ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 9, Issue 4 , April 2022

Investigation of the Influence of the Carboxymethylcellulose Brand on the Process of Sludge Depression During Potassium Chloride Flotation

Bayraeva Dildora, Erkayev Aktam, Toirov Zokir, Adilova Moxira

Doctoral student PhD, Tashkent Chemical-Technological Institute, Tashkent, Uzbekistan
Professor, Tashkent Chemical-Technological Institute, Tashkent, Uzbekistan
Associate Professor, Tashkent Chemical-Technological Institute, Tashkent, Uzbekistan
Associate Professor, Tashkent Chemical-Technological Institute, Tashkent, Uzbekistan

ABSTRACT: The possibility of obtaining potassium chloride from low-grade silvinites with the use of a flotation agent obtained on the basis of local raw materials is shown. The influence of the fractional composition of silvinite ore on its chemical composition is investigated. Installed, that when enriching low-grade silvinites using NaCMS grades 55H, 75B, 85H and starch as a depressor reagent, the degree of extraction of silvin increases while reducing the content of impurities in the concentrate due to increased depressing effect.

KEYWORDS: flotation, flotation agent, depressor, potassium chloride, starch, carboxymethylcellulose, silvinite.

I. INTRODUCTION

The production of potash fertilizers from silvinite ores of various deposits of the post-Soviet space and far abroad is carried out mainly by the flotation method from saturated solutions. The total mass fraction of soluble salts in a saturated solution changes with temperature changes, while the properties of the reagents used in the flotation suspension also change, which affects the physicochemical interaction of the reagents in the volume and on the interface of the phases, as well as the technological parameters of the process. The effect of an increase in the temperature of a saturated salt solution in the range of 30-35 °C The influence on the process of flotation of potash ores has been studied in sufficient detail [4-8].

In order to determine the effective reagents of the paraffin base, amine was used as modifiers for the flotation of silvinite ore at mother liquor temperatures above 30 ° C. In addition to the reagents currently used by the industrial factories of JSC Belaruskali, such as petroleum liquid paraffin (PG), polyethylene glycol (PEG) and previously used GACH, other paraffin-containing reagents were investigated: vacuum gas oil, oil edema (produced by JSC "Mining Wax Plant", Republic of Belarus), reagents 1C, 2C, 5C (developed by BSTU) [7].

Currently, the demand for potash fertilizers is increasing day by day. The Republic of Uzbekistan occupies one of the leading places in the world in terms of potash salt reserves (silvinite). Even one of the Tubegatan deposits has reserves of over 215 million tons of silvinite. Currently, a technology has been developed for the production of potassium chloride from the silvinite of the Tyubegatan deposit by flotation at the Dehkanabad Potash Plant, which provides for the processing of silvinite ore with a content of at least 31.93% KCI and water-insoluble residues (n.o.) of no more than 3.25%. The mechanism consists of the following stages: diffusion in solution and selective adsorption of the collector on the surface of the salt mineral; collision of mineral particles with air bubbles in the pulp; fixing the particles that collided with them on the bubbles; formation of mineralized foam, removal of particles with foam to the surface in the form of concentrate. The separation of potassium chloride (silvin) into the foam product from silvinite ores by flotation is possible due to the ability of air bubbles to adhere and carry hydrophobic particles of silvin into the foam product. The hydrophobicity property of sylvin particles is not related to the property of its crystal lattice, but is the result of processing

ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 9, Issue 4 , April 2022

them with a special selectively acting reagent collector. The selective process of hydrophobization of floated minerals is based on the phenomenon of sorption of the reagent on individual minerals, which can be considered as chemisorption or physical sorption on floated minerals.

II. SIGNIFICANCE OF THE SYSTEM

The possibility of obtaining potassium chloride from low-grade silvinites with the use of a flotation agent obtained on the basis of local raw materials is shown. The study of methodology is explained in section III, section IV covers the experimental results of the study, and section V discusses the future study and conclusion.

III. METHODOLOGY

The content of potassium ions was determined by tetraphenylborate method and flame photometry; sodium ions - by flame photometry and by calculation [1-3]. The chlorine ion content was determined by the Mohr method [1].

Determination of the mass fraction of the residue insoluble in water in raw materials and products of potash production was carried out by the gravimetric method [1], which is based on the separation of the insoluble residue by filtering the dissolved analyzed sample and subsequent drying and weighing of the resulting sediment.

