

Article

Selective Removal of Arsenic and Antimony by Alkaline Leaching with Sodium Sulfide: Remediation of Metalloids-Contaminated Concentrates from Zimapán, Hidalgo, Mexico

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Abstract

Selective alkaline leaching was evaluated to remove arsenic (As) and antimony (Sb) from a polymetallic copper concentrate from Zimapán, Mexico, where these metalloids cause environmental risk and smelter penalties. Batch tests used sodium sulfide (Na₂S) in alkaline media, varying reagent concentrations and temperature; kinetic modeling identified the rate-controlling step, and X-ray diffraction (XRD) plus scanning electron microscopy/energy-dispersive spectroscopy (SEM–EDS) assessed phase changes. The kinetic analysis indicated chemical control with a higher reaction order for Na₂S than for NaOH. A quadratic regression described the process and identified Na₂S concentration and temperature as the dominant factors. Maximum extractions reached 91.9% for As and 72.1% for Sb while limiting dissolution of value-bearing sulfides, as supported by XRD and SEM–EDS. Environmental indices (*I*_{geo}, EF) classified As and Sb as highly contaminating and geochemically enriched in the feed, underscoring the need for selective removal. Overall, alkaline leaching with Na₂S provides a technically feasible and environmentally favorable route to remediate metalloids and upgrade polymetallic concentrates.

Keywords: selective leaching; alkaline leaching; sodium sulfide; arsenic; antimony; copper concentrate; kinetics; Zimapán; chalcopyrite; environmental remediation



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1. Introduction

The Zimapán mining district (Hidalgo, Mexico) is known for producing polymetallic concentrates containing metals of interest such as copper and silver; however, they often contain arsenic (As) and antimony (Sb), elements that pose environmental, regulatory, and metallurgical challenges by increasing toxicological risk and leading to commercial penalties [1–8]. In parallel, the demand for Cu and Ag for electronics, transportation, and low-carbon energy technologies continues to rise [9,10], which is driving metal recovery from both primary materials—ores and their concentrates (process intermediates)—and

secondary resources, including tailings, slags, smelter dusts, and waste electrical and electronic equipment (WEEE), in order to reduce pressure on natural resources and mitigate environmental impacts [11,12]. Both arsenic and antimony occur in these material types and can be mobilized under acidic to neutral conditions through redox processes, facilitating dispersion into surrounding soils and waters [13–20].

This high mobility promotes accumulation in terrestrial and aquatic ecosystems, complicating remediation and posing risks to public health [21–34]. In response, international regulations have established maximum permissible limits for these elements in waters, soils, and industrial emissions [35–37]. From a metallurgical standpoint, As and Sb hinder selective flotation, complicate leaching and smelting, and depress concentrate value via market penalties [1,2,38]. Consequently, multiple strategies have been developed for their selective removal from concentrates and mining by-products to reduce environmental impact, improve product quality, and avoid penalties [38]. Among the most used methods are differential flotation, removal from industrial effluents, and selective acidic leaching with H_2SO_4 , HCl , and HNO_3 , which have shown high efficiency but can generate toxic gases [39–43].

In contrast, the Na_2S – NaOH system has been investigated for its high selectivity in extracting As and Sb from copper concentrates rich in enargite and tetrahedrite, enabling preferential dissolution of these elements without affecting copper recovery and thereby enhancing metallurgical efficiency [44–47].

In this context, this study identifies the variables that significantly influence the removal of As and Sb from a copper concentrate from the Zimapán district. We analyze the kinetic behavior of alkaline dissolution via reaction orders and activation energy (E_a) to understand how operating variables affect the reaction rate, and we develop a quadratic regression model to determine the relative influence of each factor on extraction efficiency. The study is complemented by mineralogical and morphological characterization to validate concentrate composition and the selectivity of the Na_2S – NaOH system and by estimating the geoaccumulation index (I_{geo}) and enrichment factor (EF) to contextualize the potential environmental impact.

2. Materials and Methods

2.1. Materials

The copper concentrate from Zimapán, Hidalgo, Mexico, was homogenized and classified using ASTM sieves (150, 106, 75, 53, 45, 38, and $<38\ \mu\text{m}$). Unless otherwise stated, the particle size used during experimentation was the $-149/+105\ \mu\text{m}$ fraction. The elemental composition was determined by Inductively Coupled Plasma–Optical Emission Spectrometry ICP-OES (PerkinElmer 8300) (PerkinElmer Inc., Waltham, MA, USA) after aqua regia digestion. Approximately $0.1000 \pm 0.0001\ \text{g}$ of dried sample was placed in Erlenmeyer flasks, 20 mL of freshly prepared aqua regia ($\text{HCl}:\text{HNO}_3$, 3:1) was added, and the mixture was digested at 388–393 K for 2 h on a hot plate with a reflux cap to minimize As/Sb losses by volatilization. After cooling to room temperature, the digest was quantitatively transferred and diluted to 100 mL with deionized water and then filtered. The crystalline phases present in the sample were identified by XRD (INEL EQUINOX 2000, Co- $\text{K}_{\alpha 1}$ radiation, $\lambda = 1.78901\ \text{\AA}$, (INEL S.A., Artenay, France)). Morphology was examined by SEM-EDS (JEOL JSM-5900LV), (JEOL Ltd., Tokyo, Japan), coupled with an energy-dispersive X-ray spectroscopy (EDS, Oxford) system. Thermodynamic modeling of the reactions was performed using HSC Chemistry software version 6 (Metso Outotec, Espoo, Finland). For the leaching tests of As and Sb, deionized water was used along with sodium sulfide ($\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$, purity $> 99\%$, molecular weight: 240.18 g/mol, supplied by Técnica Química S.A., Mexico City, Mexico) and sodium hydroxide (NaOH , purity $> 97\%$,

molecular weight: 40.0 g/mol, supplied by MEYER., Mexico City, Mexico). Based on the data obtained by ICP, the geoaccumulation index (I_{geo}) and enrichment factor (EF) were calculated for selected trace elements in order to assess the contaminant potential of the concentrate. The geoaccumulation index was calculated using the expression proposed by Müller (1979) (Equation (1)), with Fe considered as the normalizing element [48].

$$I_{\text{geo}} = \log_2 \frac{C_n}{1.5B_n} \quad (1)$$

where C_n is the concentration of the element of interest in the sample and B_n is the geochemical background concentration of the element in the Earth's crust. The I_{geo} index allows for the classification of contamination levels, ranging from unpolluted ($I_{\text{geo}} \leq 0$) to extremely polluted ($I_{\text{geo}} > 5$).

In addition to I_{geo} , we computed the (x/Fe) index, defined as the concentration ratio of element x to Fe. Values with $(x/\text{Fe})_{\text{sample}} \approx (x/\text{Fe})_{\text{background}}$ indicate a lithogenic behavior (no enrichment); $(x/\text{Fe})_{\text{sample}} > (x/\text{Fe})_{\text{background}}$ suggests a relative enrichment of x ; and $(x/\text{Fe})_{\text{sample}} < (x/\text{Fe})_{\text{background}}$ implies depletion. Fe is used as the denominator due to its abundance and relatively conservative behavior in sulfide-rich matrices, serving as a suitable internal normalizer to compare element-to-element variations, and was calculated using the following expression:

$$\text{EF}_x = \frac{\left(\frac{x}{\text{Fe}} \right)_{\text{sample}}}{\left(\frac{x}{\text{Fe}} \right)_{\text{Crust}}} \quad (2)$$

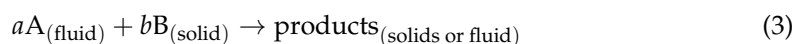
where $(x/\text{Fe})_{\text{sample}}$ is the ratio between the concentration of the element of interest and the concentration of iron in the sample, and $(x/\text{Fe})_{\text{Crust}}$ refers to the corresponding ratio in the Earth's crust. Iron was used as the reference element, as it is the most abundant economically relevant base element in the parent rock [49,50]. The enrichment factor highlights relative enrichment or depletion with respect to the chosen background and can be used to support the identification of elements with potential for recovery [51].

2.2. Leaching Test

Leaching tests were carried out in batch mode in a thermostated glass reactor with agitation ($\omega = 600 \text{ min}^{-1}$). Solutions of Na_2S ($0.25\text{--}1.75 \text{ mol} \cdot \text{L}^{-1}$) and NaOH ($0.75\text{--}2.0 \text{ mol} \cdot \text{L}^{-1}$) were prepared in deionized water. The $-149/+105 \text{ }\mu\text{m}$ fraction of the concentrate was fed at a constant solid-to-liquid ratio of $S/L = 10 \text{ g/L}$, and the pH was kept ≥ 12 throughout the reaction. The temperature was set between 343 and 373 K depending on the condition, and each test was run for 360 min. pH was monitored throughout each test and remained ≥ 12 in all cases. ORP was checked periodically to confirm operation within an alkaline-reducing window consistent with the Na_2S – NaOH system. At the end, the liquor was filtered, the solid was washed and dried for XRD, and the filtrate was analyzed by ICP–OES for As and Sb. Each condition was executed as a single run; therefore, the curves are reported without error bars.

2.3. Kinetic Parameters: Reaction Orders (α , β) and Apparent Activation Energy (E_a)

The determination of reaction orders (α , β) and activation energy (E_a) enables the identification of the controlling mechanism governing the leaching of As and Sb in heterogeneous solid–liquid systems (Equation (3)), such as their removal from metal sulfides.



In this equation, a and b are the stoichiometric coefficients, while A and B represent the fluid and solid-phase reactants, respectively. If the surface reaction occurs rapidly and mass transfer is the limiting step, the process is diffusion-controlled. Conversely, if the chemical reaction at the solid–liquid interface is slow—such as the cleavage of metal–sulfur bonds and the formation of sulfur complexes—then the process is chemically controlled. Distinguishing between these mechanisms is essential for optimizing process efficiency and selecting appropriate operating conditions. Accordingly, both control mechanisms can be expressed as a function of fractional conversion (X_{As} and X_{Sb}) and fitted to kinetic models to evaluate the nature of the process [52,53], as summarized in Table 1.

