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APROTIC REAGENTS USED IN FLOTATION PROCESSES

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Abstract: Chromatographic analysis determined and compared the qualitative and quantitative compositions of imported and obtained liquid paraffin. It is proved that liquid paraffin use obtained from secondary raw materials, flotation has the best performance, besides allows to increase the KCl extraction into concentrate and reduce the expensive amine consumption, thereby reduce the cost price.
Keywords: flotation, flotation reagents, collector, recovery, selvinite ore, potassium chloride, liquid secondary raw materials, chromatographic analysis, apolar reganet, liquid paraffins, saturated hydrocarbons, C_{14} - C_{18} composition

Introduction

One of the important ore processing stages in the chemical industry is metal compounds concentration and ores beneficiation. The main chemicals used in this technological process are flotation reagents that 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.

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

Such a wide application 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 concentrates quality, as well as enriches finely disseminated, poor and those ores that cannot be processed by other processes [3 - 5].

In the sylvite flotation, cationic reagents are used as a collector, in particular, primary aliphatic amines, which interact with the useful component KCl minerals surface, 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 when grinding ore and actively adsorb amine molecules. The greatest interest from the influence point view on the potassium salts separation process is provided by a finely dispersed 0.001 mm clay fraction. Specific clay sludge surface, available to a cationic collector - amine, is 350-370 m^2/t. Salt sludge is represented by finely dispersed sylvite and halite with 60 microns size is formed during minerals over-grinding in the mining, transportation, ore preparation process for flotation and directly in flotation chambers [6].

The main flotation reagents components used in industry are C_{11}- C_{22} paraffins. [7 - 9].

The authors of [10] propose to use kerosene as a fatty amines collector and a hydrocarbons mixture. As a rule, kerosene contains up to 30% aromatic hydrocarbons, which negatively affect the flotation beneficiation selectivity of sylvinite ores. In addition, naphthalene hydrocarbons are carcinogenic substances; therefore their involvement in the process is undesirable.

A liquid paraffin obtaining method by processing the oil fraction with crystalline carbamides is proposed in a solvent presence, followed by formed carbamide complex separation with paraffin from the dewaxed product, stepwise washing and complex decomposition with liquid paraffin release, растворит, β,β'-dichlorodiethyl ether is used as a solvent, with methyl ethyl or methyl isobutyl ketone in 1:1 mass ratio, treatment with carbomide is carried out by adding 180 - 260

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mass% solvent to the raw material and the complex is washed with the specified solvent in the first stage and methylethyl-or methyl isobutyl ketone in the second stage[11].

The literature [12] investigated the emulsions obtaining possibility based on petroleum paraffin products base with their flotation subsequent comparison activity in the sylvinite flotation process with similar industrial apolar reagents indicators. Tests were carried out with hydrotreated vacuum gas oil (OJSC «MNPZ»); residual oil product of mild hydrocracking (OJSC«NAFTAN»); residual oil from hydrocracking (OJSC «NAFTAN»); oil edema (OJSC «ZGB»); slack (OJSC « NAFTAN»).

The authors of the studies [13-15] investigate the kerosene, gas oil, etc. use as collector reagents. Depending on the feedstock and processing conditions, they have a varied composition and represent a complex organic substances mixture. Their composition variety makes it difficult to study the kerosene and gas oil effect during coal flotation. At the same time, it is difficult to establish which constituent kerosene and gas oil parts are the most flotation active and what should be guided by when selecting reagents for these conditions.

The distillation gas oil and crude oil products use is proposed as aprotic solvents. To study the oil collecting properties reagents, an apparatus for non-froth flotation was used. In the tests, the reagents were preliminarily dispersed with food in a non-foam flotation apparatus. This made it possible to exclude the emulsion stratification during the flotation process itself. Also, the initial products collecting properties of various compositions and technologies for obtaining their fractions were studied. [16-21].

Sources [22-25] describe some aprotic reagents, such as clarified, tractor and oxidized kerosene, household stove fuel, apolar aromatized reagents AAP-1 and AAP-2, activated flotation reagent AF-2 and thermogasoil.

