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Article

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Study of the enrichment of refractory oxidized lead ores from tailings from mining plants via combined electrochemical polarization

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Abstract

Man-made oxidized nonferrous metal ores accumulate in large quantities in mining plants, causing environmental problems and polluting the soil, groundwater, and atmosphere with toxic heavy metals. Traditional flotation is often ineffective and fails to yield acceptable levels of nonferrous metal recovery. This study aims to use refractory oxidized lead ore electrochemical enrichment to increase lead recovery into flotation concentrates to 93–95% from mine plant tailings. Cerussite is the primary lead mineral in the ore. Cathodic voltammetry revealed that cerussite reduction occurs partially through decomposition into Pb^{2+} ions and through direct solid-phase discharge of mineral particles to elemental lead. To convert the mineral cerussite to an easily floatable sulfide form, the traditional flotation reagent sodium sulfide was used. Under microelectrolysis conditions in a weakly alkaline solution of sodium sulfide, lead cations formed during the solid-phase decomposition of cerussite interact with sulfide ions and form lead sulfides. The electrode mechanisms of cerussite mineral sulfidation were used for the electrochemical enrichment of oxidized lead ore. Sulfidization of ore from the Alaigyr deposit in the Republic of Kazakhstan was carried out in a special electrolyzer with a pulp preparation apparatus. A multifactorial model of lead recovery into sulfide concentrate was obtained, indicating that a high degree of recovery of 98.56% was achieved under the experimentally determined optimal conditions.

Keywords: Oxidized lead ore, Cerussite, Sulfide concentrate, Linear potential sweep voltammetry, Electrochemical enrichment, Flotation.

Introduction

Today, global environmental problems are caused by man-made oxidized nonferrous metal ores accumulated in storage piles of mining plants that pollute the soil, groundwater, and atmosphere with toxic heavy metals due to dust emissions. Most large nonferrous metal deposits of the traditional sulfide type have been largely depleted worldwide. Therefore, processing low-grade, unconventional oxidized ores is a pressing issue. In upper crustal zones**,** sulfide minerals are spontaneously oxidized, leading to oxidized ores in the upper horizons of deposits. The enrichment of nonferrous ores directly depends on the mineralogical composition of the raw materials. Ores with oxidation states of 60–90% are composed primarily of refractory minerals**,** such as oxides, silicates, basic sulfates, and carbonates**,** which significantly complicate their processing **via** conventional enrichment methods. Consequently, such ores accumulate in large volumes over long periods of time and are stored in mining plants**,** which creates environmental problems. The global concept of rational, integrated use of mineral resources presupposes the widespread involvement of man-made materials in the development of these ores. Ores of industrial interest are the tailings of the Alaigyr lead deposit from mining plants in the Republic of Kazakhstan, where oxidized forms of lead minerals predominate in the upper overburden layers, accounting for 85%. The existing methods of processing refractory oxidized lead ores include flotation[1-4], chemical[5] and biological leaching[6], high-temperature sulfiding roasting[7-9], and electrochemical flotation pulp activation[10-11]. The disadvantages of the abovementioned processing methods include the following:

- Under direct flotation enrichment conditions, as the degree of oxidation increases, lead recovery decreases to 40-60%;
- Leaching methods using inorganic and organic acids are unacceptable because of the multistage process flow, equipment requirements, high temperatures, and energy consumption for heating the pulp;
- Biological leaching methods are characterized by limited capacity, lengthy processing durations, and high costs;
- The use of high-temperature sulfiding roasting for ores characterized by a low content and a variety of oxidized forms of lead minerals is energy intensive and requires a complicated dust collection system;
- A well-known electrochemical method of activating the flotation pulp of sulfide ores provides virtually no increase in recovery from refractory oxidized ores.

Electrochemical methods for preparing refractory oxidized ores for flotation are promising[12]. The main advantage of electrochemical sulfidization is the simplified process that is carried out at low temperatures while ensuring a high degree of nonferrous metal recovery into the concentrate. The adoption of electrochemical polarization increases ore leaching and the transition of poorly soluble metal oxides into solution at low solvent concentrations. Electrochemical sulfidization facilitates the conversion of oxidized minerals into an easily floatable form. There is a lack of literature on effective electrochemical methods for enriching oxidized lead ores with a high degree of lead recovery into the concentrate.