The reaction rate in a heterogeneous system can be expressed as the change in the concentration of the solid reactant B (n_B) over time (t), normalized with respect to the specific reaction surface area (S), as shown in Equation (4):

$$-\frac{1}{S} \left(\frac{dn_B}{dt} \right) = k \prod [A_i]^{n_i} \quad (4)$$

where k is the rate constant (min^{-1}), and $\prod [A_i]^{n_i}$ represents the product of the concentrations of the reactants in solution raised to their respective reaction orders, as shown below:

$$\prod [A_i]^{n_i} = [\text{Na}_2\text{S}]^\alpha [\text{NaOH}]^\beta \quad (5)$$

where α and β in Equation (5) represent the apparent reaction orders with respect to each reactant. Their experimental determination helps identify which reactant has the greatest influence on the reaction rate, thus enabling optimization of its concentration in future applications. Previous studies have shown that the leaching of As and Sb from complex sulfides such as tetrahedrite or enargite in alkaline media predominantly follows a chemically controlled mechanism [54–56].

Table 1. Integrated equations for the different rate-controlling stages.

Rate Controlling Stage	Kinetic Equation (as a Function of the Conversion $X_{As,Sb}$)	Experimental Rate Constant, k_{exp}	Symbol
Transport in the fluid film	$1 - (1 - X_{As,Sb})^{\frac{2}{3}} = k_{\text{ff}} t \quad (6)$	$k_{\text{ff}} = \frac{2bD\prod[A]}{\rho_B r_0} \quad (7)$	k_{ff} = experimental rate constant in the fluid film in min^{-1} b = stoichiometric ^a D = molecular diffusion coefficient in $\text{m}^2 \text{min}^{-1}$ $[A]$ = fluid reagent concentration in mol m^{-3a} ρ_B = molar density in mol m^{-3a} r_0 = initial radius of the particle in m
Chemical reaction	$1 - (1 - X_{As,Sb})^{\frac{1}{3}} = k_{\text{cr}} t \quad (8)$	$k_{\text{cr}} = \frac{bk_0' \prod[A]^n}{\rho_B r_0} \quad (9)$	k_{cr} = experimental rate constant in the chemical reaction in min^{-1} , k_0' = chemical rate constant in $(\text{mol m}^{-3})^{1-n} \text{min}^{-1}$ n = reaction order

^a consider in Equation (3).

On the other hand, to identify the reacted fraction of As and Sb, Equation (10) is used:

$$X = \frac{\text{mass reacted}}{\text{initial mass}} = \frac{\text{volume reacted}}{\text{initial volume}} \quad (10)$$

2.4. Statistical Analysis and Predictive Modeling

Based on the experimentally obtained extraction percentages for As and Sb, predictive statistical models were constructed using quadratic regression. These models were developed with the Statgraphics Centurion software 16.1 (Statgraphics Technologies, Inc., The Plains, VA, USA), considering Na_2S concentration, NaOH concentration, and temperature as independent variables. Quadratic terms were applied to generate predictive equations for both responses. Model validity was assessed through analysis of variance (ANOVA),

taking into account F -values, p -values, and coefficients of determination (R^2 and adjusted R^2), all of which indicated excellent goodness of fit. Using these equations, extraction values were calculated for various experimental combinations, serving as the basis for the analysis of main effects and factor interactions.

3. Results and Discussion

3.1. Characterization Details

The elemental composition of the copper concentrate (Table 2) revealed that copper (Cu), iron (Fe), and zinc (Zn) are the predominant elements in the sample. Silver (Ag), calcium (Ca), and lead (Pb) were also detected. Notably, the metalloids arsenic (As) and antimony (Sb) were identified at concentrations of 1.82 and 0.51 wt%, respectively. It is worth mentioning that the highest proportion of these elements was found in the particle size fraction between -149 and $+105\ \mu\text{m}$.

Table 2. Elemental analysis of the copper concentrate.

Mesh	(μm)	Composition (wt%)							
		Ag (gr ton^{-1})	Cu	Fe	Zn	Pb	As	Sb	Ca
>100	>149	41.67	21.17	11.42	7.37	0.89	1.19	0.34	2.18
$-100 + 140$	$-149 + 105$	144.33	22.33	11.02	7.60	3.32	1.82	0.51	0.65
$-140 + 200$	$-105 + 74$	75.61	23.09	12.21	8.11	1.60	1.72	0.44	0.67
$-200 + 270$	$-74 + 53$	111.83	24.24	13.84	8.39	2.10	1.61	0.34	0.56
$-270 + 325$	$-53 + 44$	68.06	21.43	12.37	1.00	0.69	0.96	0.32	2.71
$-325 + 400$	$-44 + 38$	60.33	22.25	12.98	8.06	0.95	0.78	0.35	2.26
< -400	<38	73.50	23.97	14.47	7.79	2.01	0.96	0.39	1.26

In the particle size distribution analysis (Figure 1), the d_{80} percentile corresponds to a particle diameter of $105\ \mu\text{m}$, indicating that 80% of the particles have a size equal to or greater than this value. It is important to note that, according to the elemental analysis, this particle size range exhibits the highest concentration (wt%) of As and Sb.

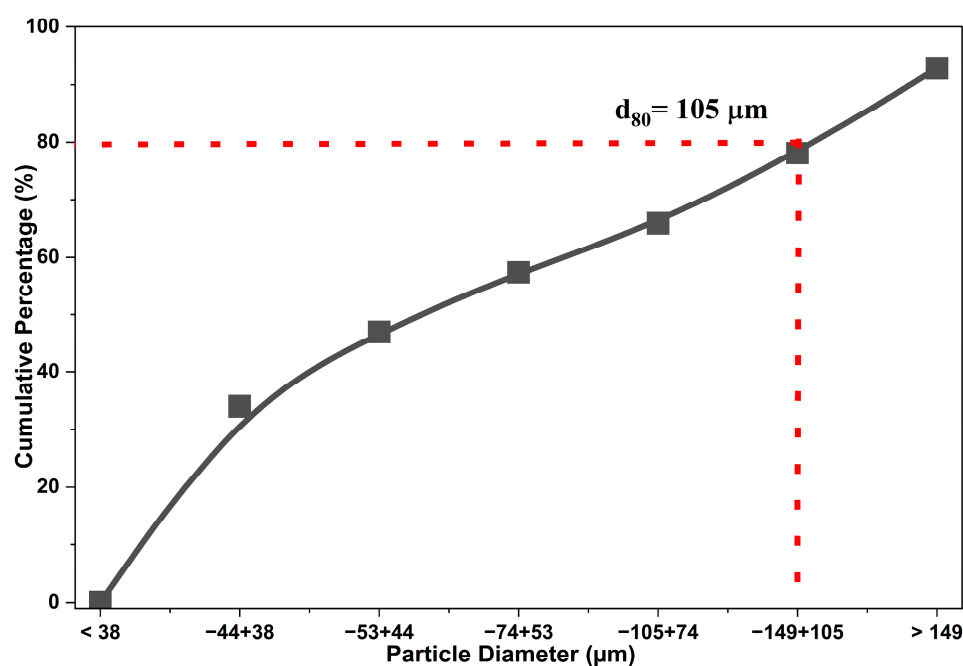


Figure 1. Particle size distribution of the copper concentrate.

The XRD pattern in Figure 2 reveals the presence of phases such as chalcopyrite (CuFeS_2), sphalerite (ZnS), anglesite (PbSO_4), and pyrite (FeS_2). Additionally, arsenic, antimony, and silver were identified as being predominantly present in the tetrahedrite phase ($\text{As}_{0.28}\text{Cu}_{10.08}\text{Fe}_{0.96}\text{S}_{13}\text{Sb}_{3.72}$). Characteristic peaks of gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and jarosite ($\text{KFe}_3(\text{SO}_4)_2(\text{OH})_6$) were also detected in minor amounts. These results are consistent with the mineralogy previously reported in the Zimapán mining district, Hidalgo, Mexico, where the presence of these minerals has been documented in studies on hydrometallurgical processes (7). Pyrite and chalcopyrite have been reported as the dominant sulfides in the mineral deposits of the region, while sphalerite and anglesite are associated with lead and zinc mineralization in the mineral concentrates [57,58]. Additionally, the presence of jarosite and gypsum is linked to metal neutralization and precipitation processes in leach residues [59,60].

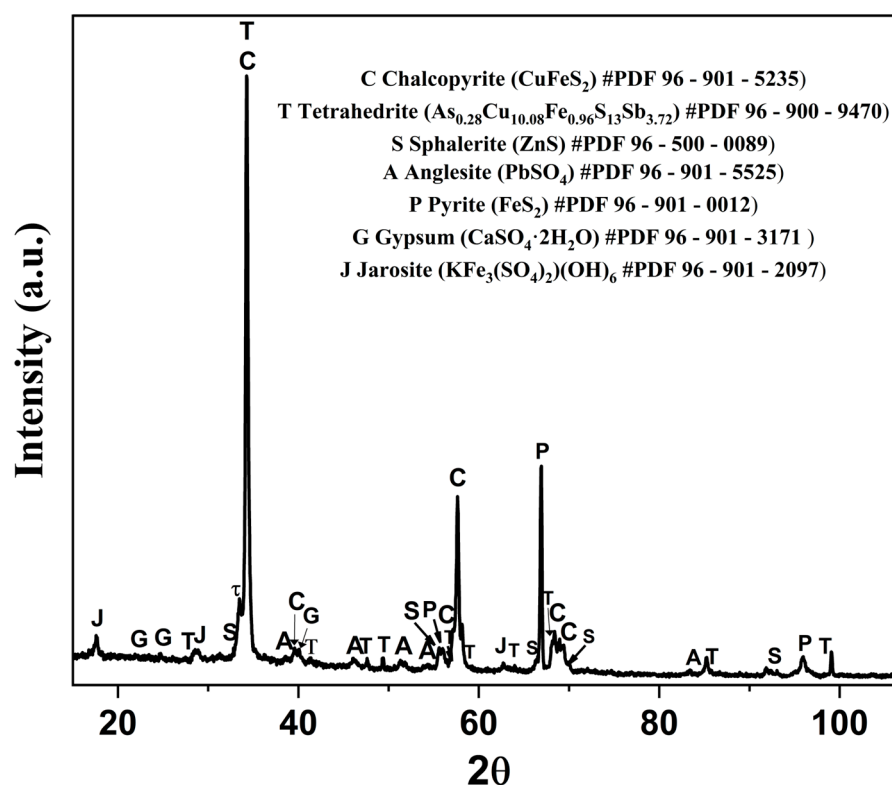


Figure 2. XRD pattern corresponding to the copper concentrate. # indicates the ICDD PDF card number used for phase matching.

