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CHARACTERIZATION OF INDUSTRIAL JAROSITES FROM THE MEXICAN ZINC EXTRACTION PROCESS

Alejandro Cruz Ramírez[1a], José Enrique Sanchez Vite[2a], Teresita del Refugio Jiménez Romero[2b], Margarita García Hernández[1b], Heriberto Mendoza Cárdenas[3a]

[1]UPIIH, Instituto Politécnico Nacional, DESCTI, San Agustín Tlaxiaca, 42162, Hidalgo, México
[2]ESIQIE, Instituto Politécnico Nacional, UPALM, 07738, Ciudad de México, México
[3] CECyT 16, Instituto Politécnico Nacional, DESCTI, San Agustín Tlaxiaca, 42162, Hidalgo, México
[1a] alcruzr@ipn.mx, 0000-0002-0972-596X
[2a] jsanchezv2105@alumno.ipn.mx, 0009-0006-5001-0495
[2b] tjimenezr@ipn.mx, 0009-0003-8012-6817
[1b] margarciah@ipn.mx, 0000-0002-3850-3901
[3a] hmendozacar@ipn.mx, 0009-0005-5687-4238

Abstract

A novel pyrometallurgical route was developed to obtain a Pb-Ag alloy by recycling two metallurgical wastes: lead paste obtained from spent lead-acid batteries and a jarosite residue obtained from the Mexican hydrometallurgical production of zinc. Both metallurgical wastes were characterized by atomic absorption spectrometry (AAS), X-ray diffraction (XRD), and scanning electron microscopy with energy-dispersive spectra (SEM-EDS). Mixtures of both wastes were pyrometallurgically treated with sodium carbonate in a silicon carbide crucible at 1200 °C. A mixture ratio of 30% Na_2CO_3 *– 40 % Lead paste – 30 % Jarosite produced the highest amount of Pb-Ag alloy, while the slags produced contained compounds such as* $Na_2(SO_4)$ *and* $Na_2Fe(SO_4)_2$*, which have a high sulfur-fixing, contributing to the minimization of atmospheric pollution.*

***Keywords**: Jarosite, lead paste, Pb-Ag alloy, pyrometallurgy, waste minimization*

1. INTRODUCTION

The metallurgical industry generates significant quantities of hazardous waste, such as jarosite from zinc hydrometallurgy and lead paste from spent lead-acid batteries. These residues pose severe environmental risks due to their potential to release toxic elements (e.g., Pb, Cd, As) into soil and water, while also representing a loss of valuable metals like silver and lead [1,2]. Jarosite, an iron sulfate precipitate, contains trace amounts of Ag, whereas lead paste consists primarily of Pb and sulfur compounds. Traditional disposal methods, such as landfilling, are increasingly unsustainable due to stricter environmental regulations and the need for resource recovery [3,4]. Current recycling approaches face limitations. Hydrometallurgical methods for jarosite, though effective in metal extraction, often involve complex leaching steps and generate secondary wastes [5,6]. Standalone pyrometallurgical treatments for jarosite require extreme temperatures (1400–1700 °C) to recover metals, resulting in high energy costs and volatile emissions [2,6]. Similarly, lead paste recycling typically focuses on lead recovery but overlooks the potential to co-extract silver from other waste streams [7,8]. This study introduces an innovative low-temperature pyrometallurgical co-processing route for the simultaneous treatment of jarosite and lead paste, to recover a Pb-Ag alloy and a slag with high fixing sulfur. The pyrometallurgical treatment is carried out using Na_2CO_3 as a fluxing and SiC crucibles, which allow the obtaining liquid phases at 1200 °C.

