Study of the material composition of lead-zinc ore of the Shalkiya deposit in order to determine the possibility of its processing

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The need for lead-zinc ore mining and processing has recently increased significantly due to the ever-increasing use of lead and zinc in various industries. Lead-acid batteries alone account for about 80% of lead consumption, while electroplated steel coatings account for about 50% of zinc use. The presence of a significant number of explored but undeveloped deposits allows the Republic of Kazakhstan to develop the mining and processing of lead-zinc ores.
The article presents the results of the material composition's study of lead-zinc ore at the Shalkiya deposit, whose total zinc reserves account for more than 30% of all reserves in the Republic of Kazakhstan and are the fifth largest in the world. Proven and probable reserves amount to 6.5 million tons of zinc by JORC classification.
The proportion of galena in the free particles is 47.42%, sphalerite — 39.52%. The particle sizes of galena and sphalerite are less than 5 and 10 μm, respectively, indicating a low prospect of separation by gravity method, which was confirmed during beneficiation at the spiral separator. Analysis of the material composition showed that the ores of the Shalkiya deposit belong to the category of hard-to-beneficiation ores.

Key words: lead-zinc ore, material composition, galena, sphalerite, particle-size composition, mineralogical analysis
DOI: 10.17580/nfm.2022.02.02

Introduction

Lead and zinc are non-ferrous metals with unique physical and chemical properties. To meet the steadily growing demand of various industries for these metals, producing companies are constantly increasing the volume of mining and processing of lead and zinc ores. Total supplies are expected to peak between 2025–2030 for lead and 2030-2050 for zinc [1].

The world leader in lead production as of 2019 was China (2.1 million tons of lead or 46.7% of global production). The contribution of other leading producers, including Australia, Peru and the United States, was less than 10% of global production in 2019 [2]. According to 2021 data, global refined zinc production (14.13 million tons) shows outstripping growth in comparison with consumption (14.09 million tons) [3]. The main natural sources of lead and zinc are ores from sulfide deposits, and the industrially important minerals of lead and zinc are galena and sphalerite [4, 5].

The main useful minerals in the ores of the Shalkiya deposit are sphalerite, galena and pyrite, which have phenocrysts from dusty to 0.1 mm and are characterized by close intergrowth between themselves, as well as with minerals of waste rock, in particular quartz and carbonates. According to the Brook Hunt research company, total zinc reserves account for more than 30% of Kazakhstan's reserves and are the fifth largest in the world. According to JORC classification proven and probable reserves amount to 6.5 million tons of zinc. Ore reserves according to categories B+C1+C2 — 127.5 million tons with zinc content of 4.27% and lead of 1.28%. The very fine phenocrysts of lead and zinc minerals are complicated by the presence of carbonaceous matter, which is present both in the host rocks and in the useful minerals. In earlier studies on the development of ore beneficiation technology of the Shalkiya deposit were proposed collective-selective and selective flotation schemes [6–8].

At the same time, the development of beneficiation technology for ores from the Shalkiya deposit is complicated by the following circumstances:

1. Complex mineral composition of ores, thin phenocrysts and close intergrowth of sulfide minerals throughout the ore body.

2. Ore of Shalkiya deposit does not contain valuable by-product components typical for polymetallic ores, but contains harmful impurities in the form of carbonaceous substances, which due to high sorption properties complicate the flotation process and increase reagent consumption. As an example, there is a lack of sufficiently effective processing technology for similar ores at the Zhayrem

(Kazakhstan) and Ozernoye (Russian Federation) deposits [9–13].

1. At the moment there are no effective technologies for beneficiation of high carbonaceous polymetallic ores, as well as selective separation of natural hydrophobic carbonaceous substances and sulfide minerals, including such minerals as galena.

2. Over the last 20 years new deposits of lead and zinc have not been practically developed, as a result of which the tendency of depletion of existing resources can be traced and the task of processing hard-to-beneficiation ores with low content of valuable components is becoming actual. [14, 15].