According to scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS) (Figure 3), arsenic (As) occurs predominantly on particle surfaces in association with Cu, Fe, Zn, S, and, locally, Ca and Mg (Figure 3a). A magnified view (Figure 3b) shows an elongated aggregate where As and Sb coexist together with Cu–Fe–Zn–Pb–S, consistent with complex sulfosalt assemblages (e.g., tetrahedrite-type or Pb–Cu sulfosalts), in line with the XRD results. Point analyses marked i. and ii. correspond to Sb-rich and As-rich microdomains, respectively. For reference, panel (c) displays a chalcopyrite (CuFeS_2) area that is essentially free of As/Sb, representative of the major sulfide matrix.

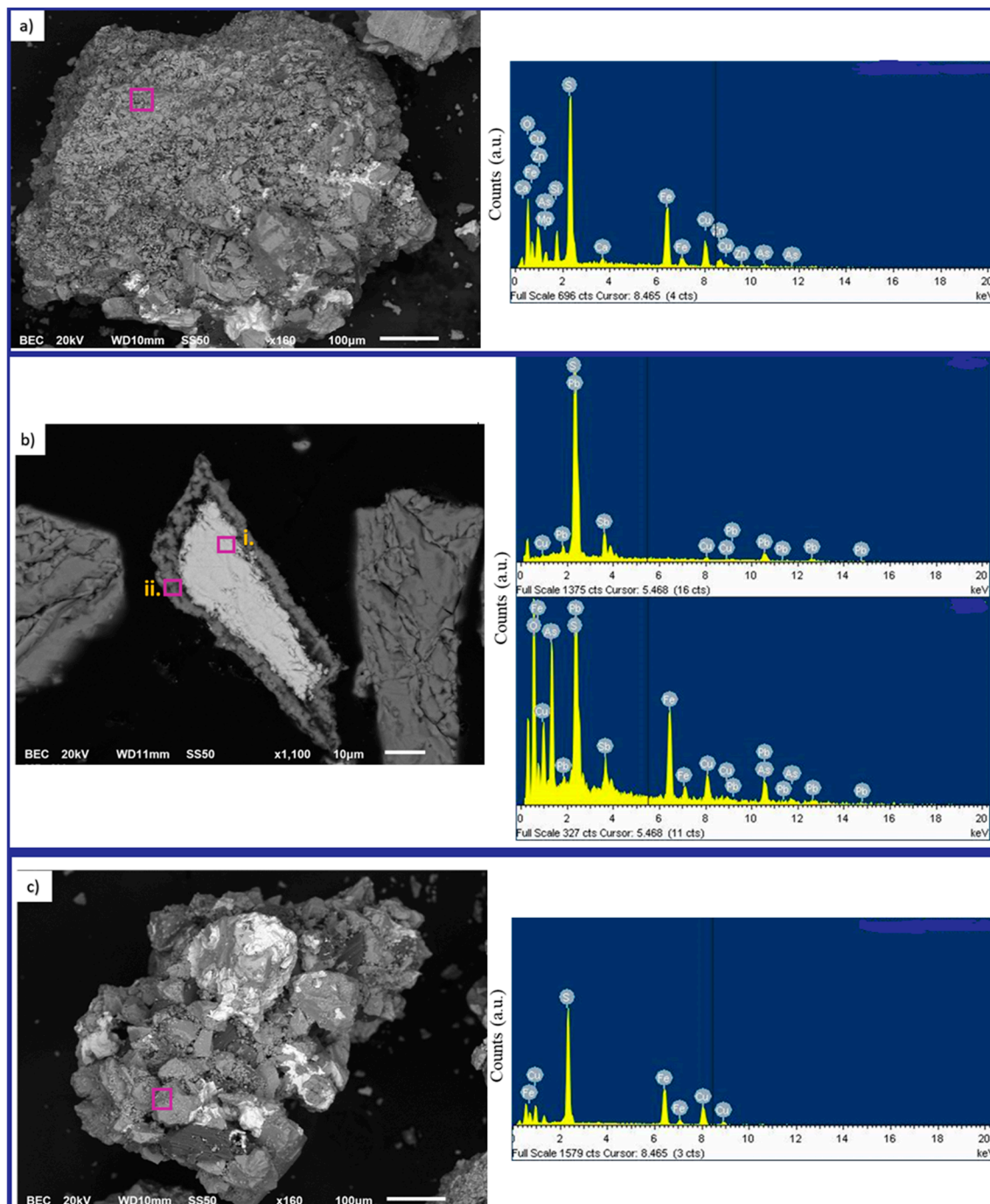


Figure 3. (a) SEM overview of the feed concentrate (scale bar = 100 μm). (b) SEM detail of a sulfide aggregate; purple boxes indicate regions of interest (ROIs) selected for EDS interrogation. Markers i. and ii. denote point-analysis locations corresponding to Sb-rich and As-rich microdomains, respectively. (c) Reference chalcopyrite (CuFeS_2) area showing the base sulfide matrix with negligible As/Sb signals.

3.2. Geoaccumulation Index and Enrichment Factors

In the Zimapán mining district, Hidalgo, a medium-scale mining company produces approximately 500 tons of polymetallic concentrate per day, which is mostly stored outdoors in areas near urban settlements. This condition facilitates exposure to erosion and weathering processes, promoting the mobility of trace elements such as arsenic (As) and antimony (Sb), particularly under sulfide oxidation and surface geochemical alteration environments [61]. In these materials, the presence of As and Sb is commonly associated with

complex sulfosalts such as pyrargyrite, proustite, xanthoconite, and tetrahedrite, which form part of the mineral matrix that also contains copper, iron, lead, and silver [27,62]. This type of mineralogical association favors the release of As and Sb during natural oxidation of the residues or their surface storage without encapsulation. To evaluate the contaminant potential of the studied concentrate, elemental compositions obtained by ICP were used to calculate the geoaccumulation index (I_{geo}) and the enrichment factor (EF), summarized in Figures 4 and 5. Figure 4 shows that elements such as iron (Fe) and calcium (Ca) exhibited I_{geo} values < 0 and $EF \approx 1$, classifying them as elements with no evidence of contamination or enrichment, and therefore not representing an immediate environmental threat. Iron (Fe), although present in high concentrations, showed intermediate values that place it within the moderately contaminated category, reflecting its primarily lithogenic origin [63,64]. In contrast, arsenic (As) and antimony (Sb) exhibited I_{geo} values greater than 5 and EF values above 10, placing them in the categories of extreme contamination and very high geochemical enrichment, respectively [65].

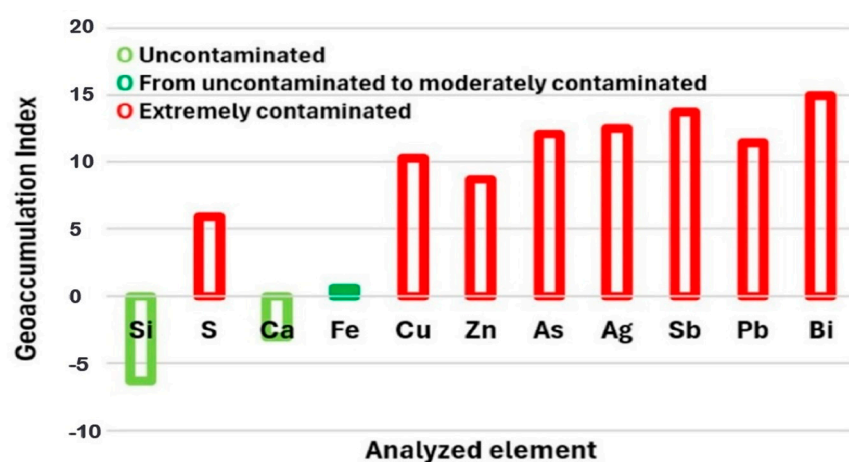


Figure 4. Contamination potential of the polymetallic concentrate based on I_{geo} and FE.

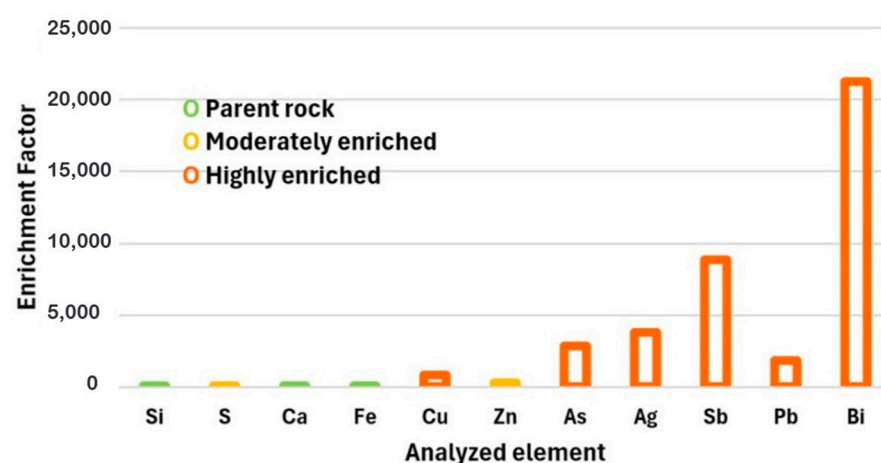


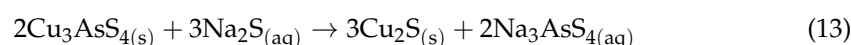
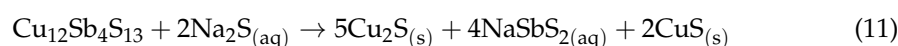
Figure 5. Geo-enrichment profile of the polymetallic concentrate.

This classification indicates not only a high relative concentration compared to background values but also a strong anthropogenic influence and high potential mobility under environmental conditions. Given the toxic nature of both arsenic (As) and antimony (Sb)—and the carcinogenicity of As, particularly in its trivalent form—these results reinforce the need for targeted mitigation measures to prevent its dispersion into the environment [66,67].

