

Search for new amide-class collecting agents and study of their flotation activity in the process of copper-nickel ore flotation

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The issue of expanding the range of effective flotation reagents remains relevant due to the deteriorating ore base and the involvement in processing of refractory ores with complex mineral composition. In recent years, complexing reagents capable of forming strong complexes with transition metal ions have been of increasing interest. At that, insertion of an additional functional group into the structure of the reagent molecule can provide a more complete extraction of the useful component. The purpose of this work is to evaluate the efficiency of using nitrogen-containing mono-derivatives of dicarboxylic acids from the amide class as collecting agents for flotation of copper-nickel ores. The synthesis of monoalkylamides of succinic and phthalic acids with different hydrocarbon radicals as well as their complexes with copper and nickel was carried out. The formation of complexes was confirmed by extraction method, chemical analysis and Raman spectroscopy. It was noted that during the extraction of copper and nickel by the reagents under consideration, an effective process of complexation in the organic phase takes place. Their interaction with the surface of copper- and nickel-containing minerals was evaluated by the method of frothless flotation in comparison with the traditional collector. It is shown that monoalkylamides are more efficient towards nickel-containing minerals. Flotation tests on a sample of finely disseminated copper-nickel ore with copper content of 0.182% and nickel content of 0.479% have shown that succinic acid monoalkylamide as an independent collector with its own foaming properties and in a mixture with butyl xanthate shows greater selectivity. The best results were obtained when it was used in a mixture with butyl xanthate at a ratio of 70 : 30.

Key words: flotation, non-ferrous metals, complexing reagents, sulfhydryl collectors, nitrogen-containing dicarboxylic acid derivatives, copper-nickel ores

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Introduction

Recently, enrichment plants have been involved in the processing of finely disseminated and refractory ores characterized by complex mineral composition. In conditions of increasingly deteriorating ore base it is an urgent task to broaden the nomenclature of applicable flotation reagents. In the process of flotation of sulfide ores, sulfhydryl collecting reagents are generally used. These are different variations of xanthates (Kx), salts of xanthogenic acid [1] and aerofloats (Af), salts of dithiophosphoric acid [2, 3]. However, these reagents, in particular xanthates, have a number of disadvantages: instability of aqueous solutions, formation of toxic products as a result of decomposition and relatively low selectivity of action [4].

Reagents of complexing action have recently attracted increased interest. It is known that the nitrogen atom, which is a part of organic compounds, is a potential reaction-active center and under certain conditions forms complex compounds with metal atoms. Therefore, such compounds have been widely used in the field of analytical chemistry for the detection and binding of metal ions in solutions, as well as in extraction and flotation processes [5–7].

Numerous studies have shown that the insertion of a nitrogen atom into the molecule of a sulfur-containing collecting agent as an additional donor center provides the possibility of formation of chelate compounds on the surface of non-ferrous metal minerals. Complexation with involvement of N – Pb – S and N – Zn – S bonds determines high selectivity of 5-(butylthio)-1,3,4-thiadazol-2-thiol at flotation separation of sphalerite and galena from pyrite [8]. The presence of S, N and O atoms in the molecule of hydroxyiminodithiocarbonate determines its high efficiency in flotation of chalcopyrite [9]. High sorption and flotation properties of dithiopyrylmethane with respect to a number of sulfide minerals have been shown by a complex of physicochemical studies [10].

Reagents with nitrogen-containing groups have also been proposed as collecting agents for sulfide ores. Carboxylic acid hydrazides and alkylhydroxamic acids have been tested as collecting agents for copper-nickel, copper-zinc and copper-molybdenum ores [11–13].

A promising direction in the search for new reagents is the insertion of an additional functional grouping, as this can provide some extra specific interaction of the reagent with the mineral surface. The use of bifunctional compounds such as amino acids with amino and carboxyl

functional groups in the extraction of copper from poor ores by bioleaching is well known [14].

Our earlier studies demonstrated the possibility of using mono-derivatives of alkenylsuccinic acids in flotation of sulfide copper-nickel ores. It is shown that monohydrazide of 2-ethylhexenylsuccinic acid has higher efficiency of action in relation to nickel-containing minerals, and its use in combination with xanthate provides an increase in nickel recovery in the rough concentrate and reduction of its losses with flotation tailings [15].

