

RESEARCH PAPER

The effect of apolar flotation reagent and additional re-grinding of lead-zinc ore processing

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ABSTRACT

Due to the involvement in the processing of increasingly complex mineralogical composition, poor, finely disseminated ores, there is a need for the correct selection of flotation reagents, their preparation for flotation, a combination of various collectors, modifiers to reduce the loss of fine particles of sulfides with flotation tailings. The article examines the effect of an apolar flotation reagent based on oil and diesel fuel and additional re-grinding on finely disseminated lead-zinc ore processing. The new apolar reagent is a dispersed emulsion consisting of a mixture of oil and diesel fuel at a ratio of 1:1. The reagent is dominated by liquid hydrocarbons of normal and isostructural composition, and sulfur compounds in the form of mercaptans are present. After optimal dispersion for 7 minutes, the content of particles in it with a grain size of less than 8.6 microns was 91.0%. It is shown that ore refinement is required to improve the disclosure of useful components from waste rock. The use of additional grinding and a new apolar reagent makes it possible to increase the extraction of zinc into the concentrate by 9.42%, while the extraction of lead increases by 12.36%.

Keywords: lead-zinc ore; flotation; additional grinding; apolar reagent; dispersion; concentrate.

INTRODUCTION

As production capacity increases, the primary reserves of high-quality lead and zinc ores are depleted. The world's mineral resources are becoming more complex in their mineralogical composition, poor and finely disseminated, complicating their processing.

Flotation is a widely used method in mineral processing based on differences in the hydrophilicity and hydrophobicity of mineral particles. In recent years, research has become widespread aimed at developing new and improving existing technological regimes that intensify the flotation process of lead-zinc ores [1-4].

Flotation collectors can selectively adsorb different mineral surfaces of sulfide minerals, increasing their hydrophobicity. Xanthate is most often used as a collector in the flotation of sulfide minerals. However, the disadvantage is its limited selectivity, which reduces the content of valuable sulfide minerals in the resulting concentrate. The extraction of fine galena slurry particles is another important problem in lead-zinc ore processing. Therefore, there is a need for proper selection of flotation reagents, their preparation for flotation, a combination of various collectors, modifiers to reduce the loss of fine particles of sulfides with flotation tailings [5-6].

A new selective collector of galena based on modified dithiophosphate in the flotation separation of Pb-Zn ores has been developed, and its adsorption mechanism has been studied [7]. The synthesized new collector S-benzoyl-O, O'-dibutyl dithiophosphate S-benzoyl-O, O'-dibutyl dithiophosphate (BDTP) has a higher collecting capacity and better selectivity for galena than traditional sulfide collectors (for example, sodium isobutyl xanthate). The new collector showed effective galena and sphalerite flotation separation at a pH of about 9.0 with ZnSO₄ and Na₂SO₃ as modifiers. Investigation of the wettability angle showed that the surface of galena treated with BDTP was more hydrophobic than the sphalerite surface. Based on the results of scanning electron microscopy, energy-dispersive X-ray spectroscopy, and X-ray photoelectron spectroscopy, it is suggested that the interaction of BDTP with galena may be dominated by chemisorption.

Selective flotation of sulfide minerals has been studied using many flotation modifiers, such as dextrin [8], polyaspartic acid [9], pectin [10], chitosan [11], and a combination of KMnO₄ and carboxylated chitosan [12]. As a depressor at a rate of 0.67 mg/l, Chitosan separates galena and sphalerite activated by copper. Amino and hydroxyl groups in chitosan are strongly influenced by sphalerite. When non-toxic and environmentally friendly pectin was used to separate galena

and sphalerite, it was shown that pectin was adsorbed on the galena surface through hydrophobic interaction and was easily replaced by xanthate. At the same time, pectin is chemically bound to zinc on the surface of sphalerite through its O atom on OH and COOH. However, organic polymer reagents such as pectin and chitosan are difficult to dissolve, so their use in industry is complicated.

The effect of 2-hydroxyethyl dibutyl dithiocarbamate on galena and sphalerite has been studied. Compared with two common collectors, sodium isobutyl xanthate and O-isopropyl-N-ethylthionocarbamate, 2-hydroxyethyl dibutyl dithiocarbamate demonstrated better selectivity for galena compared to sphalerite. The results of the adsorption mechanisms showed that 2-hydroxyethyl dibutyl dithiocarbamate could improve the galena surface by chemisorption but has little effect on the hydrophobicity of sphalerite without an obvious chemical reaction [13].