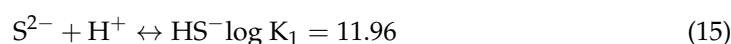
Figure 5 analyzed the calculated enrichment factors (EF) for the elements present in the concentrate. Elements such as calcium (Ca) and iron (Fe), previously identified as environmentally non-prioritary, exhibit EF values close to 1, confirming their lithogenic origin and natural abundance in the parent rock. Zinc (Zn), with a moderate enrichment value ($1 < EF < 5$), suggests limited potential for primary recovery but may be considered a byproduct if specific beneficiation stages are integrated into the metallurgical process [8,68]. In contrast, silver (Ag), arsenic (As), and antimony (Sb) exhibit the highest enrichment levels, supporting the classification of the concentrate as a viable secondary source of these strategic elements. Particularly, As and Sb stand out not only for their economic value but also for their environmental impact, highlighting the need for targeted recovery and mitigation strategies [69,70].

3.3. Extraction Profiles of As and Sb

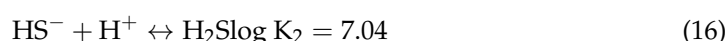
The extraction of As and Sb from mineral phases such as tetrahedrite in an alkaline medium using Na_2S proceeds according to Equations (11)–(14), where both arsenic and antimony are dissolved as thioanions (AsS_3^{3-} , AsS_4^{3-} , SbS_3^{3-} , and SbS_4^{3-}):



Accordingly, it is important to examine the stability of the sulfide ion (S^{2-}) during the leaching process. Equations (15) and (16) represent the acid–base equilibrium of the sulfide ion [71–73]. Initially, in an alkaline medium, the sulfide ion is present at a high concentration. However, as the pH decreases, protonation of the sulfide ion occurs, leading to the formation of the hydrosulfide ion (HS^-) (Equation (15)):



If the pH decreases below 7, the predominant aqueous species (Equation (16)) becomes H_2S , resulting from a second protonation step:



To ensure efficient dissolution of As and Sb and to avoid the formation of undesirable species, it is essential to maintain a controlled pH during the leaching process [74]. For this reason, an alkaline medium is critical to stabilize the thioanions formed during tetrahedrite leaching and to maintain an adequate concentration of S^{2-} in solution (Equation (17)):



In an alkaline medium, the sulfide ion plays a role as a nucleophile in the bonds formed of the metal–sulfur characteristic of the tetrahedrite structure, inciting the rupture of said bonds and the formation of thioarsenate and thioantimonate species, according to Equations (11)–(14). The reaction proceeds with the parallel formation of $\text{Cu}_2\text{S}/\text{CuS}$ as sparingly soluble phases, which “protect” the valuable sulfides (Cu, Ag) from an extensive dissolution process.

The speciation of the sulfide reagent is governed by the equilibria $\text{S}^{2-} \rightleftharpoons \text{HS}^- \rightleftharpoons \text{H}_2\text{S}$; therefore, working at a high pH is essential to maximize the free fraction of S^{2-} and stabilize

the complexes with As and Sb. Finally, to evaluate the influence of temperature on the stability of the leaching system, Eh–pH diagrams for the S–H₂O system were constructed at 343 K and 373 K (Figure 6a,b). These figures show that increasing the temperature broadens the stability region of the S^{2−} ion, favoring its participation in the process. Specifically, at 343 K, the sulfide ion remains stable within a pH range of 11.8 to 14 under reducing conditions, while at 373 K, this range extends from 11.27 to 14. Although the pH variation is minor, the results suggest that temperature not only enhances the reaction rate but also stabilizes the leaching ion within the reaction medium. Previous studies have shown that temperature is a critical factor in the leaching of sulfides, as it can improve the solubility of target species [75].

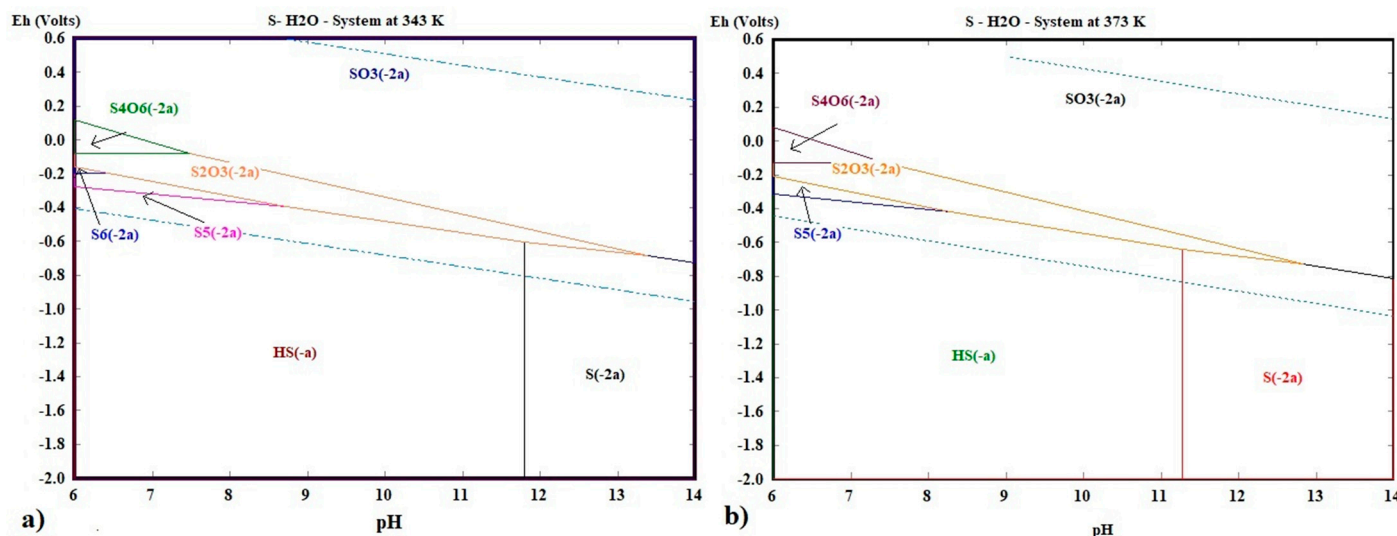


Figure 6. Eh–pH diagram of the S–H₂O system at: (a) 343.15 K and (b) 373.15 K.

1. Influence of Na₂S Concentration on the Extraction of As and Sb

The effect of Na₂S concentration on the extraction of As and Sb was evaluated over a range of 0.25 to 1.75 mol·L^{−1}, while keeping temperature (353.15 K) and NaOH concentration (1.5 mol·L^{−1}) constant (Figure 7). After 360 min of reaction, a low Na₂S concentration (0.25 mol·L^{−1}) resulted in limited extraction of As (28.3%) and Sb (15.7%). Increasing the concentration to 1.0 mol·L^{−1} improved recovery to 44.7% and 38.6%, respectively, indicating that higher Na₂S levels enhance the leaching of both elements. However, the most significant increase was observed between 1.0 and 1.5 mol·L^{−1}, where As and Sb dissolution reached 71.8% and 56.1%, respectively, highlighting the critical role of sulfur-based ligands in the process. At higher concentrations (1.75 mol·L^{−1}), As and Sb extraction reached 81.2% and 67.4%, respectively. In the absence of Na₂S (pH ≥ 12), no appreciable As/Sb leaching is anticipated because the thiocomplexation pathway is not available [46,76–78].

2. Influence of NaOH concentration on the extraction of As and Sb

Figure 8 shows the effect of NaOH concentration (0.75–2.0 mol·L^{−1}) on the extraction of As and Sb while keeping the temperature (353 K) and Na₂S concentration (1.25 mol·L^{−1}) constant. The results revealed that increasing NaOH concentration progressively enhances the dissolution of both elements. At 0.75 mol·L^{−1}, the extraction efficiencies for As and Sb were 39.1% and 33.0%, respectively, whereas at 2.0 mol·L^{−1}, the values reached 67.8% and 51.7%. Although this effect is positive, it is not as pronounced as the effect observed when varying Na₂S concentration, where the increases were more substantial. These findings suggest that while an alkaline medium is essential for the stability of soluble species,

the availability of sulfide ions plays a more decisive role in the overall efficiency of the leaching process.

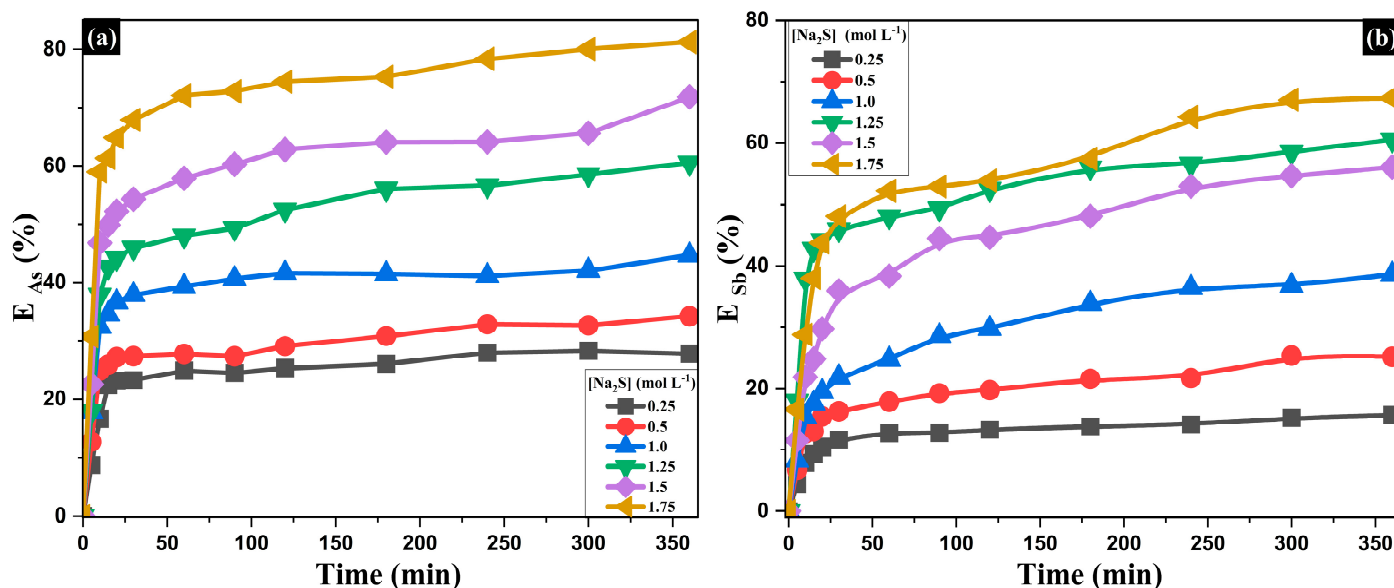


Figure 7. Influence of Na_2S concentration on the extraction of (a) arsenic and (b) antimony at 353.15 K and $[\text{NaOH}] = 1.5 \text{ mol}\cdot\text{L}^{-1}$.