3. An additional factor which complicates the development of the technology for processing such ores is that it is very difficult to recovery sphalerite from them [16].

Thus, the development of an effective technology for processing hard-to-beneficiation lead-zinc ores requires, first of all, a deep comprehensive study of their material composition and technological properties, including research into the interaction of new reagents with the surface of minerals.

Study of the material composition of lead-zinc ore of the Shalkiya deposit

A comprehensive study of the material composition of lead-zinc ore samples from the Shalkiya deposit included macro- and microscopic studies using optical methods, electron microscopy, local *X*-ray spectral (microprobe) analysis, atomic emission and mass spectrometric methods with inductively coupled plasma, titrimetric and gravimetric methods, sieve, sedimentation, chemical and assay analysis.

Nikon equipment was used for optical research methods: polarizing microscope ECLIPSE LV100-POL, optical stereo microscope SMZ-1500 equipped with digital photomicrographic system DS-5M-L1 and stereo microscope SMZ-645. The composition of minerals in the briquettes was determined on an MLA 650 (FEI Company) instrumental automatic complex, including an FEI Quanta 600 SEM scanning electron microscope equipped with an *X*-ray microanalysis system with two detectors.

The content of the main monitored components in the process sample and in individual size classes was determined by X-ray spectral analysis in the IGEM RAS laboratory of mineral matter analysis (certificate of state accreditation № POCC RU. 0001.514143). Granulometric composition of the sample material was determined by the standard wet method using a set of laboratory sieves (GOST 3584–72). The chemical composition of the Shalkiya deposit ore sample is given in **Table 1**.

The main components in the studied sample of ore material are silicon oxide 40.15%, calcium oxides 19.21% and magnesium 8.71%. The main valuable components of industrial interest in this product are lead (content 1.21%) and zinc 4.13%. The ore also contains iron (in terms of FeO 2.47%), organic carbon 1.11%.

Table 1
Chemical composition of lead-zinc ore sample

Component	Content, %
SiO_2	40.15
TiO_2	0.10
Al_2O_3	2.00
FeO_t	2.47
CaO	19.21
MgO	8.71
MnO	0.09
K_2O	0.66
Na_2O	<0.02
P_2O_5	0.05
S_t	2.80
Cl	0.08
C_{org}	1.11
CO_2	16.63
Cr	0.02
Cu	0.01
Zn	4.13
Pb	1.21
Total	***99.43***

When analyzing the particle size distribution of the examined samples and the nature of the distribution of the main valuable components into size classes, the main attention was paid to the parameters that determine the predicted technological properties of the ore:

– particle size of the beginning of the liberation of the main valuable minerals;

– optimum particle size of the separation process;

– stadiality of grinding and beneficiation;

– presence of the productive fraction and expediency of its separation;

– expediency (or inexpediency) of ore material deslurrying before the separation process.

The optimum particle size of the separation process is determined by establishing the particle size of the beginning of the liberation, characterized by the prevalence of the content of the valuable component in the class over the initial content, and the particle size of the complete liberation, determined by the maximum excess of its distribution over the yield.

The level of unavoidable losses is determined by the value of the distribution of the valuable component in the size class of less than 0.020 mm; in flotation – less than 0.010 mm.

The expediency of deslurrying is determined by the output of the slurry class, the concentration of the valuable component in it, and the value of the distribution of the valuable component in a given slurry fraction.

The behavior of the associated component in the subsequent separation is determined by the correlation of its content with the content of the main valuable component and, accordingly, by comparing the values of their distribution.

