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Original Article

Hydrometallurgical Decomposition of Zinc Sulfide Concentrate Supplied by JSC Svyatogor, Russia, via High-Temperature Sulfuric Acid Leaching with Hydrogen Sulfide Recovery

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Abstract

This study investigates the hydrometallurgical decomposition of zinc sulfide concentrate supplied by JSC Svyatogor through high-temperature sulfuric acid leaching with simultaneous hydrogen sulfide recovery. The feed contained 45.9 wt.% Zn, and X-ray diffraction showed sphalerite and pyrite as the main minerals. Leaching tests were carried out in a closed reactor using 50 g of concentrate, sulfuric acid solutions of 30–75 wt.%, an L/S ratio of 15:1, and a total leaching time of 3 h. The evolved hydrogen sulfide was absorbed in an alkaline solution, while elemental sulfur in the leach residue was determined by extraction and gravimetry. Under the optimum conditions, zinc extraction into the sulfate solution reached 94.6%, whereas dissolution of copper-, arsenic-, and pyrite-bearing phases remained negligible. Based on these results, we propose a flowsheet that includes sulfuric acid leaching, high-temperature crystallization of zinc sulfate hydrate in pulp, filtration, and redissolution of washed zinc sulfate monohydrate crystals. The process provides a potential route for producing commercial zinc sulfate and recovering hydrogen sulfide as a valuable byproduct.

.**Keywords:** Zinc sulfide concentrate; High-temperature sulfuric acid leaching; Hydrogen sulfide recovery; Zinc sulfate

1. Introduction

Zinc concentrates are traditionally processed using a technology based on roasting followed by leaching of the calcine. Metallic zinc is then recovered from the resulting zinc-bearing solution by electrowinning (Kliain, 2009). At present, only one zinc plant is operating in Russia, namely Chelyabinsk Zinc Plant JSC (CZP, Chelyabinsk). The roasting capacity of this plant is limited; therefore, the development of hydrometallurgical technologies for opening up zinc concentrates is of considerable interest, particularly since their principal valuable component is the mineral sphalerite.Sulfuric acid oxidative leaching technologies for zinc concentrates, which occur with the supply of oxygen or air, are well known (Svens et al., 2005). The decomposition process in an oxidizing atmosphere proceeds according to the following scheme (Kurniawati et al., 2024; Song et al., 2018; Karimi et al., 2021):

$$ZnS+Fe_2(SO_4)_3=ZnSO_4+FeSO_4+S^0;\downarrow$$

$$ZnS+H_2SO_4+1/2\ O_2=ZnSO_4+S^0\downarrow+H_2O.(1)$$

Based on the study of Eh-pH diagrams for the ZnS-H_2 Osystem, it can be suggested that an exchange reaction may occur at elevated temperatures and sulfuric acid concentrations in the absence of oxidizing agents (Mu et al., 2010):

$$ZnS + H_2SO_4 = ZnSO_4 + H_2S \uparrow. (2)$$

Thermodynamic equilibrium calculations for the non-oxidative sulfatization of sphalerite with sulfuric acid were carried out using HSC Chemistry 6.12 (Reaction Equations module) across the temperature interval of 100–180°C (373.15–453.15 K). At the baseline leaching temperature of 150°C(423.15 K), the calculated standard Gibbs free energy change is $\Delta G^{\circ}_{423} = -18.42$ kJ/mol(with ΔG°remaining negative, decreasing from -11.2 kJ/mol at 100°C to -24.8 kJ/mol at 180°C), and the standard enthalpy change is $\Delta H^{\circ}_{423} = +29.74$ kJ/mol. The negative ΔG°_{T}values confirm that the direct exchange reaction is thermodynamically spontaneous across the entire operational temperature window under continuous removal/scrubbing of gaseous H_2S. Therefore, this reaction is thermodynamically feasible. Interest in reaction (2) is driven by the possibility of recovering hydrogen sulfide as a valuable byproduct. Hydrogen sulfide can be used to prepare sodium sulfide or sodium hydrosulfide solutions, which are used as flotation activators.