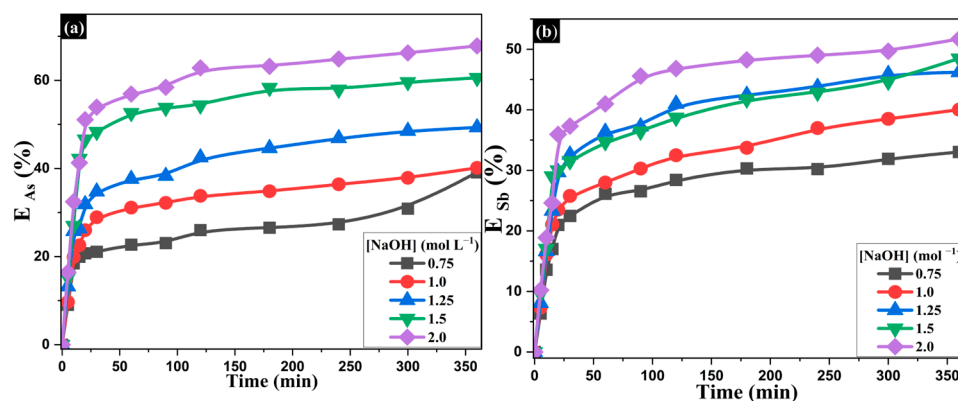


Figure 8. Influence of NaOH concentration on the extraction of (a) arsenic and (b) antimony at 353 K and $[\text{Na}_2\text{S}] = 1.25 \text{ mol}\cdot\text{L}^{-1}$.

3. Influence of Temperature on the Extraction of As and Sb

Based on the results shown in Figure 9, the influence of temperature (343–373 K) on the extraction of As and Sb was evaluated while keeping the concentrations of Na_2S ($1.25 \text{ mol}\cdot\text{L}^{-1}$) and NaOH ($1.5 \text{ mol}\cdot\text{L}^{-1}$) constant. A direct correlation was observed between increased temperature and enhanced extraction of both elements. At 343 K, extraction rates reached 47.2% for As and 36.9% for Sb, while at 373 K, they increased to 87.5% and 66.4%, respectively. This behavior is attributed to the acceleration of dissolution kinetics at higher temperatures, as well as to the enhanced stability of sulfurous species (S^{2-}) in alkaline media, which promotes the formation of soluble complexes with As (Figure 9a) and Sb (Figure 9b). When comparing the three evaluated parameters (Na_2S concentration, NaOH concentration, and temperature), it is confirmed that although all positively influence the leaching process, Na_2S concentration and temperature exert a significantly greater effect than alkalinity alone. This underscores the importance of a comprehensive design of leaching conditions to optimize metalloid recovery. The results from all three variables are summarized in Table 3.

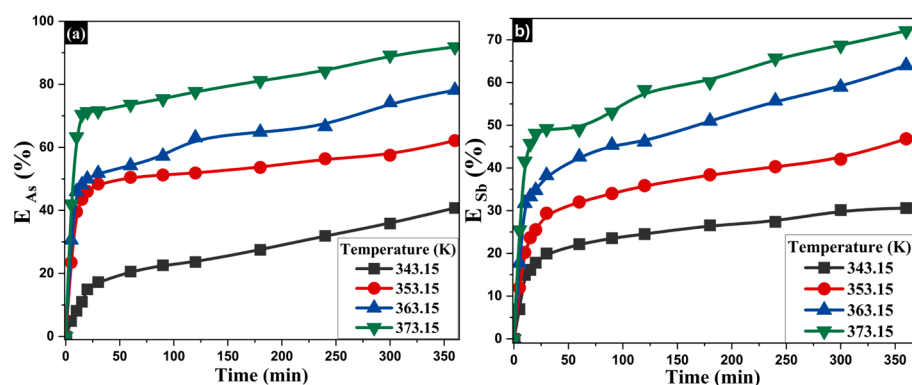


Figure 9. Influence of temperature on the extraction of (a) arsenic and (b) antimony.

Table 3. Conditions and results of As and Sb extraction experiments in alkaline medium with S^{2-} .

Na_2S (mol·L ⁻¹)	NaOH (mol·L ⁻¹)	Temperature (K)	E_{As} (%)	k_{exp} (min ⁻¹)	E_{Sb} (%)	k_{exp} (min ⁻¹)
0.25	1.5	353	28.29	0.0059	15.67	0.0027
0.5	1.5	353	34.27	0.0091	25.36	0.0044
1	1.5	353	44.66	0.0123	38.61	0.0054
1.25	1.5	353	60.54	0.0147	47.00	0.0075
1.5	1.5	353	71.85	0.0190	56.09	0.0079
1.75	1.5	353	81.22	0.0257	67.39	0.0107
1.25	0.75	353	39.11	0.0066	33.04	0.0048
1.25	1	353	40.15	0.0071	40.02	0.0057
1.25	1.25	353	46.20	0.0095	46.20	0.0059
1.25	1.5	353	60.58	0.0100	48.49	0.0060
1.25	2	353	67.82	0.0123	51.72	0.0067
1.25	1.5	343	40.78	0.0028	30.61	0.0053
1.25	1.5	353	62.13	0.0145	46.80	0.0072
1.25	1.5	363	78.14	0.0186	63.99	0.0119
1.25	1.5	373	91.87	0.0284	72.09	0.0164

3.4. Reaction Orders (α , β) and Activation Energy (E_a)

Considering Equation (10), which enables a quantitative assessment of the leaching progress as a function of time, Figure 10a shows the evolution of As and Sb conversion at $[Na_2S] = 1.25 \text{ mol}\cdot\text{L}^{-1}$, $[NaOH] = 1.5 \text{ mol}\cdot\text{L}^{-1}$, and 353 K. Figure 10b presents linearized fits to the three classical shrinking-core limits: external film diffusion ($1 - (1 - X)^{2/3} = k_{ff}t$), diffusion through the product layer [$1 - 3(1 - X)^{2/3} + 2(1 - X) = k_{pf}t$], and surface chemical-reaction control [$1 - (1 - X)^{1/3} = k_{ct}t$]. The chemical-reaction model provides the best description of the data for both As and Sb (e.g., $R^2 = 0.9991$ and 0.9978 , respectively), whereas the diffusion-controlled models (film and product layer) yield lower determination coefficients and more structured residuals. Thus, under these conditions the rate-limiting step is dominantly the surface chemical reaction, while a diffusional contribution cannot be completely ruled out.

Once it was confirmed that the kinetic model governing the removal of As and Sb corresponds to a chemically controlled reaction (Equation (8)), the influence of reactant concentration on the reaction rate was evaluated. Experimental rate constants (k_{exp}) were determined from linear fits of the integrated chemical control model equation, using various Na_2S concentrations ($0.25\text{--}1.75 \text{ mol}\cdot\text{L}^{-1}$) while keeping $[NaOH]$ and temperature constant. The results obtained for As and Sb are shown in Figures 11a and 11b, respectively.

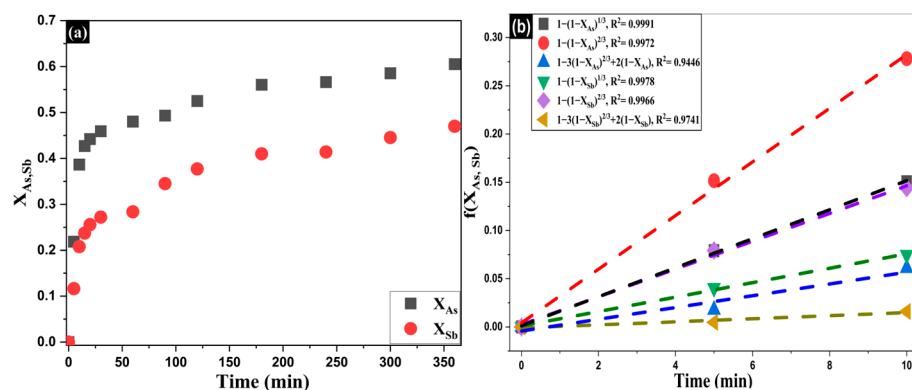


Figure 10. (a) $X_{As,Sb}$ vs. time using Equation (10); (b) Linearized fits to external film diffusion, product-layer diffusion, and surface reaction control.

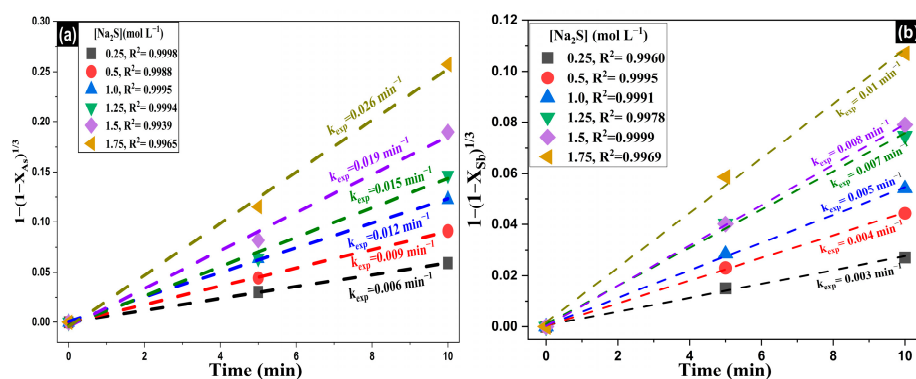


Figure 11. Experimental data fitted to the integrated shrinking core model under chemical reaction control for the leaching of (a) arsenic and (b) antimony, using varying concentrations of Na_2S .

