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Selective Removal of Arsenic from Copper-Arsenic Complex Ore via NaHS-NaOH Leaching

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Abstract – *The increasing global demand for copper has driven the exploitation of non-traditional copper resources, including low-grade ores with elevated concentrations of deleterious elements such as arsenic. This study investigated the selective removal of arsenic from a Cu-As complex ore through leaching in alkaline conditions using sodium hydrosulfide (NaHS). The complex ore was subjected to leaching to obtain a high-Cu low As residue and high As pregnant leach solution. A mineralogical analysis identified enargite (Cu_3AsS_4) and scorodite (FeAsO4·2H2O) as arsenic-bearing phases, corresponding to approximately 1.06 wt% As and 1.09 wt% Cu in the ore. Scorodite readily dissolved under alkaline conditions and contributed up to 33% As dissolution. Arsenic dissolution was strongly influenced by high solvent concentration, elevated temperature, and reduced particle size. In contrast, copper dissolution remained consistently below 0.5%, indicating a high selectivity for arsenic over copper. The leaching kinetics exhibited an initial rapid rate potentially due to the dissolution of soluble species such as scorodite followed by an abrupt decrease as the more refractory species were consumed. The leaching kinetics can be effectively described using the Avrami model where the activation energy was calculated to be 18.1 kJ/mol. These results demonstrate the potential of NaHS–NaOH media for the selective removal of arsenic from a Cu-As complex ore under alkaline leaching conditions.*

Keywords: *Enargite dissolution, scorodite, sodium hydrosulfide, arsenic leaching*

I. INTRODUCTION

Copper is a critical metal that will support the move to a more sustainable low-carbon future. This shift is expected to drive the copper demand in sectors such as vehicles, wind turbines, and other green energy [1]. To meet this rising demand, the copper industry will have to increase current production or develop new copper projects. However, due to this demand, supply might be sourced from non-traditional copper resources particularly ores with lower grades, more complex mineralogy, and increasing levels of deleterious elements [2].

One of the most common toxic impurities found in copper concentrates is arsenic. This element has been shown to exhibit toxic potential with serious health implications including carcinogenic properties. Copper ores associated with arsenic occur commonly as enargite (Cu_3AsS_4) or tennantite ($Cu_{12}As_4S_{13}$). Other rare copper-arsenic minerals include luzonite (Cu_3AsS_4), and sinnerite ($Cu_6As_4S_9$) [3].

Mineral processing does not always achieve complete separation of Cu-As minerals such as enargite from the other Cu minerals. These Cu-As minerals commonly report to the flotation concentrates posing significant challenges not only for smelters but also for mining operations. Elevated impurity levels reduce the marketability of the concentrate and often result in penalties, thereby increasing overall processing costs. The presence of high arsenic in copper concentrates imposes additional costs on the smelter operators. These include environmental and occupational health control costs, disposal costs, smelting and refining costs [2, 3].

It is more advantageous to remove the arsenic impurity at an early stage of processing compared to handling high arsenic wastes or extracting arsenic from metallic products. Since arsenic and its oxides are highly volatile, roasting is the conventional process for arsenic removal. However, due to the required facilities and toxic emissions, such a method is uneconomical, non-environmental, and non-health friendly.

Hydrometallurgical methods have been investigated recently and considered as a viable alternative means for selective removal of arsenic. Among current technologies, alkaline leaching processes demonstrated successful results in the removal of arsenic from copper concentrates. Furthermore, these methods are generally more environmentally benign and less capital-intensive [4].

Selective leaching of arsenic under alkaline leaching has shown to be an effective method to treat arsenic. Enargite is considered a refractory mineral since this cannot be easily digested in aqueous media [5]. Different authors have studied the sulfidic leaching of arsenic from copper concentrates [6-8]. These authors employed alkaline media such Na2S and NaHS which demonstrated promising selectivity and effectiveness in leaching arsenic from concentrates.

