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Original scientific paper

MINERALOGICAL EVOLUTION IN INDUSTRIAL JAROSITE THROUGH FLUX-ASSISTED ROASTING

Jose Enrique Sanchez-Vite[1a], Alberto Hernandez-Casimiro[1b] , Teresita del Refugio Jiménez Romero[1c], Alejandro Cruz-Ramírez[2a], Margarita García-Hernández[2b]

[1] ESIQIE, Instituto Politécnico Nacional, UPALM, 07738, Ciudad de México, México
[2] UPIIH, Instituto Politécnico Nacional, DESCTI, San Agustín Tlaxiaca, 42162, Hidalgo, México
[1a] jsanchezv2105@alumno.ipn.mx, 0009-0006-5001-0495
[1b] ahernandezc2200@alumno.ipn.mx, 0009-0004-2097-9305
[1c] tjimenezr@ipn.mx, 0009-0003-8012-6817
[2a] alcruzr@ipn.mx, 0000-0002-0972-596X
[2b] margarciah@ipn.mx, 0000-0002-3850-3901

Abstract
Industrial jarosite residues from zinc hydrometallurgy are both an environmental liability and a secondary resource for valuable metal recovery (Zn, Pb, Ag). The mineralogical evolution of an ammonium-jarosite residue during oxidizing roasting was evaluated, comparing (i) pure residue and (ii) a flux-assisted/reducing formulation (45 wt% jarosite + 40 wt% Na_2CO_3 + 15 wt% SiC). Thermogravimetric analysis shows decomposition events related to dehydration, dehydroxylation, and sulfate breakdown with an overall mass loss of ~49%. X-ray diffraction measurements to temperature in the range from 600 to 1400 °C evidence the pathway of spinel formation: jarosite → hematite + ZnO → $ZnFe_2O_4$ (spinel) for pure jarosite roasting, which immobilizes Zn. In contrast, the addition of Na_2CO_3 and SiC to jarosite suppresses $ZnFe_2O_4$, fixes sulfur predominantly as Na_2SO_4 and $CaSO_4$, while iron is contained in oxide and sulfate compounds. SEM/EDS reveals a locally Pb-rich droplet domain at elevated temperatures, indicating partial metal liberation under optimized redox conditions.

Keywords: *(Jarosite; Thermal decomposition; Flux-assisted roasting; Sulfur fixation; Zinc ferrite)*

1. INTRODUCTION

Jarosite-type residues produced during iron removal in zinc hydrometallurgy are widely produced and incorporate valuable and hazardous elements (e.g., Zn, Pb, Ag), thus posing both an environmental liability and a secondary resource opportunity [1,2]. Thermogravimetric studies typically reveal a multistage pathway dehydration, dehydroxylation and (for ammonium jarosite) NH_4^+ release, followed by sulfate breakdown between ~100–700 °C [3]. At higher temperatures, hematite and zincite emerge and may couple to form $ZnFe_2O_4$ spinel, a refractory phase that immobilizes zinc and hinders hydrometallurgical recovery [3,4]. To circumvent this, strategies such as leaching, sulfidization–flotation, stabilization, and reducing/flux-assisted roasting have been explored [2,5,6]. Building on this body of work, an industrial jarosite was treated with Na_2CO_3 (flux) and SiC (reducing agent) to promote sulfur fixation in sulfates and to suppress $ZnFe_2O_4$ formation [4–8].

2. EXPERIMENTAL

An industrial ammonium jarosite residue (−75 µm) was thermally treated in static air at 600, 800, 1000, 1200, and 1400 °C (10 °C min^{-1}; 2 h hold) using high-purity alumina crucibles. Two compositions were studied: Case 1: 100 wt% jarosite and Case 2: 45 wt% jarosite + 40 wt% Na_2CO_3 + 15 wt% SiC (10 g per trial). Characterization included TGA/DTG (RT–1000 °C), XRD (10–70° 2θ), and SEM/EDS. The industrial jarosite contained Fe (~8 wt%), total S (~17 wt%), Zn

(~5 wt%), Pb (~0.45 wt%) and Ag (~143 ppm). XRD of the as-received residue showed ammonium jarosite with minor gypsum, ZnS and $ZnSO_4 \cdot 7H_2O$ compounds (Figure 1).