Aim of the Study: To investigate the feasibility of decomposition (decomposition/leaching) of ZnS concentrate according to reaction (2).

2. Materials and Methods

The object of study was a zinc concentrate supplied by JSC Svyatogor, with the following composition (wt.%): Zn – 45.9, S – 34.2, Fe – 8.11, Cu – 2.32, SiO_2– 1.0, Pb – 0.1, As – 0.022; along with Au – 0.86 g/t and Ag – 71 g/t. X-ray diffraction (XRD) analysis performed on a Shimadzu diffractometer revealed that the primary minerals in the concentrate were sphalerite (68 wt.%) and pyrite (17 wt.%). Copper minerals were predominantly represented by chalcopyrite.

The raw material utilized in this investigation was industrial zinc flotation concentrate provided by JSC Svyatogor (Urals, Russia). The chemical composition of the concentrate (determined by ICP-AES and XRF) was: Zn: 45.90 wt% , S: 34.20 wt% , Fe: 8.11 wt% , Cu: 2.32 wt% , Pb: 0.10 wt% , As: 0.022 wt% , Ag: 71.0 g/t , and Au: 0.86 g/t . Mineralogical phase analysis by XRD demonstrated that zinc is primarily hosted in sphalerite (ZnS, ≈ 68 wt%), iron in pyrite (FeS_2, ≈ 17 wt%), and copper predominantly in chalcopyrite ($CuFeS_2$, ≈ 1.5 wt%). The sphalerite leaching process was investigated in sulfuric acid solutions at elevated temperatures. A schematic diagram of the experimental setup is shown in Fig. 1. Batch digestion tests were conducted in a hermetically sealed 500 mLfour-neck borosilicate glass reactor equipped with a Teflon-coated overhead mechanical stirrer (operated at 400 rpm), a PTFE-sheathed Pt100 thermocouple coupled to a PID-controlled external heating mantle (±1°C), and a reflux condenser. The gaseous H_2Sgenerated during the reaction was continuously swept through an intermediate safety trap (to prevent back-suction) into two series-connected gas absorbers packed with glass Raschig rings (6 × 6 mm), each charged with 250 mLof a 10 wt% NaOHaqueous absorbent solution (2.75 M). The scrubbing efficiency surpassed 99.2%, converting off-gas H_2S into sodium hydrosulfide/sulfide ($H_2S + NaOH \rightarrow NaHS + H_2O$ and $NaHS + NaOH \rightarrow Na_2S + H_2O$), suitable for direct reuse as a sulfide mineral activator in flotation.The experimental program was structured into two distinct stages:(i) Parametric and kinetic decomposition runs: To accurately determine the apparent activation energy and the reaction orders without being masked by acid depletion or mass transfer starvation, initial kinetic experiments were conducted under an excess-acid regime with a liquid-to-solid mass ratio (L/S) of 15: 1over a total duration of 3 h. This ensured that the bulk sulfuric acid concentration remained essentially constant ($< 3\%$ variation) throughout each kinetic run.(ii) Technological optimization and cycling experiments: Following the kinetic determination, parametric optimization and 10-cycle locked tests were carried out to establish technologically feasible and industrially viable parameters. In this stage, the L/S ratio was systematically varied from 15: 1down to 3: 1, and the residence time at the target temperature (130–160°C) was reduced from

180 min down to 15 min to maximize volumetric throughput while maintaining high zinc recovery.The experiments were conducted according to the following procedure: A 50 g sample of the concentrate was placed into a 500 cm^3reactor (4) at room temperature. Subsequently, a measured volume of a 30–75 wt.% H_2SO_4solution was dosed into the sealed reactor from a constant-head tank (1) using a peristaltic pump (2). Mixing was provided by a magnetic stirrer. The liquid-to-solid (L/S) mass ratio was kept at 15:1, ensuring that the H_2SO_4concentration remained virtually constant during the experiment. The slurry was then heated to the target temperature over 30 min. The total leaching duration was 3 h. Upon completion of the experiment, the resulting reaction mixture was diluted with a large volume of water, filtered, and thoroughly washed. The washings and filtrate were combined, and their total volume was measured. The residue yield, zinc concentration in the liquid phase, and zinc content in the unreacted solid residue were determined, and the zinc extraction into solution was calculated. If required, the hot reaction mixture was filtered using a synthetic filter cloth.