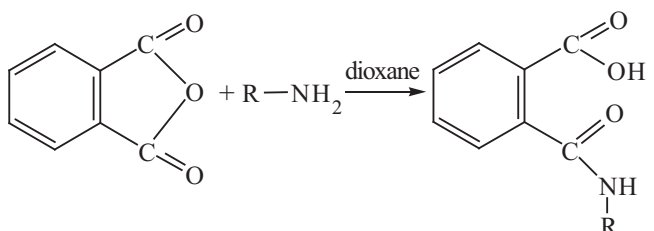
The results obtained gave reason to consider monoamide derivatives of dicarboxylic acids as collecting agents. Perspective of such class of compounds is also confirmed by the data on high extraction ability of amide derivatives of carboxylic acids in relation to platinum metals [16] and *f*-elements [17].

In addition, the synthesis of dicarboxylic acid monoalkylamides based on the opening of the anhydride ring under the effect of the corresponding alkylamide is simple and does not require complicated apparatus design.

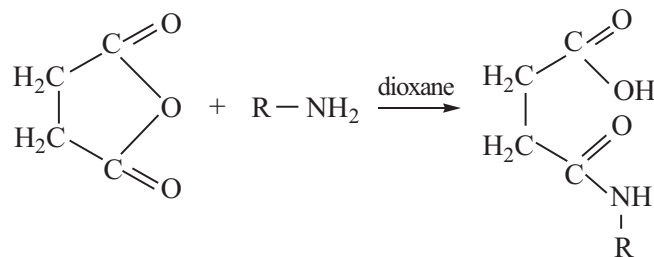
The present work presents the results of the study of complexing, surface and collecting properties of monoalkylamides of succinic and phthalic acids in relation to copper- and nickel-containing minerals.

Materials and methods

Bifunctional compounds representing nitrogen-containing mono-derivatives of dicarboxylic acids: phthalic acid monododecylamide (C12-PAA) (I) and succinic acid monododecylamide (C10-SAA) (II) were synthesized by interaction of equimolar amounts of the corresponding dicarboxylic acid anhydride and alkylamine. The synthesis was carried out at room temperature for 1 h according to the following reactions:



where $\text{R} = \text{C}_{12}\text{H}_{25}$



where $\text{R} = \text{C}_{10}\text{H}_{21}$

The obtained compounds were analyzed and identified on the basis of acid-base titration and melting point determination. For C10-SAA the mass fraction of carboxyl groups was $w_{\text{COOH}} = 18.0\%$ ($w_{\text{COOH}}^{\text{theor}} =$

Table 1
Chemical analysis of the samples

Ore sample	Content, %				Ni/Cu ratio
	Cu	Ni	Fe	S	
CN-1	1.4	5.81	52.64	34.62	1/4.15
CN-2	16.95	3.66	41.55	31.52	4.63/1

$= 17.5\%$), $t_m = 87^\circ\text{C}$; for C12-PAA – $w_{\text{COOH}} = 13.7\%$ ($w_{\text{COOH}}^{\text{theor}} = 13.5\%$), $t_m = 88^\circ\text{C}$.

The interaction of synthesized reagents with the surface of sulfide minerals was evaluated by the method of frothless flotation in a Hallimond tube on samples of copper-nickel ore rich in pentlandite and pyrrhotite (CN-1) and chalcopyrite (CN-2) with a grain size of minus $0.09 + 0.063$ mm (Table 1).

A 0.5 g ore suspension was agitated with pH regulator (0.1% NaOH solution) for 1 min, then 2 min with collecting agent. The flotation time was 3 min at $20 \pm 1^\circ\text{C}$. The rate of air supply was $5.3 \text{ cm}^3/\text{min}$. Flotation with the studied compounds was carried out at $\text{pH} \approx 10$. The efficiency of flotation was estimated by the recovery of nickel and copper in the froth product.

A sample of copper-nickel ore from the Pechenga ore field was used for flotation studies. Nickel and copper content in the sample amounted to 0.479 and 0.182%, respectively. The mineralogical and petrographic analysis showed that the sample is represented by partially serpentinized olivinites and metaperidotites with a small proportion of fine crystalline phyllites.

In olivinites serpentine together with magnetite develops in the intergranular space and in the form of rims around olivine grains. In metaperidotites (rocks of olivine-pyroxene composition) the primary minerals are replaced by serpentine, magnetite, and to a lesser extent chlorite and talc. Minor carbonatization occurs in certain places. Phyllites are schistose rocks consisting of actinolite-tremolite type amphiboles, quartz, feldspars, serpentine and chlorite. In general, the degree of serpentinization of the sample rocks can be assessed as moderate (about 50%).