Clarified kerosene was widely used in the early flotation days and is still used in a small number of factories. The limiting compounds predominance in kerosene gives it a more selective effect in coal flotation in comparison with aromatized kerosene, but the flotation rate is somewhat lower. The lighting kerosene advantages are the specific smell absence, ease of use, calm process management, low cost. Kerosene has no foaming ability, and at high consumption it exhibits foaming properties. For coal flotation, lighting kerosene is used only in conjunction with a heteropolar reagent. Its consumption is within 1 - 3 kg/t.

Tractor kerosene by chemical composition is a mixture of saturated, unsaturated and aromatic hydrocarbons. The increased content (17%) in it in comparison with the lighting kerosene of unsaturated and aromatic hydrocarbons containing 16 - 24 carbon atoms in the molecule, gives tractor kerosene increased flotation ability. Its consumption in the middle stage coals flotation of metomorphism is 1 - 1.5 kg/t, in the low-metamorphosed flotation - 1.5 - 2.5 kg/t.

Oxidized kerosene is an oily homogeneous liquid of light yellow color. It is obtained by oxidizing kerosene containing saturated hydrocarbons at 140 - 150°C temperature by blowing air for 2-6 hours in a catalyst - manganese naphthenate presence. The oxidized kerosene contains carboxylic and naphthenic acids, oxo acids, organic substances containing hydroxyl, ether and other groups. As a result, it has some foaming properties and is a more active reagent than lighting kerosene. In practice, it has not received proper implementation due to the need to organize specialized production to obtain it.

Apolar flavored reagent AAP-1 is a specialized substance for flotation, obtained from oil condensate. AAP-1 is a dark brown liquid with a pungent unpleasant odor. Density at 20°C not less than 0.85 g/cm^3, kinematic viscosity not less than 2·10-6 m^2/s, refractive index not less than 1.46. The reagent is insoluble in water, but readily soluble in alcohol, ether, benzene. AAP-1 belongs to the group of rather effective reagents and is used in some Donbass factories. Its widespread use is prevented by an unpleasant odor, supplied by AAP-1 in undiluted form (in an aerosol form) to the pulp preparation apparatus. Consumption is 1 - 3 kg/t.

Apolar flavored reagent AAP-2 - a specialized reagent for flotation - is a fraction with 195 – 300°C boiling point, obtained by catalytic cracking of vacuum distillates of oils mixture. In appearance, it is a homogeneous brown liquid with a specific odor. Density at 20°C is 0.900 g/cm^3, kinematic viscosity not less than $2 \cdot 10^{-6}$ m^2/s, refractive index not less than 1.5000, flash point in an open crucible 265°C, vapors ignition limits +40- 80°C, vapor pressure - at a temperature 20°C 66,6 Pa, pour point no higher than - 30 °C.

The chemical composition of AAP-2 is very complex,%: aromatic hydrocarbons 75; paraffin-naphthenic 21; unsaturated 1. The AAP-2 advantages are the relative composition constancy, the action effectiveness during flotation, lack of deficiency, low cost. Negative properties are considered to be an increased capacity for foaming and an unpleasant odor. Its consumption is 1 - 3 kg/t.

Activated flotation reagent AF-2 - a thermal reforming product of gas gasoline, is a homogeneous oily liquid. The pour point is not higher than -50°C. AF-2 does not dissolve in water, but we will easily dissolve in alcohol, ether, benzene. The chemical composition of the reagent is a mixture of hydrocarbons,%: paraffinic 60.8, naphthenic 23.6, unsaturated and aromatic 15.6. MPC of reagent vapors in the working rooms' air is driven by consciousness loss. It is supplied to the pulp preparation apparatus together with a heteropolar reagent.