As shown in Figure 11, the rate constants (k_{exp}) for both As and Sb increase progressively with rising Na_2S concentration. However, the increase is more pronounced for As, indicating a higher sensitivity of its removal rate to sulfide ion concentration compared to Sb. Finally, by plotting $\log [Na_2S]$ vs. $\log(k_{exp})$ (Figure 12), the reaction orders (α) for As and Sb were determined to be 0.6751 and 0.6357, respectively, confirming that the removal rates of both elements are influenced by sodium sulfide concentration. Nevertheless, the magnitude of the reaction orders suggests that the rate does not depend exclusively on the reactant concentration but is also affected by complementary phenomena [79,80].

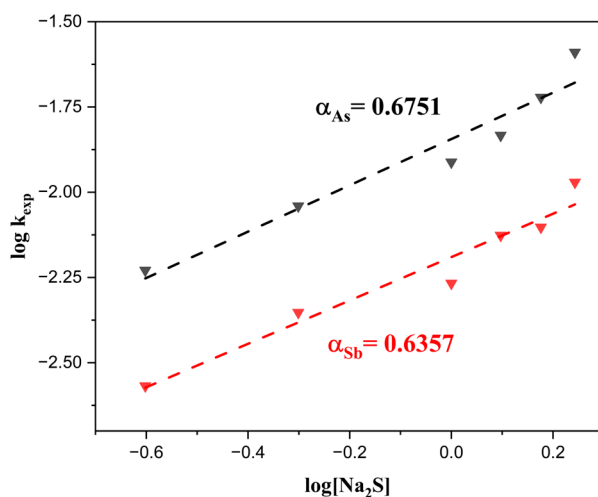


Figure 12. Reaction orders (α) with respect to Na_2S concentration in the extraction of As and Sb.

Once the reaction order with respect to Na_2S was determined, the influence of NaOH concentration on the removal rate of As and Sb was evaluated. Figure 13a,b show the fitting of the experimental X_{As} and X_{Sb} data to Equation (8) for different NaOH concentrations. Although the rate constants (k_{exp}) increase with NaOH concentration, the variation is more moderate compared to that observed for sodium sulfide. For As, k_{exp} increases from 0.006 to 0.012 min^{-1} , while for Sb it rises from 0.005 to 0.0067 min^{-1} . Additionally, a linear fit with correlation coefficients above 0.995 was observed in all cases, further confirming the validity of the chemical reaction control model in describing the system.

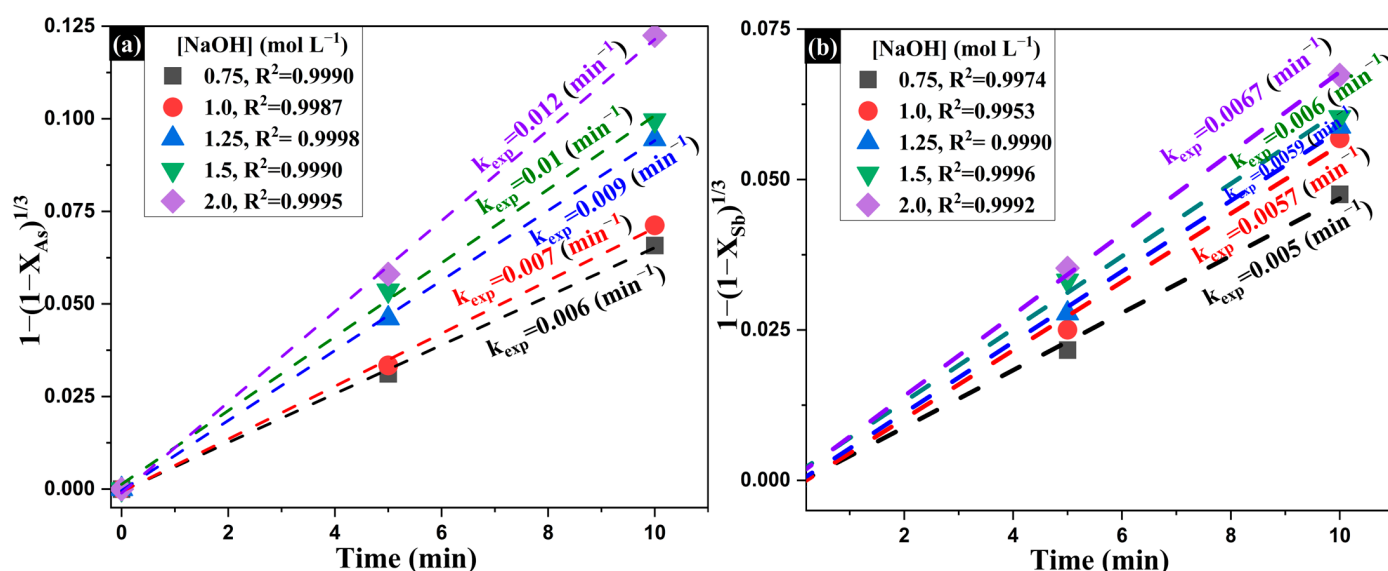


Figure 13. Experimental data fitted to the integrated shrinking core model under chemical reaction control for the leaching of (a) arsenic and (b) antimony, using varying concentrations of NaOH.

Finally, the obtained k_{exp} values were plotted against $\log [\text{NaOH}]$ for both As and Sb, allowing the determination of the reaction orders β , which were 0.6637 for As and 0.3252 for Sb (Figure 14). These results confirm that although the hydroxide ion participates in the surface dissolution mechanism, its influence is less significant compared to that of the sulfide ion.

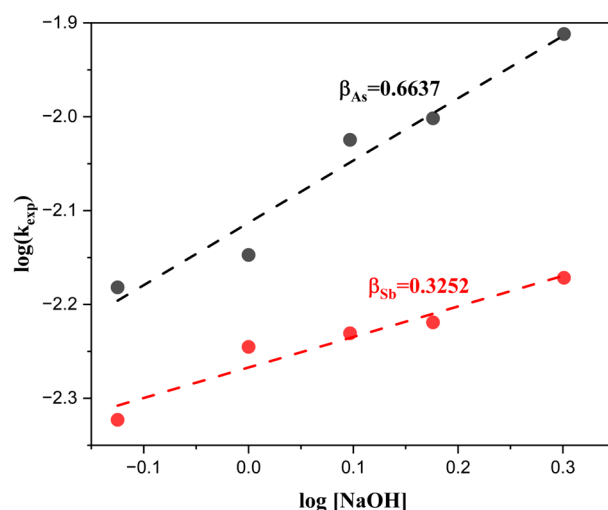


Figure 14. Reaction orders (β) with respect to NaOH concentration in the extraction of As and Sb.

Once the reaction orders (α and β) were determined, the influence of temperature on the removal rate of As and Sb was evaluated. For this purpose, the experimental

rate constants (k_{exp}) were calculated at different temperatures under constant reagent concentrations (Figure 15). To determine the activation energy (E_a), the effect of temperature on the removal process was assessed by calculating the values of k_{exp} at four different temperatures (343.15 to 373.15 K), while keeping Na_2S and NaOH concentrations constant. Figure 15a,b show the linear fittings obtained for As and Sb, respectively, consistent with the chemically controlled model previously validated. In both cases, a significant increase in k_{exp} was observed with temperature: from 0.003 to 0.028 min^{-1} for As, and from 0.005 to 0.016 min^{-1} for Sb. This progressive increase in the kinetic constant is characteristic of a process limited by chemical reaction at the solid–liquid interface, as this type of mechanism is highly temperature-dependent, a behavior widely reported in kinetic studies on the leaching of complex minerals [81–83].

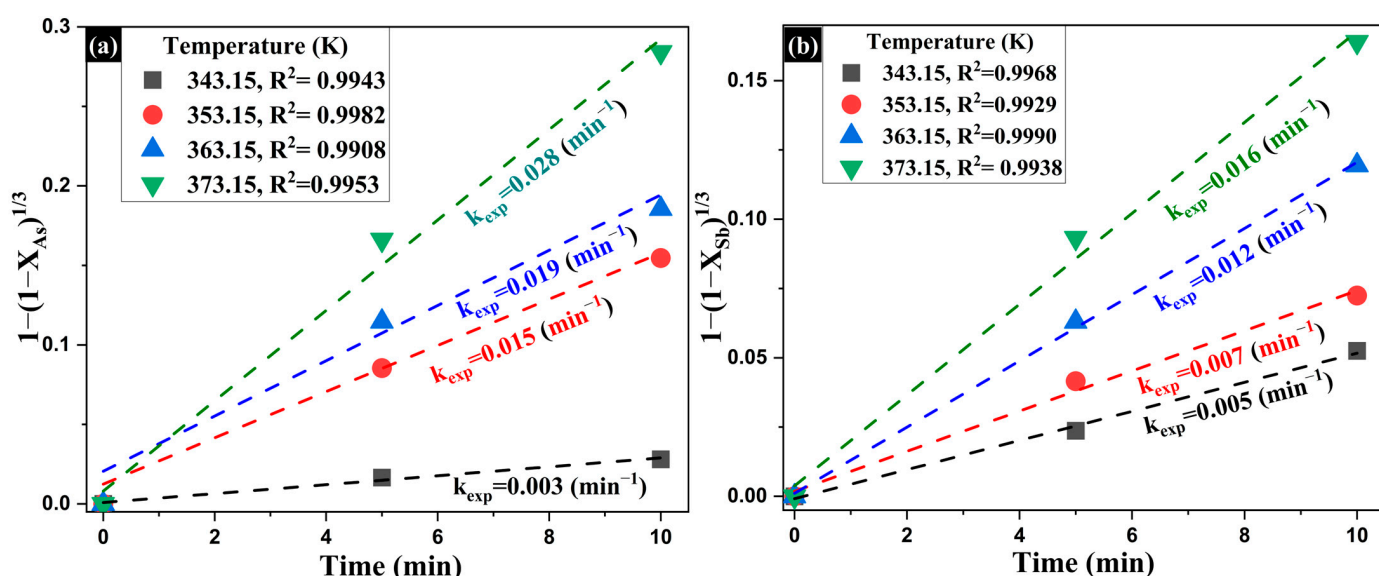


Figure 15. Experimental data fitted to the integrated shrinking core model under chemical reaction control for the leaching of (a) arsenic and (b) antimony, using varying temperature.