IV. EXPERIMENTAL RESULTS

Therefore, in this work, the possibility of using low-grade silvinites and flotation reagents obtained on the basis of raw materials of local origin was studied. In order to determine the relationship between the water-insoluble residue (n.o.) and potassium chloride in the layers, the influence of the fractional composition of the ore on its chemical composition was studied. For this purpose, the average crushed ore with a known concentration was divided into fractions with a particle diameter, mm: 1,60-1,25; 1,25-1,00; 1,00-0,50; 0,50-0,10; -0,10. The chemical composition of each fraction was studied. The results of the studies are shown in Table 1.

Table 1
Influence of the fractional composition of ore on its chemical composition

Silvinite ore, mm	Mass fraction of components, %			
	KCl	NaCl	н.о.	H_2O
+1,60-1,25	9,42	83,17	5,01	0,14
+1,25-1,00	10,32	75,73	12,56	0,15
+1,00-0,50	7,85	66,97	24,54	0,24
+0,50-0,10	6,32	60,11	32,20	0,28
-0,10	4,78	47,56	44,60	0,16

The results of experiments show that when grinding low-grade ore, the main part of the insoluble residue in the form of carbonates, sulfates and soil passes into the composition of a fine fraction, and potassium and sodium chlorides due to the complexity of grinding remain in the composition of a large fraction. Based on the data obtained, it was found that when the silvinite samples were divided into fractions, the optimal fraction in terms of the amount of KCl and n.o. can be considered fractions in the range of +1.60-1.25 and +1.25-1.00. In this interval, the amount of KCl ranges from 9.42-10.32%, and n.o. - 5.01-12.56%, and these fractions can be further enriched by flotation, and the remaining fractions by gallurgy.

Clay-carbonate impurities are easily slimed in the liquid phase (saturated salt solution) of KCl- NaCl-H2O with a MgCl2 content of 0.1-1.0%, actively sorb the cationic collector - amine and worsen the floatability of silvin. To eliminate the negative effect of clay-carbonate sludge in the flotation of potassium chloride, depressant reagents are used, supplied to the power of the sylvin flotation before the collector reagent. Depressant reagents are organic polymers and oligomers. Being fixed on the surface of helmets, they prevent the sorption of amine on the surface of slurries and thereby create favorable conditions for fixing the required amount of amine on the surface of KCl particles and their successful flotation.

It is known that the products of the synthesis of urea, formaldehyde and polyethylene - polyamine with a molar ratio of urea - formaldehyde – polyethylene – polyamine are used as a depressor reagent in the flotation of potash ores

ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 9, Issue 4 , April 2022

1:1,12:0,05-1:2,70:0,30 [18]. The disadvantage of this method is the high cost, instability of the technological properties of the resins of the claimed composition for a relatively short time of their storage (after 6 months, the resin begins to polymerize and loses the properties of the depressor).

CMS monocarboxycellulose (МКЦ), cellulose acetates and xanthogenates, cellulose sulfoesters (СЭЦ), urea-formaldehyde resins (МФС) are proposed as sludge modifiers. The sodium salt of carboxymethylcellulose, used as a sludge modifier at the «Belaruskali» production association, has found the most widespread use.

At optimal КМЦ costs, both a 20-30% reduction in the sorption of the collector of clay –carbonate sludge is achieved, providing conditions for successful flotation of silvin, and effective depression of clay sludge.

The effectiveness of КМЦ is determined by the degrees of its substitution (the number of one hundred carboxyl groups in which hydrogen is replaced by sodium ion) and polymerization (the number of links in the КМЦ molecule equal to n).

As can be seen from the results of [20,21] sorption experiments on the flotation of the silvinite Starobin deposit, in parallel with a decrease in the sorption of amine on slurries, the extraction of KCl into concentrate also increases. The greatest extraction of KCl into the concentrate is achieved by using a modifier that reduces the sorption of amine on slurries to the greatest extent. Flotation experiments also showed that during the enrichment of silvinites with a KCl content of 25-30% and about 4%, the collector consumption (without the addition of a foam regulator) is 100 g/t, the modifier (КМЦ) is 500-800 g/t of ore, i.e. in terms of clay-carbonate sludge, the amine consumption is 2.5 mg/g, the modifier is 15-20 mg/g. This amount of amine is almost completely sorbed on slurries, and when their surface is treated with a modifier - 20-30%, which corresponds to amine consumption standards. As experiments on monomineral flotation of KCl have shown, this amount of amine is sufficient for the complete extraction of KCl into the foam product.

Currently, starch is used as a depressor reagent at the UP "Dehkanabad Potash Plant".

The disadvantages of this method are:

- high depressor consumption - 250 g/t of ore;
- low efficiency of the presence of insoluble residue and magnesium chloride in the liquid phase - more than 2%;
- certain difficulties in the preparation of starch paste.