This study aims to use refractory oxidized lead ore electrochemical enrichment to increase lead recovery into flotation concentrates to 93–95% from mine plant tailings.

Materials and methods

To understand the behavior of the electrochemical polarization of refractory lead ore, the properties of its main component, cerussite PbCO3, were studied under linear potential sweep voltammetry conditions. Voltammmetric measurements were carried out on an Autolab PGSTAT302N potentiostat/galvanostat from the Metrohm (Netherlands) using a specially designed electrode for studying ore monominerals[12].

The phase compositions of the oxidized monomineral cerussite, lead sulfide, original oxidized lead ore, and sulfide concentrate were determined via X-ray diffraction analysis on an Empyrean Malvern Panalytical X-ray diffractometer. The diffractometer is equipped with an X-ray tube and an anode material of Cu (Kα1 = 1.541874 Å). Measurements were conducted at room temperature in the 2θ range from 0° to 90° in step-scan mode with a resolution of 0.013 degrees.

Electron microscopy studies of the above objects were conducted on a ZEM20 (ZepTools, China) 2024 scanning electron microscope with an Oxford energy-dispersive spectroscopy attachment (Oxford Instruments, UK). Figure 1 shows a Pb distribution map, the total spectrum of the map and the X-ray diffraction pattern of the monomineral cerussite, with a major component content of 95.7%. According to electron microscopic analysis, the lead content of cerussite is 58.08%.

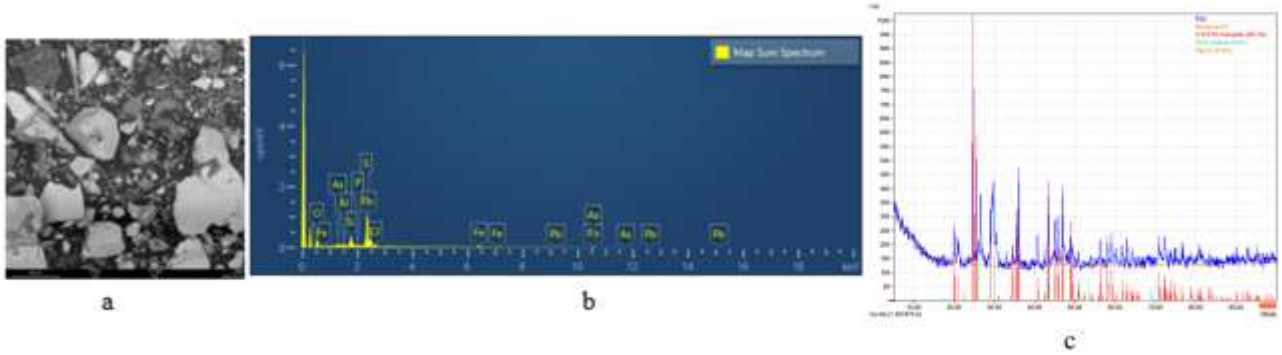

Figure 1. Lead distribution map in a sample of the natural mineral cerussite (a), map total spectrum (b) obtained via a ZEM20 scanning electron microscope, and X-ray diffraction pattern of the cerussite (c)

Microelectrolysis at a controlled potential was used to convert cerussite to lead sulfide.

A representative sample of oxidized refractory lead ore from the Alaigyr deposit in the Republic of Kazakhstan was selected as the object for electrochemical sulfidization. The chemical composition of the ore, wt.% was as follows: total lead 3.82; oxidized lead 3.12; copper 0.016; zinc 0.027; total sulfur 0.80; iron 1.13; bismuth 0.005; nickel 0.005; cobalt 0.005; molybdenum 0.005; sodium oxide 0.47; silicon oxide 70.58; calcium oxide 0.390; aluminum oxide 13.20; magnesium oxide 0.49; barium sulfate 0.33; manganese oxide 0.027; potassium oxide 8.70. According to the results of the X-ray phase and chemical analyses of the ore material composition, the main oxidized lead mineral is cerussite. Nonmetallic minerals are mainly represented by quartz.