Table 2
Granulometric composition and distribution of main elements in the Shalkiya deposit ore sample, crushed to a –1 mm grain size

Size grade, mm	Yield	Content, %					Distribution, %				
		Zn	Pb	S	Fe	SiO_2	Zn	Pb	S	Fe	SiO_2
–1+0.5	44.73	3.26	0.92	2.35	1.54	43.77	39.51	38.89	40.86	40.32	47.75
–0.5+0.25	16.65	3.45	0.88	2.54	1.54	41.66	15.58	13.88	16.43	14.94	16.92
–0.25+0.1	15.85	3.90	1.09	2.77	1.78	42.42	16.77	16.34	17.05	16.49	16.40
–0.1+0.074	2.92	4.56	1.22	3.26	1.94	39.73	3.61	3.39	3.70	3.31	2.83
–0.074+0.044	4.24	4.98	1.30	3.60	2.14	38.45	5.72	5.25	5.93	5.28	3.98
–0.044+0.020	6.28	6.05	1.26	1.76	2.15	29.95	10.31	7.49	4.29	7.89	4.59
–0.020+0.010	3.01	5.15	0.94	1.69	1.98	28.32	4.20	2.70	1.97	3.47	2.08
–0.010+0	6.32	5.41	1.51	1.99	2.04	27.08	9.27	9.07	4.89	7.53	4.17
Initial sample (by balance)	100.00	3.69	1.05	2.57	1.71	41.00	100.00	100.00	100.00	100.00	100.00
Direct measuring		4.12	1.21	2.79	1.91	40.12					

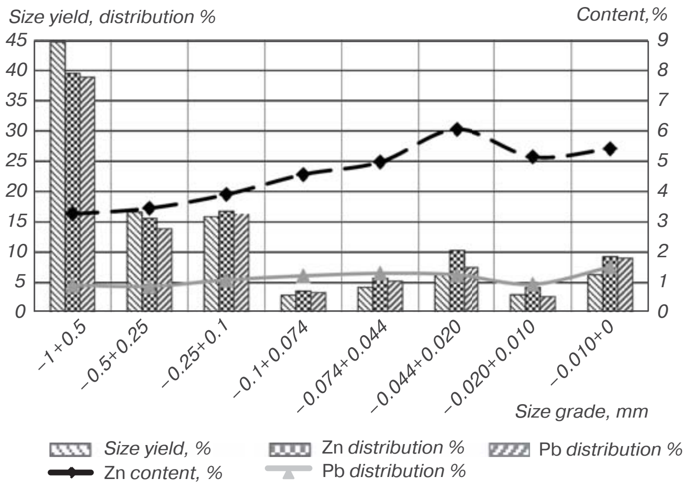

Fig. 1. Distribution by size of Shalkiya deposit ore sample, crushed to 1 mm size; content and distribution in size classes of lead and zinc

Analysis of the particle size distribution of the original ore sample (**Table 2, Fig. 1**), crushed to a size of –1 mm, showed that the bulk of the sample accounts for a large class –1+0.5 mm (44.73%), a significant yield have classes –0.5+0.25 and –0.25+0.1 mm (16.65 and 15.85%, respectively). The yield of the slurry class –0.044+0 is 15.62%.

According to the results of the analysis it was found that the content of lead and zinc increases with decreasing the size of the ore material. The beginning of liberation of galena is observed in the class –0.25+0.1 mm and has a bimodal distribution character with peaks in the classes –0.074+0.044 and –0.010+0 mm. For sphalerite we can note a unimodal distribution character with a peak in the size class –0.044+0.020 mm.

The Mineral Liberation Analysis (MLA) was carried out on a briquette made from the sample material of the initial ore of the Shalkiya deposit, crushed to a particle size of –0.25 mm. Microphotographs of the surface of the anslip briquette are shown in **Fig. 2**.

During the automated mineralogical analysis of samples were identified mineral phases grouped into separate mineral groups, depending on their chemical composition and physical properties **(Table 3)**.

Further description of the analysis results is given according to the final grouping of minerals.

The mineral composition of the sample, calculated on the basis of the data of microscopic studies, *X*-ray spectral and mass spectrometric analysis, is given in **Table 4**.

Ore minerals are represented by galena, sphalerite, and pyrite. The host rocks are represented by quartz and carbonates (dolomite and calcite), the ore also contains small amounts of carbonaceous matter, feldspars, and muscovite.

