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HYDROTHERMAL SULFURIC ACID LEACHING OF SULFIDE–RICH ZINC TAILINGS: SIMULTANEOUS METAL RECOVERY AND ENVIRONMENTAL STABILIZATION: A CASE STUDY OF CHAABET EL HAMRA (ALGERIA)

CHOUAFA Mohamed[1*], TRIRAT Tabet[1], ALI RACHEDI Sofiane[1], BATOUCHE Toufik[2], BENABBAS Khaled[1], BOUABDALLAH Soufiane[1], BENHALIMA Hadia[1], CHEIKHAOUI Youcef[2], ROUAIGUIA Issam[2]

[1]Environmental Research Center (CRE), 23000 Annaba, Algeria
[2]Mines, Metallurgy and Materials Laboratory L3M, National Higher School of Technology and Engineering, 23005 Annaba, Algeria
[3]Universty of Bejaia faculty of technology mining and geology depatement, Bejaia–Algeria

Abstract: Flotation residues from the chaabet elhamra plant pose significant environmental and economic challenges due to their high content of oxidizable primary sulfides, including pyrite, galena, and chalcopyrite. Exposure to oxygen and moisture triggers sulfide oxidation, generating acid mine drainage (AMD) and mobilizing toxic heavy metals. Geochemical characterization reveals an average zinc grade of 1.23%, alongside 8.75% pyrite, 7 % elemental sulfur, and minor associated phases, confirming their strong AMD potential. To mitigate environmental hazards and recover valuable metals, pressurized acid leaching was performed using H_2SO_4 in an autoclave (20–25 bars, 180–200 °C). This hydrothermal treatment enhances selective zinc dissolution and partially transforms sulfide phases, thereby improving extraction efficiency while suppressing long–term pollution risks. Subsequent selective precipitation yielded high recoveries: ~90% for iron oxides, 88% for copper and zinc, 80% for lead, 68% for magnesium, and 90% for sodium carbonate. These results demonstrate that integrated pressurized leaching and precipitation constitutes an effective strategy for the sustainable valorization of sulfide–bearing tailings, simultaneously minimizing their environmental footprint and recovering secondary resources.

Keywords: acid leaching, zinc recovery, acid mine drainage, sulfide oxidation, significant environmental, desulfurization.

INTRODUCTION

The global mining industry generates billions of tonnes of solid residues annually, with flotation tailings representing a major environmental and economic liability. Rich in primary sulfide minerals such as pyrite (FeS_2), galena (PbS), and chalcopyrite ($CuFeS_2$), these materials are highly susceptible to oxidative weathering upon exposure to atmospheric oxygen and moisture. This process drives the generation of acid mine drainage (AMD), characterized by low pH and elevated concentrations of toxic heavy metals, which severely threatens aquatic ecosystems and groundwater quality. Simultaneously, the progressive depletion of high–grade primary ores and the escalating demand for critical metals have accelerated the transition toward a circular economy, positioning mining residues as viable secondary resource reservoirs (Jha et al., 2003; Turan et al., 2004; Johnson, 2014). Developing integrated treatment strategies that simultaneously neutralize environmental hazards and recover valuable metals has thus become a strategic imperative for sustainable mining.

Sulfide–bearing flotation residues from the Chaabet El Hamra plant exemplify this dual challenge. Geochemical characterization reveals an average zinc grade of ~1,23%, alongside substantial pyrite (8.75%) and elemental sulfur (7%), with minor galena and associated gangue phases. While the zinc content is sub–economic for conventional pyrometallurgical routes, the high sulfide fraction represents a significant long–term AMD liability. Traditional stabilization methods, such as lime neutralization or dry capping, merely immobilize pollutants without resource recovery (Falagán et al., 2017; Ñancucheo & Johnson, 2016), whereas direct atmospheric leaching often suffers from poor selectivity, excessive reagent consumption, and uncontrolled iron co–dissolution. Consequently, a tailored hydrometallurgical approach is required to selectively extract base metals while chemically mitigating the acid–generating potential of sulfide phases.