The effect of the new sphalerite depressant hyaluronic acid on the flotation of galena and sphalerite has been studied. The XPS results showed that hyaluronic acid is chemisorbed on sphalerite by coordinating carboxyl and N-acetyl groups with Zn sites. In contrast, on the galena surface, it is physically adsorbed by hydrophobic interaction [14].

The beneficiation of lead-zinc ores is often hampered by ultrafine carbonaceous substances, easily crushed calcite minerals, and difficult-to-grind silicified dolomites. The characteristics of carbonaceous minerals in the flotation of lead-zinc ores have rarely been studied. However, the surface properties of carbonaceous minerals greatly influence the flotation of lead and zinc. In coal flotation, nonpolar hydrocarbon oil collectors are adsorbed on naturally hydrophobic coal particles, making them more selectively hydrophobic [15].

Research is underway on the effectiveness of apolar reagents containing aromatic hydrocarbons and their homologues. There has been a noticeable increase in interest in hetero-organic oil compounds' use in the flotation of polymetallic sulfide ores. Technological processes are being developed and mastered separate organic sulfur-containing compounds from oil products, mainly consisting of mercaptans, sulfides, and thiophenes [16]. Oil-based emulsified apolar reagents are particularly effective in flotation complex sulfide finely grained ores. For example, the high floatability of galena with the introduction of a hydrocarbon collector is due to the high density of the hydrocarbon sorption layer, and it is significantly higher than the sorption of hydrocarbons on molybdenum and pyrite.

This work aims to study the effect of apolar flotation reagent and additional re-grinding on the processing of lead-zinc ore from one of the Kazakh deposits. Due to the very fine dispersion of galena and sphalerite minerals in the ore under study, their close interpenetration with each other and the rock-forming minerals,

there is a need for finer grinding of the ore before the beneficiation processes. It is known from the flotation practice that mineral particles less than 10 microns in size do not participate in selective flotation processes. This leads to significant losses of useful components during the flotation processing of ores. Losses with fine grades for some polymetallic sulfide ores reach 30-40 % of the total metal losses during beneficiation. At the same time, obtaining high-quality lead and zinc concentrates is very difficult. In this regard, studying the effect of apolar reagents on the beneficiation of refractory ores requiring fine regrinding is relevant. This will improve the technological performance of the process of flotation beneficiation. Of raw mineral materials, reducing the loss of useful components with flotation tailings.

MATERIAL AND METHODS

Materials

The research objects were lead-zinc ore from one of Kazakhstan's deposits of polymetallic refractory ores, an apolar reagent obtained from natural oil from the Kumkol deposit, and diesel fuel.

Material composition of lead-zinc ore

The nature of the refractory ore under study is associated with the very fine dispersion of galena, sphalerite and pyrite minerals, their close interpenetration with each other and rock-forming minerals, the presence of ultrathin carbonaceous substances, as well as the presence of easily crushed calcite minerals with difficult-to-grind silicified dolomites. The average total content of valuable metals in the deposit is about 5 %, of which 70-80 % is zinc and 20-30 % is lead. The results of the chemical analysis of the ore under study are shown in **Table 1**.

Table 1 Chemical composition of lead-zinc ore

Element, connection	Content, %
Lead	1.3
Zinc	3.7
Copper	0.05
Iron	3.42
Common sulfur	5.52
Organic carbon	1.02
Calcium	5.35
Magnesium	3.58
Aluminum	0.80
Potassium	0.56
Sodium	0.033
Quartz	45.00
Silver, g/t	5.4

A phase analysis, shown in **Table 2**, was performed to determine the form of lead and zinc in the initial ore.

The phase analysis results show that most of the lead and zinc in the ore (about 90%) are in sulfide form in galena and sphalerite. According to the results of X-ray fluorescence analysis (**Table 3**), the ore contains 1.475 % lead, 3.575% zinc, 2.592% iron, 21.887% silicon, and 7.684 % calcium.