The main mass of sulfides in the studied sample is represented by pyrrhotite, pentlandite and chalcopyrite of syngenetic and epigenetic origin. Pyrite is present in single grains. The total sulfide content in the sample is 3%. Other ore minerals are represented by magnetite (12%) and rare grains of ilmenite.

A syngenetic phenocryst of sideronite-type sulfides is associated with unaltered and weakly serpentinized olivine clasts, which is characterized by the location of ore minerals between olivine grains (Fig. 1, a). Rounded dark gray grains are olivine, gray areas between olivine grains are serpentine, and light gray and bright areas are sulfide-magnetite phenocrysts. Such phenocrysts are closely associated with magnetite, and the composition of phenocrysts is often pentlandite-pyrrhotite, with the predominance of the latter (Fig. 1, b). Less frequently occur

phenocrysts with predominance of pentlandite. The pentlandite-chalcopyrite-pyrrhotite and chalcopyrite-pyrrhotite composition of phenocrysts is also characteristic of the phenocrysts. The grain size of individual sulfides in sideronite phenocrysts is most often 0.10–0.30 mm, significantly decreasing when they are replaced by magnetite or fine-needle serpentine (Fig. 1 *c, d*).

Epigenetic phenocrysts are associated with areas of deep serpentinization of olivinites, with metaperidotites and fine crystalline schistose phyllites. The size of phenocrysts varies from 0.05–0.10 mm to 1.0–1.5 mm and usually has irregular outlines and monomineral composition (Fig. 2, *a, b*). This sample is dominated by pyrrhotite with flame-like inclusions of pentlandite (Fig. 2, *b*).

For flotation, the ore was crushed to a size of –2.0 mm, then the suspension of 1.2 kg was ground in a laboratory ball mill with a volume of 7 liters to a size of grade –0.071 mm 80%. The experiments were carried out in an open cycle on tap water with the rougher and scavenger flotation operations according to the scheme presented in Fig. 3. The necessary pH value of the medium was created by adding Na₂CO₃ soda ash. For activation of sulfide minerals copper sulfate CuSO₄ was used. The investigated reagents were compared with the traditional reagent regime, providing the use of Kx (butyl potassium xanthate) and Af (butyl aerofloat) as collecting agents.

Results and discussion

The formation of complex compounds of copper and nickel with monoamides of phthalic and succinic acids was evaluated by the extraction method. Extraction was carried out with reagent dissolved in chloroform from aqueous solutions of copper sulfate and nickel chloride in the acidic and alkaline medium: pH = 4–5.5 and pH = 9.3–9.5. The extraction was performed under constant stirring for 1 hour. The transition of metal ions into the organic phase was qualitatively recorded by the color change of the aqueous and organic layer and confirmed by the results of chemical analysis.

At the natural pH value of aqueous solutions of metal salts metal ions did not pass into the organic phase, while at pH values of the aqueous phase ~9.5 the transition of