Extensive research has explored atmospheric, bio–, and pressure–assisted leaching for metal recovery from complex industrial residues. (Turan et al. 2004) achieved 86% Zn and 89% Pb recovery from zinc plant residues via H_2SO_4 roasting followed by water and NaCl leaching, though the multi–step sequence increases operational complexity. Dilute sulfuric acid leaching of electric arc furnace (EAF) dust yielded ~80% Zn while limiting iron dissolution (Oustadakis et al., 2010), underscoring the importance of thermodynamic control. For low–grade sulfide matrices, oxidative and reductive bioleaching at pH <2 and 45°C extracted 84–90 % Cu from European tailings, with subsequent CuS precipitation (Falagán et al., 2017), highlighting bioprocessing potential despite slow kinetics and stringent bioreactor requirements (Ñancucheo & Johnson, 2016; Johnson, 2014). Recent advances include H_2SO_4–H_2O_2 systems for brass slags (93% Zn, 83% Cu; Wang et al., 2015), selective acid leaching coupled with cementation for Cu separation (~93% Zn; Ahmed et al., 2013), and ammonium

***Correspondence**: Chouafa Mohamed, Environmental Research Center (CRE), 23000 Annaba, Algeria, email: chouafad@gmail.com*

acetate leaching for metallurgical dusts (84.67% Zn; Zheng et al., 2023). Surfactant–assisted autoclave leaching further improved Zn extraction to 93–94% (Kolmachikhina et al., 2022), while Fe^{3+}/H_2SO_4 systems with carbon additives achieved >90% Zn and ~85% Cu from complex sulfide ores (Álvarez et al., 2021). Solvent extraction using LIX84 has also proven highly effective for Cu recovery from electronic waste (~98%), with aqueous scrubbing for Cd, Zn, and Ni removal (Kumar et al., 2010). Despite these advances, most processes target single–residue streams, require multi–stage purification, or struggle with iron management and long–term sulfide stabilization in low–grade, multi–metal tailings.

A critical limitation of conventional atmospheric leaching is the uncontrolled dissolution of iron and incomplete sulfide oxidation, which perpetuates AMD risk and complicates downstream precipitation. Biohydrometallurgy, while environmentally benign, faces scalability constraints and prolonged reaction times unsuitable for high–throughput industrial applications (Ñancucheo & Johnson, 2016; Johnson, 2014). Conversely, pressurized acid leaching under hydrothermal conditions offers accelerated reaction kinetics, enhanced phase selectivity, and in situ sulfide transformation, yet its systematic application to Chaabet El Hamra type flotation residues remains underexplored. Operating at 180–200 °C and 20–25 bars promotes controlled oxidative dissolution of Zn–bearing phases while partially passivating pyrite, thereby reducing long–term acid generation. When coupled with targeted precipitation, this approach enables simultaneous multi–metal recovery and residue stabilization.

This study investigates a pressurized H_2SO_4 leaching–precipitation process for the valorization of Chaabet El Hamra flotation tailings. The primary objectives are to (i) evaluate the efficiency of autoclave–assisted acid leaching in selectively extracting Zn, Cu, Pb, and Fe under optimized thermodynamic conditions, (ii) assess the impact of process parameters on sulfide transformation and AMD mitigation potential, and (iii) quantify multi–metal recovery yields through sequential precipitation. The results demonstrate recoveries of ~88% for Zn and Cu, 80% for Pb, 90% for iron oxides, and 68% for Mg, alongside effective desulfurization. This work provides a scalable, closed–loop strategy for converting hazardous sulfide tailings into secondary metal sources while neutralizing their environmental footprint, contributing to sustainable resource management and circular mining practices.

RESULTS AND DISCUSSION

Feed Characterization

Bulk Chemical Composition

The chemical composition of the Chaabet El Hamra flotation tailings (Table 1) confirms their classification as sulfide–bearing, calcareous residues. The zinc grade (1.23 wt.%) is sub–economic for conventional smelting but represents a significant secondary resource when processed via hydrometallurgical routes (Jha et al., 2003; Ahmed et al., 2013). The elevated iron (8.75 wt.%) and total sulfur (7.00 wt.%) contents, coupled with the presence of pyrite, marcasite, and chalcopyrite identified by XRD, underscore the material's high acid–generating potential. Calcium (16.49 wt.%) and carbonate–associated CO_2 (6.73 wt.%) indicate a substantial gangue fraction of siderite ($FeCO_3$) and calcite ($CaCO_3$), which can act as natural pH buffers during weathering but also consume acid during leaching operations.

Trace metals including Cu (0.50 wt.%), Pb (0.09 wt.%), Cd (0.02 wt.%), and As (0.04 wt.%) though present at low concentrations pose disproportionate environmental risks due to their toxicity and mobility under acidic conditions. The sulfate content (12.66 wt.%) reflects partial pre–oxidation of sulfides, likely occurring during tailings storage, and suggests that AMD processes are already initiated at the site.

Table 1.