The galvanostatic electrolysis method was used for preliminary electrochemical polarization of oxidized lead ore before flotation. To prepare the pulp, a sample of crushed ore (grade -0.074 mm in 70% ore) was mixed with the alkaline electrolyte solution at a liquid:solid ratio of 3:1. Sodium sulfide was used as a sulfidizing agent that was introduced into the pulp to promote the formation of easily floatable lead sulfides. Electrochemical polarization of the ore pulp was performed via a setup consisting of a pulp preparation apparatus and an electrolyzer (Figure 2).

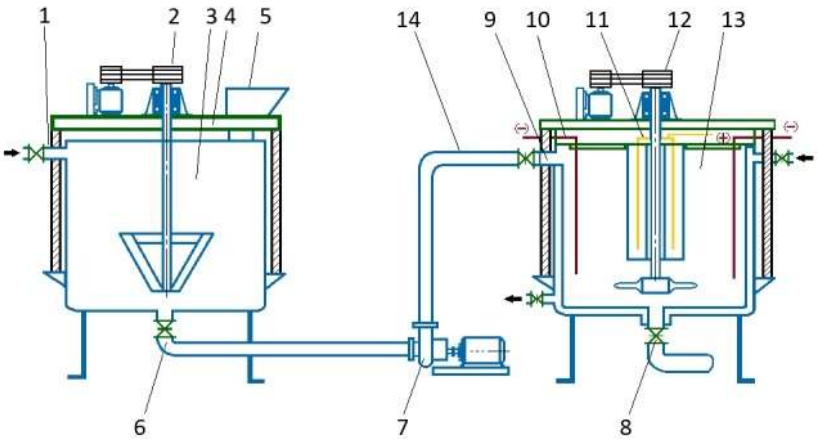

Figure 2. Schematic diagram of the setup of a thermostatically controlled electrolyzer with a pulp preparation apparatus for the oxidized ores enrichment: 1 - branch pipe with a valve for the feeding electrolyte; 2 - stirring device with a drive (stirrer); 3 - ore pulp preparation tank with a lid; 4 - device for measuring and controlling the volume of liquid in the apparatus; 5 - special bin for loading dry mixture (ore, sulfidizer); 6 - valve for discharging pulp; 7 - pump for pumping the pulp; 8 - valve for discharging the pulp; 9 - branch pipe for connecting the pipeline; 10 - cathode (stainless steel); 11 - anode (lead); 12 - stirring device with a drive; 13 - electrolyzer tank with a lid; 14 - pipeline.

To prepare the pulp, the calculated amount of alkaline electrolyte was initially pumped through pipe (1) into the pulp preparation apparatus (3), and then the stirring mixer (2) was turned on. The required amounts of crushed ore (class -0.074 mm in 70% ore) and sodium sulfide sulfidizing agent were loaded into the apparatus through a special bin (5). The pulp preparation apparatus mixer was operated until the electrolyte was completely mixed with the crushed ore and sulfidizing agent. Then valve (6) was opened and the pulp was pumped through pipeline (14) via pump (7) through pipe (9) into the electrolyzer tank (13). The stirring device of stirrer (12) of the electrolyzer (13) was started after the valve of the pulp preparation apparatus (6) was closed and the pump (7) was switched off. After the electrolyzer stirrer (12) was started, before the electrodes were connected to the power source, the correct pole signs (+) of the anode (11) and (-) of the cathode (10) were checked. Current was then applied to the electrodes from an AC rectifier. At the end of the calculated electrochemical sulfidation duration, the current to the electrodes was disconnected, with the stirrer (12) running, the valve (8) was opened, and the pulp was pumped into the flotation machine. After the pulp, was pumped, the valve (8) was closed and the electrolyzer stirrer (12) was turned off. The pulp was fed to the laboratory flotation machine of the F237 FL-A brand. Then flotation was carried out with the consumption of 120 g t^{-1} butyl xanthate flotation reagent and 80 g t^{-1} T-92 frother. The duration of the main flotation was 8 minutes, and that of the control was 11 minutes.

Seidel-Gauss method of traditional experimental design was used to establish optimal conditions for the preliminary electrochemical sulfidation of oxidized lead ores prior to flotation.