Table 5 shows the distribution of zinc and lead by mineral groups. According to MLA data, the main mineral concentrators of elements are:

– zinc: sphalerite and carbonates with a distribution of 87.44% and 8.04%, respectively;

– lead: galena and carbonates with a distribution of 86.99% and 10.64%, respectively.

The distribution of the main minerals by particle size in the initial sample is shown in **Table 6**.

Analysis of the distribution of clusters by the number of mineral phases showed that the degree of liberation of lead and zinc sulfides is rather low; the proportion of galena accounted for free particles is 47.42%, sphalerite – 39.52%.

The predominance of the stable mineral association of carbonates with galena and sphalerite among binary clusters should be noted. Among the polymineral clusters with galena and sphalerite, quartz prevails.

Galenite and sphalerite are characterized by a low proportion of fully disclosed particles (no more than 48%).

Analysis of the quality distribution of galena-containing intergrows showed that the mineral in the sample is present in 11.86% of all particles analyzed, of which 9.23% are particles with sphalerite content less than 10%, 1.62% of particles are poor and ordinary clusters, 0.27% are rich clusters and 0.74% are free particles. The main distribution of the mineral falls on free particles (47.42%), rich (15.60%) and ordinary clusters (12.17%), amounting in total to 75.19%. But it should be noted that the grain size of free galena particles is less than 5 μm and this reduces the contrast for the gravitational separation process. Sphalerite (up to 6.16%) and carbonates (up to 14.41%) are present in the mineral composition of galena-rich matrixes, which can lead to higher zinc, calcium and magnesium contents in the lead concentrate

An analysis of the quality distribution of sphalerite-containing particles showed that the mineral is present in the sample in 33.87% of all particles analyzed, 21.30% of which are particles with sphalerite content less than 10%, 8.18% of particles are poor and ordinary clusters, 1.65% are rich clusters and 2.74% are free particles. The main distribution of the mineral falls on free particles (39.52%) and rich (21.00%) and ordinary clusters (17.56%), amounting in total to 78.08%. The size of free sphalerite

