoflot 619 (200 g/t) is 2.86%, while the NaCl grade in carnallite concentrate by kimiaflot 619 (100 g/t) is 2.75%. Figure 4 also shows that the NaCl grade in carnallite decreases with increase in the collector concentrate, and the NaCl grade in the carnallite concentrate by kimiaflot 619 (125 g/t) is 2.02%. The flotation tests were repeated trice.

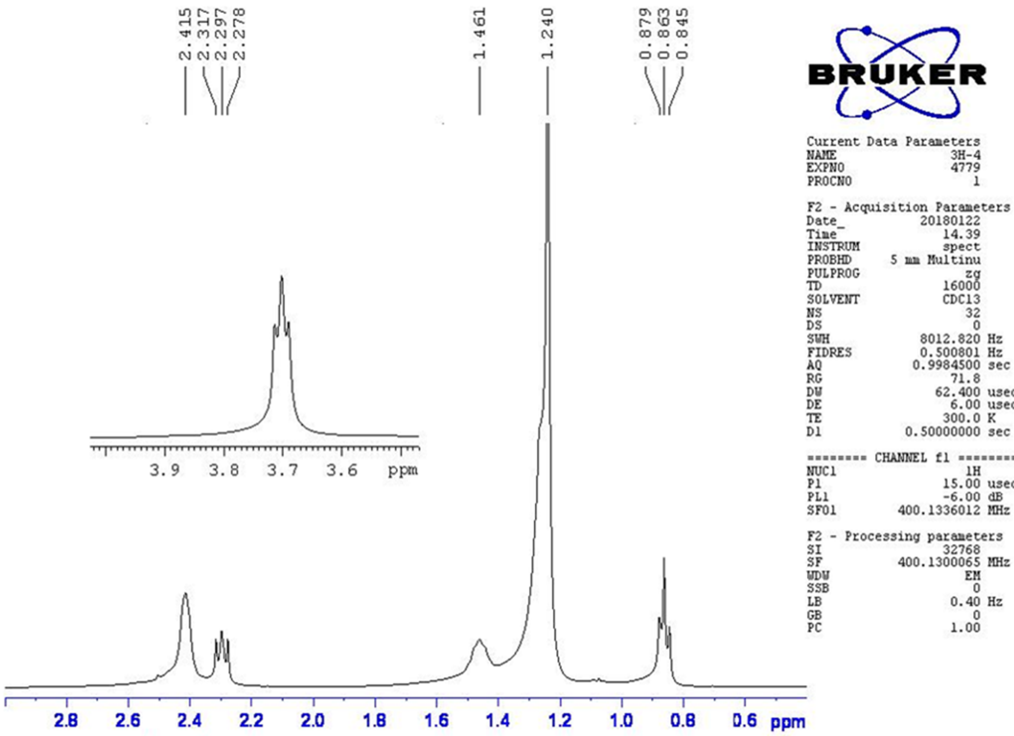

Figure 3. ^{1}H-NMR spectrum of kimiaflot 619 in $CDCl_3$.

Table 1. Design of experiments for flotation with kimiaflot 619.

Exp. name	Condition	Type collector	Collector concentration (g/t)	pH	H_2O (%)	$CaCl_2$ (%)	$MgCl_2$ (%)	KCl (%)	NaCl (%)
Feed					33.65	0.32	29.45	19.11	17.13
TA	Flotation tailings	Armoflot 619	200	0.2 ± 5	38.98	0.24	34.17	23.29	2.86
CA-1	Flotation concentrate after 1.5 min of foaming	Armoflot 619	200	0.2 ± 5	7.45	0.25	6.59	4.01	81.01
CA-2	Flotation concentrate after 4.5 min of foaming	Armoflot 619	200	0.2 ± 5	31.96	0.21	27.95	19.1	20.12
Ta	Flotation tailings	kimiaflot 619	50	0.2 ± 5	38.21	0.11	33.51	23.23	4.45
Ca-1	Flotation concentrate after 1.5 min of foaming	kimiaflot 619	50	0.2 ± 5	10.08	0.25	9.67	6.31	73.1
Ca-2	Flotation concentrate after 4.5 min of foaming	kimiaflot 619	50	0.2 ± 5	32.8	0.19	28.8	19.12	18.6
Tb	Flotation tailings	kimiaflot 619	75	0.2 ± 5	38.89	0.1	34.16	23.25	3.02
Cb-1	Flotation concentrate after 1.5 min of foaming	kimiaflot 619	75	0.2 ± 5	8.32	0.24	6.93	4.14	79.92
Cb-2	Flotation concentrate after 4.5 min of foaming	kimiaflot 619	75	0.2 ± 5	27.85	0.19	24.41	15.28	31.79
Tc	Flotation tailings	kimiaflot 619	100	0.2 ± 5	38.95	0.17	34.2	23.32	2.75
Cc-1	Flotation concentrate after 1.5 min of foaming	kimiaflot 619	100	0.2 ± 5	8.51	0.25	6.67	4.59	79.5
Cc-2	Flotation concentrate after 4.5 min of foaming	kimiaflot 619	100	0.2 ± 5	33.15	0.24	28.97	20.31	16.85
Td	Flotation tailings	kimiaflot 619	125	0.2 ± 5	38.98	0.26	34.23	23.95	2.02
Cd-1	Flotation concentrate after 1.5 min of foaming	kimiaflot 619	125	0.2 ± 5	7.47	0.25	6.67	4.11	81.08
Cd-2	Flotation concentrate after 4.5 min of foaming	kimiaflot 619	125	0.2 ± 5	31.97	0.21	27.89	19.15	20.15