Results and discussion

Voltammetric measurements

Voltammetric measurements of mineral cerussite were conducted in sodium hydroxide solutions using stainless steel and lead electrodes at room temperature. Figure 3 shows that the voltammetric curves during cerussite reduction exhibit only a single wave, the potential of which varies slightly depending on the electrode substrate material and ranges from -1.0 to -1.1 V. The surfaces of all electrodes at the point of contact with the test sample were coated with a black precipitate of freshly formed elemental lead.

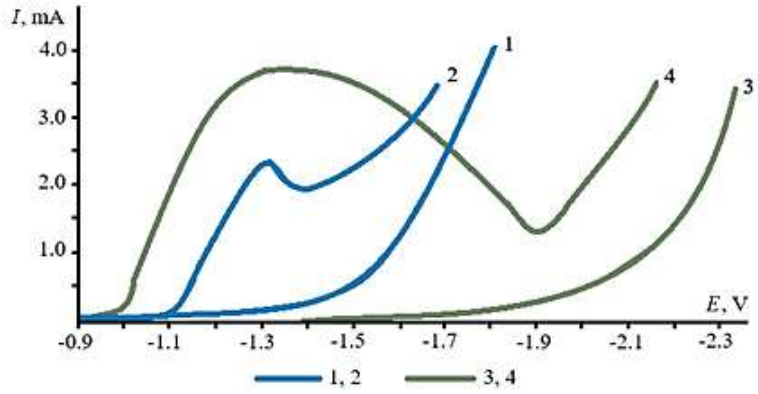

Figure 3. Cathodic polarization curves of the monomineral cerussite sample on steel (2) and lead (4) electrodes in alkaline solution, 20 g L^{-1}; 1 and 3 background curves.

According to work[13], the reduction of solid metal salts or oxides occurs via the following electrode reactions: direct discharge of the solid particles (mechanism I); the electron carrier is an intermediate substance formed at the cathode (mechanism II); the solid particles undergo decomposition into cations and anions that are then reduced at the electrode (mechanism III). The voltammogram shapes (Figure 3, curves 2 and 4) revealed that the peaks of the limiting current drop sharply at potentials of -1.3 to -1.4 V. This usually occurs when the near-electrode layer is significantly depleted of the electroactive substance. It can be concluded that cerussite reduction occurs via mechanisms III and I.

To convert the mineral cerussite into an easily floatable sulfide form, the traditional flotation reagent sodium sulfide was used. Under microelectrolysis conditions of automatic maintenance at a given potential, a sample of the monomineral was polarized in a weakly alkaline sodium sulfide solution on a steel electrode. The potential was set

according to the voltammetric measurements (Figure 3). After microelectrolysis, the product was rinsed with distilled water from the electrolyte solution and dried. Electron microscopy and X-ray diffraction analysis confirmed that the microelectrolysis product was lead sulfide (Figure 4).

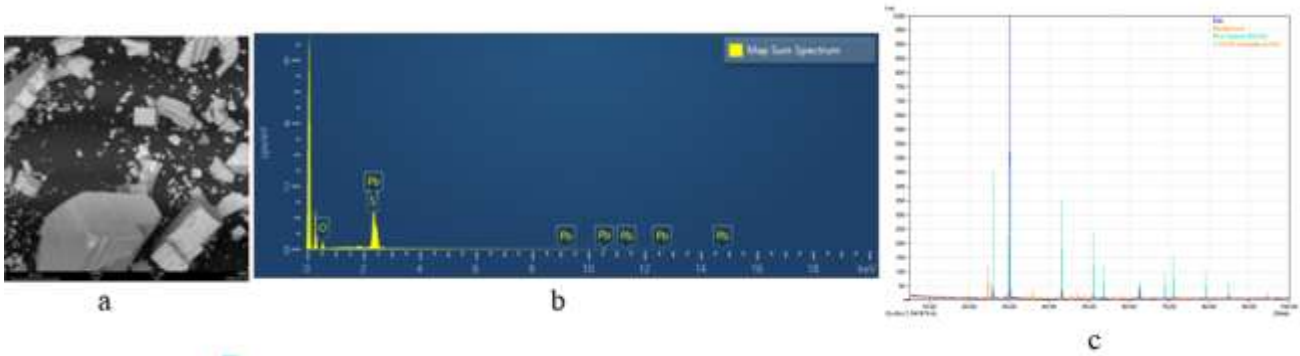

Figure 4. Distribution map of lead sulfide obtained by microelectrolysis (a) and its map total spectrum (b) obtained via a scanning electron microscope, and an X-ray diffraction pattern (c) of PbS