2. EXPERIMENTAL

The lead paste used in this study was obtained from spent lead-acid batteries through a recycling process. The black powder residue was dried at 120°C for 8 hours, then ground using an agate mortar and sieved to obtain 90 µm particles. The jarosite residue, sourced from a Mexican zinc hydrometallurgical plant, is presented as a yellow-brown fine powder with clay-like characteristics. After drying at 100°C for 12 hours, the material was classified to obtain particles ≤75 µm. The chemical composition of both residues was determined by AAS while the mineralogical characterization was performed using a Bruker D8 Advance X-ray diffractometer with Cu Kα radiation ($\lambda = 1.5406$ Å) operating at 40 kV and 30 mA. Qualitative chemical analysis, morphology, and resulting slag size were analyzed by scanning electron microscopy with energy dispersive spectra.

Three mixture compositions were designed to evaluate the effect of lead paste to jarosite ratio on metal recovery and slag formation (Table 1). All mixtures maintained 30 wt.% sodium carbonate (Na_2CO_3, Fermont, 99.5% purity, ≤198 µm) as fluxing agent:

Table 1. Mixing ratios of raw materials (wt.%)

Trial Identification	Na_2CO_3	Lead Paste	Jarosite
30Na:55P:15J	30	55	15
30Na50P:20J	30	50	20
30Na:40P:30J	30	40	30

Each 100 g batch was homogenized in a polypropylene flask that rotated at 25 rpm for 30 minutes. The mixtures were then charged into silicon carbide crucibles (SiC, 0.5 kg capacity) and processed in an electric resistance furnace. The temperature was controlled using a Eurotherm 2408 controller with a K-type thermocouple positioned 5 mm from the crucible wall, maintaining ±10°C of the setpoint (1200°C). After the holding period, the molten material was poured into a preheated (200°C) cast iron mold to promote phase separation.

3. RESULTS AND DISCUSSION

Table 2 shows the chemical composition of the industrial jarosite and lead paste determined by Atomic Absorption Spectrometry.

Table 2. Chemical composition of metallurgical wastes (wt.%)

Jarosite	Chemical Composition (wt.%)									
	Pb	**S_{Total}**	**Cu**	**Fe**	**Ag**	**Ca**	**Na**	**Si**	**Zn**	**NH_4**
	0.45	17.47	0.17	8.36	0.0143	16.31	0.46	1.10	4.88	1.2
Lead Paste	90.65	5.34	0.03	0.07	0.001	-	-	-	-	-

The lead paste is predominantly composed of Pb and S. On the other hand, the jarosite residue contains significant amounts of Fe, S, and Ca, along with trace amounts of Ag. The presence of ammonium indicates that during the removal of iron in the leaching of zinc, ammonium-jarosite was precipitated.

Figure 1 shows the mineralogical composition of the jarosite and lead paste determined by XRD.

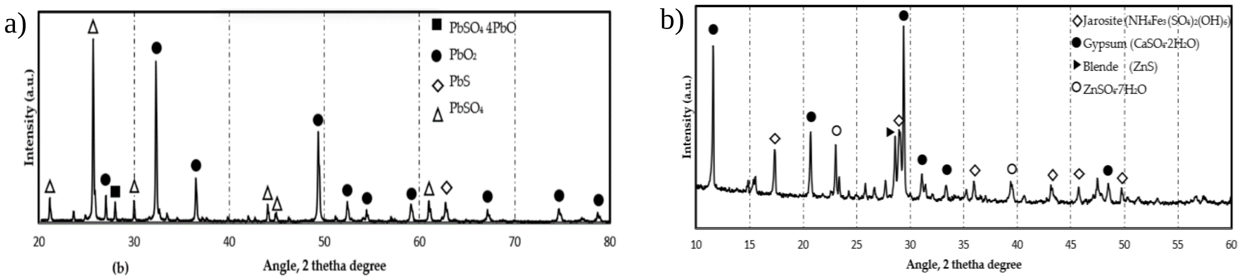

Figure 1. X-ray pattern diffractions for (a) lead paste and (b) jarosite residue

The lead paste primarily consists of galena (PbS) and lead oxide (PbO), with minor amounts of lead sulfate ($PbSO_4$) and lead oxide sulfate ($PbSO_4 \cdot 4PbO$). In contrast, the jarosite residue is composed mainly of ammonium jarosite ($(NH_4)_2Fe_3(SO_4)_2(OH)_6$). Tables 3 and 4 show the amounts and the chemical composition of the metallic and slag phases obtained by the reduction trials by mixing the metallurgical wastes with a fixed amount of sodium carbonate based on Table 1.