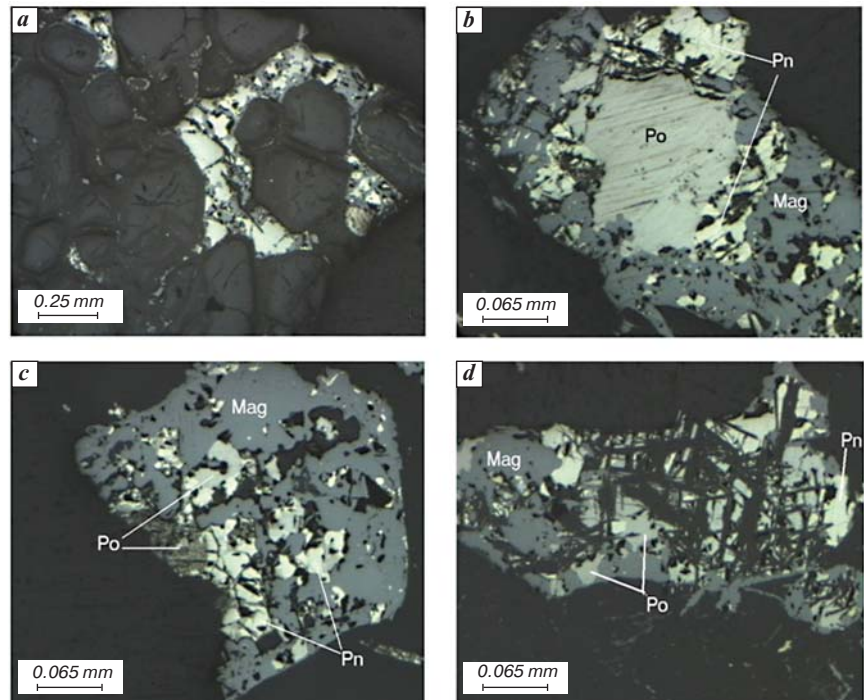


Fig. 1. Syngenetic phenocrysts of sideronite-type sulfides in olivinites (*a*); syngenetic phenocrysts of pentlandite-pyrrhotite-magnetite composition (*b*); intensive replacement of syngenetic sulfide phenocrysts by magnetite (*c*) and fine-needle serpentine (*d*): Po – pyrrhotite; Pn – pentlandite; Mag – magnetite

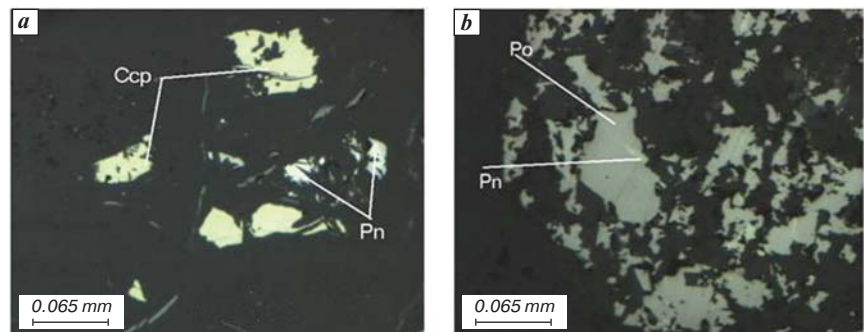


Fig. 2. Epigenetic phenocrysts of sulfides: monomineral composition of phenocrysts and their replacement by needle serpentine (*a*); flame-shaped pentlandite phenocrysts in pyrrhotite (*b*): Po – pyrrhotite; Pn – pentlandite; Ccp – chalcopyrite

metal ions into the organic phase was observed almost immediately. Table 2 shows the calculation of the extraction degree of copper and nickel.

In order to confirm complexation, copper and nickel complexes with unsubstituted monoamides of phthalic and succinic acids were synthesized and identified by Raman spectroscopy using an EnSpectr R532 Raman analyzer. Aqueous solutions of metal sulfate and sodium salt of monoamide were mixed in 1 : 2 ratio. The precipitate was separated from the solution by centrifugation, washed with water, then dried in air [18].

Stretching vibrations of the characteristic absorption bands of NH– и C=O bonds in the amide group appear in the region of 3320 and 1647 cm⁻¹ in the case of C10-SAA and 3305 and 1698 cm⁻¹ in the case of C12-PAA,

respectively. Stretching vibrations of C–H bonds in the benzene ring appear at 3065 cm^{-1} (Fig. 4).

The absence of peaks at 3320 and 3305 cm^{-1} in the spectra of copper and nickel complexes, as well as the shift of the absorption bands of the C=O bond by $5\text{--}10\text{ cm}^{-1}$ in the low-frequency region in the case of complexes with