During microelectrolysis, solid particles of cerussite decompose into lead cations (mechanism III), which interact with sulfide ions and form lead sulfide according to the following reaction:

$$Pb^{2+} + S^{2-} \rightarrow PbS. \quad (1)$$

The established electrode mechanisms of cathodic reduction and sulfidation of the mineral cerussite with sodium sulfide were used for the electrochemical enrichment of oxidized lead ore. Under direct flotation conditions, lead recovery into the concentrate from the ore under study is only 40–60%.

Electrochemical enrichment

The results of electron microscopy, X-ray phase and chemical analysis show that the studied ore, in addition to the oxidized minerals anglesite and plumboyarosite, contains finely disseminated refractory mineral cerussite (Figure 5).

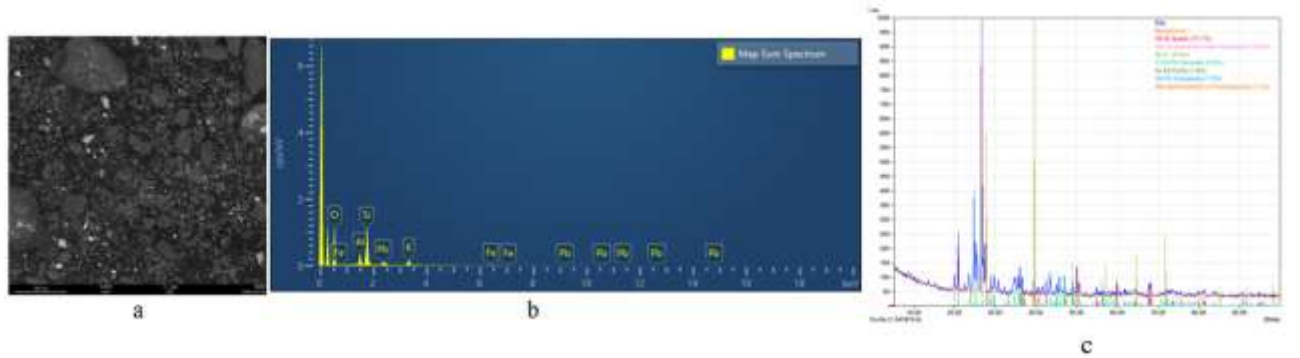

Figure 5. Lead distribution map in a sample of the initial oxidized lead ore of the Alaigyr deposit (a) and the map total spectrum (b) obtained via a scanning electron microscope, and an X-ray diffraction pattern (c) of the sample

The essence of the electrochemical enrichment process is that during cathodic polarization, the reduction of dispersed oxidized minerals such as cerussite ($PbCO_3$), anglesite ($PbSO_4$) and plumboyarosite ($Pb_{0,5}Fe^{3+}{}_3(SO_4)_2(OH)_6$ or the doubled formula $PbFe_6(SO_4)_4(OH)_{12}$) can occur partially through decomposition into Pb^{2+} ions and through the stage of direct solid-phase discharge of mineral particles to elemental lead:

cerussite:	$PbCO_3 + 2e \rightarrow Pb^0 + CO_3^{2-}$	(2)
anglesite:	$PbSO_4 + 2e \rightarrow Pb^0 + SO_4^{2-}$	(3)
plumboyarosite:	$½\ Pb(OH)_2 + e \rightarrow ½\ Pb^0 + OH^-$	(4)

The lead ions then react with the sulfidizing agent sodium sulfide to form easily floatable lead sulfide (reaction 1).