Table 2 Phase analysis of lead and zinc in lead-zinc ore

Component	Minerals	Content, %	
		absolute	relative
Lead	anglesite, cerussite,	0.085	6.5
	galena	1.157	89.0
	residual forms	0.058	4.5
	Total lead:	1.3	100.0
Zinc	oxygen-containing compounds	0.433	11.7
	sphalerite	3.267	88.3
	Total zinc:	3.7	100.0

Table 3 Results of X-ray fluorescence analysis of lead-zinc ore

Analyte	Compound formula	Concentration, %	Status
O	O	43.964	BgC; DC
Mg	Mg	3.521	BgC; DC
Al	Al	1.272	BgC; DC
Si	Si	21.887	BgC; DC
P	P	0.042	BgC; DC
S	S	3.734	BgC; DC
Cl	Cl	0.033	BgC; DC
K	K	0.360	BgC; DC
Ca	Ca	7.684	BgC; DC
Ti	Ti	0.114	BgC; DC
Mn	Mn	0.053	BgC; DC
Fe	Fe	2.592	BgC; DC; LoR
Cu	Cu	0.014	BgC; DC
Zn	Zn	3.575	BgC; DC
As	As	0.021	BgC; DC
Sr	Sr	0.007	BgC; DC
Cd	Cd	0.010	BgC; DC
Pb	Pb	1.475	BgC; DC

A sample of the initial lead-zinc ore was analyzed on a JEOL JXA-8230 electron probe microanalyzer (**Figs. 1, 2**).

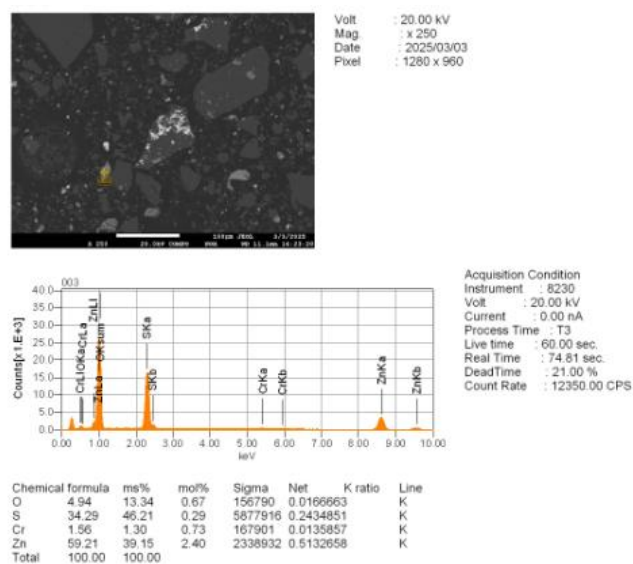


Fig.1 Analysis of the initial tailings of the flotation beneficiation lead-zinc ore on an electron probe microanalyzer JXA-8230 from JEOL (sphalerite)

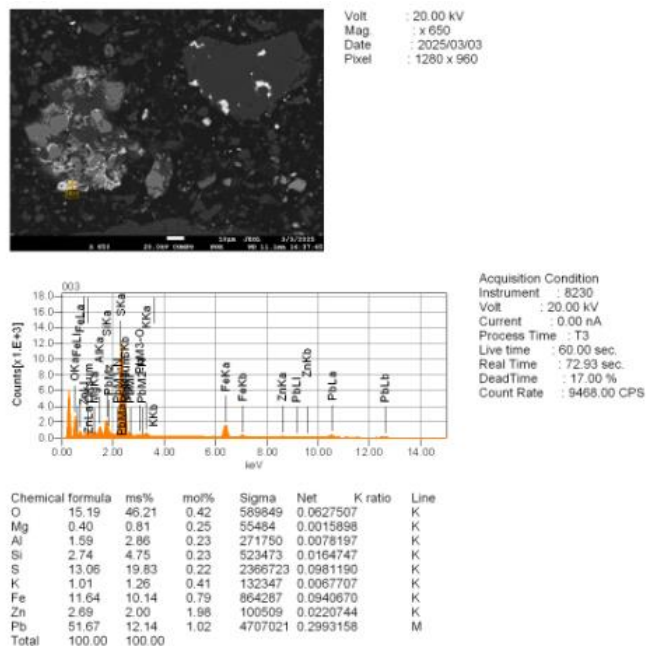


Fig.2 Analysis of the initial tailings of the flotation beneficiation of the lead-zinc ore on an electron probe microanalyzer JXA-8230 from JEOL (galena)

Preparation of the apolar reagent emulsion

A new apolar reagent—a mixture of oil and diesel fuel in the form of a direct emulsion—is proposed as a replacement for expensive kerosene. Together with the final grinding of the beneficiation products, this makes it possible to intensify the flotation process of mineral slurry particles and thereby improve ore beneficiation rates for lead and zinc.