Both Na2S and NaHS have similar leaching behavior as represented by Eq 1 and Eq 2. enargite (Cu_3AsS_4)reacts with the sulfide ions (S2-) to leach out arsenic in the form of AsS43- while copper will remain in the residue as Cu2S. Simulations by Awe and Sandstrom indicate that the stability of the S2- at 298K is confined at conditions that are both strongly reducing (Eh< -600mV) and highly alkaline (pH>13) [9]. Furthermore, under these Eh-pH conditions, Cu2S and S2− emerge as the dominant species which confirms that transformation of Cu3AsS4 to Cu2S can be theoretically achieved [10]. To ensure the presence of the S2- ions in Eq 1, enough NaOH must be added to prevent its hydrolysis to hydrosulfide (HS-) as shown in Eq 2, which was reported to decrease the leaching efficiency [6].

$$Cu_3AsS_{4\,(s)} + 3S^{2-}{}_{(aq)} \leftrightarrow \; 2Cu_2S_{\,(s)} + \; 2AsS_4^{3-} \qquad \text{Eq 1}$$

$$S^{2-} + H_2O \; \leftrightarrow \; HS^- + \; OH^- \qquad \text{Eq 2}$$

Fewer studies have been done on using NaHS as a solvent in arsenic leaching. Tongamp et al. (2009) and Ruiz et al. (2015) studied enargite leaching using NaHS as a source of S^{2-} [11, 12]. NaHS was selected due to its higher sulfur content (~43%) compared to Na2S (~13%). Leaching in NaHS-NaOH media is described by Eq 3, as proposed by Tongamp et al. (2009)

wherein NaHS dissociates to provide S^{2-} ions to substitute arsenic in enargite.

$$2Cu_3AsS_{4\,(s)} + 3NaOH_{(aq)} + 3NaHS_{(aq)} \leftrightarrow 3Cu_2S_{\,(s)} + 3H_2O_{\,(aq)} + 3Na_3AsS_{4\,(aq)} \qquad \text{Eq 3}$$

Majority of the existing studies have concentrated on the use of sodium sulfide as the primary leaching agent for arsenic removal and only a limited number have explored the potential of sodium hydrosulfide (NaHS) in similar applications [11, 12]. Moreover, these studies have primarily focused on copper concentrates, aiming to reduce the arsenic content prior to pyrometallurgical processing. Notably, limited studies has been done on the direct leaching of copper–arsenic ores, which differ significantly from concentrates in terms of mineralogical characteristics and processing. Thus, there is the need for further studies that evaluate the effectiveness of alkaline sulfide media, particularly NaHS, in treating arsenic-bearing copper ores.

II. METHODOLOGY

2.1 Materials

Raw High-arsenic copper ore samples were collected to carry out the experimental work. The ore underwent crushing and grinding to obtain samples with 80% passing (P80) of 37 and 75 µm. Reagents for leaching were prepared from technical-grade reagents (NaHS and NaOH) using deionized water for mixing and dilution.

2.2 Ore Characterization

Determination of elemental composition was done using the Inductively Coupled Plasma (ICP). Mineralogical and particle size analysis was obtained using the Mineral Liberation Analyzer from the Julius Kruttschnitt Mineral Research Centre (JKMRC) of The University of Queensland.

2.3 NaHS-NaOH Leaching

Leaching of the ore was performed under alkaline conditions using NaHS while NaOH was added to maintain the alkalinity of the system. Furthermore, a setup without any addition of NaHS but with the same alkalinity was also conducted as a reference.

Leaching media was prepared by initially creating an alkaline solution through the addition of NaOH followed by the addition of the solvent. Once the desired temperature was reached, the ore sample was then added to the solution. Leaching conditions such as pH, NaOH concentration, time, percent solids, and agitation rate were set constant as listed in Table 1. Tests were carried out in 500mL two-necked flasks with a Liebig condenser in a temperature-controlled oil bath, with a thermometer, oxidation-reduction potential (ORP) tester and pH meter to constantly monitor and record the temperature, potential and pH, respectively.

