ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 7, Issue 11 , November 2020

A new source of liquid paraffins for flotation enrichment of potassium chloride from natural silvinite

Sh.B. Bukhorov, S.S. Vakkasov, I.D. Eshmetov, X.E. Kadirov

Candidate of chemical sciences, associate professors of Tashkent Chemical Technological Institute, Republic of Uzbekistan
Jizzakh Polytechnic Institute, Uzbekistan
Doctor of Technical Sciences, Professor, Institute of General and Inorganic Chemistry,Academy of Sciences of the Republic of Uzbekistan, Republic of Uzbekistan
Doctor of Technical Sciences, associate professors of Tashkent Chemical Technological Institute, Republic of Uzbekistan

ABSTRACT: Chromatographic analysis determined and compared the qualitative and quantitative compositions of the imported and obtained liquid paraffin. It is proved that the use of liquid paraffin obtained from liquid secondary raw materials, flotation is the best performance, in addition, it allows you to increase the extraction of KCl into concentrate and reduce the consumption of expensive amine, thereby reducing the cost. It has been determined that a decrease in the concentration of potassium chloride in the composition of the mixture leads to an increase in the yield of its extraction from this mixture, for the flotation of sylvinite ores with a mass content of the main essential component of more than 20%, the required amount of apolite reagent is more than 10 g per ton ore. In this case, as a result of an increase in the temperature of the solution from 20 to 30 °C, an increase in the extraction of KCl from the apolite reagent by 20% is observed.

KEYWORDS: flotation, flotation reagents, collector, recovery, selvinite ore, potassium chloride, liquid secondary raw materials, chromatographic analysis, apolar reganet, liquid paraffins, saturated hydrocarbons**.**

I. INTRODUCTION

In recent years, great changes have been taking place in the chemical industry of the Republic of Uzbekistan. Promising directions for the development of chemical production are the implementation of the localization program, the production of import-substituting products based on the processing of secondary raw materials from the local industry.

Flotation is the main process of mineral processing. It is used for the beneficiation of 95% of mined non-ferrous metal ores, and not metal (phosphorites, graphite, talc, small grades of coal) - almost 100% [1,2].

Such a widespread use of this process is explained by the fact that flotation allows you to extract valuable components in the most complete and complex way with the required quality of concentrates, as well as to enrich finely disseminated, poor and those ores that cannot be processed by other processes [3 - 5].

One of the important stages of ore processing in the chemical industry is the concentration of metal compounds and ore beneficiation. The main chemicals used in this technological process are flotation reagents, which are used through imports. Many compounds are used as flotation reagents, however, flotation reagents used in industry are usually relatively high molecular weight aliphatic amines and liquid paraffins. The main components of flotation reagents used in industry are paraffins with C_{11}-C_{22} [6].

ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 7, Issue 11 , November 2020

In the flotation of sylvite, cationic reagents are used as a collector, in particular, primary aliphatic amines, which interact with the surface of minerals of the useful component KCl, form a shell on it, imparting hydrophobicity. This allows the mineral to combine with air bubbles and float into the concentrate foam. However, in addition to KCl and NaCl, the original ore contains clay impurities with a developed surface. They are able to strongly disperse during ore grinding and actively adsorb amine molecules. The greatest interest from the point of view of influence on the process of separation of potassium salts is provided by a finely dispersed clay fraction of 0.001 mm. The specific surface area of clay sludge available to the cationic collector amine is 350-370 m^2/t. Salt sludge is represented by finely dispersed sylvite and halite with a particle size of 60 microns, it is formed during the overgrinding of minerals during mining, transportation, preparation of ore for flotation and directly in flotation chambers [7].

The authors of work [8] propose to use kerosene as a collector of fatty amines and a mixture of hydrocarbons. As a rule, kerosene contains up to 30% aromatic hydrocarbons, which negatively affect the selectivity of flotation concentration of sylvinite ores. In addition, naphthalene hydrocarbons are carcinogenic, so their involvement in the process is undesirable.

Currently, in the Republic of Uzbekistan, the main users of flotation processes are the NavoiMining and Metallurgical Combine, Almalyk Mining and Metallurgical Combine, Dekhkanabad Potash Plant JSC, etc., on which ores of non-ferrous, rare, ferrous metals, coal, phosphate ores float, sulfur, feldspar, boric ores, potash salts and other minerals.

JSC “Dekhkanabad Potash Plant” in the process of flotation enrichment of potassium chloride from natural sylvinite in the composition of the collective mixture of flotation reagents uses liquid paraffin. Liquid paraffin are mainly saturated hydrocarbons with a C_5 - C_{18} carbon number of normal structure.