To obtain an emulsion, the initial reagents were dispersed on an ultrasonic device “UZDN-A1200T” at different ratios of oil and diesel fuel (1:1, 1:2, 1:3) and different dispersion times (3, 5; 7 min.) at a concentration of 0.1% reagents.

To select the emulsion with the smallest dispersed particles, the particle size of the emulsion was analyzed under different conditions of its preparation using a Winner2000E laser particle size analyzer. The particle sizes of the diesel fuel emulsion, oil and their mixtures were determined in various ratios and at different dispersion times. The histograms of the particle distribution of the prepared emulsions showed that the smallest particles are contained in an emulsion of a mixture of oil and diesel fuel in a ratio of 1:1, dispersed for 7 minutes. The content of particles smaller than 8.6 microns was 91.0% (Fig. 3). With a further increase in the dispersion time, the dispersion of the apolar reagent did not increase.

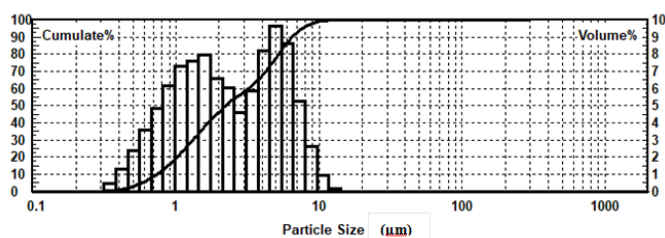


Fig.3 Distribution of apolar collector particles with a dispersion time of 7 minutes

The ultrasound power during emulsion processing at UZDN-A1200T was 1200 watts, the operating frequency was 20 kHz, and the operating voltage was 220 V, 50 Hz. The emulsion prepared in this way was used to flotation lead-zinc ore.

Research methods and equipment

The following research methods were used in this work: IR spectroscopy using the Avatar-370 CsI Tegmo Nicolet IR Fourier spectrometer (USA), electron probe microanalysis using the JXA-8230 analyzer from JEOL (Japan), X-ray fluorescence (Axios spectrometer, Malvern Panalytical Ltd., Great Britain), X-ray phase (D8 diffractometer ADVANCE, Bruker, Billerica, Massachusetts, USA) analyses, atomic absorption (AA-7000 spectrometer, Shimadzu, Tokyo, Japan). The emulsion of the flotation reagent was obtained using the “UZDN-A1200T” ultrasound machine from the Ukrospribor NPP (Ukraine). The particle size of the apolar flotation agent emulsion was determined using a Winner2000E laser particle size analyzer (China). Ore crushing was carried out on a DMD160/100 jaw crusher (Kyrgyzstan). After crushing, before flotation, the ore was crushed in a 40ML-000PS ball mill (Russia). Ore flotation was carried out on laboratory flotation machines of the FL series (Russia) with a chamber volume of 3; 1.0; 0.5; 0.25 dm³.

RESULTS AND DISCUSSION

Studies have been conducted on the flotation of lead-zinc ore according to the basic direct selective flotation scheme. The ore grinding was 90% class of size - 0.074 mm. The lead cycle consisted of rough and scavenge lead flotation and three cleanings of lead concentrate. The zinc cycle consisted of rough and scavenge zinc flotation and three cleanings of zinc concentrate.

To determine the content of lead and zinc in different grain classes, a dispersion analysis of ore grinding to 90% class of size -0.074 +0 mm was performed before flotation, the results of which are shown in Table 4.

Table 4 Results of dispersion analysis of lead-zinc ore grinding to 90% class of size less than 0.074 mm

Size class, mm	Yield, %	Content, %		Distribution, %	
		Zn	Pb	Zn	Pb
+ 0.074	10.0	3.1	1.5	8.75	9.36
- 0.074 +0.050	5.2	3.8	1.8	5.58	5.84
- 0.050 +0.040	10.9	3.3	1.5	10.17	10.21
- 0.040 +0.030	10.1	4.0	1.8	11.41	11.35
- 0.030 +0.020	11.6	3.9	1.7	12.78	12.31
- 0.020 +0.010	10.5	3.4	1.5	10.08	9.83
- 0.010 +0.005	16.1	3.5	1.7	15.91	17.1
-0.005 +0	25.6	3.5	1.5	25.32	24.0
Total:	100.0	3.65	1.6	100.0	100.0

The results of dispersion analysis of grinding ore show that the ore is finely disseminated, the majority of useful components (50.93% lead, 51.31% zinc) are concentrated in the -20 + 0 microns fraction, which belongs to the refractory slurry size.