Table 1. Constant Leaching Parameters

Parameters	Units	Value
NaOH	mole/L	3.5
Leaching Time	hrs	2
Solids	%	5
Agitation Rate	RPM	500

To evaluate the influence of leaching temperatures, solvent concentrations and ore particle size, a series of batch experiments were designed using a factorial approach, as summarized in Table 2. These solvent concentration levels were selected based on prior studies, where similar NaHS concentrations and leaching temperatures were shown to be effective in selectively leaching arsenic from copper concentrates [11, 12]. The target coarse particle size (P80 = 75 µm) was selected to represent the typical grinding output in mineral processing operations. On the other hand, the fine particle size (P_{80} = 37 µm) was selected based on the mineralogical analysis, which indicated that arsenic-bearing minerals predominantly occur in the finer fractions of the ore.

The leaching solution was filtered to separate the leached residue and the pregnant solution. The arsenic and copper concentration of the pregnant solution was determined using ICP.

Table 2. Leaching Parameters

Setup	Solvent Conc, M	Temp, °C	Particle Size (P_{80})
A	0.75	60	75
B	0.75	60	37
C	0.75	90	75
D	0.75	90	37
E	1.50	60	75
F	1.50	60	37
G	1.50	90	75
H	1.50	90	37

2.4 Kinetic Study

A series of tests were carried out to determine the kinetics of the arsenic leaching using NaHS using the same experimental setup previously discussed. Each run was conducted in a 500mL flask containing a leaching solution of 1.5M NaHS, and ore sample with particle size (P80) of 37 µm. These conditions were selected based on preliminary experiments, which showed that the combination of high reagent concentration and fine particle size resulted in the highest arsenic dissolution. Kinetic leaching tests were performed at three different temperatures (30°C, 60°C and 90°C). The aliquot solution was collected for characterization at a predetermined time interval.

III. RESULTS AND DISCUSSION

3.1 Ore Characterization

The composition of the main elements of the ore is summarized in Table 3. A high concentration of copper in the ore is noted and as reference, this level is already considered as high grade. However, the ore also contains a relatively large amount of arsenic, which must be effectively removed to mitigate challenges associated with downstream processing. Additionally, based on the Mineral Liberation Analyzer data as illustrated in Figure 1, arsenic occurs primarily as 4% enargite (Cu_3AsS_4)and 1% scorodite ($FeAsO_4.2H_2O$). The ore is also comprised of 9% pyrite, and <0.12% copper minerals and is dominated by quartz as the gangue mineral at 83% SiO_2.

Table 3. Main elemental composition of the ore

Element	%As	%Cu	ppm Au	%Fe
Composition	1.06	1.93	3.8	4.97

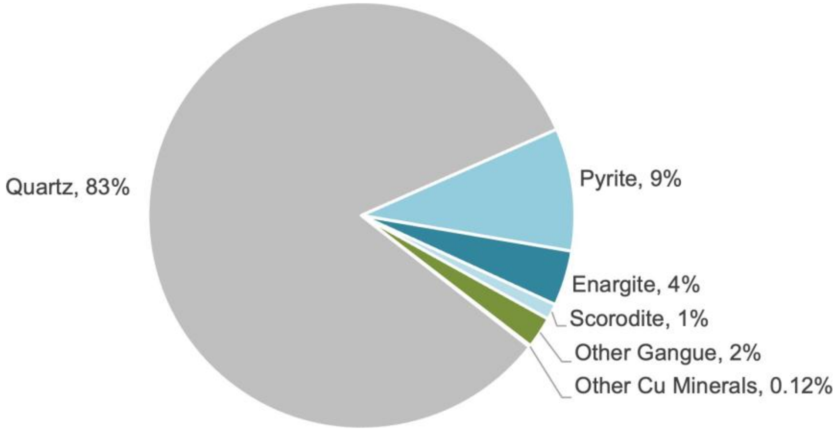

Figure 1. Overall modal mineralogy of the ore using Mineral Liberation Analyzer

The presence of scorodite in the ore indicates that the deposit underwent oxidation. The formation of scorodite can be depicted by Eq 4 [13]. As pyrite gets oxidized, Fe ions will react with enargite to leach out arsenic forming scorodite. The ore also contains pyrite which serves as the source of Fe to facilitate the oxidation process of enargite.