Electrochemical enrichment of the oxidized lead ore was carried out in the electrolyzer (Figure 2). Using the Seidel–Gauss method[14], the effects of the current density (i, 25-150 A m^{-2}), alkali concentration (C_{NaOH}, 3-24 g L^{-1}), sodium sulfide concentration (C_{Na_2S}, 0-0.9 g L^{-1}), electrolyte temperature (t, 25-65 °C), and electrolysis duration (τ, 20-120 min) on lead recovery into the concentrate were studied. Partial dependencies were constructed (Figure 6). The approximating functions were selected considering the physical meaning of the dependencies studied. Figure 6a shows

that high lead recovery rates are achieved at current densities of 50 A m^{-2} and higher. The degree of lead recovery into the sulfide concentrate reached a maximum value of 96.8% at a sodium hydroxide concentration of 24 g L^{-1} (Figure 6b). The concentration of sodium sulfide, the sulfidizing agent (Figure 6c), has a significant effect on lead recovery and tends to be saturated. The most complete sulfidization of lead minerals occured with a sodium sulfide concentration of 0.60 g L^{-1}. With this concentration, the amount of sulfidizing agent is sufficient to sulfidize all the oxidized lead minerals. Increasing the temperature (Figure 6d) accelerated the rate of reduction of oxidized minerals and their interaction with sodium sulfide, resulting in the formation of easily floatable lead sulfide. In the range of electrolysis duration of 30–120 min (Figure 6e), lead recovery increases consistently, which means that the process of lead sulfide formation is complete.

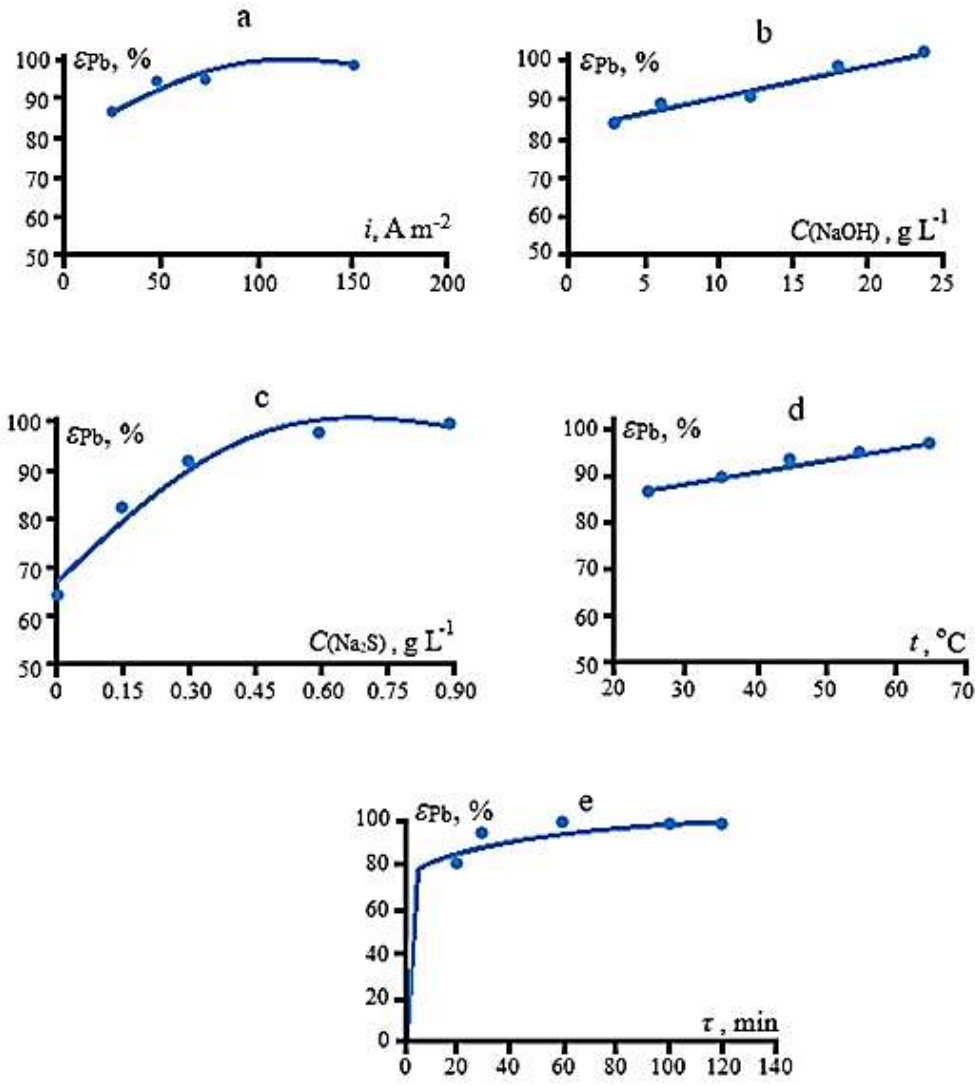

Figure 6. Lead recovery as a function of various factors: a – current density; b – sodium hydroxide concentration; c – sodium sulfide concentration; d – electrolyte temperature; e – electrolysis duration.
Dots – experimental data; line – approximating function