The main lead flotation was supplied with liquid glass for the depression of pyrite and waste rock, sodium sulphate, zinc sulfate and sodium cyanide for the depression of zinc minerals, collector butyl sodium xanthate, foaming agent T-80. A collector and a frother were fed into the control lead flotation, zinc sulfate and sodium cyanide were added to the purifiers for additional zinc depression. The main zinc flotation was supplied with a medium regulator lime to create pH = 10-11, liquid glass, zinc mineral activator copper sulfate, collector butyl sodium xanthate, frother agent T-80. An activator, collector, and frother were supplied to the control zinc flotation, lime was used to create pH = 10-11, liquid glass was used to depress pyrite, and waste rock was added to the purifiers. Kerosene was added to the main and control flotations to improve the floatability of the thin classes. The results of ore flotation according to the basic reagent regime in a closed cycle are presented in Table 5.

The above analyses show that 34.54% of zinc and 44.78% of lead are lost in flotation tailings. Dispersion analysis of zinc flotation tailings was performed to determine the reason for the low recoverability of useful components (Table 6).

Table 5 Results of flotation of the lead-zinc ore according to the basic reagent conditions

Products	Yield,%	Content, %		Recovery, %	
		Zn	Pb	Zn	Pb
Zn concentrate	4.5	54.07	1.96	63.2	4.82
Pb concentrate	2.1	4.15	43.92	2.26	50.4
Tails	93.4	1.42	0.87	34.54	44.78
Ore	100.0	3.85	1.83	100.0	100.0

Table 6 Results of the dispersion analysis of ore flotation tailings obtained by the basic reagent conditions

Size class, mm	Yield,%	Content, %		Distribution, %	
		Zn	Pb	Zn	Pb
+ 0.074	6.1	1.6	1.2	6.84	8.44
- 0.074 +0.050	3.6	1.6	1.2	4.03	4.98
- 0.050 +0.040	8.2	1.5	1.2	8.62	11.35
- 0.040 +0.030	8.2	1.4	1.2	8.04	11.35
- 0.030 +0.020	9.1	1.4	1.1	8.92	11.55
- 0.020 +0.010	12.8	1.4	0.7	12.55	10.34
- 0.010 +0.005	19.4	1.4	0.7	19.02	15.67
-0.005 +0	32.6	1.4	0.7	31.97	26.32
Total:	100.0	1.43	0.87	100.0	100.0

As a result of dispersion analysis of flotation tailings, it is shown that zinc content in all size classes is approximately 1.4 - 1.6 %, and lead content varies in different size classes from 0.7 to 1.2 %. Lead content is higher in coarse grades than in fine grades. The highest distribution of lead (52.33%) and zinc (63.54%) in the flotation tailings is observed in the fine refractory size fractions of -20 +0 μm. Fig. 4 shows a micrograph of the ore flotation tailings obtained using a JEOL JXA-8230 electron probe microanalyzer. It can be seen that the losses in the tailings are due to the fine dissemination of lead and zinc within the gangue minerals and the insufficient degree of ore grinding.

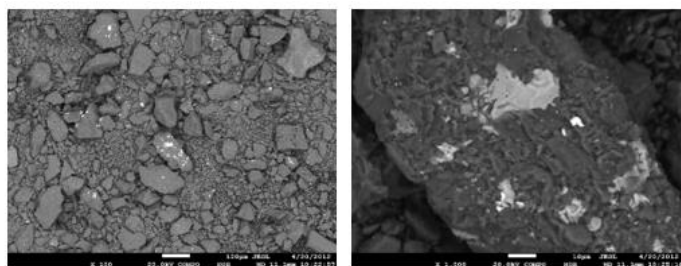


Fig. 4 Micrograph of lead-zinc ore flotation tailings

As a result of the research, it is shown that improving the recoverability of useful components from the waste rock requires re-grinding ore. To increase lead and zinc recovery in the technological scheme (flowsheet) of beneficiation, a different grinding mode was adopted in contrast to the basic mode. The initial ore was grinded to 80 % of class -0,074 mm, the foam product of the first lead retreatment was grinded to 97 % of class - 0,074 mm and the chamber product of the control lead flotation - to 99 % of class - 0,074 mm. To increase the flotability of fine size classes -0.020 mm, a new apolar reagent (dispersed emulsion of a mixture of oil and diesel fuel in the ratio 1:1) was used instead of paraffin (in the basic mode).