Object of study: the process of flotation of enrichment of potassium chloride from natural sylvinite as part of the collective mixture of Dekhkanabad Potash Plant JSC.

The subjects of research are liquid paraffin with C_{14}-C_{18} carbons of normal structure, liquid secondary raw materials, production of polyethylene and polypropylene by polymerization in the presence of a Ziegler-Natta catalyst in a hexane solution.

II. RESEARCH METHODS

I. We used chromium-mass spectroscopy: an Agilent Technology GC 6890 / MS 5973N gas chromatograph-mass spectrometer using a capillary column 30 m × 0.25 mm in size with 5% phenylmethylsiloxane in dimethylsiloxane, carrier gas hydrogen, injector temperature 280 °C, MS temperature of the source - 230°C, MS temperature of the quadrupole - 180°C, when programming the temperature of the column oven from 100 to 280°C, temperature rise rate 10°C min, sample size 1 μL, in splitless mode , in addition to this, standardized test methods, etc. have been studied to determine the physical, mechanical and technological properties.

III. RESEARCH RESULTS AND DISCUSSIONS

To select a source of raw materials and conduct further research, chromatographic analysis was carried out and the qualitative and quantitative composition of imported liquid paraffin was determined (Fig. 1).

ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 7, Issue 11 , November 2020

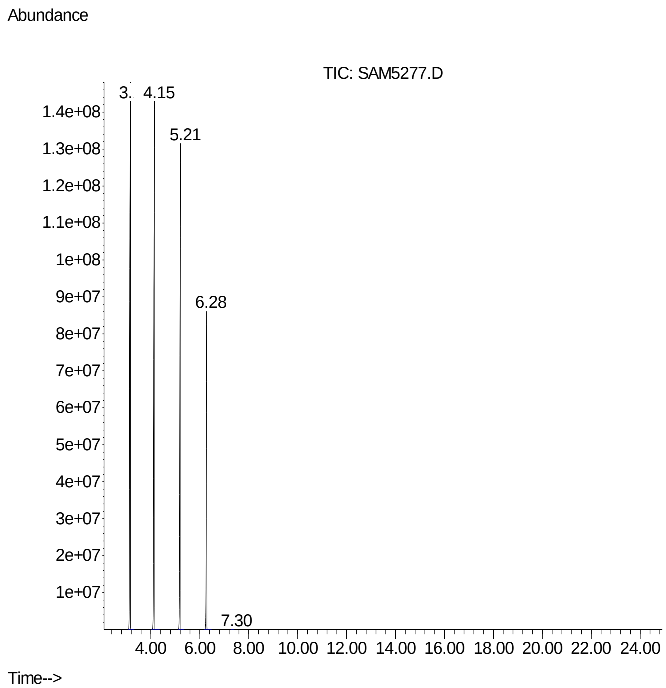

Fig. 1. Chromatogram of imported liquid paraffin of flotation concentration of potassium chloride from natural sylvinite in the composition of the collective mixture of flotation reagents
1 - Tetradecane; 2- Pentadecane; 3- n-Cetan; 4 - n-Heptadecane; 5 - n-octadecane

The obtained results show that the basis of the composition of imported liquid paraffin is saturated hydrocarbons with C_{14} - C_{18} carbons of normal structure, and this is the basis for the search and selection of a raw material source.

The Ustyurt gas chemical complex (Republic of Karakalpakistan) produces polyethylene and polypropylene by polymerization reaction in the presence of a Ziegler-Natta catalyst in a hexane solution. In this process, in addition to the main polymer product, liquid secondary raw materials are also formed. The residual product is an oligomer of the monomers used, the bulk of which are paraffin from C_6 to C_{20}, the qualitative and quantitative composition of which was determined by the chromatographic method (Fig. 2).

Chromatographic analysis shows that the composition of the spent hexane mainly consists of saturated hydrocarbons of the C_{14} - C_{18} fraction of normal structure.

The separation of a mixture of liquid secondary raw materials was carried out on a laboratory vacuum distillation apparatus equipped with a cube, a reflux condenser, a thermometer, a Liebig refrigerator connected to a vacuum pump: distillation was carried out until a thermometer reading of 135 °C under a vacuum of 650 mm Hg. From one liter of a sample of spent hexane, 0.450 liters of C_{12} - C_{20} fraction of saturated hydrocarbons of normal structure were obtained. The density of the obtained sample of liquid paraffin at 20 ° C is 745 kg/m^3.

ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 7, Issue 11 , November 2020

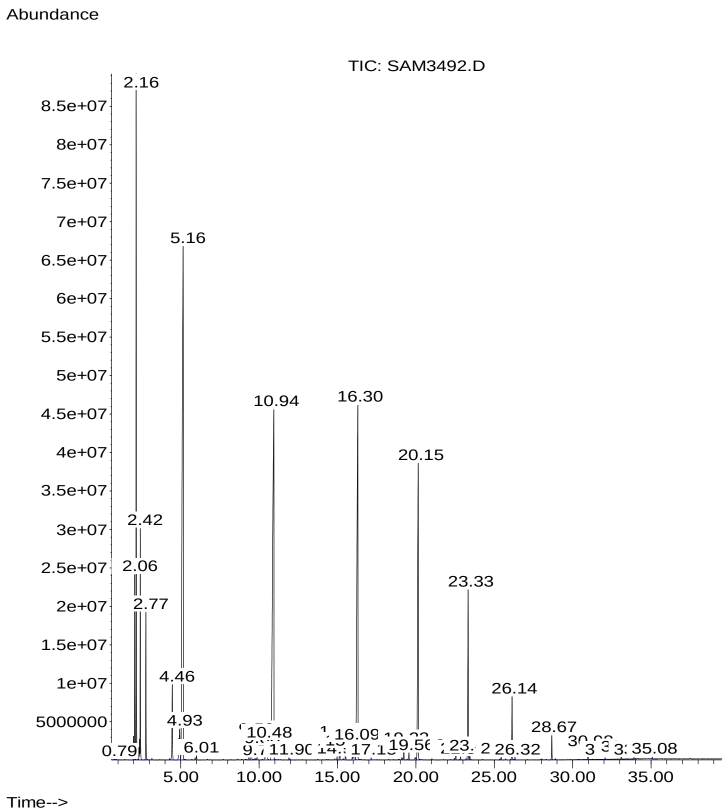

Fig.2. Chromatogram of liquid secondary raw materials for the production of polyethylene and polypropylene by polymerization reaction in the presence of a Ziegler-Natta catalyst in a hexane solution

1 - 2-methylpentane; 2 - 3-methylpentane; 3 - hexane; 4 - cyclopentane; 5 -cyclohexane; 6 - 2-ethyl-hexane; 7 - trans-1-ethyl-3-methylcyclopentane; 8 - n-octane; 9 - ethylcyclohexane; 10 - octane; 11 - 5-methylnonan; 12 -9-methyleicosane; 13 - 3-methylnonan; 14 - 2-heptenal; 15 -decane**;** 16 -1-cyclohexyl; 17 - 4-ethyldecane; 18 -undecane; 19 - 3-methyl-undecane; 20 - 1-hexyl-3-methylcyclo-pentane; 21 -dodecane**;** 22 - 1-hexylcyclohexane; 23 -tridecane; 24 - 3-methyl-tridecane; 25 - n-tetradecane; 26 - 5-methyltetradecane; 27 -pentadecane; 28 -n-hexadecan; 29 -nonyl-cyclohexane; 30 - 5-methyl-pentadecane; 31 - n-octadecane; 32 -cyclohexylmethane; 33 - n-eicosane; 34 - n-docosane; 35 - 2-methyl-cyclodecanone; 36 - n-Tetracosane; 37 - Z,Z-3,13-octadecedien-1-ol; 38 -hexacosane

When separating 1 liter of a mixture of liquid secondary raw materials, on a laboratory vacuum distillation apparatus equipped with a cube, a reflux condenser, a thermometer, a Liebig refrigerator connected to a vacuum pump: distillation was carried out to a thermometer reading of 135°C under a vacuum of 650 mm Hg, 0.450 liters of fraction was obtained C_{12} - C_{20} saturated hydrocarbons of normal structure, the density of the obtained sample of liquid paraffin at 20 °C is 745 kg/m^3.

On the basis of liquid paraffin obtained in laboratory conditions, sylvinites were floated in artificially prepared 3 different concentrations of model solutions, in which the mass ratio of NaCl and KCl is: 1: 1 (P1); 1: 0.5 (P2) and 1: 0.25 (P3), and the total salt content in the solution does not exceed 50%.