During the experiment, the evolved hydrogen sulfide was absorbed by an alkaline solution in absorber (6). To prevent mixing (or back-siphonage) of the alkaline solution with the reaction mixture, a safety bottle (5) was placed between them. Upon completion of the experiment, the residual hydrogen sulfide was purged with air using a laboratory air compressor (3).In the resulting leach cake, the formed elemental sulfur was quantified using an acetone extraction method, followed by acetone distillation and gravimetric weighing of the precipitated elemental sulfur (Busev, 1975). The concentration of sulfide ions in the alkaline solution was determined by a method that involves solution acidification, precipitation of ZnS or CdS, and subsequent iodometric titration of sulfur in the precipitated sulfide (Lurie, 1984). Based on the analytical results, the yields of elemental sulfur and hydrogen sulfide per unit mass of reacted zinc sulfide were calculated.

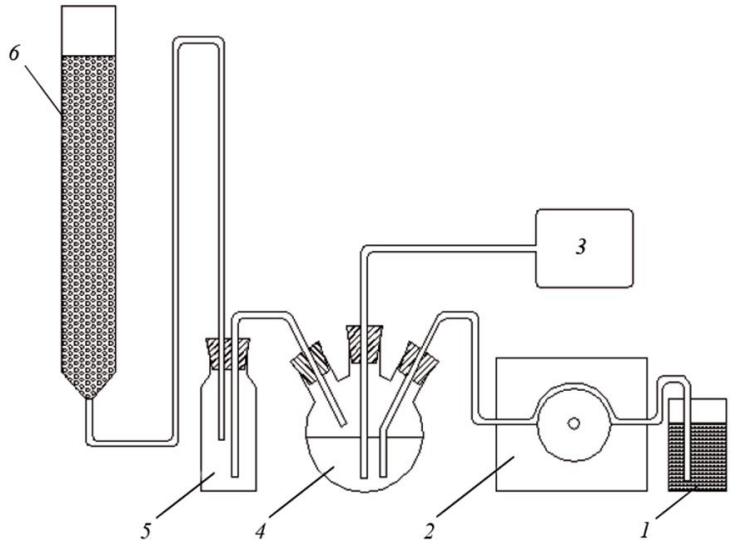


Figure 1. Schematic diagram of the experimental apparatus for zinc concentrate sulfuric acid digestion: 1 – compressed air feed; 2 – flow rotameter; 3 – three-way valve; 4 – 500 mLjacketed/heated glass reactor with mechanical stirrer and temperature sensor; 5 – intermediary safety buffer trap; 6 – gas absorption column packed with glass Raschig rings charged with 10 wt% NaOH aqueous solution for H_2S scrubbing."

3. Results

During the experiments, it was observed that sphalerite decomposition proceeds intensively, accompanied by the evolution of a significant amount of hydrogen sulfide.

The dependence of zinc extraction into the aqueous phase on temperature is illustrated in Fig. 2. In the temperature range of 80 to 200 °C, this dependence exhibits an extremal behavior. With an initial sulfuric acid concentration of 65 wt.%, the maximum extraction reaches 93.05–95.65% within the temperature interval of 130–160 °C, respectively. High zinc extraction,

comparable to traditional technologies (Kliain, 2009; Svens et al., 2005), is achieved over a wide temperature range.

According to reaction (2), the theoretical mass ratio of evolved hydrogen sulfide to reacted zinc should be 0.52. The experimentally determined yields of hydrogen sulfide and elemental sulfur as a function of temperature are presented in Figure 2b.

The data indicate that in the temperature range of 80–180 °C, the hydrogen sulfide yield reaches a mass fraction of 0.49–0.50 (remaining practically constant), which represents 94–96% of the theoretical value. As the temperature increases to 180 °C and above, a noticeable evolution of elemental sulfur is observed (up to a mass fraction of 0.04–0.09), accompanied by a decrease in the hydrogen sulfide yield to 0.48–0.45 mass fraction (92.3–86.5% of the theoretical yield).