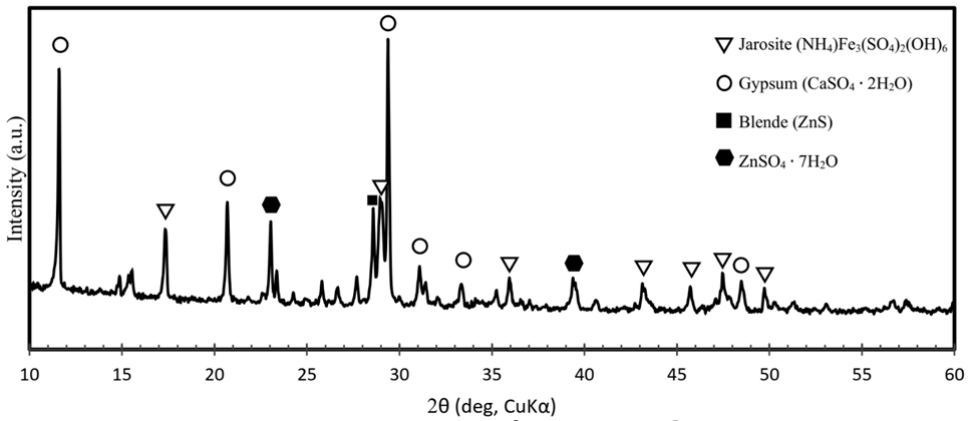

Figure 1. XRD pattern of jarosite residue.

3. RESULTS AND DISCUSSION

3.1 Thermal decomposition (TGA/DTG)

The residue exhibits a multistage mass-loss sequence (total ≈49%): initial dehydration (<100 °C), dehydroxylation and deammoniation (≈140–230 °C), lattice collapse with onset of sulfate breakdown (≈380 °C), and a minor event above ≈750–780 °C associated with conversion of residual sulfates to oxide/slag species fully consistent with literature on ammonium jarosite [3]. Multistage behavior is clearly illustrated in the TGA profile (Figure 2). These landmarks define roasting windows for phase steering and for avoiding extensive $ZnFe_2O_4$ growth [3,4].

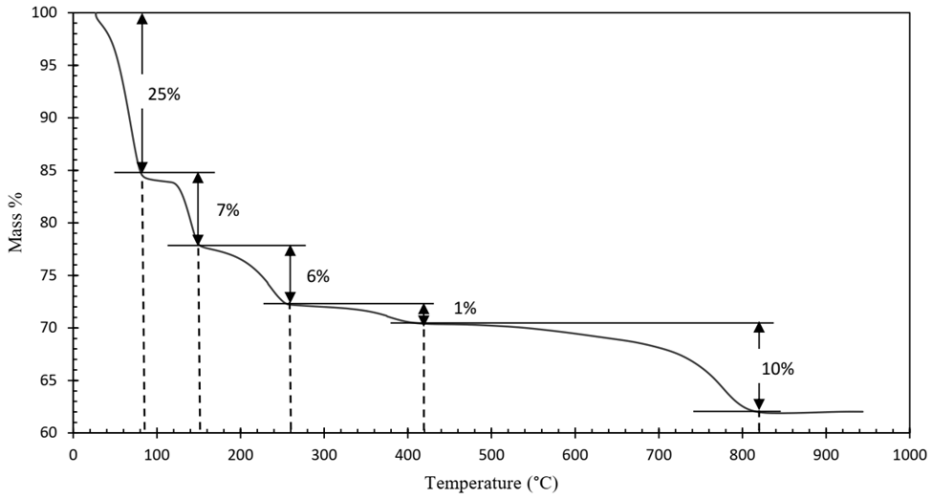

Figure 2. Thermogravimetric analysis (TGA) of the industrial ammonium jarosite sample.

3.2 Unfluxed roasting (100% jarosite)

XRD evolution follows jarosite → α-Fe_2O_3 + ZnO → $ZnFe_2O_4$ as temperature rises in air. The tendency of ZnO to react with hematite to form zinc ferrite is pronounced at ≥800–1000 °C and is a well-known barrier to zinc recovery due to the spinel's chemical robustness [3,4]. The corresponding phase evolution identified by XRD is shown in Figure 3.