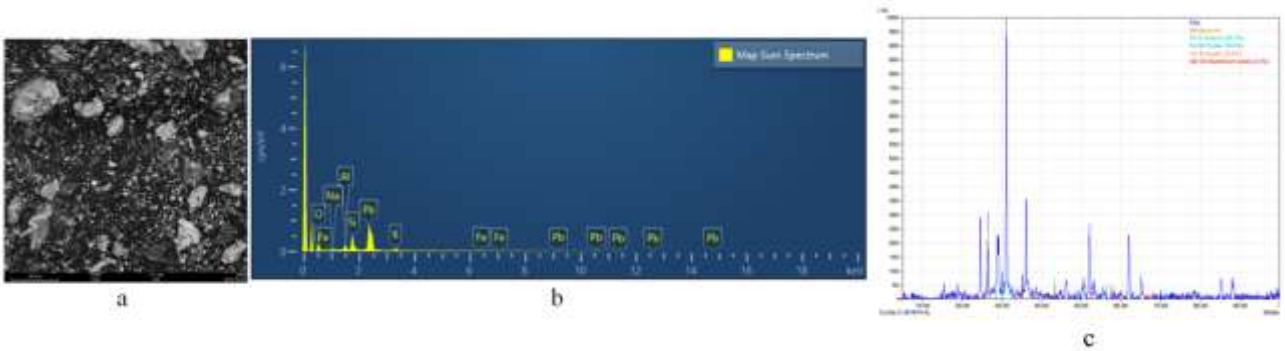

Figure 7. Lead distribution map in a sample of the obtained sulfide lead concentrate (a) and the map total spectrum (b) obtained via a scanning electron microscope, and an X-ray diffraction pattern (c) of the sample

Correlation coefficients and their significances were calculated for all the obtained partial dependences (Table 1).

Table 1. Correlation coefficient R and its significance t_R for partial functions

Equation	t_R	R	Significance
$\mathcal{E}_{Pb} = -0.0012i^2 + 0.3089i + 79.943$	8.10>2	0.966	significant
$\mathcal{E}_{Pb} = 0.788\, C_{NaOH} + 79.804$	167.4>2	0.994	significant
$\mathcal{E}_{Pb} = -25.656\, C^2_{Na_2S} + 59.512 C_{Na_2S} + 66.193$	264.2>2	0.987	significant
$\mathcal{E}_{Pb} = 0.3092t + 79.212$	53.7>2	0.984	significant
$\mathcal{E}_{Pb} = 66.823\tau^{-12}$	183.5>2	0.994	significant

The resulting equations for lead recovery into sulfide concentrate (Table 1), taking into account the significant functions for describing the set of factors involved, were generalized according to[14] for conditions common to all the functions (i = 150 A m^{-2}, C_{NaOH} = 24 g L^{-1}, C_{Na_2S} = 0.6 g L^{-1}, τ = 60 min, t = 65 °C) for the value of $\mathcal{E}_{Pb}$ = 98.56%. The generalized equation was then expressed as follows:

$$\varepsilon_{Pb} = \frac{(-0.0012\, i^2+0.3089\, i+79.943)(0.788C_{NaOH}+79.804)(0.3092t+79.212)}{8873769594\left[(-25.656\, C^2{}_{Na_2S} + 59.512\, C_{Na_2S} + 66.193)(66.823\tau^{-12})\right]^{-1}} \quad (5)$$

The comparison with the data calculated directly by substituting experimentally determined values yielded R = 0.96 and t_R = 61.64>2, σ_{abs} = 0.32%. The confidence interval calculated via t_R[14] was ±0.55%.