To prepare solutions, the salts, dried to constant weight at a temperature of 120 °C, were quantitatively transferred into volumetric flasks with a capacity of 1000 cm^3, dissolved in distilled water, bringing the solution to the mark. Experimental flotation processes were carried out on an FML 240 flotation machine. Consumption of aliphatic amine for all samples was 10 mg/kg of salt mixture. The research results are shown in Table 2.

ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 7, Issue 11 , November 2020

Table 2
Results of flotation of model solutions at 22 ± 1 °C using liquid paraffin

Index		Model solutions		
		P1	P2	P3
Output,%:	concentrate	41,2	30,5	19,6
	tail	58,8	69,5	80,4
MassfractionofKCl, %:	concentrate	97,2	91,3	90,8
	tail	17,0	7,8	4,3
ExtractionofKCl, %:	concentrate	80	83,6	89
	tail	20	16,4	11

As the data in Table 2 shows a decrease in the concentration of potassium chloride in the mixture leads to an increase in the yield of its extraction from this mixture. Probably, the lower recovery factor P1 is due to the lack of amounts of the apole reagent and amine for the adsorption of its molecule on the entire surface of the floated material. Under the influence of the apolic reagent, P3 floats more fully, which indicates that the amount of the collector is sufficient. Consequently, for the flotation of sylvinite ores with a mass content of the main required component of more than 20%, the required amount of apolite reagent is more than 10 g per ton of ore (this amount is calculated using the KKM value).

It is generally known that the temperature of the solution will be a key factor in the flotation of potassium chloride from sylvinite ore. An increase in temperature will have a greater effect on the solubility of potassium chloride than sodium chloride. Consequently, an increase in temperature favors an increase in the yield of flotation processes. Figure 2 shows curves characterizing the effect of the temperature of extracting potassium chloride from the mixture.

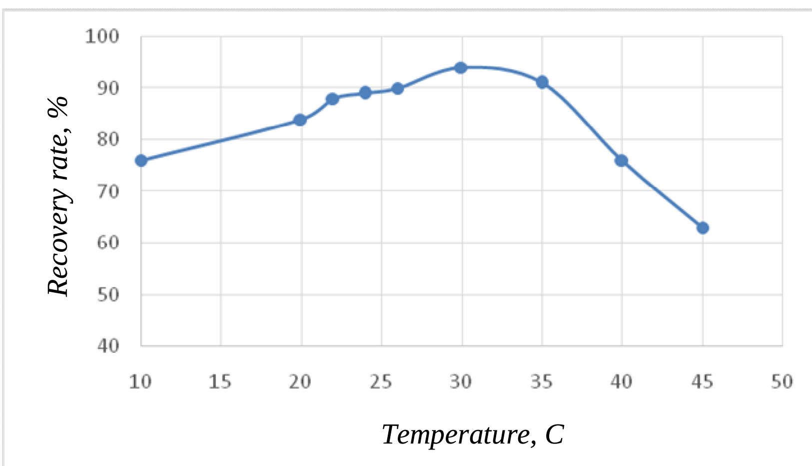

Fig.2. Change in the degree of extraction of KCl from P3 with temperature

As the curves of the diagram show, as a result of an increase in the temperature of the solution from 20 to 30 °C, an increase in the extraction of KCl from P3 by 20% is observed. A decrease in this indicator with a further increase in temperature is associated with the structural features of surfactants and an increase in the fraction of their desorption at the interface.

On the basis of the experimental data obtained, sylvinite flotation was carried out in the plant laboratory of Dekhkanabad Potash Plant JSC on a pilot flotation unit. The experiments were carried out in parallel with an imported sample of liquid paraffin. The ore supplied to beneficiation contains a significant amount of water-insoluble impurities, which during processing turn into a fine (fine), practically non-filtering material - clay sludge. The content of clay

ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 7, Issue 11 , November 2020

materials in the ore ranges from 3 to 12%. The useful component sylvite (KCl) in its content in the ore also varies considerably and accounts for 18-35% of the total mass of the ore. The rest of the ore is represented by sodium chloride (NaCl), which does not cause any particular complications during the flotation of sylvite (KCl). Salt flotation is carried out in a solution saturated with the components of the rock (potassium and sodium). The mortar is characterized by strong foaming, high viscosity and surface tension. In this case, the floatability of salts depends on changes in the composition of the solution, selective coagulation of a number of salts, intensive micelle formation and salting out of reagents. This leads to a weakening or even complete loss of the collecting and foaming properties of a number of reagents used in the beneficiation of other minerals. TheresultsareshowninTable3below.