The aim of the study was to select a depressor reagent that eliminates the above disadvantages.

In accordance with this, flotation was carried out using the sodium salt of carboxymethylcellulose (NaNACMS) as a depressor reagent, providing a degree of polymerization of 500-1500, to obtain a product that meets the requirements of technical specifications-88.2-12-2005 . NaNACMS is obtained by leaching cotton cellulose with alkaline solutions followed by carboxymethylation.

The process involves the preparation of a 0.5-1% NaNACMS solution and its supply to the sylvin flotation feed before adding a collector reagent in the amount of 30-250 g per ton of feedstock.

A distinctive feature of the proposed method is the use of NaNACMS, which does not have a toxic, irritating effect; it is not explosive and is produced from the waste of the textile industry – cotton lint.

The proposed method was tested in laboratory conditions on a sample of potash ore from the Tyubegatan deposit of the following composition: KCl-28.7%, n.o.-4%.

The liquid phase of flotation is a saturated salt solution (KCl-10.3%, NaCl-20.3%, MgCl2-0.7%).

We have conducted a series of experiments on the enrichment of silvinite ores in laboratory conditions using grades 55H, 75B, 85H and starch as a depressor reagent of NaNACMS. NaNACMS has the property of dissolving in water with the formation of colloidal solutions and suppression of flotation of silvinite ore waste rocks. The technical result is a reduction in the consumption of the depressor reagent during the flotation of silvinite ores, an increase in the degree of extraction of silvin while reducing the content of impurities in the concentrate by increasing the depressing effect. The depressor reagent is a product of aqueous solutions with concentrations in the range of 0.5-1.0%. The starch consumption rate according to the approved technological regulations was set at 250 grams per 1 ton of ore, and the consumption of samples of NaNACMS ranged from 30 to 150 mg per 1 ton of silvinite ore. During the tests, the consumption of other flotation reagents and technological parameters were maintained in accordance with the current technological regulations.

The installation consisted of a 240 ФЛ-4 flotation machine with a chamber volume of 1.5 liters and a sprayer for preparing an aerosol of the tested reagents. The nozzle of the sprayer was inserted into the nozzle connecting the block-impeller and the circulation pocket. The amount of air supplied from the compressor was regulated using a rotameter. Flotation experiments with aerosol and conventional dosage of reagents were carried out with the same parameters: flotation time, air flow, impeller speed and others.

ISSN: 2350-0328

International Journal of Advanced Research in Science, Engineering and Technology

Vol. 9, Issue 4 , April 2022

We have conducted a series of experiments on the enrichment of silvinite ores in laboratory conditions using a depressor reagent based on NaNACMS grades 75-400, 85-600, 85-800 and starch. The technical result is a reduction in the consumption of the depressor reagent during the flotation of silvinite ores, an increase in the degree of extraction of silvin while reducing the content of impurities in the concentrate by increasing the depressing effect. The starch consumption rate according to the approved technological regulations is set at 200-250 g, and the consumption of NaNACMS samples ranged from 30 to 100 g per 1t of silvinite ore. During the tests, the consumption of other flotation reagents and technological parameters were maintained as in the current technological regulations. Distilled and process water, mother liquor, brine and a mixture of brine and process water at mass ratios of 1:1 were used as a solvent. When using distilled and process water (Table 2), the KCl content in the rough concentrate increases to more than 94% with the potassium chloride yield reaching 89.52%. However, when using NaNACMS in salt solutions, the content of potassium chloride in the rough concentrate decreases to 92% and the yield of the latter decreases to 26.35%. This shows that in order to ensure the required quality and yield of the product, an increase in the number of chambers of the flotation machine to 10 is required. Thus, when using 75/400 grade NaNACMS as a sludge depressor with the latter dissolved in distilled or industrial water and at its consumption of 100-120 g/ t, the yield of the product increases by 0.2 and 12%, respectively, compared with the use of starch.

Of the tested depressant reagents, the most effective is 75/400 grade NaNACMS at a consumption of 100-120 g per 1t of silvinite ore. Based on the above, it was recommended to conduct pilot tests in the existing workshop of the UP "DKZ".