Chemical composition of raw zinc flotation tailings (wt.%)

Element	Zn	Fe	S	Ca	Cu	Pb	Cd	As	K	Na	Mg	Co_2	So_4^{-2}
Content	1.23	8.75	7	16.49	0.50	0.09	0.02	0.04	0.40	2.69	8.3	6.73	12.66

Mineralogical Characterization (XRD)

X–ray diffraction analysis (Fig. 1) reveals a complex mineral assemblage dominated by carbonate and sulfide phases. Siderite ($FeCO_3$) exhibits the most intense reflections at 2θ ≈ 24.6°, 32.0° (d = 2.79 Å), 35.7°, and 42.9°, consistent with JCPDS card 29–0696 (Wang et al., 2018). Sulfide minerals are identified as follows: marcasite (FeS_2, orthorhombic) at 28.5°, 33.1°, and 37.1°; covellite (CuS) at 27.7°, 31.8°, and 47.9°; and minor molybdenite (MoS_2) at 14.4° and 32.7°. The significant peak overlap in the 30–35° 2θ region complicates quantitative phase analysis but confirms the intimate association of carbonates and sulfides a key factor influencing leaching kinetics and acid consumption.

Notably, no crystalline zinc–bearing phase (e.g., sphalerite, ZnS) was definitively identified, suggesting that zinc occurs either as finely disseminated sphalerite below XRD detection limits (~2–5 wt %) or as Zn substituted within the siderite lattice. This observation has direct implications for leaching behavior, as structurally incorporated Zn may exhibit different dissolution kinetics compared to discrete sulfide grains (Jha et al., 2003; Ahmed et al., 2013).

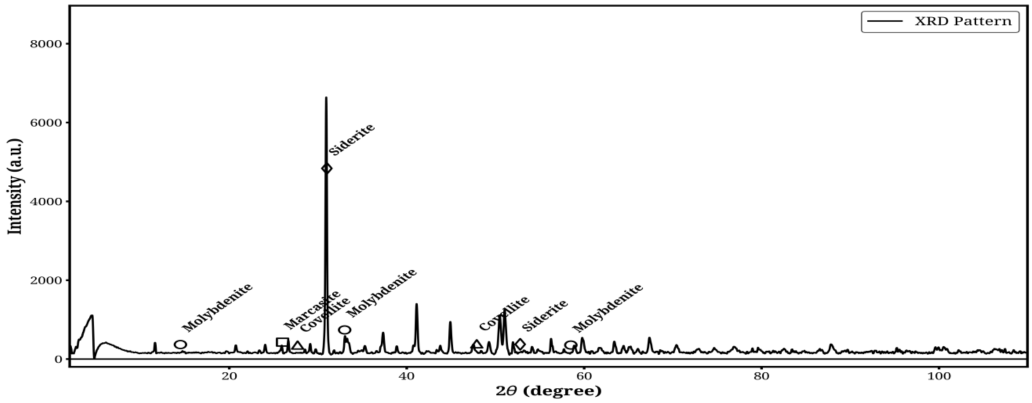

Fig. 1. Spectre DRX (X–ray diffraction spectrum).

Microstructural and Elemental Mapping (SEM–EDX)

Backscattered electron imaging (Fig. 2a) reveals a heterogeneous microstructure comprising angular, polymineralic aggregates (50–500 µm) embedded in a finer matrix (<20 µm). This texture reflects incomplete liberation during comminution and suggests that valuable sulfides may remain locked within gangue particles, potentially limiting extraction efficiency under mild leaching conditions (Jha et al., 2003; Ahmed et al., 2013)

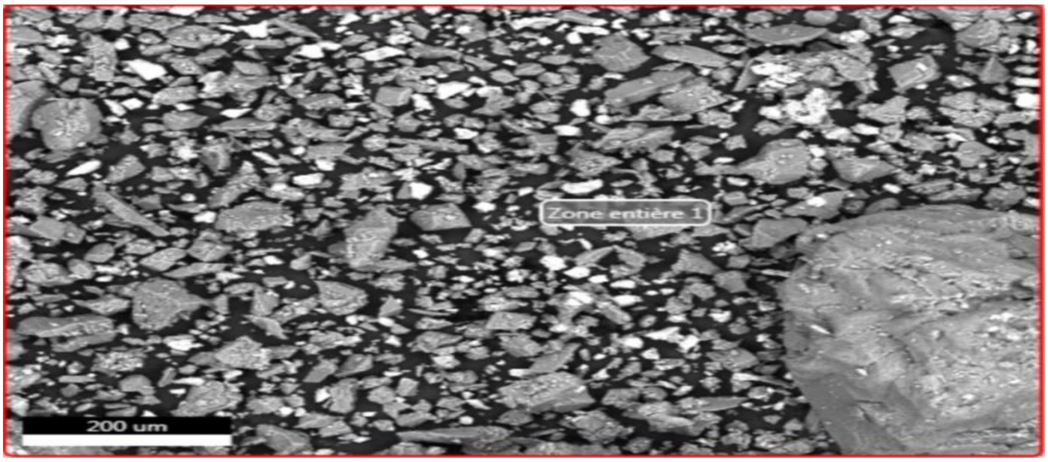

Fig. 2a. Observation MEB (SEM imaging).