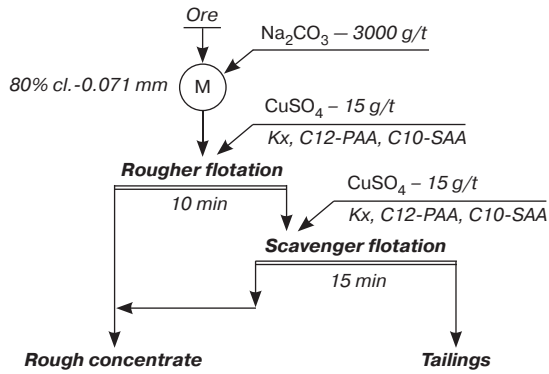


Fig. 3. Scheme of laboratory flotation tests

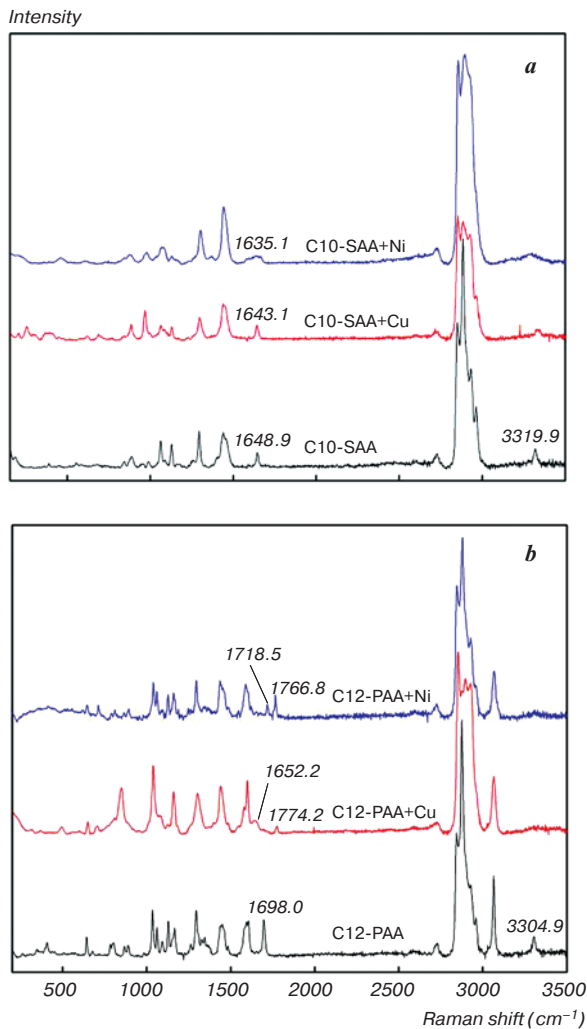


Fig. 4. Raman spectra: C10-SAA and its Cu- and Ni-complexes (a); C12-PAA and its Cu- and Ni-complexes (b)

C10-SAA (Fig. 4, a) and in the high-frequency region in the case with C12-PAA (Fig. 4, b) suggests the formation of a bond between metal cations and anions of functional groups.

Comparative evaluation of the interaction of monoalkylamides with the surface of nickel- and copper-containing minerals and their hydrophobizing ability was carried out by the method of frothless flotation. This method implies conversion into a “foam” product of mineral particles, the surface of which is hydrophobized as a result of preliminary interaction with the reagent in the solution. The unflotated material is a “chamber” product. The use of ore with a higher content of copper- or nickel-bearing minerals allows to conduct the experiment taking into account the mutual influence of minerals on each other. The effect of the investigated reagents was compared with the traditional collecting agent Kx in terms of copper and nickel recovery into the “foam product”. The obtained concentration dependences are presented in Fig. 5.

Table 2
Extraction parameters of Cu^{2+} and Ni^{2+} ions by C12-PAA reagent

Metal	pH	$\text{C}(\text{Me}^{n+})_{\text{init}}$, mg/l	$\text{C}(\text{Me}^{n+})_{\text{res}}$, mg/l	Extraction, %
Cu^{2+}	4.35	589.8	588.8	0.17
	9.30		77	86.9
Ni^{2+}	5.55	545.4	544.0	0.26
	9.45		178.7	67.2

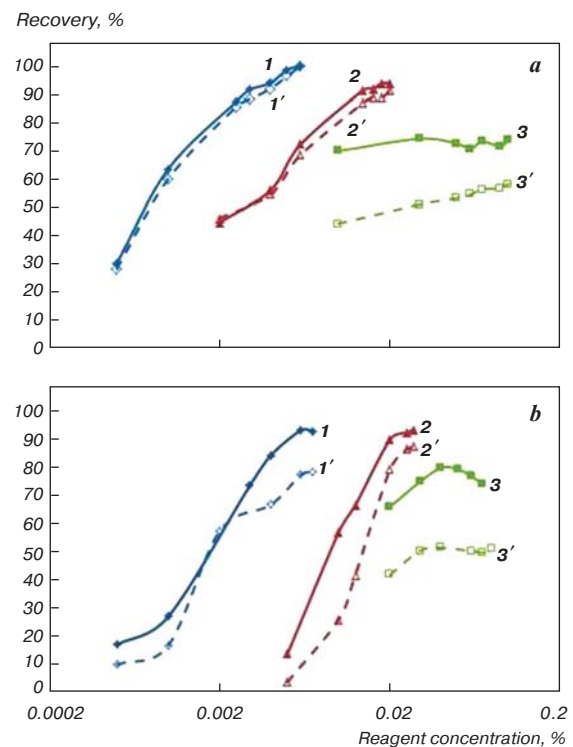


Fig. 5. Recovery of copper (I–3) and nickel (I’–3’) into the foam product during the flotation of CN-1 (a) and CN-2 (b) ores with reagents: I – C12-PAA, 2 – C10-SAA, 3 – Kx