Thermogasoil is a mixture of kerosene-gasoil fractions. It is a dark brown liquid with a specific odor. The density at 20°C is not less than 0.810 g/cm^3, the kinematic viscosity at 20°C is 5-610-6 m^2/s, the refractive index is not less than 1.4500. The reagent has a complex chemical composition, including hydrocarbons,%: aromatic 41, paraffin-naphthenic 30, unsaturated 29. The iodine number is 35 g/100g MPC 10 mg/m^3 composition variability; high pour point and unpleasant odor do not contribute to the widespread use of this aprotic reagent.

JSC "Dekhkanabad Potash Plant" in the flotation enrichment process of potassium chloride from natural sylvinite, liquid paraffins are used as part of the collective flotation reagents mixture. Liquid paraffins are mainly saturated hydrocarbons with a C_5 - C_{18} carbon number of normal structure.

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

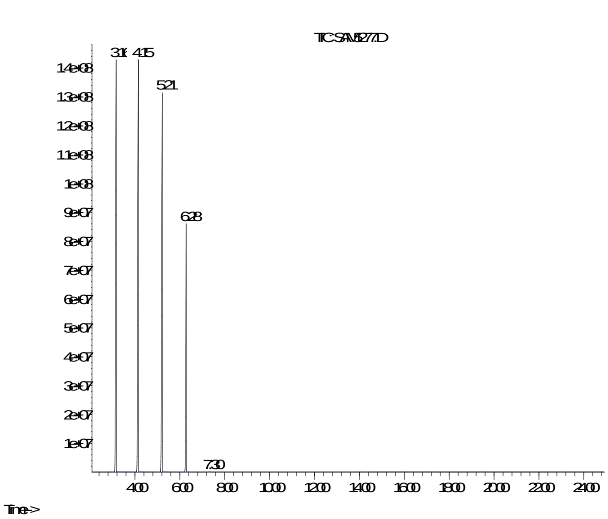

Fig.1. Chromatogram of imported liquid paraffin of flotation concentration of potassium chloride from natural sylvinite as part of a collective mixture of flotation reagents:
1 - tetradecane; 2- pentadecane; 3- n-cetan; 4 - n-heptadecane; 5 - n-octadecane

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

In the gas chemical complex Ustyurt (Republic of Karakalpakstan) polyethylene and polypropylene are produced by polymerization reaction in a Ziegler-Natta catalyst presence 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 the monomers oligomer used, which bulk are paraffins from C_6 to C_{20}, the qualitative and quantitative composition of which was

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determined by the chromatographic method (Fig. 2).

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

Separation of a liquid mixture 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 the thermometer reading 135°C under vacuum - 650 mm Hg. From one liter of spent hexane sample, 0.450 liters of C_{12}-C_{20} saturated hydrocarbons fraction of normal structure were obtained. In this case, the obtained sample density of liquid paraffin at 20°C is 745 kg/m^3.

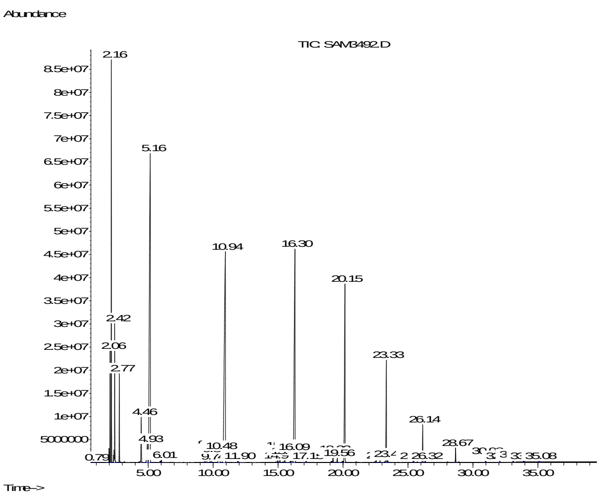

Fig.2. Chromatogram of liquid secondary raw materials of polyethylene production and polypropylene by polymerization reaction in a Ziegler-Natta catalyst presence in hexane solution

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

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

For salt solutions preparation, dried to constant weight at 120°C temperature, quantitatively transferred into volumetric flasks with 1000 cm^3 capacity, dissolved in distilled water, bringing the solution to the mark. Experimental flotation processes were carried out on an FML 240 flotation machine. The aliphatic amine consumption for all samples was 10 mg/kg of the salt mixture. The research results are shown in Table 1.