$$Cu_3AsS_4 + FeS_2 + 12.5O_2 + 5H_2O \rightarrow FeAsO_4 \cdot 2H_2O + 3Cu^{2+} + 6SO_4^{2-} + 6H^+ \qquad \text{Eq 4}$$

Based on the elemental distribution shown in Figure 2, 67% of arsenic is from the enargite mineral while the remaining 33% is from scorodite. Although scorodite comprises only about 1% of the total mineral content, its arsenic contribution remains significant due to its relatively higher stoichiometric composition. Scorodite contains 32% As by weight which is higher than the 19% As in enargite.

Based also on the same figure, majority of the copper is from enargite at 97%. The other copper sulfide minerals such as chalcopyrite ($CuFeS_2$) and bornite (Cu_5FeS_4) occur in trace amounts. This implies that the usual process of separating Cu-As-S minerals from Cu-S through froth flotation with pulp potential adjustments would be ineffective for this type of ore. Thus, to obtain a high-Cu but low-As concentrate, arsenic should be selectively removed from the enargite mineral.

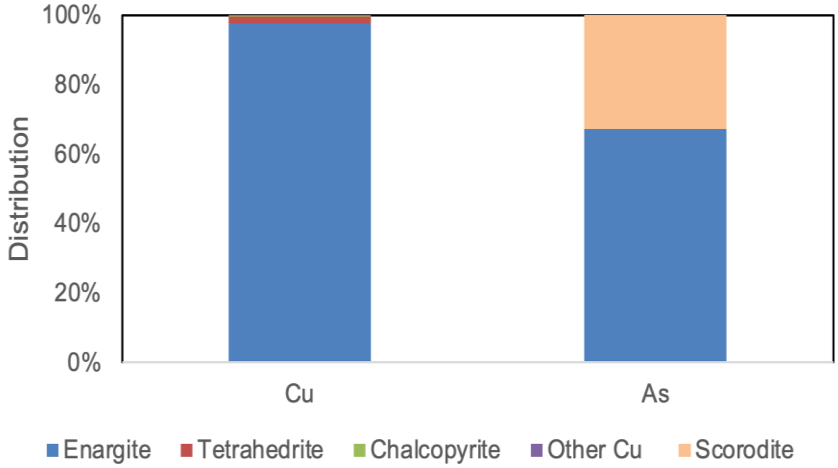

Figure 2. Elemental copper and arsenic distribution

The mineral distribution across particle size fractions is shown in Figure 3. Enargite, scorodite, and pyrite are present in various particle sizes. However, majority of the arsenic minerals are more concentrated in the finer particles (<75 microns). This suggests that targeting finer size fractions could improve arsenic dissolution and kinetics due to increased surface area and higher exposure of the target mineral phases. These findings provide the basis of investigating this size fraction in the study.