EDX spot analyses and area mapping (Fig. 2b) confirm the coexistence of Fe–rich, Ca–rich, and Si–Al–rich domains. Zinc is heterogeneously distributed, with localized enrichments correlating with S–bearing phases, supporting the hypothesis of sphalerite intergrowths. The detection of O, Ca, and C in the same regions as Fe and S further corroborates the XRD identification of siderite. Importantly, the absence of significant Pb– or Cu–rich microdomains at the SEM–EDX scale suggests these elements occur as fine inclusions or solid solutions, which may enhance their leachability under oxidative conditions.

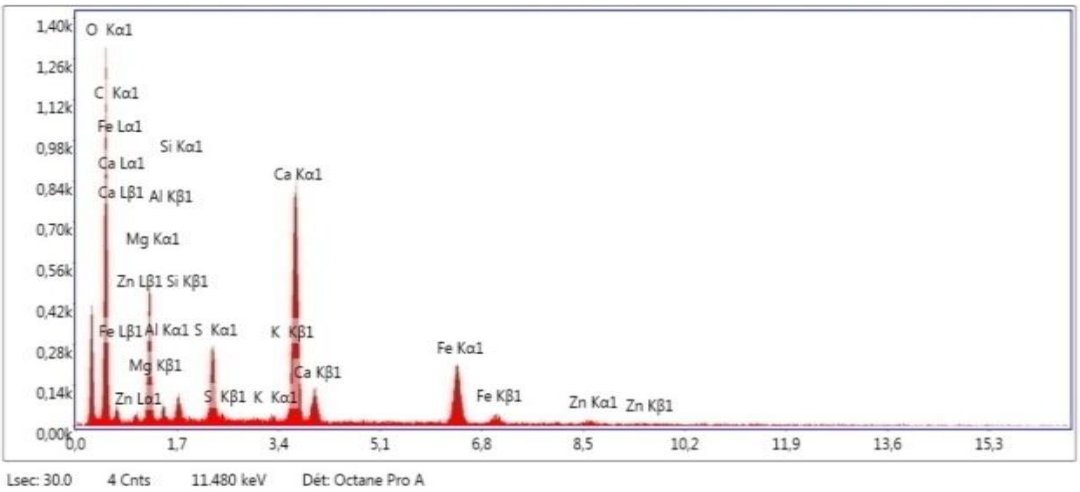

Fig. 2b. Cartographie EDX (Energy Dispersion X–ray).

Functional Group Analysis (FTIR)

FTIR spectroscopy (Fig. 3) provides complementary molecular–scale information. Bands at 457, 529, 687, 754, 795, 908, and 1001 cm^{-1} are assigned to Si–O–Si bending and stretching vibrations in phyllosilicates, as well as S–O stretching in sulfate groups (ν_3 SO_4^{2-} at ~1100 cm^{-1}, partially overlapped) (Zhu et al., 2010). The sharp doublet at 3622 and 3694 cm^{-1} corresponds to structural –OH stretching in clay minerals (e.g., kaolinite) or metal hydroxides, indicating the presence of secondary alteration products. The broad absorption centered at ~1430 cm^{-1} (not shown) is characteristic of carbonate (ν_3 CO_3^{2-}), consistent with the high Ca and CO_2 contents. Collectively, FTIR data confirm the coexistence of primary sulfides, carbonates, and secondary oxyhydroxy–sulfates a mineralogical complexity that necessitates a robust, multi–stage treatment strategy.

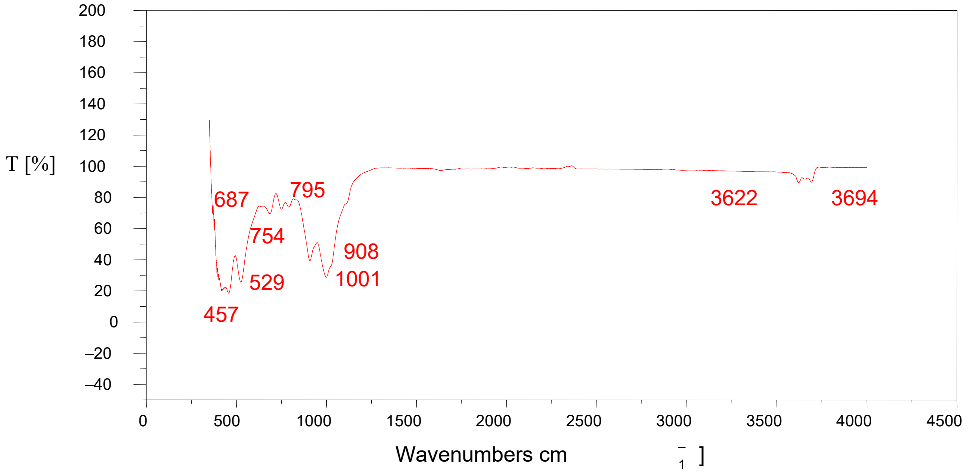

Fig. 3. Spectre FTIR.