Table 2

Test results of solutions of NaNACMS brand 75/400 and starch as a depressor reagent in laboratory conditions

Type of solvent	Brand of depressor	Depressor concentration, mass.%	Depressor consumption, g/t	Ratio T:J	Chemical composition of silvinite, mass.%				Chemical composition of the rough concentrate, mass.%				Exit KCl, %
					KCl	K_2O	H.O	W	KCl	K_2O	H.O	W	
Distilled water	NaCMS	0,8	120	1,15	32,0	20,21	2,9	0,48	94,1	59,5	6,08	1,04	84,63
		1	100	0,89	32,0	20,21	2,9	0,48	93,0	58,75	6,1	1,08	72,94
Technical water		0,8	120	1,01	26,8	16,93	3,1	0,36	94,5	59,72	6,07	1,01	89,52
		1	100	1,65	30,3	19,14	2,93	0,5	93,7	59,23	6,09	1,32	59,70
Mother liquor		0,8	120	0,48	32,0	20,21	2,94	0,48	93,7	59,25	6,08	1,03	34,03
		1	100	0,49	32,0	20,21	2,9	0,48	93,7	59,21	6,10	1,01	28,02
Brine		0,8	120	0,61	30,3	19,14	2,9	0,5	93,5	59,1	6,11	1,31	26,35
		1	100	0,77	30,3	19,14	2,9	0,51	92,9	58,7	6,12	1,26	30,75
Technical water: Brine =1:1		0,8	120	0,52	30,3	19,14	2,9	0,52	92,5	58,43	6,10	1,25	33,91
		1	100	0,87	30,3	19,14	2,9	0,5	92,0	58,16	6,12	1,09	35,39
technological water	Corn starch	2,5	250	0,73	26,8	16,93	3,1	0,36	94,4	59,67	7,08	1,18	77,22

ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 9, Issue 4 , April 2022

Table 3
Examples of the implementation of the method in the laboratory

№	Starch and NaNACMS obtained from cotton pulp	Depressor consumption, g/t	Dry cake concentrate, g	Content of oxides, %		Output of potassium chloride, %
				K_2O	Na_2O	
1	Starch	30	5,69	48,56	4,68	4,42
2		60	51,19	54,22	3,14	44,44
3		210	50,17	59,83	2,75	48,06
4		300	44,19	53,21	1,44	37,65
5	CMS-85/800	30	4,12	43,86	2,91	3,02
6		60	46,78	53,07	0,53	39,67
7		210	14,61	54,83	0,81	12,83
8		300	1,71	33,96	2,78	0,96
9	CMS-85/600	30	7,60	50,71	2,13	6,15
10		60	50,72	48,08	1,57	39,06
11		210	30,80	55,52	1,31	27,38
12		300	16,98	56,51	0,59	15,33
13	CMS-75/400	30	20,64	59,81	0,98	19,76
14		60	44,53	54,07	0,95	38,55
15		210	3,07	57,80	0,99	2,83
16		300	34,41	44,46	6,79	

In further experiments, 200g of sylvinite and 600g of mother liquor were used.

200 or 400 g of ore were crushed to a size of 1 mm, added to 600 or 1200 g of mother liquor and flotation of KCl was carried out using primary amines with a length of hydrocarbon radical C20-C22 as a collector at a consumption of 50 g/ton of ore. As a depressor reagent, various grades of NaNACMS and starch were used.

Table 3 shows examples of execution and their results.

As can be seen from the presented data, flotation using NaNACMS as a depressor reagent with polymerization degrees of 300-1000 at a depressor consumption of 30, 60, 120 g / t of ore improves the flotation of potassium chloride (increases the extraction of KCl into the finished product).

Tables 3 and 4 present comparative data on the implementation of the method in laboratory and laboratory – model conditions, respectively. The data show that in order to achieve the maximum yield of potassium chloride, the costs of the depressor reagent are sharply reduced, since 60 g of NaNACMS is used for 1 ton of ore in the experiments instead of 250 g of starch.

ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 9, Issue 4 , April 2022

Table 4
Examples of the implementation of the method on the model installation of the ЦЗЛ УП "Dehkanabad potash plant".

№	Type of depressor	Depressor consumption, g	Wet cake concentrate, g	Dry cake concentrate, g	Content, %			Output of potassium chloride,%
					K_2O	Na_2O	н.о.	
1	Starch	200	125,63	109,18	54,99	1,70	0,94	82,89
2	CMS 55	60	104,88	93,72	59,10	0,81	0,87	76,47
3	CMS 75	60	89,32	81,7	59,20	0,91	0,14	66,48
4	CMS 85/900	60	123,91	109,13	52,76	0,55	1,03	79,49

A slight decrease in the yield of potassium chloride when using NaNACMS is compensated by a decrease in the Na_2O content in the product, which makes it possible to reduce the number of devices in subsequent operations. The higher efficiency of NaNACMS is expressed in the fact that, compared with starch, it can be used for flotation enrichment of silvinites with an impurity content of more than 2.5%, whereas in the case of starch this is impossible.