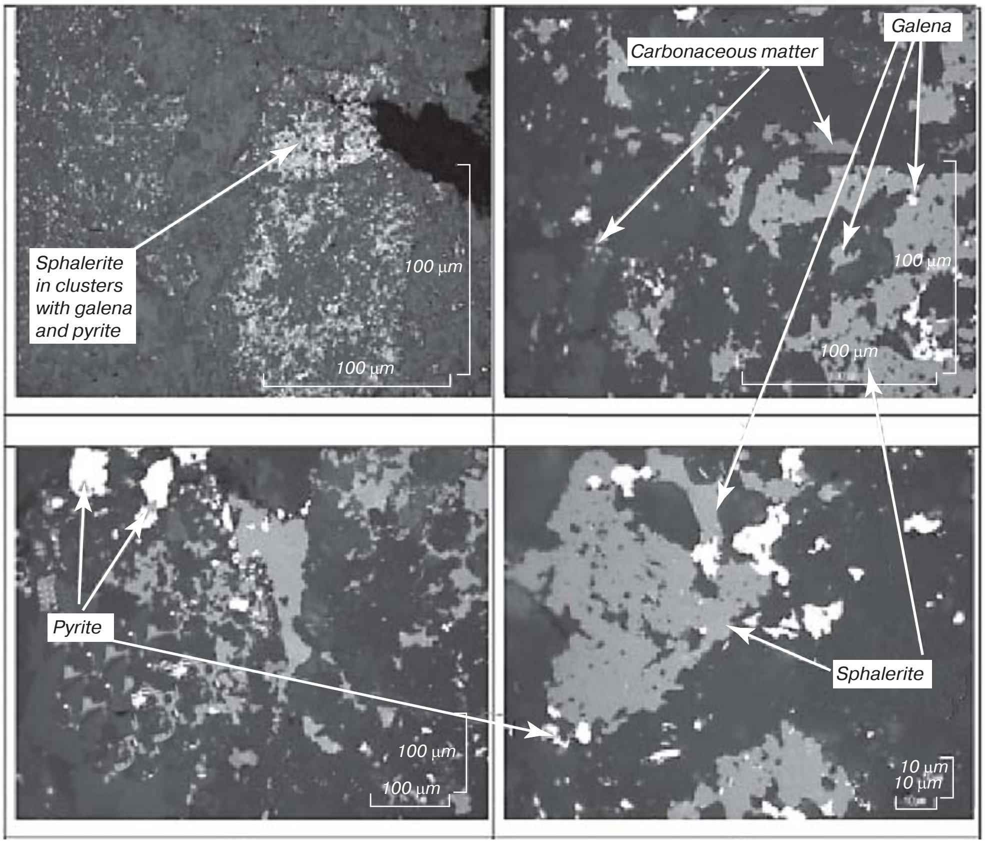

Fig. 2. Image of the polished section of an ore sample from the Shalkiya deposit in reflected light

Table 3
Mineral groups after mineral combining (according to MLA)

Mineral group	Minerals in the group
Galena	Galena
Sphalerite	Sphalerite, sphalerite + muscovite
Sulfides (pyrite, chalcopyrite, tennantite)	Pyrite, pyrite + sphalerite, pyrite + quartz, chalcopyrite, tennantite
Smithsonite	Smithsonite
Quartz	Quartz, quartz + smithsonite + siderite, orthoclase, oligoclase, quartz + muscovite, oligoclase, quartz + dolomite, quartz + calcite, quartz + sphalerite, quartz + siderite + muscovite + dolomite, quartz + orthoclase + galena.
Carbonates	Calcite, dolomite, calcite + feldspars, ankerite, calcite + quartz, dolomite + muscovite, dolomite + quartz + muscovite + galena + smithsonite, dolomite + orthoclase, dolomite + quartz, calcite + smithsonite, dolomite + muscovite + siderite, calcite + dolomite, smithsonite + rhodochrosite.
Mica and clay minerals (muscovite, kaolinite, chlorite)	Muscovite, muscovite + calcite, kaolinite, muscovite + sphalerite, muscovite + galena + sphalerite, chlorite, muscovite + galena, smithsonite + muscovite + galena, muscovite + pyrite.
Iron hydroxides	Goethite + galena + siderite + smithsonite, hydrogoethite + kaolinite + carbonates, hydrogoethite + carbonates.
Other barren minerals	Apatite, baryte, iron scrap, epidote, rutile, aluminum scrap

particles is less than 10 μm. The mineral composition of sphalerite-rich clusters contains quartz (up to 8.43%), carbonates (up to 6.67%), sulfides (up to 4.94%) and mica (up to 3.31%), which may lead to higher concentrations of quartz, calcium, magnesium and iron in zinc concentrate.

Table 4
Mineral composition of the sample

Mineral Group	Content, wt. %
Galena	1.57
Sphalerite	6.94
Sulfides (FeS_2, $CuFeS_2$, $Cu_{12}As_4S_{13}$)	4.28
Smithsonite	0.02
Quartz	38.13
Carbonates	43.97
Mica and clay minerals (muscovite, kaolinite, chlorite)	3.38
Iron hydroxides	0.23
Other barren minerals	0.17
Carbonaceous matter	1.30
Total	***100.00***

Table 5
Distribution of zinc and lead by main mineral groups (according to MLA data)

Mineral group	Distribution, %	
	Zn	Pb
Galena	–	86.99
Sphalerite	87.44	–
Sulfides (FeS_2, $CuFeS_2$, $Cu_{12}As_4S_{13}$)	0.30	–
Smithsonite	0.24	–
Quartz	0.02	0.13
Carbonates	8.04	10.64
Mica and clay minerals (muscovite, kaolinite, chlorite)	3.87	1.98
Iron hydroxides	0.09	0.26
Total	***100.00***	***100.00***

Analysis of the distribution of clusters by the open surface share of galena showed that almost all particles containing galena are closed and almost closed (9.94% out of 11.86% with a mineral distribution of 19.66%). The bulk of the mineral (up to 80.33) is distributed in open and partially open clusters, which account for only 1.92% of the 11.86% of particles containing galena. It should be noted that fully exposed galena particles (with a mineral distribution of up to 47.53%) have a particle size of less than 5 μm, which can lead to lower flotation recovery.