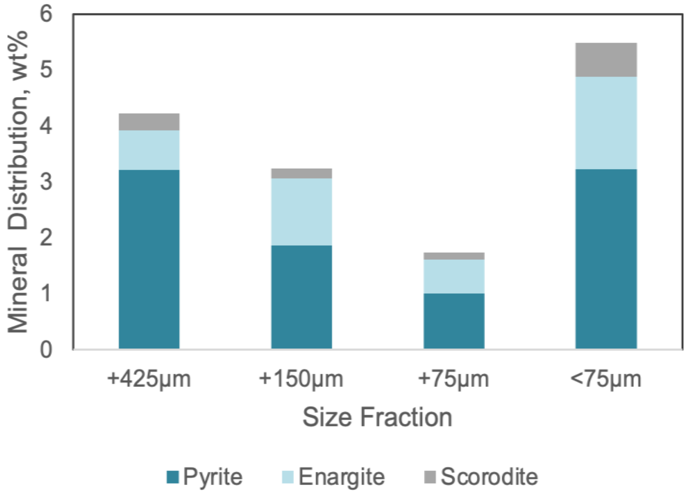

Figure 3. Mineral distribution per size fraction

3.2 NaHS-NaOH Leaching

The resulting arsenic and copper dissolution across different solvent concentrations, temperature, and ore particle size (P80) is illustrated in Figure 4. Arsenic dissolution ranges from 75% to as high as 85% leached out to the solution while copper dissolution is consistently below 0.5%. The trend shows that most treatments resulted in low to negligible copper dissolution with only minor observed increase in treatments E and F. Furthermore, the different particle size, solvent concentration and temperature had minimal influence on copper dissolution. This dissolution behavior reflects the selectivity of the NaHS-NaOH media to arsenic over copper.

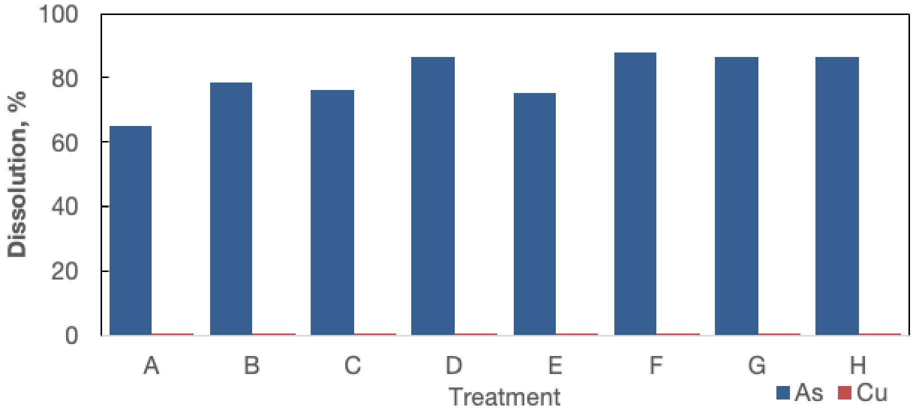

Figure 4. Arsenic and copper dissolution at different treatments

As previously discussed, S2- are noted to be stable under highly alkaline and strongly reducing conditions. The measured potential for this system at 30oC was approximately -650mV with the pH maintained in the range of 13.5-14. These conditions favor the presence of sulfide ions which ensures the reduction of enargite reaction shown in Eq 1. Furthermore, Tongamp obtained Eh values ranging from -500 to -550mV with pH consistently over 13.5

which closely align with those observed in the present study [10].

Figure 5 illustrates the different effects of the parameters on arsenic dissolution. The figure shows that arsenic dissolution is directly proportional to the NaHS concentration, and solution temperature, and inversely proportional to the particle size of the ore. Observations in this study correspond with the findings of Tongamp et al. (2009)[11]. Higher solvent concentration would indicate more S2- is available for the reaction thus expecting higher As dissolution.

An increase in particle size resulted in a decrease in arsenic dissolution. Increasing the particle size decreases the surface area which leads to fewer available sites for leaching to occur. This observation conforms with the study of Viñals et al. (2003) [14]. However, it should be noted that obtaining a P80 of 37 µm already requires fine grinding. This type of grinding is energy-intensive and entails higher grinding media costs. Furthermore, fine grinding usually corresponds to using a stirred mill and may not be easily achieved using the typical ball or SAG mills [15].