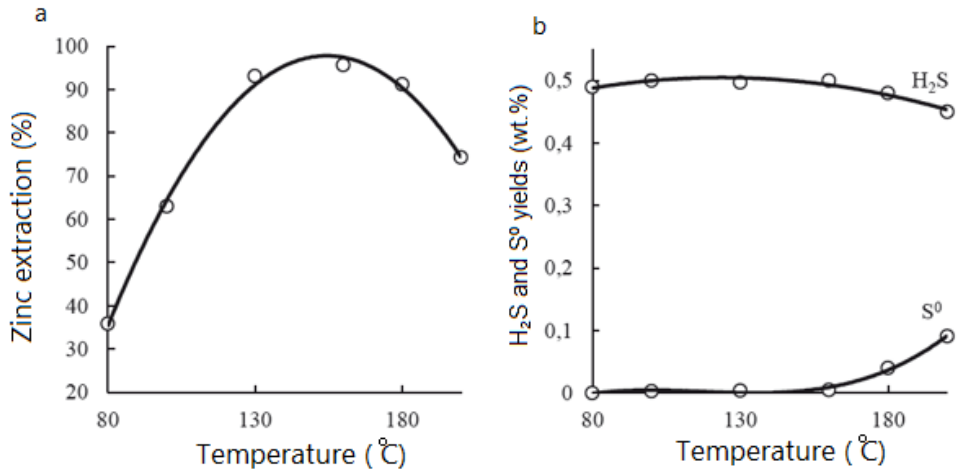


Figure 2. Effect of temperature during leaching with 65 wt.% H_2SO_4on: (a) zinc extraction from the concentrate; and (b) the yields of elemental sulfur and hydrogen sulfide.

Based on the results, it can be concluded that the process proceeds according to reaction (2) throughout the entire temperature range studied. The decrease in extraction with increasing temperature above 180 °C can be explained as follows: apparently, the sulfur yield via reaction (1) increases. Elemental sulfur, in turn, forms an impermeable layer on the surface of sphalerite particles, thereby passivating the surface and inducing internal diffusion resistance against the reagent (Karimi et al., 2021). Consequently, the overall extraction efficiency decreases. At a sulfuric acid concentration of 65 wt.%, boiling begins at 169.2 °C (Amelin and Iashke, 1980), which further reduces the driving force of the process.

Safety and Industrial Off-Gas Management**:** Hydrogen sulfide (H_2S) is an acutely toxic, flammable gas with a low permissible exposure limit (IDLH = 100 ppm). To mitigate operational hazards during testing:

(i) The hermetic integrity of the digestion apparatus was continuously monitored, with all runs conducted inside a dedicated high-capacity fume hood equipped with continuous electrochemical H_2S sensors;

(ii) An intermediate vacuum buffer trap (safety vessel 5, Figure 1) was placed between the reactor and the scrubber to eliminate any risk of alkaline liquid suckback during transient thermal fluctuations or pressure drops;

(iii) Off-gases were passed through two counter-current scrubbing columns packed with Raschig rings containing 10 wt% NaOH solution ($pH > 12.5$). Under these conditions, $H_2S$was quantitatively converted into sodium hydrosulfide (NaHS) and sodium sulfide (Na_2S):

$$H_2S + NaOH \rightarrow NaHS + H_2O$$

Quantitative gas chromatography and potentiometric titrations confirmed that the H_2Sabsorption efficiency consistently exceeded 99.4%, preventing hazardous emissions while generating a commercially valuable NaHS co-product."

Figure 3 presents the dependence of zinc extraction into the aqueous phase on the initial sulfuric acid concentration during sulfation. The dependence of extraction on sulfuric acid concentration exhibits extremal behavior. Maximum extraction is achieved in the 65–70

wt.% sulfuric acid concentration range, reaching 95.65–94.92%, respectively (at $t = 160$°C).