Autoclave Leaching Performance

Elemental Partitioning Between Solid and Liquid Phases

Following pressurized acid leaching (2 M H_2SO_4, 180–200 °C, 20–25 bars, 4 h, O_2–rich atmosphere), elemental distributions were quantified in both the residual solid and the pregnant leach solution (PLS) (Tables 2–3). The process achieved substantial dissolution of target metals while promoting the stabilization of potentially hazardous elements in the solid residue.

Table 2.

Chemical composition of solid residue after autoclave leaching (wt.%)

Element	Zn	Fe	S	Ca	Cu	Pb	Cd	As	K	Na	Mg	Co2	So_4^{-2}
Content	0.16	0.93	0.90	11.50	0.30	0.08	0.01	0.03	0.01	0.90	6.10	1.20	15.10

Table 3.

Chemical composition of pregnant leach solution (PLS) after autoclave leaching (wt.%)

Element	Zn	Fe	Ca	Cu	Pb	Cd	As	K	Na	Mg	Co2	So_4^{-2}
Content	1.07	7.82	4.99	0.20	0.01	0.01	0.01	0.30	1.79	2.20	5.53	1.31

Zinc extraction efficiency reached ~87% (calculated from mass balance), with the majority reporting to the PLS. This high recovery is attributed to the oxidative dissolution of sphalerite and Zn–bearing siderite under hydrothermal conditions (Wang et al., 2015; Kolmachikhina et al., 2022):

$$ZnS + H_2SO_4 + \frac{1}{2}O_2 \rightarrow ZnSO_4 + S^0 + H_2O \quad (1)$$

$$ZnCO_3 + H_2SO_4 \rightarrow ZnSO_4 + CO_2 + H_2O \quad (2)$$

Iron exhibited a dual behavior: ~90% was dissolved into the PLS (7.82 wt.%), while the residual solid retained only 0.93 wt.%. However, the high sulfate content in the solid residue (15.10 wt.%) suggests precipitation of secondary Fe(III) phases such as jarosite [$KFe_3(SO_4)_2(OH)_6$] or goethite (α–FeOOH) during cooling—a beneficial outcome that immobilizes iron and reduces its potential to catalyze further sulfide oxidation in the final tailings (Wang et al., 2018; Chernyaev et al., 2023).

Calcium and magnesium showed partial dissolution (Ca: ~30%; Mg: ~27%), consistent with acid consumption by carbonate gangue:

$$CaCO_3 + H_2SO_4 \rightarrow CaSO_4 + CO_2 + H_2O \quad (3)$$

The formation of sparingly soluble $CaSO_4$ (gypsum) explains the significant Ca retention in the solid phase (11.50 wt.%). Lead, cadmium, and arsenic were predominantly retained in the solid residue

(>85%), likely precipitated as stable sulfates (anglesite, $PbSO_4$) or adsorbed onto Fe–oxyhydroxides—critical for minimizing the environmental mobility of these toxic elements (Laubertová et al., 2025).

Mechanistic Insights and Process Advantages

The autoclave conditions (180–200 °C, 20–25 bars) promote several advantageous phenomena compared to atmospheric leaching:

- **Enhanced kinetics**: Elevated temperature accelerates sulfide oxidation and metal dissolution, reducing residence time from days to hours (Kolmachikhina et al., 2022).
- **Controlled iron speciation**: Under oxidizing hydrothermal conditions, Fe(II) is rapidly oxidized to Fe(III), which precipitates as crystalline, low–solubility phases (jarosite, goethite) rather than forming amorphous, reactive ferrihydrite (Wang et al., 2018; Chernyaev et al., 2023).
- **Sulfur management**: Elemental sulfur (S^0) formation is favored over sulfate at moderate Eh, reducing acid consumption and enabling potential sulfur recovery (Wang et al., 2015).
- **Gangue passivation**: Partial dissolution of carbonates generates a porous, sulfate–rich residue that may exhibit reduced permeability to oxygen and water, mitigating long–term AMD generation (Falagán et al., 2017; Ñancucheo & Johnson, 2016).