In addition, Na CMC is distinguished by a longer shelf life. All these advantages make it possible to reduce costs by 1.8 times compared to using starch.

V. CONCLUSION AND FUTURE WORK

A series of experiments on the enrichment of silvinite ores in laboratory conditions with the use of a depressor reagent based on NaKMC of various brands were carried out. It is shown that the use as a depressor reagent of 0.5 - 1.0% sodium salt solution of carboxymethylcellulose - NaKMC with a degree of polymerization of 600-1200 in an amount of 60-80 g per 1 ton of enriched initial silvinite gives the following advantages:

- low consumption of the depressor reagent – 60-160 g/t of ore;
- increasing the stability of the solution during long-term storage;
- more effective action with an increased content of n.o. and magnesium chlorides in the ore than with the use of starch;
- waste from the textile industry is being disposed of;
- the costs are reduced by 1.8 times compared to the known method

REFERENCES

[1]. Pozin M.E. Guide to practical exercises on the technology of inorganic substances. - L.: Chemistry, 1980. - 368 p.
[2]. Zhukov A.F., Kolosova I.F., Kuznetsov V.V. Analytical chemistry. Physical and physico-chemical methods of analysis. Moscow: Chemistry, 2001. 496 p.
[3]. Flame photometry: guidelines for laboratory work.// Samara. SGTU. 2013. -13 p.
[4]. Alexandrovich Kh. M. Fundamentals of the use of reagents in the flotation of potash ores. Moscow: Science and technology, 1973. 412 p.
[5]. Aliferova S.N. dis. … cand. tech. Sciences: 25.00.13. Yekaterinburg, 2007. 198 sheets.
[6]. Abramov, A. A. Enrichment of ores // Proceedings of scientific and technical. conf. Institute of Mechanobr. Leningrad, 1969, p. 304.
[7]. Podlesnaya, Z. S. Investigation of the regularities of flotation enrichment of sylvin from clay-salt suspensions using apolar collectors: dis. … Ph.D. Sciences: 05.15.08 Leningrad, 1979. 167 sheets. [9]. [8]. ISSN 1683-0377. Proceedings of BSTU. 2014 No. 3. Chemistry and technology of inorganic materials and substances S-71 UDC622.7.017.2 “Sylvin flotation from potash ores at elevated temperatures.” M. R. Turko, O. B. Dormeshkin, E. M. Miskov, L. A. Solovieva.
[9]. News of the National Academy of Sciences of Belarus. Series of chemical sciences. 2019. V.55, No. 3. P.277-287 “Flotation enrichment of potash ores”. L.V. Dikhtievskaya, L.F. Shlomina, E.O. Osipova, V.V. Shevchuk, F.F. Mozheiko
[10]. Patent No. 2327525, B03D 1/02, Publ. 06/27/2008, Bull. No. 18.
[11]. Patent No. 2245742, B03D 1/02, Publ. 10.02.2005, Bull. No. 4.
[12]. Titkov S.N., Mamedov A.I., Solovyov E.I. Enrichment of potash ores.-M.: Nedra, 1982.

ISSN: 2350-0328

International Journal of Advanced Research in Science, Engineering and Technology

Vol. 9, Issue 4 , April 2022

[13]. Alexandrovich Kh.M., Mozheiko F.F., Korshuk E.F., Markin A.D. Physicochemistry of selective flotation of potassium salts, pp. 156-164 – prototype.
[14]. US patent No. 3017028 dated 01/16/62.
[15]. Patent No. 2165798 dated 09/01/1999 (prototype).
[16]. Methods for the analysis of brines and salts. Under the editorship of Morchevsky Yu.V. and Petrova E.M., All-Russian Research Institute of Gallurgy. Chemistry - 404 p.
[17]. Deposits of potassium salts. Methods of their search and exploration. Ed. Raevsky V.I. and M.P. Viehweg. L .: Nedra, 1989
[18]. Patent RU216G798 Method for flotation concentration of potash ores. CM. Titkov, A.M. Vakhrushev, A.A. Tsistenov. Claimed 09/01/1999. Application No. 99118841/03. Published on 04/27/2001.
[19]. Permanent technological regulation. UE Dekhkanabad Plant of Potash Fertilizers. 2012 p.81.
[20]. Skvirsky L.Ya., Titkov S.N. Features of the action of reagents - modifiers in the cationic flotation of potash ores. – Enrichment of ores. 1974, No. 2, pp. 25-30.
[21]. Titkov S.N. Flotation of clayey sylvinite ores using organic depressants. Abstract dis. for the competition uch.step. Candidate of Technical Sciences, L., 1972. (LGI).