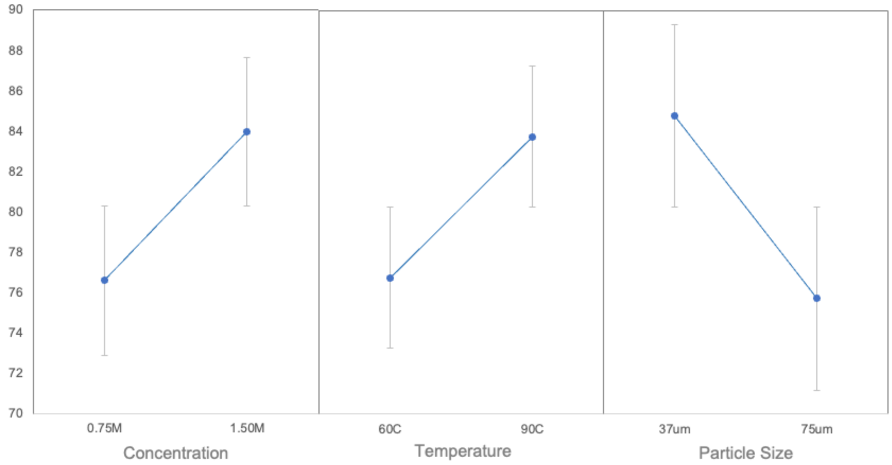

Figure 5. Main effects plot for arsenic dissolution via NaHS-NaOH leaching

3.3 NaHS-NaOH Leaching Kinetics

Figure 6 presents the arsenic alkaline leaching profile over 120 minutes at 30 °C, comparing the dissolution behavior in systems with and without NaHS. Blank leaching was performed to establish a baseline for arsenic dissolution in the absence of the solvent. Both conditions exhibit a rapid initial increase in As dissolution within the first few minutes. However, after five minutes, there is a further increase in the As dissolution with the NaHS system. In contrast, As dissolution stabilized at approximately 33% and remained unchanged for the remainder of the duration. This indicates that while the presence of NaHS enhances arsenic dissolution, the observed trend also reflects the presence of highly soluble arsenic species under alkaline conditions. Furthermore, this behavior reflects a two-stage dissolution mechanism, an initial rapid release of As from soluble minerals, followed by a slower dissolution rate.

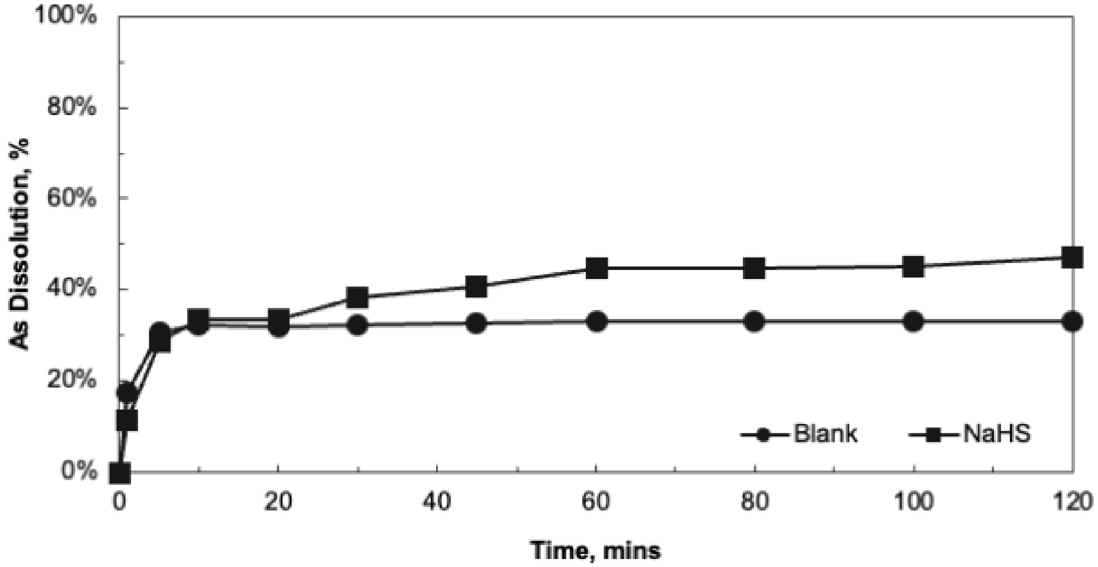

Figure 6. Comparison of arsenic dissolution in alkaline leaching at 30oC with (at 1.5M) and without NaHS solvent using ore with particle size (P80) of 37 um