The final leaching residue retained approximately 4.8–5.5% of the initial zinc. Mineralogical and XRD evaluations revealed that this residual zinc loss is primarily governed by two mechanisms: (i) mechanical entrainment/encapsulation of ultra-fine sphalerite inclusions within the refractory pyrite matrix ($FeS_2 \approx 17\%$), which remains largely inert under these non-oxidative sulfuric acid conditions; and (ii) minor isomorphous incorporation or surface adsorption of zinc onto co-precipitated lead sulfate (Anglesite, $PbSO_4$) and elemental sulfur traces. Further ultra-fine regrinding could potentially liberate these encapsulated grains but would drastically compromise subsequent solid-liquid filtration kinetics.

Analysis and Discussion

By varying the liquid-to-solid (L:S) ratio during the experiments, it was established that the optimal conditions for the decomposition of zinc concentrate are: a sulfuric acid concentration of 65–70 wt.%, an L:S ratio of 5:1 (which corresponds to approximately 3.1–3.2 m^3 of solution per 1 ton of concentrate), an operating temperature of 130–160 °C, and a process duration of 30 min (the reaction essentially reaches completion within thirty minutes of residence time at the target temperature). Under these conditions, the conversion of zinc into sulfate form reaches its maximum level of 94–95%. The hydrogen sulfide yield during the process is 0.22–0.23 t per 1 ton of concentrate. A further reduction in the L:S ratio is impractical, as it leads to a decrease in zinc extraction.

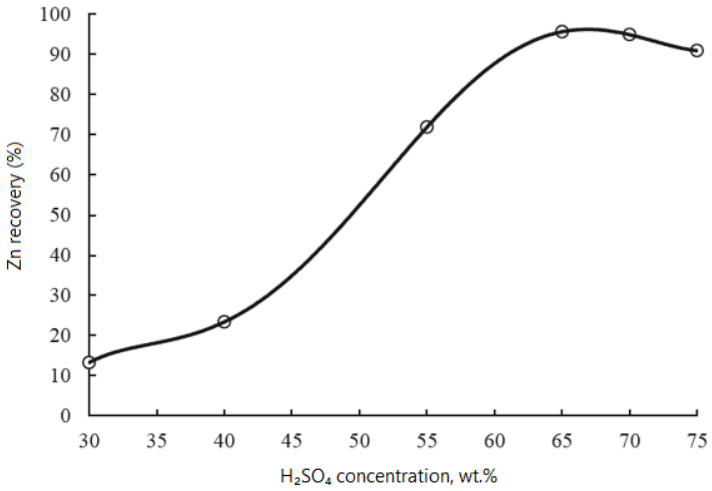


Figure 3. Effect of initial H_2SO_4 concentration on zinc extraction from the concentrate (t = 160 °C).

As evidenced by the time-dependent leaching profiles at 140–150°C, sphalerite digestion exhibits rapid initial kinetics, with zinc extraction surpassing 92% within the first 25–30 min of residence time; extending the duration to 3 h yields only a negligible increase (< 2.5%). Similarly, reducing the liquid-to-solid ratio from 15:1 to 5:1 had no statistically significant impact on the final zinc conversion (94.8% vs. 95.2%), whereas further reduction to 3:1 caused a noticeable drop in recovery (< 87%) due to slurry thickening and local acid transport resistance. Consequently, $L/S = 5:1$ and an isothermal residence time of 30 min were designated as the optimum technological parameters for industrial design, offering an optimal balance between maximal reactor productivity and minimal acid volume.

It is not recommended to reach the boiling point during the decomposition process, as this leads to the flotation of the zinc concentrate and subsequently impairs the contact with the acid solution. Upon completion of the experiment, the acid concentration in the liquid phase drops to 55–56 wt.%, which corresponds to the stoichiometric consumption according to reaction (1). It was established that virtually all of the decomposed zinc converts into the form of salts that are insoluble in the given medium. The solubility of zinc in the liquid phase of the slurry is negligible, varying between 0.6 and 3.9 g/L depending on the ambient temperature (in the range of

130–180 °C, respectively). The specific acid consumption, determined by its depletion in the solution, is approximately 0.65 t per 1 ton of concentrate, which closely matches the stoichiometry of reaction (2) presented earlier. At the end of the process, a white crystalline phase forms, which can be identified as a crystalline hydrate with the formula $ZnSO_4 \cdot H_2O$ (Akhmetov, 2002).