Fig. 5 shows IR spectra of diesel fuel and oil samples from Kumkol deposit. According to IR spectra it was found that in the samples under study liquid hydrocarbons of normal and isostructure predominate, sulfur compounds in the form of mercaptans are present. The presence of naphthenic and aromatic structures was noted. Carbonyl group is absent, oil is not oxidised. As can be seen from Figure 5 the compositions of liquid hydrocarbons in both samples practically coincide. Therefore, to obtain a qualitative direct emulsion of oil in water, diesel fuel served as a solvent of oil.

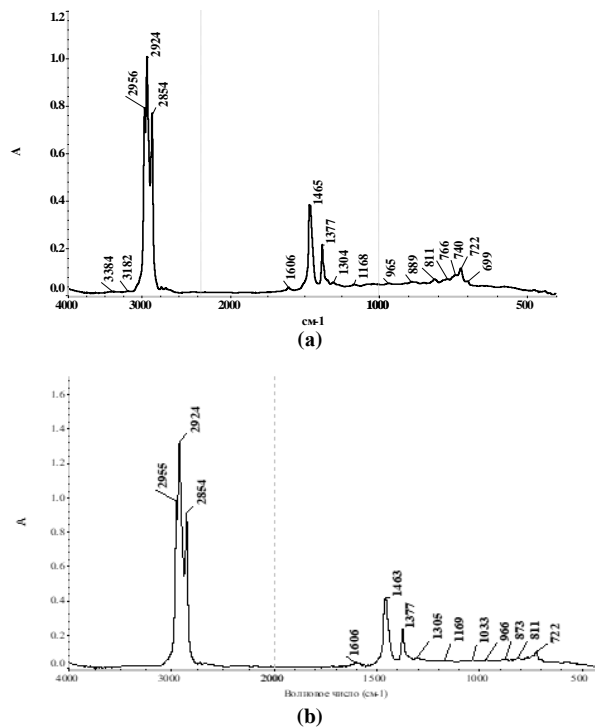


Fig. 5 IR spectra of diesel fuel sample (a) and oil from Kumkol deposit (b)

Table 6 shows the research results on the effect of re-grinding and new apolar reagent on the beneficiation of lead-zinc ore. Flotation was carried out according to the basic mode without re-grinding, with re-grinding and with the use of kerosene to improve the flotation of slurry particles and according to the proposed mode with re-grinding and the use of modified apolar reagent. **Table 7** shows the flotation scheme with reagent mode.

Table 7 Results of studies on the impact of re-grinding and a modified apolar reagent on the beneficiation of lead and zinc ore.

Products	Yield %	Content, %		Recovery, %		Note
		Zn	Pb	Zn	Pb	
Zn concentrate	45	54.07	1.96	63.2	4.82	Basic mode without additional re-grinding
Pb concentrate	2.1	4.15	43.92	2.26	50.4	
Tails	93.4	1.42	0.87	34.54	44.78	
Ore	100.0	3.85	1.83	100.0	100.0	With additional re-grinding
Zn concentrate	4.7	56.2	4.1	68.33	10.21	
Pb concentrate	2.3	4.7	47.8	2.80	58.25	
Tails	93.0	1.2	0.64	28.87	31.54	With additional re-grinding and using kerosene
Ore	100.0	3.87	1.89	100.0	100.0	
Zn concentrate	4.65	57.3	4.9	70.03	12.17	
Pb concentrate	2.35	5.0	48.2	3.09	60.51	With additional re-grinding and using an apolar reagent
Tails	93.0	1.1	0.55	26.89	27.32	
Ore	100.0	3.80	1.87	100.0	100.0	
Zn concentrate	4.8	57.9	4.9	72.62	12.53	With additional re-grinding and using an apolar reagent
Pb concentrate	2.4	5.0	49.1	3.14	62.76	
Tails	92.8	1.0	0.5	24.25	24.71	
Ore	100.0	3.83	1.88	100.0	100.0	