The analysis of the distribution of clusters according to the open surface share of sphalerite showed that the bulk of the particles containing sphalerite are closed (22.80%) and almost closed (33.87%); the distribution of the mineral in them is 12.65%. The bulk of the mineral (up to 87.35%) is distributed in open and partially open clusters, which account for 11.07% of the 33.87% of sphalerite-containing particles. Up to 41.68% of sphalerite is distributed in completely open particles, but their particle size is less than 10 μm.

The results of mineralogical analysis showed that the ore of the Shalkiya deposit is very likely to be difficult to beneficiation. Mainly, it is caused by very fine embedding of particles of valuable minerals, significant number of clusters and, to a lesser degree, the presence of carbonaceous material. When grinding to the size required for the liberation of clusters, the reciprocal activation of galena and sphalerite surface is very likely, which can lead to deterioration of the selective flotation process. The direction of further research should be chosen taking into account the use of new highly selective reagents and the development of combined beneficiation schemes.

To assess the quantitative characteristic of recoverable sufficiently rich in galena clusters we carried out experiments on preliminary beneficiation of ore after coarse crushing to the size of -0.25 mm by gravity method on the “СВШ-500” spiral separator. The indicators are given in **Table 7**.

We managed to recover 6.16% of lead (product content 30.61%) into gravity concentrate with a yield of 0.27%, but

Table 6
Distribution of minerals by particle size (according to MLA data)

Mineral	Distribution (%) by particle size (μm)								
	up to 5	5–10	10–20	20–44	44–74	74–100	100–250	250–500	over 500
Galena	33.94	16.23	12.67	13.51	7.91	4.03	8.38	3.33	–
Sphalerite	15.01	18.86	16.38	14.71	11.50	7.30	13.15	3.09	–
Sulfides (FeS_2, $CuFeS_2$, $Cu_{12}As_4S_{13}$)	17.75	22.00	17.13	18.72	13,97	8.95	1.48	–	–
Smithsonite	8.14	10.23	17.07	0.92	–	40.35	23.29	–	–
Quartz	3.58	6.55	8.00	10.63	11.43	10.10	40.38	9.14	0.19
Carbonates	9.85	13.10	14.41	14.38	10.09	7.31	25.30	5.56	–
Mica and clay minerals (muscovite, kaolinite, chlorite)	21.29	20.59	13.43	11.50	10.09	6.20	15.69	1.21	–
Iron hydroxides	8.63	14.82	19.13	10.23	31.00	5.60	10.59	–	–
Other barren minerals	12.98	20.84	27.44	25.96	5.52	2.88	4.38	–	–

Table 7
Results of preliminary beneficiation at the spiral separator

Product	Yield, %	Content, %		Recovery, %	
		Zn	Pb	Zn	Pb
Concentrate	0.27	10.51	30.61	0.59	6.16
Middlings 1	10.07	10.97	2.16	22.66	15.98
Middlings 2	50.19	3.76	1.04	38.73	38.21
Light suite	39.46	4.70	1.37	38.02	39.65
Initial ore	100.00	4.88	1.36	100.00	100

Table 8
Flotation results

Product	Yield, %	Content, %		Recovery, %	
		Pb	Zn	Pb	Zn
Main Pb concentrate	12.67	4.38	4.35	52.75	14.84
Control Pb concentrate	6.38	2.42	5.03	14.71	8.65
Main Zn concentrate	15.31	0.82	14.89	11.89	61.45
Control Zn concentrate	4.41	0.62	8.15	2.59	9.69
Tailings	61.23	0.31	0.32	18.06	5.36
Initial ore	100.00	1.05	3.71	100.00	100.00
Total lead concentrate	19.05	3.72	4.58	67.46	23.50
Total zinc concentrate	19.72	0.78	13.38	14.48	71.14