To verify the possibility of implementing continuous recycling of the acidic filtrate, an experiment was conducted using a closed-loop scheme: in accordance with the previously established regime, a 100 g sample of the concentrate was decomposed using 320 cm^3 of 70 wt.% H_2SO_4. Following the concentrate decomposition, the acidic slurry was vacuum-filtered through a synthetic cloth using a steel filter funnel to obtain an acidic recycled filtrate. The filter cake was washed with water at a ratio of approximately 3.8 m^3 per 1 ton of the initial concentrate, aiming to obtain a practically saturated zinc solution (110–120 g/L Zn (Kliain, 2009)).

The slurry was filtered, and the resulting filtrate with a density of 1.46 g/cm³ (corresponding to 55 wt.% H_2SO_4) was fortified by adding the consumed amount of acid and adjusted with water to the initial volume to obtain a final solution of 70 wt.% H_2SO_4 (corresponding to a density of 1.61 g/cm³). Subsequently, the high-temperature leaching cycle with filtrate recycling was repeated on a fresh concentrate sample. The required volume of concentrated 92.5 wt.% acid for replenishment was 35–40 cm^3 per cycle (for a 100 g concentrate charge). In total, 10 successive approximation cycles were carried out until a steady-state regime was established. Upon completion of the 10th cycle, the filter cake was washed with hot water at a ratio of 3.8 m^3 per 1 ton of concentrate charge. Steady-state conditions in the recirculating acid stream were achieved by cycle 5–6, as evidenced by the stabilization of residual free H_2SO_4 concentration ($\approx$ 55 wt%) and dissolved metal levels. Mass balance calculations over the closed-loop sequence indicated that water losses through evaporation and filter cake retention were fully compensated by the wash-water recycle stream and acid adjustment (supplemented with 92.5% H_2SO_4 at 35–40 mL per 100 g charge). Regarding impurity accumulation, iron and copper remained largely in the insoluble leach residue/sludge (Table 2; 20.5% Fe and 6.5% Cu) with dissolved fractions reporting at controlled steady concentrations ($<$ 1.2 g/L Fe and $<$ 0.8 g/L Cu) in the spent mother liquor. Importantly, recrystallization and subsequent hot-water washing of the recovered zinc sulfate monohydrate crystals ($ZnSO_4 \cdot H_2O$) prevented the co-crystallization and entrainment of these impurities, thereby maintaining high product purity ($>$ 98.5%) without altering crystal habit or morphology.

Table 1. Composition of the obtained acidic solutions (scaled-up locked-cycle experiment)

Product	Zn (g/L)	Fe (g/L)	Cu (mg/L)	Pb (mg/L)	Ag (mg/L)	Au (mg/L)	As (mg/L)	H_2SO_4 (wt%)
Recycled filtrate	6.41	0.46	0.22	0.60	<0.01	<0.01	<0.05	55
Zinc sulfate solution	114.00	1.48	9.55	2.50	<0.01	<0.01	<0.05	11

The high selectivity of the process toward zinc extraction (94.6%) over iron and copper sulfides is governed by both thermodynamic stability and passivation kinetics under concentrated sulfuric acid (60–70 wt% H_2SO_4) at 130–160°C. While sphalerite (ZnS) undergoes rapid oxidative/acidic breakdown, pyrite (FeS_2, $\approx$ 17 wt% in feed) and chalcopyrite ($CuFeS_2$, $\approx$ 1.5 wt%) exhibit pronounced refractoriness. Quantitative elemental accounting shows that only 6.5–7.2% of total iron dissolved into the leach liquor, while $>$ 92% remained unreacted, concentrating directly into the leach residue (20.5 wt% Fe, Table 2). Copper dissolution was even more negligible, yielding merely 9.55 mg/L Cu in the pregnant zinc sulfate solution (Table 1), representing $<$ 0.15% total Cu extraction.