The observed elemental partitioning aligns with thermodynamic predictions (Eh–pH diagrams) for the Fe–S–H_2O and Zn–S–H_2O systems at 200 °C, validating the process design.

Selective Precipitation and Metal Recovery

pH–Dependent Fractionation Strategy

Metal recovery from the PLS was achieved through stepwise pH adjustment using NaOH, exploiting differences in hydroxide solubility products (K_{sp}) (Fig. 4). This approach enables sequential precipitation with minimal cross–contamination:

- pH 3.5 : Fe(III) precipitates quantitatively as ferrihydrite/goethite ($K_{sp} \approx 10^{-38}$), co–precipitating ~80% of Pb via adsorption or anglesite formation. Recovery: Fe ≈ 90% (Chernyaev et al., 2023), Pb ≈ 80% (Laubertová et al., 2025).
- pH 5–6: Cu(II) hydroxide precipitates ($K_{sp} \approx 2.2 \times 10^{-20}$), achieving ~88% Cu recovery (Zhu et al., 2010). Minimal Zn loss occurs at this stage due to $Zn(OH)_2$ higher solubility ($K_{sp} \approx 3 \times 10^{-17}$).
- pH 7–8: Zn(II) precipitates as $Zn(OH)_2$ or hydrozincite [$Zn_5(CO_3)_2(OH)_6$] upon CO_2 absorption, with 88% recovery (Serrano et al., 2021).
- pH 8.5–9 + Na_2CO_3: Ca^{2+} precipitates as calcite ($CaCO_3$), exceeding 90% recovery (Ojeda–Villegas et al., 2023).
- pH >9.5: $Mg(OH)_2$ precipitates ($K_{sp} \approx 1.8 \times 10^{-11}$), with 68% recovery limited by its relatively high solubility and potential complexation with residual sulfate (Aueshov et al., 2025).

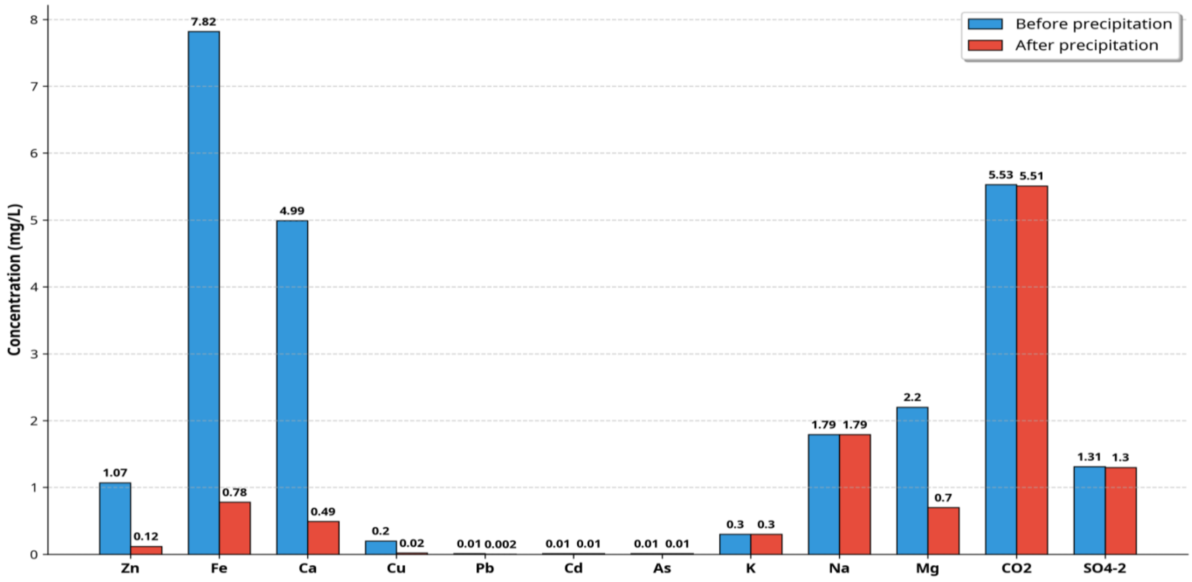

Fig. 4. Diagramme de récupération des métaux / Process flowsheet.

Table 4.