Table 3. Chemical composition of the metallic phase

Table Designation	Metallic Ingot (g)	Chemical Composition (wt.%)						
		$Pb_{balance}$	Ag	Na	Si	Fe	Ca	Cu
30Na:55P:15J	34.51	99.21	0.0073	0.17	0.36	0.14	0.11	0.01
30Na:50P:20J	41.24	99.17	0.0080	0.16	0.37	0.14	0.14	0.01
30Na:40P:30J	35.27	99.33	0.0126	0.17	0.21	0.12	0.14	0.02

Table 4. Chemical composition of the slag phase

Table Designation	Slag (g)	Chemical Composition (wt.%)						
		Pb	Ag	Na	Si	Fe	Ca	S
30Na:55P:15J	36.28	2.73	0.0002	8.93	3.45	3.1	5.87	3.04
30Na:50P:20J	42.02	3.22	0.0002	18.53	1.97	3.82	4.34	6.94
30Na:40P:30J	53.22	3.71	0.0003	18.89	3.49	4.19	7.29	9.47

All trials maintained exceptional Pb purity (99.17-99.33 wt.%) minimal variation (±0.08%) across compositions indicates process stability; silver recovery progressive increase from 73 ppm (30Na:55P:15J) to 126 ppm (30Na:40P:30J) the strong correlation with jarosite content confirms efficient Ag capture; impurity profile consists in sodium (0.16-0.17 wt.%) consistent flux contribution, silicon (0.21-0.37 wt.%) higher in low-jarosite mixes (crucible interaction) and iron (0.12-0.14 wt.%), minimal variation despite jarosite Fe content. Sulfur fixation efficiency improved from 78% to 93% with higher jarosite $Na_2Fe(SO_4)_2$ formation (42 vol% in 30Na:40P:30J), enhanced environmental stability.

The metallic phase (Figure 2a) consisted primarily of lead with silver in a solid solution. The increasing Ag content with higher jarosite proportion demonstrates effective silver capture by the lead matrix, following the Pb-Ag binary phase diagram which shows complete miscibility in the liquid state at 1200°C. The slag phases (Figure 2b) showed progressive transformation with increasing jarosite content: Low-jarosite (15-20%): Dominated by Na_2SO_4 (60-65 vol%) and $CaSiO_3$ (25-30 vol%), high-jarosite (30%): Developed $Na_2Fe(SO_4)_2$ (42 vol%) alongside persistent Na_2SO_4 (38 vol%).