The minor fraction of dissolved iron partitioned predominantly as ferrous iron (Fe^{2+}) due to the strongly reducing conditions maintained by evolving H_2S/SO_2 off-gases during sulfide digestion, with minimal

ferric (Fe^{3+}) oxidation. Importantly, the low equilibrium concentration of dissolved Fe(1.48 g/L in the final zinc sulfate stream and 0.46 g/Lin the recycle filtrate; Table 1) remained well below the solubility limit of iron sulfates under the salting-out/cooling crystallization regime. Consequently, co-precipitation or isomorphic substitution of Fe^{2+} into the $ZnSO_4 \cdot H_2O$crystal lattice was strictly suppressed, safeguarding both crystal purity and phase homogeneity.

The washed insoluble residue was dried and weighed, yielding 35.7% relative to the initial concentrate. The insoluble zinc-depleted residue, containing 7.0% Zn, 6.5% Cu, and 60% S, can be directed to copper smelting.

The concentrations of gold, silver, and arsenic in the solutions are below the detection limits of the analytical methods used. The compositions of the resulting liquid products and sludge are presented in Tables 1 and 2. The presence of acid in the zinc sulfate solution is attributed to its carryover in the mother liquor trapped within the crystalline hydrate precipitate. The decomposition of copper and arsenic minerals is practically negligible (trace amounts in solutions). Similarly, the degree of pyrite decomposition is insignificant. The zinc recovery into the sulfate solution reached 94.6%.

Table 2. Composition of the obtained sludge based on chemical analysis (scaled-up locked-cycle experiment)

Product	Zn (%)	S (%)	Fe (%)	Cu (%)	Pb (%)	Ag (g/t)	Au (g/t)	As (%)
Sludge (insoluble residue)	7.0	60.0	20.5	6.5	0.26	205	2.40	0.07

The process flowsheet for the locked-cycle experiment, including the material balance, is illustrated in Fig. 4. This figure provides a graphical representation of the recommended scheme for sphalerite decomposition across various products (where βdenotes the concentrations of the valuable components—copper and zinc).

Based on the investigation conducted using zinc concentrate produced by JSC “Svyatogor”, a hydrometallurgical process scheme for processing zinc sulfide raw materials is proposed. The process is based on sulfuric acid leaching accompanied by hydrogen sulfide evolution, elevated-temperature crystallization of zinc sulfate hydrate directly within the slurry, filtration, and subsequent dissolution of acid-washed zinc sulfate monohydrate crystals.

The evolved hydrogen sulfide can be effectively utilized for precipitating non-ferrous metals from industrial wastewater or used to synthesize sodium hydrosulfide (NaHS) using standard procedures (Pozin, 1974). The resulting purified zinc sulfate solution can be further processed into commercial zinc sulfate heptahydrate (vitriol) or utilized for metallic zinc recovery via electrowinning (Rudnic, 2019; Kaya et al., 2020).

It has been experimentally demonstrated that the zinc sulfate solution obtained via this flowsheet—following neutralization of excess sulfuric acid (which can be achieved using zinc dust or Waelz oxide)—complies with the requirements of TU 2141-003-41848759-2008 (“Zinc Sulfate Aqueous Solution”, Grade A/B) for the zinc sulfate solution produced by JSC Mednogorsk Copper-Sulfur Plant (MMSK, Mednogorsk). This solution can either serve as a commercial product or as a feedstock for preparing the electrolyte for zinc electrowinning. The resulting zinc sulfate and sodium hydrosulfide can be successfully utilized in pyritic ore flotation/beneficiation technologies (Mamonov et al., 2023).

4. Conclusions

The proposed hydrometallurgical process enables efficient decomposition of zinc concentrate while simultaneously recovering valuable products, including zinc sulfate and hydrogen sulfide. The process produces a purified zinc sulfate solution that meets industrial requirements and can be further used for zinc recovery or conversion to commercial zinc sulfate hydrates. In addition, the by-products are suitable for further utilization in non-ferrous metal precipitation and mineral processing applications, making the process technically and industrially promising.