Based on the obtained k_{exp} values, Arrhenius plots were constructed by representing $\ln(k_{\text{exp}})$ as a function of $1000/T$. The slopes of the linear regressions allowed for the calculation of the activation energy (E_a) using the Arrhenius equation:

$$k_q = k_0 e^{\frac{-E_a}{RT}} \quad (18)$$

where R is the universal gas constant ($8.314 \text{ J} \cdot \text{mol}^{-1} \text{ K}^{-1}$). The activation energy (E_a) values obtained were $76.6 \text{ kJ} \cdot \text{mol}^{-1}$ for As and $41.6 \text{ kJ} \cdot \text{mol}^{-1}$ for Sb, confirming that the rate-limiting step in both cases corresponds to a chemically controlled mechanism. The higher activation energy associated with arsenic leaching suggests a greater sensitivity of the process to temperature, which may be attributed to a higher energy barrier required for bond cleavage and the formation of soluble species (Figure 16).

The global kinetic expression describing the removal rate of As and Sb was formulated by integrating the experimentally determined kinetic parameters (α , β , E_a , and k_0) into the shrinking core model under chemical reaction control. The pre-exponential factor k_0 was calculated from the intercept of the linear Arrhenius plots obtained using the rate constants at different temperatures. This integration enabled the development of a general equation that quantitatively models the system behavior under the evaluated conditions. The resulting expression is shown below:

$$1 - (1 - X)^{\frac{1}{3}} = \frac{bk_0}{\rho_B r_0} \cdot e^{\frac{-E_a}{RT}} \cdot [\text{Na}_2\text{S}]^\alpha \cdot [\text{NaOH}]^\beta \cdot t \quad (19)$$

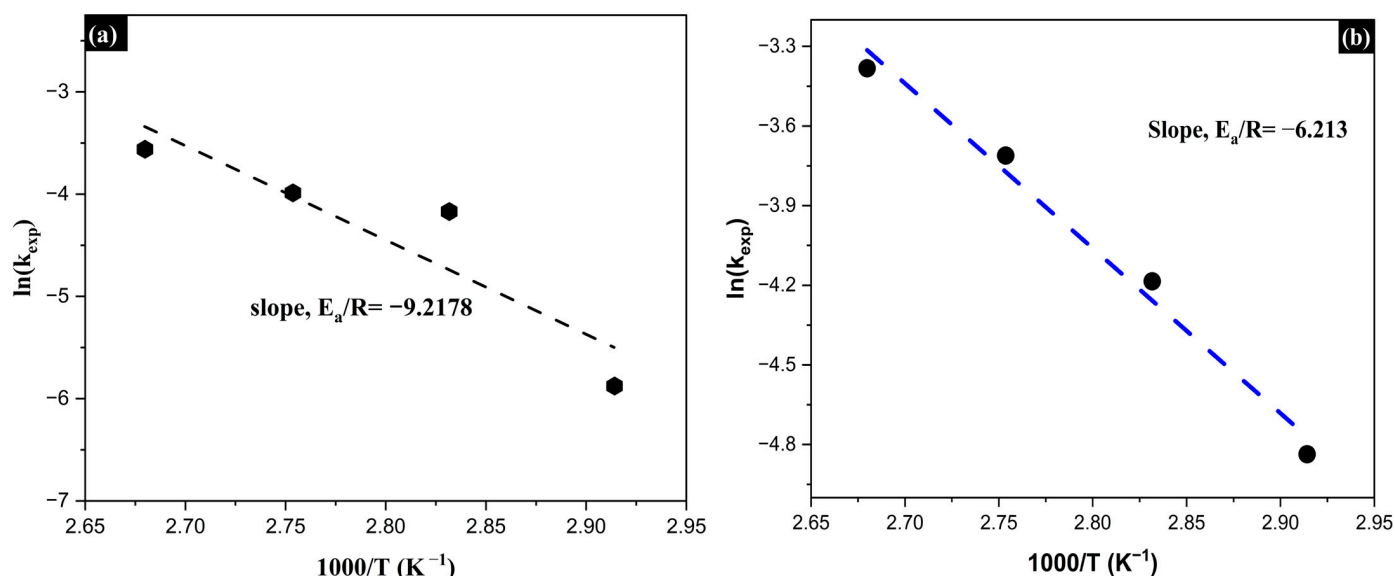


Figure 16. Calculation of the activation energy (E_a) for the removal of As (a) and Sb (b) using the Arrhenius model.

Using the kinetic parameters determined experimentally (α , β , E_a) and the pre-exponential factor k_0 from Equation (18), Equations (20) and (21) were derived. These expressions can be used to calculate k_{calc} based on different values of X and t :

$$1 - (1 - X_{\text{As}})^{\frac{1}{3}} = 1.77 \times 10^9 \cdot e^{\frac{-76.6}{RT}} \cdot [\text{Na}_2\text{S}]^{0.67} \cdot [\text{NaOH}]^{0.66} \cdot t = k_{(\text{calc})} t \quad (20)$$

$$1 - (1 - X_{\text{Sb}})^{\frac{1}{3}} = 1.12 \times 10^4 \cdot e^{\frac{-41.66}{RT}} \cdot [\text{Na}_2\text{S}]^{0.63} \cdot [\text{NaOH}]^{0.32} \cdot t = k_{(\text{calc})} t \quad (21)$$

3.5. Exploratory Quadratic Modeling of As and Sb Extraction

Based on the data obtained during the experimental campaign (Table 3), we conducted an exploratory statistical analysis to model the extraction of arsenic (As) and antimony (Sb) as a function of sodium sulfide concentration $[\text{Na}_2\text{S}]$, sodium hydroxide concentration $[\text{NaOH}]$, and temperature (T). Quadratic regression (Statgraphics) was applied to derive compact response equations (Equations (22) and (23)). The resulting fits show high coefficients of determination ($R^2 = 99.99\%$ for As and $R^2 = 99.93\%$ for Sb) within the tested data set. We emphasize that this analysis is non-confirmatory: experiments were single-run per condition, no independent validation was performed, and the data set does not originate from a full factorial DoE; therefore, the models are intended to check mechanistic consistency and guide operating ranges, rather than to provide general predictive capability or to resolve potential cooperative/competitive effects among variables outside the explored space.

$$E_{\text{As}}(\%) = -2523.3 + 23.8484 \cdot [\text{Na}_2\text{S}] + 55.1552 \cdot [\text{NaOH}] + 12.2424 \cdot T + 5.7171 \cdot [\text{Na}_2\text{S}]^2 - 11.7021 \cdot [\text{NaOH}]^2 - 0.0147123 \cdot T^2 \quad (22)$$

$$E_{\text{Sb}}(\%) = -2438.91 + 23.6027 \cdot [\text{Na}_2\text{S}] + 42.6246 \cdot [\text{NaOH}] + 12.1096 \cdot T + 5.43867 \cdot [\text{Na}_2\text{S}]^2 - 10.4188 \cdot [\text{NaOH}]^2 - 0.0149817 \cdot T^2 \quad (23)$$

The statistical significance of the terms included in the models was evaluated using analysis of variance (ANOVA) (Table 4).

Table 4. Analysis of variance (ANOVA) for the quadratic regression models of arsenic (As) and antimony (Sb) extraction as a function of Na₂S, NaOH, and temperature.

	Source	DF	Sum of Squares	Adj MS	F-Value	p-Value
As	Na ₂ S	1	41.58	41.58	484.34	0.0021
	NaOH	1	66.16	66.16	770.61	0.0013
	Temperature	1	13.91	13.91	162.07	0.0061
	Na ₂ S·Na ₂ S	1	8.97	8.97	104.54	0.0094
	NaOH·NaOH	1	22.01	22.01	256.39	0.0039
	T·T	1	10.32	10.32	120.24	0.0082
	Error	2	0.1717	0.08585		
	Total	8	3660.42			
Sb	Na ₂ S	1	41.78	41.78	49.31	0.0197
	NaOH	1	40.53	40.53	47.83	0.0203
	Temperature	1	12.78	12.78	15.08	0.0604
	Na ₂ S·Na ₂ S	1	8.31	8.31	9.82	0.0885
	NaOH·NaOH	1	17.88	17.88	21.11	0.0442
	T·T	1	10.1	10.1	11.92	0.0746
	Error	2	1.69	0.8473		
	Total	8	2430.42			

For arsenic (As), all main factors—[Na₂S], [NaOH], and temperature—were statistically significant ($p < 0.05$), together with their quadratic terms, as supported by high F-values within the tested data set. For antimony (Sb), both [Na₂S] and [NaOH] were also significant ($p = 0.0197$ and 0.0203 , respectively), whereas temperature yielded a p -value of 0.0604 , indicating a less pronounced yet near-significant effect. In this case, the quadratic interaction of [NaOH] was statistically significant ($p = 0.0442$), while other quadratic terms showed trends but did not reach statistical significance. The mean absolute error (MAE) of the models was low, particularly for As (MAE = 0.0934), indicating minimal deviation between observed and predicted values. To assess the influence of the variables on the extraction of As and Sb, a single-block data set design was carried out, considering different combinations of sodium sulfide concentration, sodium hydroxide concentration, and temperature. Based on the quadratic regression models (Equations (22) and (23)), the extraction percentages were estimated for each experimental point. These predicted values, presented in Table 5, served as the basis for generating main effects plots and response surface diagrams, which were used to analyze the behavior of the system.

Table 5. Experimental design combinations and estimated extraction percentages of As and Sb based on the regression equations.

[Na ₂ S] (mol·L ^{−1})	[NaOH] (mol·L ^{−1})	T (K)	%As	%Sb
1	1.375	358	57.74	44.16
0.25	0.75	358	15.57	8.56
1.75	0.75	358	68.49	60.28
0.25	2	358	44.29	26.03
1.75	2	358	97.21	77.75
0.25	1.375	343	5.54	4.75
1.75	1.375	343	58.46	48.97
1	1.375	358	57.74	44.16
0.25	1.375	373	56.84	38.73
1.75	1.375	373	99.00	90.45
1	0.75	343	9.85	7.25
1	2	343	38.57	24.72
1	0.75	373	61.15	48.73
1	2	373	89.87	66.20
1	1.375	358	57.74	44.16

The analysis of main effects reveals that, for both metalloids, the concentration of $[\text{Na}_2\text{S}]$ exerts a clear positive influence on extraction efficiency—consistent with the monotonic trends observed in Figures 7–9. In the case of arsenic (As), increases in both $[\text{Na}_2\text{S}]$ and temperature significantly enhance recovery, consistent with kinetic parameters such as reaction order and activation energy (Figure 17a) and the time–conversion profiles in Figures 7 and 9. For antimony (Sb) (Figure 17b), increasing $[\text{Na}_2\text{S}]$ from 0.25 M to 1.75 M leads to a notable rise in extraction, which is attributed to the formation of soluble complexes. Regarding $[\text{NaOH}]$, its effect exhibits a curvilinear behavior, with an optimal concentration at which the extraction efficiency is maximized before stabilizing or slightly decreasing at higher concentrations. As for temperature, a positive correlation was observed between rising temperatures and Sb leaching efficiency, aligning with the known acceleration of dissolution reactions at elevated temperatures.