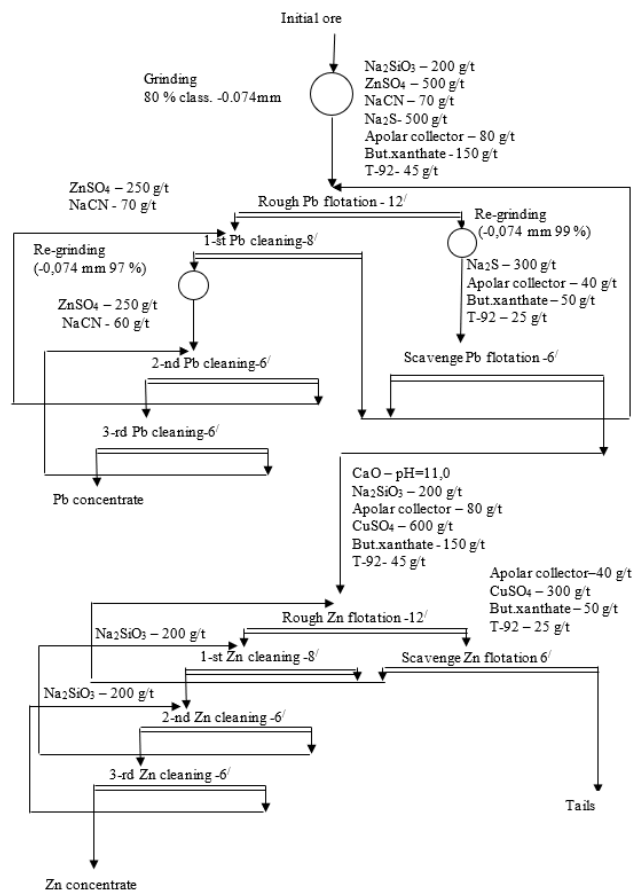


Fig. 6 Technological scheme and reagent mode of lead-zinc ore flotation using re-grinding and a new apolar reagent

The results of the conducted studies showed that adding the re-grinding operation to the basic technology improves the quality and extraction of lead and zinc into concentrates of different names. The zinc content in the zinc concentrates increases from 54.07 to 56.2% (by 2.13%), and its extraction increases from 63.2% to 68.33% (by 5.13%). The lead content in the lead concentrate rises from 43.92% to 47.8% (3.88%), and lead extraction increases from 50.4% to 58.25% (7.85%). The additional use of kerosene to improve the floatability of sludge particles of useful components allows for an increase in the extraction of lead and zinc into concentrates by approximately another 2%.

The flotation analysis showed that using a new apolar reagent compared to the base mode increases the zinc content in the zinc concentrate by 3.83%, and its extraction increases by 9.42%. The lead content in the lead concentrate rises by 5.18%, and lead extraction increases by 12.36%.

Lead content in flotation tailings decreases from 0.87 to 0.5 %, zinc content decreases from 1.42 to 1.0 %. Losses of lead and zinc with flotation tailings are reduced by 20.07 % and 10.29 %, respectively.

CONCLUSION

Thus, to increase the extraction of lead and zinc in the technological scheme (flowsheet) of beneficiation of finely disseminated difficult-to-enrich lead-zinc ore, in contrast to the basic mode, a different grinding mode was adopted, and a new apolar reagent was added to the reagent mode. The original ore was grinding to 80% of the -0.074 mm class, the foam product of the first lead re-cleaning was grinded to 97% of the -0.074 mm class and the chamber product of the scavenge lead flotation - to 99% of the -0.074 mm class.

The new apolar reagent is a dispersed emulsion consisting of a mixture of Kumkol oil and diesel fuel in a 1:1 ratio. After optimal dispersion for 7 minutes, the content of particles smaller than 8.6 microns in size was 91.0%.

The studies conducted on lead-zinc ore showed that the use of re-grinding and a new apolar reagent, compared to the basic mode, allows increasing the zinc content in zinc concentrate by 3.83% and increasing its extraction by 9.42%; the lead content in lead concentrate rises by 5.18%, and lead extraction increases by 12.36%. The lead content in flotation tailings obtained according to the beneficiation scheme (flowsheet) using re-grinding and a new reagent decreases from 0.87 to 0.5%, and zinc - from 1.42 to 1.0%. Losses of lead and zinc with flotation tailings are reduced by 20.07% and 10.29%, respectively.

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