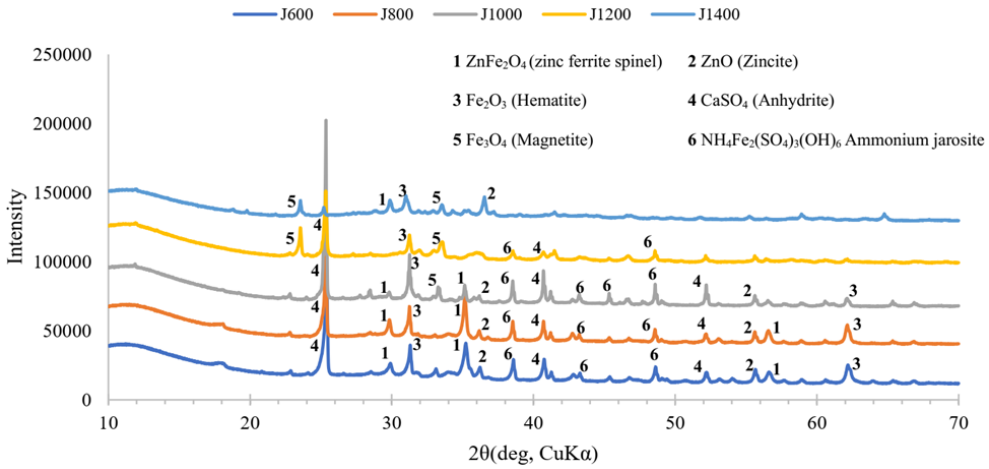

Figure 3. XRD pattern of phase evolution of pure jarosite roasted in air at different temperatures.

3.3 Flux-assisted roasting (jarosite + Na_2CO_3 + SiC)

The Na_2CO_3 and SiC additions modify the compounds formation: by 600–800 °C, jarosite has decomposed, and thenardite (Na_2SO_4) dominates, indicating efficient sulfur fixation in condensed phases. Fe partitions mainly into magnetite (Fe_3O_4) and hematite (Fe_2O_3). $ZnFe_2O_4$ is suppressed or delayed relative to the pure jarosite within 600–1200 °C. Fe–Si–O frameworks may appear via reaction of FeO-bearing domains with SiO_2 formed from SiC oxidation. At 1400 °C, sintering/incipient liquid is evident, with Na_2SO_4 still detectable and Fe_3O_4/α-Fe_2O_3 prevailing [4–8]. These transformations are confirmed by the XRD patterns displayed in Figure 4.

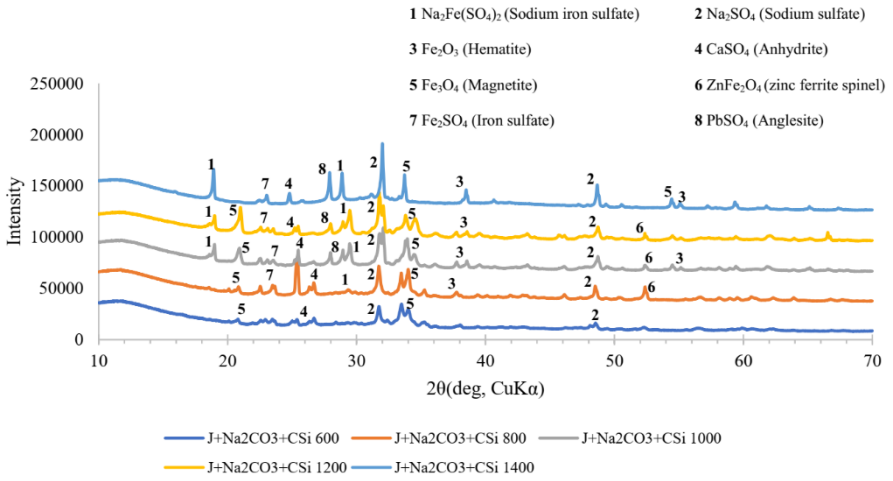

Figure 4. Phase evolution of roasted jarosite with fluxing and reducing agent at different temperatures.