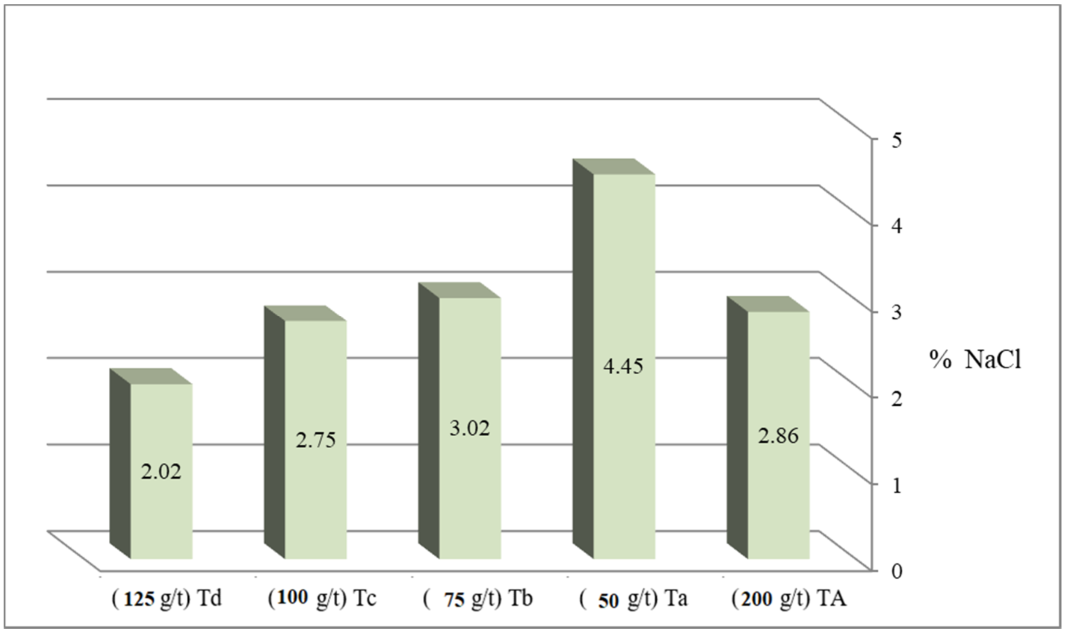

Figure 4. NaCl grade in carnallite flotation using Kimiaflot 619 and Armonflot 619.

Figure 5 shows the potassium chloride grade in the carnallite flotation with the kimiaflot 619 and Armoflot 619 collectors. The potassium chloride grade in carnallite concentrate by Armoflot 619 (200 g/t) is 23.29%, while the potassium chloride grade in carnallite concentrate by kimiaflot 619 (100 g/t) is 23.32%. The higher the potassium chloride, the higher the grade of carnallite because the chemical formula is carnallite ($KCl.MgCl_2.6H_2O$). Figure 5 also shows that the KCl grade in carnallite increases with increasing of kimiaflot 619 concentrate, and the KCl grade in carnallite concentrate by kimiaflot 619 (125 g/t) is 23.29%.