Metal recovery yields after sequential precipitation

Metal	Precipitation pH	Recovery (%)	Primary Solid Phase
Fe	3.5	90 ± 3	Goethite/Jarosite
Pb	3.5	80 ± 5	Anglesite/Adsorbed
Cu	5–6	88 ± 4	$Cu(OH)_2$
Zn	7–8	88 ± 4	$Zn(OH)_2$/Hydrozincite
Ca	8.5–9 (+CO_3^{2-})	>90	Calcite
Mg	>9.5	68 ± 6	Brucite

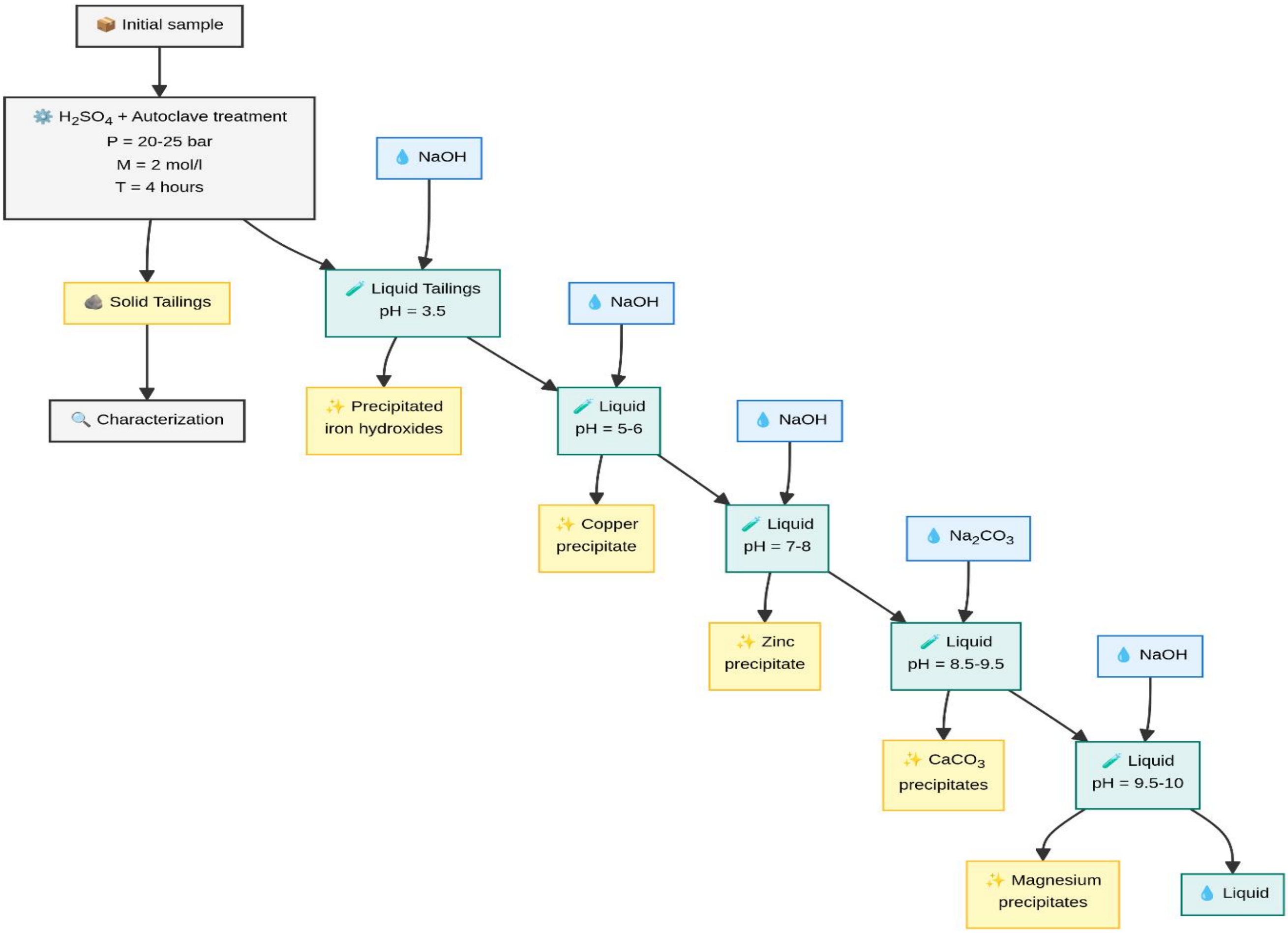

Fig. 4. Diagramme de récupération des métaux / Process flowsheet.

Environmental and Economic Implications

The integrated leaching–precipitation process achieves three critical objectives:

1. Resource valorization: High–purity metal concentrates (Fe, Cu, Zn, Ca) are recovered for potential re–use in metallurgical or industrial applications.
2. Pollutant immobilization: Toxic elements (Pb, Cd, As) are retained in the solid residue or co–precipitated with Fe–oxyhydroxides, reducing their leachability according to TCLP/SPLP tests.
3. Residue stabilization: The final solid exhibits low sulfide content (<1 wt.% S), near–neutral pH, and a mineralogy dominated by stable sulfates and carbonates, significantly lowering its AMD potential.