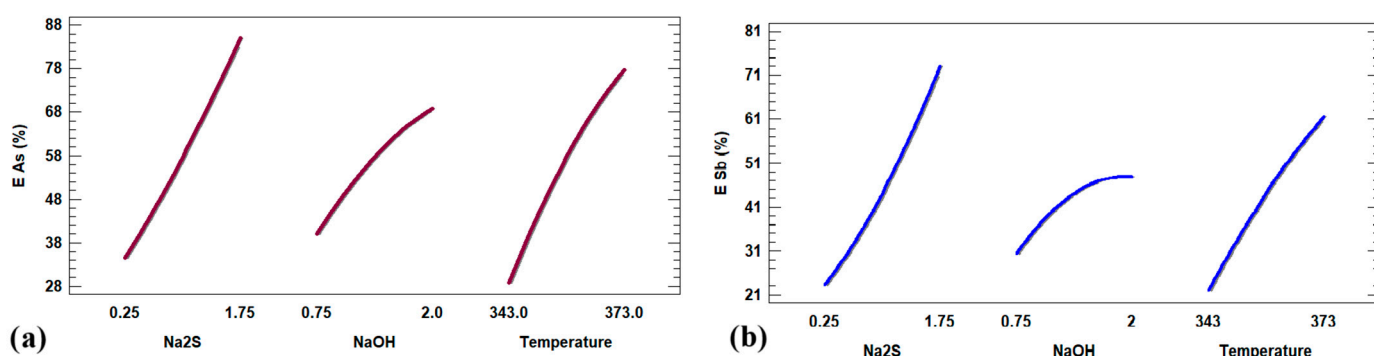


Figure 17. Effect of independent variables on the extraction efficiency of As (a) and Sb (b).

3.6. Solid Residue Characterization by XRD

To assess the mineralogical changes induced by the selective leaching of As and Sb, the solid residues were characterized by X-ray diffraction (XRD) (Figure 18). The diffractogram exhibits intense peaks corresponding to chalcopyrite, galena, wurtzite, and acanthite, confirming that the valuable metallic phases of the concentrate remained virtually unaltered after treatment. This observation supports the high selectivity of the Na_2S – NaOH system, as it demonstrates minimal interaction with Cu, Pb, and Ag sulfides. Additionally, signals corresponding to stibnite, parasymplectite, and realgar were identified, indicating that a residual fraction of Sb and As remained in the solid post-leaching [84]. These phases may be associated with less accessible or partially encapsulated structures, suggesting opportunities for process optimization or the need for an additional pretreatment stage.

Accordingly, the Eh–pH diagram in Figure 19 indicates that, within the alkaline–reducing interval of the leaching system employed, the stable Cu phases are sparingly soluble solids ($\text{Cu}_2\text{S}/\text{CuS}$ y $\text{Cu}_2\text{O}/\text{Cu}(\text{OH})_2$, under oxidizing environments) and, therefore, the process shows high selectivity toward As and Sb with null or minimal co-dissolution of copper.

The absence of well-defined copper sulfide peaks in the XRD pattern (Figure 18) is consistent with amounts below the detection limit or with peak overlap.

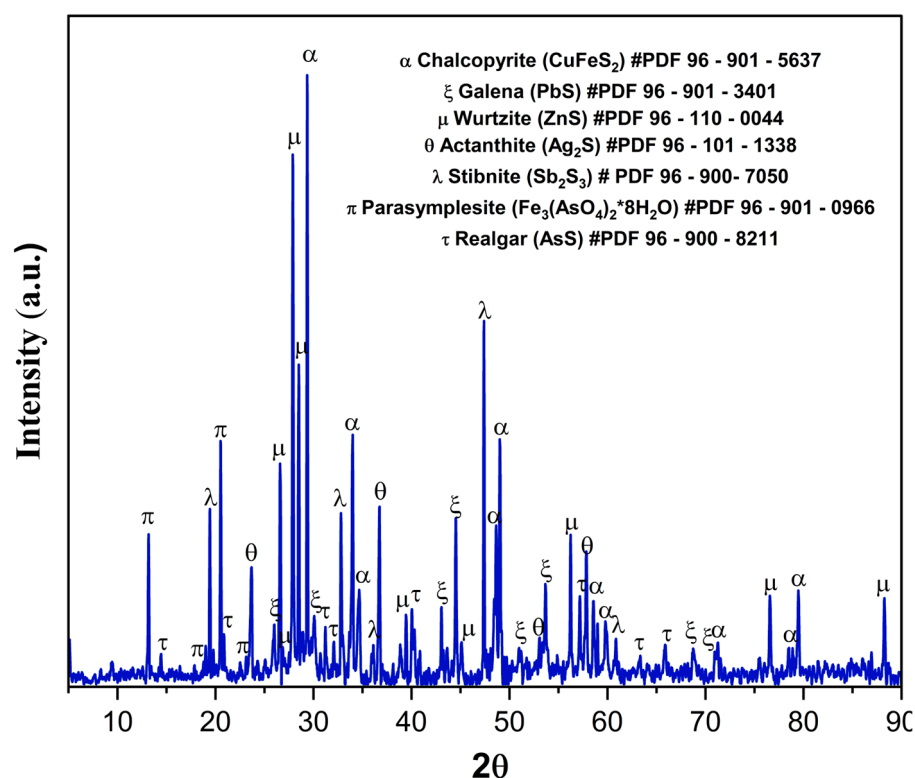


Figure 18. X-ray diffraction (XRD) pattern of solid residues. # indicates the ICDD PDF card number used for phase matching.

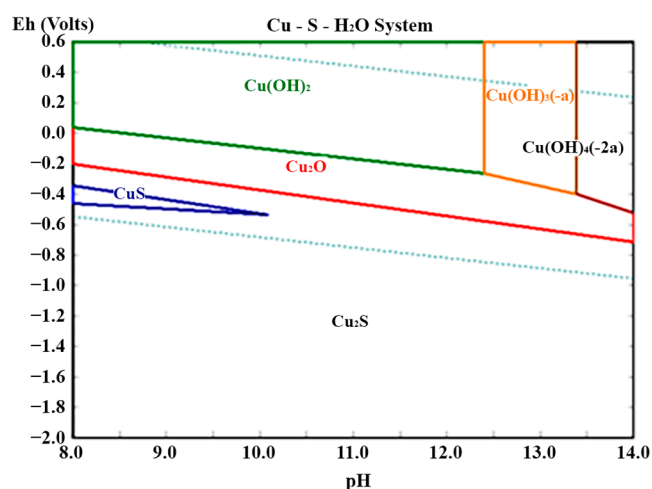


Figure 19. Eh-pH Diagram for the Cu-S-H₂O System at 353 K.

3.7. Feasibility and Scale-Up Conditions

The sulfide required for the leaching process can be supplied as commercial Na₂S (e.g., Na₂S·9H₂O) or generated in situ in an alkaline medium; this flexibility facilitates plant supply. Based on the dissolution reactions toward thiosalts (Equations (11)–(14)), the removal of As and Sb requires approximately 1.5 mol of Na₂S per mol of As + Sb. Using the head grades of 1.82 wt% As and 0.51 wt% Sb as reference, this corresponds to ≈ 0.427 kmol or 55.5 kg of 60% Na₂S (flakes) per metric ton of concentrate. Considering a typical cost range of the reagent (630–720 US\$/t) and the stoichiometric ratio above, the direct sulfide expense is 35–40 US\$ per ton of concentrate [85]. It is worth noting that NaOH does not have a fixed stoichiometric consumption (it depends on pulp density and make-up strategy), because it is mainly used to maintain pH ≥ 12 . Likewise, liquor recirculation with

a minimal purge (oxidized to sulfate before disposal) reduces the net sulfide consumption and the effluent load, reinforcing the circularity of the process [76].

4. Conclusions

This study demonstrated the effectiveness of a selective alkaline leaching system based on Na_2S – NaOH for the controlled removal of arsenic (As) and antimony (Sb) from a polymetallic concentrate originating from the Zimapán mining district in Hidalgo, Mexico. From an environmental perspective, the geoaccumulation index (Igeo) and enrichment factors (EF) classified As and Sb as extremely contaminating and highly enriched elements, underscoring the need for their removal in materials derived from mining activities. High extraction efficiencies were achieved for both metalloids while preserving valuable metal phases such as chalcopyrite, galena, and acanthite, as confirmed by XRD analysis. These results support the use of this leaching system as an effective treatment strategy to reduce and control the adverse effects associated with the presence of contaminant metalloids in mineral concentrates.

The kinetic analysis of the process revealed that the dissolution of As and Sb follows a chemically controlled mechanism, with notable sensitivity to temperature changes, suggesting the presence of significant energy barriers related to the cleavage of metal–sulfur bonds. This insight contributes valuable information for the potential industrial scalability of the system, as it relies on adjustable operational parameters such as temperature and reagent concentration.

Statistical modeling through quadratic regression enabled the identification of the relative influence of operational variables on extraction efficiency. For both As and Sb, the Na_2S concentration and temperature were found to be the most impactful factors, followed by NaOH concentration. The generated models exhibited excellent predictive capability and statistical significance ($p < 0.05$), validating their application for optimizing the proposed leaching process.

Finally, the system's high selectivity was confirmed through the characterization of the leaching residues, which showed the presence of secondary phases such as stibnite and realgar, while the main metal-bearing phases of the concentrate remained unaltered. These findings confirm the potential of the process as a technically and environmentally viable alternative for the treatment of polymetallic concentrates.

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Conflicts of Interest: The authors declare no conflicts of interest.

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