The obtained results show that reagents with amide group — monoamides of dicarboxylic acids in comparison with xanthate show a higher tendency to interact with nickel-containing minerals. In flotation of the ore of sample CN-1 for both tested reagents, a close degree of nickel and copper recovery in the froth product is observed, while xanthate shows higher selectivity towards copper (Fig. 5, a).

For ore with high chalcopyrite content (CN-2), copper recovery by monoalkylamide action is expectedly slightly higher than nickel recovery (Fig. 5, b). However, this difference appears to a lesser extent for monoalkylamides in comparison to xanthate.

Reagents C10-SAA and C12-PAA were tested both as independent collectors and as additives to the traditional

Table 3
Flotation results when using monoalkylamides in combination with Kx

Product	Yield, %	Content, %		Recovery, %		Total reagent concentration, g/t
		Ni	Cu	Ni	Cu	
Base test						
Froth (rough)	17.19	1.92	0.785	65.94	71.62	Kx – 135 Af – 95
Froth (scavenger)	13.41	0.438	0.14	11.74	11.74	
Rough concentrate	30.6	1.27	0.50	77.68	81.58	
Tailings	69.4	0.161	0.05	22.32	18.42	
Initial	100.00	0.501	0.188	100.00	100.00	
Kx : C12-PAA = 70 : 30						
Froth (rough)	7.61	2.29	1.04	35.72	45.93	Kx – 161 C12-PAA – 69
Froth (scavenger)	11.59	1.20	0.315	28.51	21.20	
Rough concentrate	19.20	1.70	0.60	64.23	67.13	
Tailings	80.80	0.216	0.070	35.77	32.81	
Initial	100.00	0.488	0.172	100.00	100.00	
Kx : C12-PAA = 50 : 50						
Froth (rough)	22.95	1.43	0.598	65.72	69.98	Kx – 115 C12-PAA – 115
Froth (scavenger)	8.86	0.454	0.149	8.06	6.73	
Rough concentrate	31.81	1.16	0.473	73.78	76.71	
Tailings	68.19	0.192	0.067	26.22	23.29	
Initial	100.00	0.499	0.196	100.00	100.00	
Kx : C12-PAA = 30 : 70						
Froth (rough)	24.82	1.38	0.500	67.84	71.33	Kx – 69 C12-PAA – 161
Froth (scavenger)	8.42	0.51	0.140	8.50	6.78	
Rough concentrate	33.24	1.16	0.400	76.34	78.10	
Tailings	66.76	0.179	0.057	23.66	21.89	
Initial	100.00	0.505	0.174	100.00	100.00	
Kx : C10-SAA = 70 : 30						
Froth (rough)	11.48	2.05	0.830	48.04	56.29	Kx – 161 C10-SAA – 69
Froth (scavenger)	12.55	0.860	0.215	22.03	15.89	
Rough concentrate	24.03	1.43	0.510	70.07	72.18	
Tailings	75.97	0.193	0.062	29.93	27.82	
Initial	100.00	0.490	0.169	100.00	100.00	
Kx : C10-SAA = 50 : 50						
Froth (rough)	21.61	1.48	0.560	63.43	69.26	Kx – 115 C10-SAA – 115
Froth (scavenger)	13.5	0.52	0.139	13.92	10.76	
Rough concentrate	35.11	1.11	0.380	77.35	80.02	
Tailings	64.89	0.176	0.054	22.65	19.98	
Initial	100.00	0.504	0.175	100.00	100.00	
Kx : C10-SAA = 30 : 70						
Froth (rough)	26.91	1.26	0.460	69.09	72.40	Kx – 69 C10-SAA – 161
Froth (scavenger)	12.64	0.430	0.120	11.08	8.89	
Rough concentrate	39.55	0.995	0.350	80.17	81.29	
Tailings	60.45	0.161	0.053	19.83	18.71	
Initial	100.00	0.491	0.171	100.00	100.00	