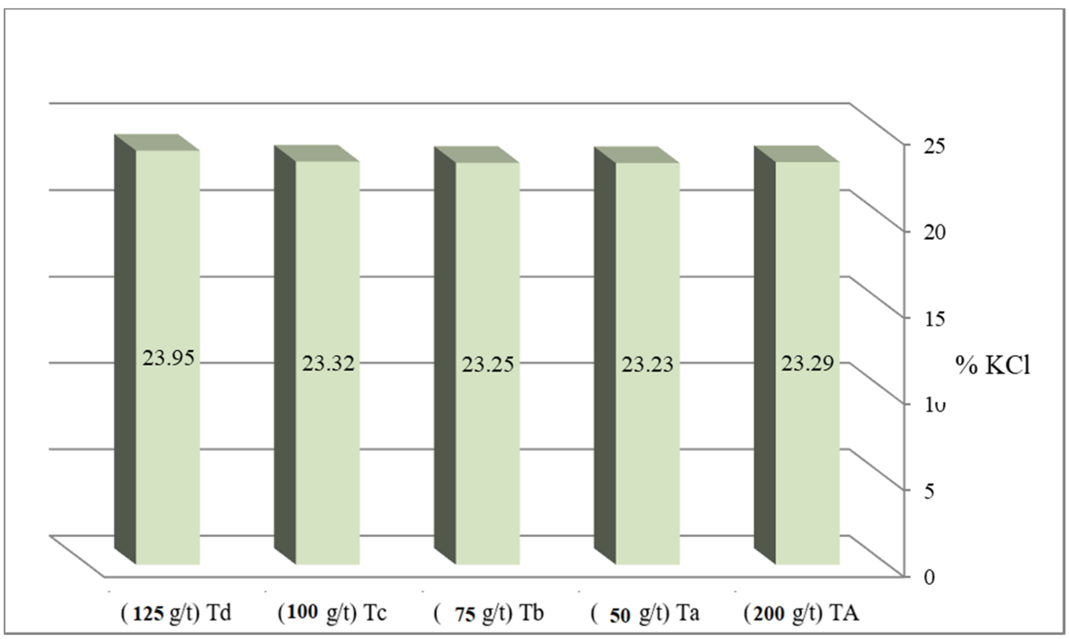

Figure 5. KCl content in carnallite flotation using Kimiaflot 619 and Armonflot 619.

4. Conclusions

Currently, more than 80% of the world's potassium production is carried out by the selective flotation of KCl from NaCl using the alkyl morpholine collector. In this research work, kimiaflot 619, as a new collector, was used, and the flotation results showed that kimiaflot 619 had better results than Armoflot 619 so that the sodium chloride grade in the carnallite concentrate with Armoflot 619 (200 g/t) was 2.86%, and the sodium chloride grade in the carnallite concentrate with kimiaflot 619 (100 g/t) was 2.75%. The frother's stability of the Armoflot 619 collector after flotation is very high and uncontrollable, and if a pump or flotation mixer fails, it will foam in the whole factory, and makes a difficult maintenance, while kimiaflot 619 solves this problem, and it is completely controllable. Kimiaflot 619 can be useful for the potash industry with improved grade in sodium chloride in the carnallite concentrate and less chemical usage.

Acknowledgments

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فلوتاسیون کارنالیت مجتمع پتاس خور بیابانک با استفاده از Kimiaflot 619 به عنوان کلکتورجدید

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چکیده:

کارنالیت، با فرمول شیمیایی $KMgCl_3.6H_2O$، یک ماده معدنی است که برای اولین بار در کوههای اورال در روسیه کشف شد. فلوتاسیون معکوس برای پردازش کارنالیت در دهه‌های اخیر ایجاد شده است و از کلکتور آلکیل مورفولین برای حذف NaCl از کارنالیت با استفاده از فلوتاسیون معکوس استفاده می شود. روش فرآوری کارنالیت شامل فلوتاسیون معکوس با کلکتور دودسیل مورفولین و سپس سانتریفیوژ و تبلور سرد است. در این کار تحقیقاتی ، Kimiaflot 619، به عنوان یک کلکتورجدید، سنتز شد و فلوتاسیون در مقیاس آزمایشگاهی نشان داد که Kimiaflot 619 انتخاب پذیری بهتری را برای بلورهای NaCl در pH اسیدی با دوزهای کلکتور کمتر (نصف کلکتور Armoflot 619) نشان می‌دهد. نتایج فلوتاسیون نشان داد که نمک NaCl در کارنالیت فراوری شده توسط Armoflot 619 (۲۰۰ گرم در تن) ۲/۸۶ است، در حالی که نمک NaCl در کارنالیت با غلظت کلکتور Kimiaflot 619 (۱۰۰ گرم در تن) ۲/۷۵ است. ضمنا ثبات کف پس از فلوتاسیون با کلکتور Armoflot 619 بسیار زیاد و غیر قابل کنترل است، در حالی که Kimiaflot 619 این مشکل را حل کرده است و کاملاً قابل کنترل است.

کلمات کلیدی: کارنالیت ، فلوتاسیون ، پتاس ، کلکتور، مجتمع پتاسخور بیابانک.