Na_2CO_3 ties sulfur into stable alkali/alkaline-earth sulfates ($Na_2SO_4 \pm CaSO_4$), diverting Fe away from Zn–ferrite coupling; SiC fosters $Fe^{3+} \rightarrow Fe^{2+}$ and magnetite stabilization under globally oxidizing but locally reducing conditions. The combined effect yields magnetite-rich, spinel-lean assemblages advantageous for downstream processing or immobilization, consistent with prior thermogravimetric/thermodynamic demonstrations for jarosite residues [4–8].

3.4 Microstructure and elemental speciation (SEM/EDS)

Figure 5 shows a SEM-EDS analysis of the jarosite roasted with the addition of Na_2CO_3 and SiC reagents at 1200 °C. The image obtained using backscattered electrons of the fluxed products reveals alkali–silicate domains (Si–Al–Na), sulfate-rich grains (Na_2SO_4/$CaSO_4$), and oxide/silicate

matrices that host Fe. EDS confirms strong S and Na signals in sulfate grains (sulfur fixed in condensed phases). At the same time, Zn and Pb show low counts in the matrix consistent with (i) dispersion into submicrometric phases below SEM resolution, (ii) partial incorporation into amorphous silicate/sulfate matrices, or (iii) redistribution among minor sulfide/silicate hosts. Ag remained below EDS detection in the surveyed fields. These observations corroborate the XRD-inferred sulfur fixation and spinel suppression along the flux-assisted route [4–7] and are consistent with thermodynamic and experimental evidence that, at ~1200 °C under Na_2CO_3–SiC conditions, liquid-assisted segregation can promote Pb–Ag alloy droplet formation and matrix differentiation in jarosite-bearing systems [8,9].

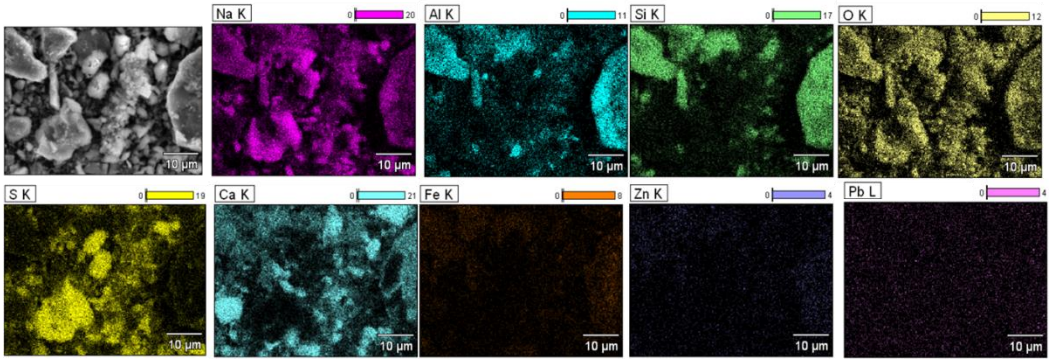

Figure 5. SEM / EDS analysis for jarosite with fluxing and reducing agent at 1200°C.

3.5 Process implications

Pure roasting efficiently removes structural water/NH_4^+ but traps Zn in $ZnFe_2O_4$, limiting leachability. In contrast, Na_2CO_3 + SiC simultaneously fixes sulfur, suppresses $ZnFe_2O_4$, and steers Fe into magnetite/oxide–silicate matrices. This phase tailoring is a promising platform for selective recovery (e.g., Zn via targeted leaching; Pb–Ag coalescence under optimized, more reducing co-roast conditions) or for environmental immobilization [4–8].

4. CONCLUSIONS

Flux-assisted roasting of industrial ammonium jarosite with Na_2CO_3 + SiC (600–1400 °C, air) delivers sulfur fixation (Na_2SO_4/$CaSO_4$), suppresses $ZnFe_2O_4$, and favors magnetite/oxide–silicate matrices, in agreement with recent reports on jarosite valorization. These outcomes define processing windows to minimize gaseous S, reduce refractory spinel formation, and condition residues for metallurgical recovery or stabilization.

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