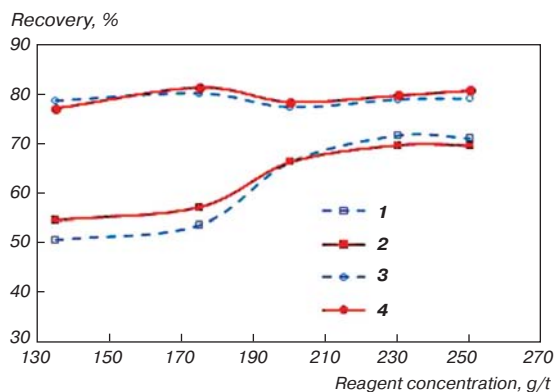


Fig. 6. Dependence of nickel and copper recovery in rough concentrate on reagent concentration:
 1 – C12-PAA, Ni recovery; 2 – C12-PAA, Cu recovery; 3 – C10-PAA, Ni recovery; 4 – C10-PAA, Cu recovery

reagent regime in flotation of copper-nickel sulfide ore. Due to good own foaming ability of the reagents, the experiments were carried out without the addition of foaming agent. Flotation activity of reagents as independent collecting agents was studied at different flow rates (**Fig. 6**).

The results show that when using phthalic acid monoalkylamide the extraction of non-ferrous metals reaches the maximum (~70%) at the consumption rate of 230 g/t. Application of succinic acid monoalkylamide allows to achieve recovery of about 80% already at a reagent consumption of 135 g/t. And if for C12-PAA copper recovery at lower flow rates exceeds nickel recovery, then in the case of C10-SAA at any flow rate the metals are extracted almost simultaneously, which may indicate the specificity of the reagents' action in relation to nickel minerals. When using a basic mixture of xanthate and aerofloat as a collecting agent at the rate of 135 g/t and 95 g/t, respectively, the difference in nickel (79%) and copper (83.4%) recovery in the rough concentrate for this type of ore is 4.5%.

It should be noted that, despite the sufficiently high recovery of nickel and copper, the use of monoalkylamides does not achieve the same content of non-ferrous metals in the flotation tailings as in the traditional mode. Comparable recovery is achieved, in particular, by increasing the yield of the froth product, which on average is 10% more than in the basic experiment. Thus, the minimum content in the tailings obtained using C12-PAA was 0.244% nickel and 0.100% copper. In experiments with application of C10-SAA the process proceeds more efficiently. The use of succinic acid monoalkylamide at the same flow rate with the collecting mixture in the basic experiment allows to obtain rough concentrate with sulfide recovery of about 80% and tailings with nickel and copper content of 0.170 and 0.058%, respectively.

The use of monoalkylamides in combination with xanthate is more effective. **Table 3** presents the results of flotation at different ratios of the tested reagents and xanthate. The total consumption of collectors in all experiments

remained constant and corresponded to the consumption in the baseline experiment – 230 g/t.

The obtained results showed that the increase in the proportion of monoalkylamides in the mixture with xanthate from 30 to 70% favorably affects the flotation of sulfides – the recovery of nickel and copper increases and their content in the flotation tailings decreases. When used in a mixture with xanthate reagent C12-PAA, as well as in the case of its independent application, flotation proceeds less efficiently. Apparently, this is due to the fact that the benzene ring in the structure of the reagent C12-SAA creates steric hindrances in the fixation of the reagent molecule on the mineral surface. Higher efficiency of copper-nickel ore flotation was achieved when using C10-SAA in the ratio with xanthate 70 : 30. When using C10-SAA, the content of nickel and copper in the tailings was also obtained close to the basic mode.

Conclusion

Monoalkylamides of phthalic and succinic acids are able to form complex compounds with copper and nickel ions, which determines the efficiency of their interaction with the surface of nickel- and copper-containing minerals.

Monoalkylamides of phthalic and succinic acids can be used as reagents-collectors for flotation of sulfide ores and extraction of transition metal ions.

Of the two reagents studied, succinic acid monoalkylamide shows the highest selectivity both as an independent collecting agent with its own foaming properties and in a mixture with xanthogenate. The best results were obtained when it was used in a mixture with xanthate at a ratio of 70 : 30.

The obtained results expand the range of possible reagents-complexing agents for flotation of sulfide ores of non-ferrous metals. The simplicity of the synthesis and availability of domestic raw material base makes this class promising for practical use.

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