The developed process flowsheet can be applied to the de-zincing of a wide range of high-zinc copper

concentrates, thereby enhancing their quality. Its implementation for zinc concentrates will also prevent copper losses in the clinker (leach residue).

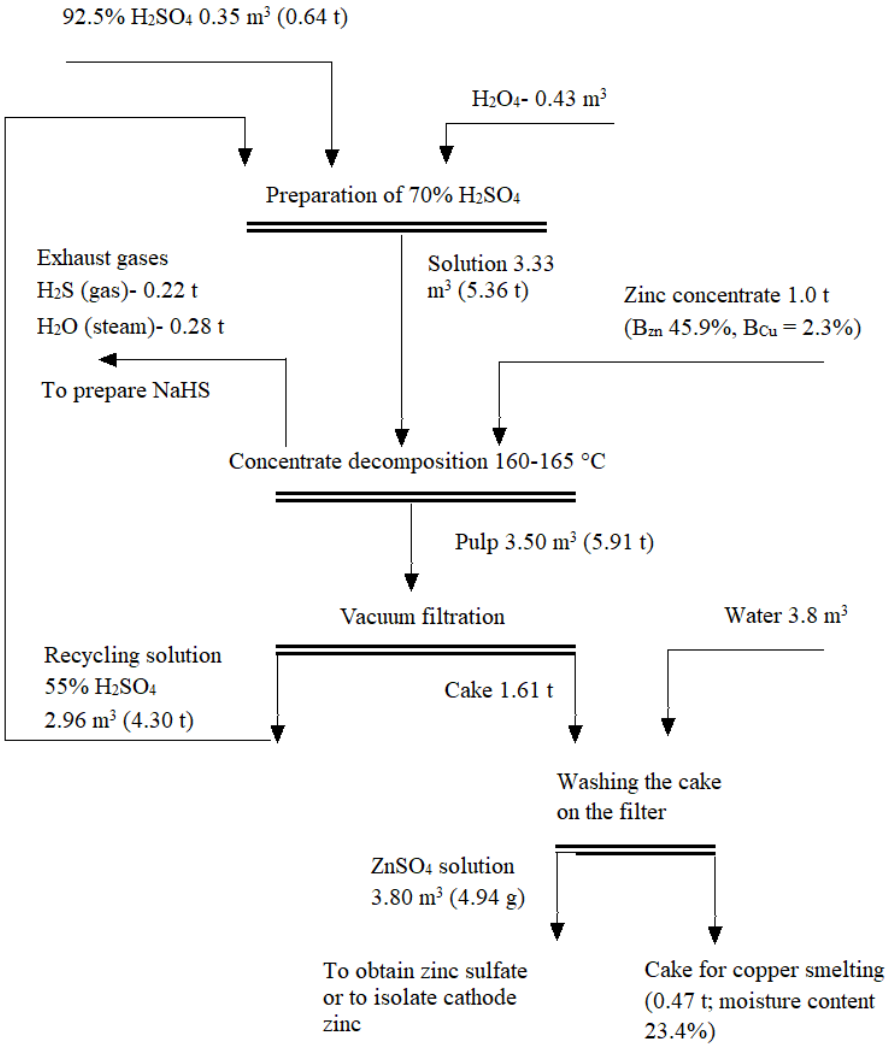


Figure 4. Integrated process flowsheet and material balance for the atmospheric leaching of zinc concentrate (Svyatogor JSC) based on a feed rate of 1 t.

Acknowledgment

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Declarations

Data and code availability

All data generated or analyzed during this study are available from the corresponding author upon reasonable request. No computational code was used in this study.

Conflicts of interest

The authors announced that they have no known conflicts of interest or personal relationships that could have appeared to influence the work reported in this manuscript.

Ethical approval

The authors declare no ethical issues; the research was carried out in full agreement with ethical standards. Also, this paper is neither under Review nor published elsewhere.

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