Table 9
Reagent flotation mode

Process	Reagents, consumption (g/t)
Grinding up to 88% of the 0.074 mm class	CaO (150); Na_2S (200); $ZnSO_4$(150)
Main lead flotation (pH = 9), time – 10 min.	CaO (30); Na_2SiO_3 (150); Na_2S (400); FlotentGL3G (reagent substitute of cyanide (NaCN), 450); Basf DP-OMC-1078 (carbonaceous material depressor, 150); $ZnSO_4$ (500); Potassium butylxanthate (150); Oxal (30)
Control lead flotation, time – 7 min.	CaO (10); Na_2SiO_3 (50); Na_2S (200); FlotentGL3G (50); Basf DP-OMC-1078 (30); $ZnSO_4$ (60); Potassium butylxanthate (5); Oxal (5)
Basic zinc flotation (pH = 10.5), time – 8 min.	CaO (400); $CuSO_4$ (700); Potassium butylxanthate (80); Aeroflot (20)
Control zinc flotation, time – 10 min.	CaO (500); $CuSO_4$ (100); Potassium butylxanthate (70); Aeroflot (20)

zinc recovery was only 0.59% (at 10.51% content). Almost the same zinc content was observed in the first middlings (10.97%) with a recovery of 22.66%. Thus, it is shown low efficiency of processing lead-zinc ore, crushed to a particle size of -0.25 mm, using gravity pre-beneficiation on the "СВШ-500" spiral separator, but the feasibility of gravity beneficiation should be proved or disproved in further experiments on beneficiation of Shalkiya deposit ore and technical and economic calculations, taking into account the complexity of the technological scheme, the need for additional equipment, etc.

Experiments were also carried out according to the direct selective flotation scheme to assess the quantitative characteristic of lead and zinc recovery into the respective concentrates and to estimate the level of losses of target metals with flotation tailings. The results of preliminary flotation studies are shown in **Table 8**, the reagent mode — in **Table 9**.

As a result of flotation of ore crushed to a particle size of 87–88% –0.074 mm, up to 67.46% of lead is extracted in the total lead concentrate, and up to 71.14% of zinc is extracted in the zinc concentrate. Based on the results of the analysis of the material composition, gravity and flotation studies it is proposed to use the scheme of direct selective flotation for beneficiation of lead-zinc ores of Shalkiya deposit.

Conclusion

The main components in the studied sample of lead-zinc ore are silicon oxide – 40.15%, calcium oxides – 19.21% and magnesium 8.71%. The main valuable components of commercial interest in this raw material are lead (content 1.21%) and zinc – 4.13%. According to the results of sieve analysis, the content of lead and zinc increases with a decrease in the particle size of the ore material. The beginning of galena liberation is observed at the –0.25+0.1 mm class and has a bimodal distribution with peaks at the –0.074+0.044 and –0.010+0 mm classes. For sphalerite, we can note a unimodal distribution with a peak at the particle size class –0.044+0.020 mm.

According to MLA data, the main element-concentrating minerals are:

– zinc: sphalerite and carbonates, 87.44% and 8.04% distribution, respectively;

– lead: galena and carbonates, 86.99% and 10.64% distribution, respectively.

The fraction of galena in the free particles is 47.42%, sphalerite – 39.52%. The particle sizes of galena and sphalerite in free particles are less than 5 and 10 microns, respectively, which indicates a low perspective of separation by gravity methods, which was confirmed by the results of beneficiation on the spiral separator.

A high fraction of galena and sphalerite has a completely open surface, with most of the particles being less than 10 μm in size. A complex analysis of the material composition of the Shalkiya deposit ores made it possible to characterize them as hard-to-beneficiation ores.

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