Table 1

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

Indicator		Model solutions		
		P1	P2	P3
Output, %:	concentrate	41,2	30,5	19,6
	stern	58,8	69,5	80,4
Mass fraction of KCl, %:	concentrate	97,2	91,3	90,8
	stern	17,0	7,8	4,3
Extraction of KCl, %:	concentrate	80	83,6	89
	stern	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 apolite reagent and amine for the adsorption of its molecule on the entire surface of the floated material. Under the apolic reagent influence, P3 floats more fully, which indicates that the amount of the collector is sufficient. Consequently, for the sylvinite ores flotation with main required component mass content of more than 20%, the required amount of apolic reagent is more than 10 g per ton of ore (this amount is calculated using the CMC value).

It is generally known that the

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temperature of the solution will be a key factor in the potassium chloride flotation from sylvinite ore. An increase in temperature will have a greater effect on the potassium chloride solubility than sodium chloride. Consequently, an increase in temperature favors an increase in the flotation processes yield. Figure 3 illustrates the curves characterizing the extracting potassium chloride temperature effect from the mixture.

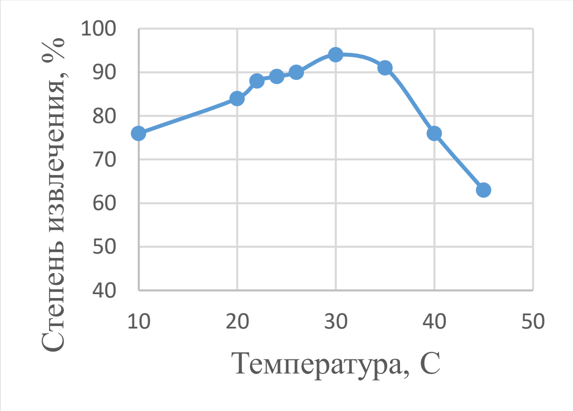

Fig.3. Change in KCl extraction degree from P3 with temperature

As the curves diagrams show, As a result of an increase in the solution temperature from 20 to 30ºC, an increase in KCl extraction from P3 by 20% is observed. A decrease in this indicator with a further increase in temperature is associated with the structural features of surface-active substances and an increase in their desorption proportion at the interface.

On the basis of the obtained experimental data, the 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 liquid paraffin sample. The ore supplied for concentration contains a significant amount of water-insoluble impurities, which in the processing course turn into a fine (fine), practically non-filtering material - clay sludge. The clay materials content in 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 ore mass. The ore rest is represented by sodium chloride (NaCl), which does not cause any particular complications during the sylvite flotation (KCl). Salts flotation is carried out in a solution saturated with the rock components (potassium and sodium). The mortar is characterized by strong foaming, high viscosity and surface tension. In this case, the salts floatability depends on changes in the solution composition, selective coagulation of a number of salts, intense 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 other minerals beneficiation. The results obtained are shown in Table 2 below.

Table 2

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

№	Options	Imported ЖП		Received ЖП		Norm for НТД
		Mass	KCl, %	Mass	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.	Stern	277,5	3,5	275,69	3,5	≤ 3,8

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

Conclusion.

The literature analysis devoted to the flotation processes study is carried out. It has been established that the use of fatty amines and hydrocarbons mixture as a collector has a positive effect on the flotation concentration selectivity of sylvinite ores. Chromatographic analysis determined and compared the qualitative and quantitative compositions of imported and obtained liquid paraffin. It has been proven that the use of liquid paraffin obtained from liquid secondary raw materials flotation passes the best performance, moreover, allows increasing the KCl recovery into concentrate and reducing expensive amine consumption 6g/1t of ore, thereby reduce the cost.

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