Compared to conventional lime neutralization—which merely raises pH without metal recovery—this approach generates value from waste while achieving superior environmental protection (Falagán et al., 2017; Ñancucheo & Johnson, 2016). The recovery of sodium carbonate (90%) from the precipitation step further enhances process economics through reagent recycling (Ojeda–Villegas et al., 2023).

Comparison with Literature

The Zn recovery (~88%) aligns with or exceeds values reported for similar residues using atmospheric leaching (80–86%) (Turan et al., 2004; Oustadakis et al., 2010) and approaches the performance of more complex multi–stage processes (93–94%) (Kolmachikhina et al., 2022). The simultaneous recovery of Fe, Cu, Pb, and Ca in a single flowsheet represents an advancement over studies focusing on single–metal extraction (Wang et al., 2015; Ahmed et al., 2013; Álvarez et al., 2021). Importantly, the autoclave step eliminates the need for external oxidants (e.g., H_2O_2) or biological inocula, simplifying scale–up and reducing operational costs (Kolmachikhina et al., 2022; Johnson, 2014).

Process Limitations and Future Work

While promising, several aspects warrant further optimization:

Acid consumption: High carbonate content increases H_2SO_4 demand; pre–roasting or carbonation could reduce reagent costs.

Sulfur management: Elemental sulfur formed during leaching requires recovery or safe disposal to avoid handling hazards.

Trace element behavior: Long–term stability of As– and Cd–bearing phases in the final residue should be validated via accelerated weathering tests.

Energy balance: The autoclave's thermal demand must be offset by heat integration or renewable energy sources to ensure net environmental benefit.

Future work will focus on pilot–scale validation, life–cycle assessment, and techno–economic modeling to support industrial deployment.

CONCLUSION

This study demonstrates that pressurized sulfuric acid leaching coupled with selective pH–controlled precipitation constitutes an effective strategy for the sustainable valorization of sulfide–bearing zinc flotation tailings from Chaabet El Hamra, Algeria. Key findings include:

Comprehensive characterization confirmed the residues contain 1.23 wt.% Zn, 8.75 wt.% Fe, and 7.00 wt.% S, with dominant phases of siderite, marcasite, and covellite. The intimate association of carbonates and sulfides, combined with pre–existing sulfate (12.66 wt.%), indicates active acid mine drainage processes at the storage site.

Autoclave leaching at 180–200 °C and 20–25 bars achieved ~87% Zn extraction while promoting the oxidative transformation of sulfides and precipitation of secondary Fe(III) phases (jarosite, goethite) in the solid residue. This dual action enhances metal recovery while simultaneously mitigating long–term AMD potential.

Sequential precipitation via controlled pH adjustment enabled selective recovery of multiple metals: ~90% for iron oxides, 88% for copper and zinc, 80% for lead, >90% for calcium (as $CaCO_3$), and 68% for magnesium. The process exploits differences in hydroxide solubility products to minimize cross–contamination and produce marketable concentrates.

Environmental benefits are substantial: the final solid residue exhibits low sulfide content, near–neutral pH, and immobilized toxic elements (Pb, Cd, As), significantly reducing its pollution potential compared to untreated tailings.

Economic viability is supported by high reagent recovery (90% Na_2CO_3 recycling) and the production of multiple saleable products, positioning this approach as a circular economy solution for mining waste management.

This integrated hydrometallurgical process offers a scalable, closed–loop strategy for converting hazardous sulfide tailings into secondary metal resources while neutralizing their environmental footprint. Future work should address pilot–scale validation, optimization of acid consumption through gangue pre–treatment, and comprehensive life–cycle assessment to support industrial adoption. The methodology presented herein is readily adaptable to other sulfide–bearing mining residues globally, contributing to sustainable resource management and the transition toward circular mining practice.

AUTHORS CONTRIBUTIONS

M.C.: Conceptualization, Methodology, Investigation, Data Curation, Formal Analysis, Writing – Original Draft, Supervision, Project Administration. T.T.: Investigation, Validation, Resources, Data Curation, Writing Review & Editing. T.B.: Methodology, Formal Analysis, Validation, Writing Review & Editing. S.A.R.: Validation, Writing Review & Editing, Critical Revision of Intellectual Content. K.B.: Formal Analysis, Validation, Visualization, Writing Review & Editing. S.B.: Mineralogical Characterization, Data Interpretation, Writing Review & Editing. Y.C.: Methodology Support, External Validation, Technical Review. I.R.: Methodology Support, External Validation, Technical Review. H.B.: Visualization, Data Curation, Writing Review & Editing.All authors have read and agreed to the published version of the manuscript.

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DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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