Table 3
Comparative results of laboratory studies of sylvinite flotation in the presence of imported and obtained liquid paraffins

№	Parameters	Imported ZhP		Received ZhP		Norm according to NTD
		Weight	KCl, %	Macca	KCl, %	
1.	Raw materials	408	31,53	408	31,53	
2.	The resulting product (potassium chloride)	130,5	89	132,31	89.2	≥ 83
3.	Tail	277,5	3,5	275,69	3,5	≤ 3,8

The test results show that the obtained liquid paraffin from the waste of the gas-chemical complex of the Uz-KorGasChemical JV LLC of spent hexane at the Dekhkanabad Potash Plant JSC is responsible for all parameters obtained on the basis of imported raw materials. It has been established that the reagent mode is very important determining the success of the flotation process, which means the range of reagents used, their consumption, the order of feeding into the process and the contact time. For effective control of the flotation process, it is necessary to investigate the factors affecting the consumption of reagents, and for this, in turn, to study the principles of constructing a technological scheme of enrichment, to identify control goals, disturbances and control actions. In addition, without stabilizing some parameters or bringing them to the optimal mode, measures to control the reagent mode will be ineffective due to the imposition of many disturbing factors.

A process flow diagram has been developed for the production of liquid paraffin based on spent hexane, which consists of the following main stages:

Preparation of raw materials. All raw materials necessary for the preparation of liquid paraffin, after visual inspection for compliance with the requirements of ND for marking and packaging, are weighed on a scale in quantities required according to the recipe.

Composite preparation. The product is prepared by mechanical mixing of the components in a batcher with a stirrer. The mixing of the components is carried out at the temperature of the production room and atmospheric pressure.

With the stirrer turned on, the following is loaded into the reactor: the calculated amount of spent hexane or cyclohexane. Stirred by heating with water vapor through the jacket of the reactor, distillation is carried out to the temperature of the residual substance in the reactor of 140 °C, for 4 hours. After settling and decantation, pumping into another reactor with a stirrer and filtered through a Nutsch filter. The end of the process is determined by the temperature, observing the thermometer.

IV. CONCLUSION

The analysis of literature devoted to the study of flotation processes is carried out. It has been found that the use of fatty amines and a mixture of hydrocarbons as a collector has a positive effect on the selectivity of flotation concentration of sylvinite ores. The quantitative and qualitative compositions of imported and obtained liquid paraffin were determined and compared by chromatographic analysis. It has been proven that the use of liquid paraffin obtained

ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 7, Issue 11 , November 2020

from liquid secondary raw materials, flotation has better performance, in addition, it allows to increase the extraction of KCl into concentrate and reduce the consumption of expensive amine 6g/1t of ore, thereby reducing the cost.

REFERENCES

[1] Bragin V.I. Flotation beneficiation methods: lecture notes for students of specialty 130405.65 “Mineral processing”. - Krasnoyarsk: IPK SFU, 2010. –123 p.
[2] Abramov A.A. Theoretical foundations of increasing the selectivity of the action of modifying reagents during flotation.2013. No. 7. P.23-29.
[3] Matveeva T.N., Ivanova T.A., Gromova N.K. Sorption and flotation properties of reagents of plant origin for selective flotation of sulfide minerals containing noble metals // Non-ferrous metals. 2012. No. 12. P.16-20.
[4] Abramov A.A. Theoretical foundations for creating innovative technologies. Part 1. Theoretical foundations of modern flotation // Non-ferrous metals. 2013. No. 2. P.41-45.
[5] ShubovL. Ya., Ivankov S.I., Shcheglova N.K. Flotation reagents in mineral processing – M.: Nedra, 1990. P.5-26.
[6] Oliferovich D.S., Shilin L.Yu., Batyukov S.V., Prigara V.N. Analysis and accounting of factors affecting the technological process of flotation of potash ores. BSUIR reports. 2009 No. 2 (40) P. 59-66.
[7] Pat. 2424855. Russia. The process of flotation using an organometallic complex as an activator. Vilhonen D.M.-L., Lubbe L. Appl. 23.01.2007. Publ. 27.07.2011. Bul. No. 21.
[8] Pat. No. 925793.Republic of Belarus. Collector for flotation of potassium-containing ores. E. I. Shcherbina, E. I. Grushova, A. E. Polyakov, N. I. Vorobyov, I. B. Makhlyankin, 3. C. Podlesnaya, V. G. Zelenkina, A. S. Malakhov, M. A. Gamilov, V. T. Bohr. Appl. 28.01.80; Publ. 05.07.82. Bulletin 17.