The sufficiently high accuracy of the resulting generalized equation allowed us to carry out kinetic analysis[15]. Since this requires taking a partial derivative with respect to the duration (τ), i.e., the remaining variables are constant, this allows them to be designated in equation (5) along with the other coefficients by the common value:

$$K = \frac{66.823(-0.0012\, i^2+0.3089\, i+79.943)(0.788C_{NaOH}+79.804)(0.3092t+79.212)}{8873769594\left[(-25.656\, C^2{}_{Na_2S} + 59.512\, C_{Na_2S} + 66.193)(66.823\tau^{-12})\right]^{-1}}$$

The generalized equation is expressed as follows:

$$\varepsilon_{Pb} = K \cdot \tau^{-12}. \quad (6)$$

Replacing the original equation τ with $\mathcal{E}_{Pb}$, the following expression is obtained:

$$\tau = \left(\frac{\varepsilon_{Pb}}{K}\right)^{12}. \quad (7)$$

Substituting expression (7) into equation (6), the following rate equation is obtained:

$$\frac{d\varepsilon_{Pb}}{d\tau} = \frac{K}{12}\left[\left(\frac{\varepsilon_{Pb}}{K}\right)^{12}\right]^{-\frac{11}{12}} = \frac{K}{12}\left(\frac{\varepsilon_{Pb}}{K}\right)^{-11} \quad (8)$$

The resulting process rate model can be processed with any combination of various factors. All isotherms were calculated for a fixed lead recovery value of 52%. The process rate values obtained from equation (8) for five temperatures (25, 35, 45, 55, and 65°C) were converted into Arrhenius coordinates $\ln\frac{d\varepsilon_{Pb}}{d\tau} - T^{-1}$, with a linear relationship. Using this relationship, the apparent activation energy was determined.. The obtained apparent activation energy is 33.405 kJ mol^{-1}, which indicates a transient process; that is, the diffusion rate and the reaction rate are approximately comparable.

Conclusions

Experimental studies indicate that oxidized lead minerals (cerussite, anglesite and plumboyarosite) can be converted into easily floatable lead sulfide by electrochemical polarization in a weakly alkaline solution of sodium sulfide.
Cerussite is the primary lead mineral in refractory oxidized lead ore. The mechanism of cerussite solid-phase reduction followed by its sulfidation was in detail via studied by linear voltammetry and microelectrolysis at a controlled potential methods. The essence of the electrochemical enrichment process is that during cathodic polarization, the reduction of cerussite, anglesite and plumboyarosite can occur partially through decomposition into Pb^{2+} ions and through the stage of direct solid-phase discharge of mineral particles to elemental lead. The lead ions then react with the sulfidizing agent sodium sulfide to form easily floatable lead sulfide. The electrode mechanisms of cerussite mineral sulfidation were used for the electrochemical enrichment of oxidized lead ore. Sulfidization of ore from the Alaigyr deposit in the Republic of Kazakhstan was carried out in a special electrolyzer with a pulp preparation apparatus. It indicated that the high recovery rate of 98.56% was achieved at current densities of 100-150 A m^{-2}, with the addition of sodium sulfide (0.6-0.9 g L^{-1}) as a sulfidizing agent, sodium hydroxide concentration of 20-24 g L^{-1}, and electrolysis duration of 30-40 minutes. A multifactorial model of lead recovery into sulfide concentrate and a generalized equation combining partial dependencies were obtained. A kinetic analysis of the generalized equation was used to calculate the apparent activation energy. The thermodynamic calculations are in good agreement with the mechanism of cerussite solid-phase reduction and interaction with the sulfidizer. The phase composition of cerussite, the original oxidized lead ore, the products of electrochemical enrichment, lead sulfide and sulfide concentrate, was determined via X-ray diffraction and electron microscopic analysis.

Author contributions statement

M.D. and I.F. conceived the project, designed the experiments and wrote the manuscript. A.J. and D.D. performed the experiments. All the authors discussed the results.

Additional Information

Competing Interests: The authors declare that they have no competing interests.

Data availability statements

The authors declare that the data supporting the findings of this study are available within the paper. Should any raw data files be needed in another format they are available from the corresponding author upon reasonable request. Source data are provided with this paper.
No datasets were generated or analysed during the current study.

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