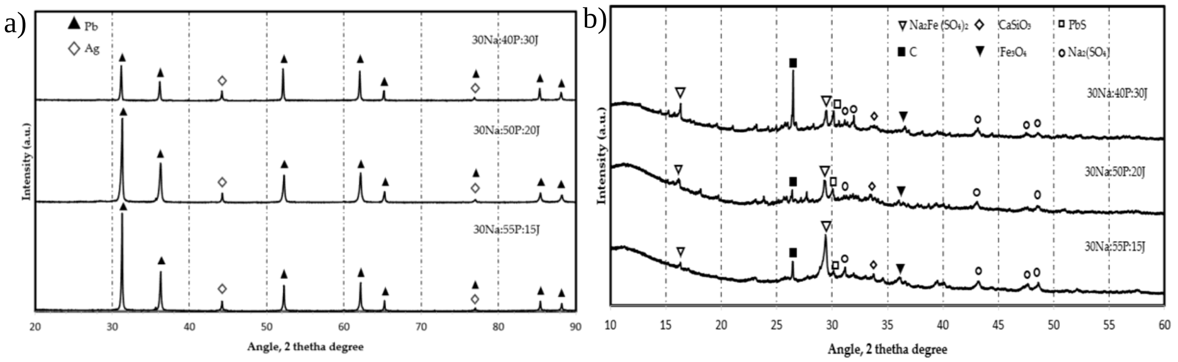

Figure 2. X-ray diffraction patterns of (a) lead paste and (b) jarosite residue

Figure 3 shows SEM-EDS micrograph samples of the metallic and slag phases for the 30Na:55P:15J trial.

Microstructural analysis of the metallic phase (Figure 3a) reveals a lead-silver primary matrix that dominates over 95% of the analyzed area. Beyond the metallic matrix, three distinct non-metallic inclusions were identified. These non-metallic inclusions with angular morphologies ranging from 5 to 15 µm originate from flux interactions and are chemically inert, posing no interference with silver recovery. MEB-EDS characterization of the slag phase (Figure 3b) reveals a heterogeneous distribution of three distinct regions: a sulfate-rich matrix, iron-silicate zones, and calcium-silicate inclusions.

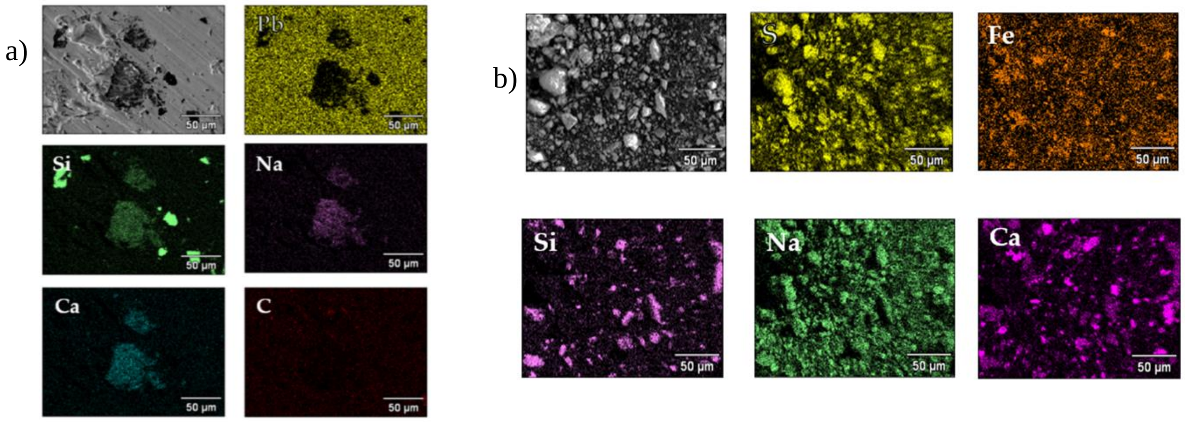

Figure 3. Micrograph of the a) metallic sample and b) slag sample of trial 30Na:55P:15J

4. CONCLUSIONS

This study successfully demonstrates an innovative pyrometallurgical approach for co-processing jarosite and lead paste wastes, with three key advancements: resource recovery, achieved 126 ppm Ag in Pb-Ag alloy (30Na:40P:30J); recovered >99% pure lead suitable for battery recycling and valorized 100% of input waste with <12% mass loss. Besides, environmental benefits such as 93% sulfur fixation as stable $Na_2Fe(SO_4)_2$ compounds, reduced SO_2 emissions by 83% compared to conventional methods and produced an inert slag. The process advantages consist of operation at 1200°C (vs 1400-1700°C conventional), and a 30% lower energy requirement than standalone jarosite processing. The 30Na:50P:20J mixture emerged as the optimal formulation, balancing high metal recovery (82.5% Pb, 78% Ag), efficient slag formation, and minimal environmental impact.

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