The observed arsenic dissolution without the addition of solvent may be attributed to the mineralogy of the ore. The presence of scorodite, as shown in Figure 1, likely contributed to the As dissolution. Fujita et al. (2009) reported significant amount of As dissolution from scorodite under alkaline conditions when leached with NaOH at pH 12 [16]. Several authors have also noted that scorodite remains highly stable under acidic conditions, however, its dissolution increases with higher pH [17, 18]. Merkel further observed that the reaction in Eq 5 proceeds at pH values above 11.5 [19].

$$FeAsO_4 \cdot 2H_2O + 4OH^- \leftrightarrow Fe(OH)_4^- + AsO_4^{3-} + 2H_2O \qquad \text{Eq 5}$$

In contrast, the dissolved arsenic is unlikely to be derived from enargite. Li et al. (2018) proposed that leaching of arsenic with NaOH is possible with the presence of pyrite at significantly higher NaOH concentration, elevated solution temperatures, and extended leaching duration. The pyrite is believed to react with NaOH and produce Na2S which would then leach out arsenic from enargite as shown in Eq and Eq [20].

$$3FeS_2 + 6NaOH \leftrightarrow 3FeS + Na_2SO_3 + 2Na_2S + 3H_2O \qquad \text{Eq 6}$$

$$(2-x)Cu_3AsS_4 + 3Na_2S \leftrightarrow 3Cu_{2-x}S + (2-x)Na_3AsS_4 \qquad \text{Eq 7}$$

In the present study, the measured potential for the leaching system was less than -100mV which falls outside the region where S2- is stable. As previously discussed, the stability of S2- requires more highly reducing conditions (Eh < –600 mV) to remain stable in solution, indicating that under the conditions of this study, there is not enough S2- to react with enargite.

The 33% arsenic that was leached out even in the absence of a solvent closely aligns with the estimated arsenic contribution from scorodite which also constitutes around 33% of the ore's total arsenic content. The consistency between the leached amount and mineral composite suggests that scorodite was potentially the primary mineral responsible for the arsenic dissolution under blank alkaline leaching conditions. It could be inferred that for the succeeding alkaline leaching, at least 33% of the leached As may originate from the scorodite mineral. This potential release of As from scorodite dissolution in NaHS-NaOH leaching could pose a problem given that scorodite is used as a stable phase for arsenic immobilization [21]. Nonetheless, further mineralogical characterization of the residue is necessary to validate the specific contributions of scorodite dissolution.

The leaching profile of arsenic dissolution at different temperatures is presented in Figure 7 7. The trend shows a clear temperature dependent behavior. An increase in solution temperature from 30oC to 90oC significantly increased the arsenic dissolution. This behavior further confirms the trend observed in Figure 5. The leaching curve at all temperatures exhibits a rapid initial rate followed by an abrupt decrease over time. These rapid dissolution rates were observed within the first 10 minutes of leaching. However, as the reaction proceeds, only minimal additional dissolution was observed, suggesting a two-stage dissolution mechanism. In the early stage, readily soluble arsenic species, potentially associated with scorodite, are rapidly leached. As leaching continues, the rate proceeds at a much slower pace potentially due to the dissolution of the more refractory type of arsenic minerals including enargite.

Experimental arsenic dissolution is evaluated using the shrinking core models as presented in Figure 8. The shrinking core model was initially selected since previous studies on enargite leaching conformed to this model. However, all three models showed a low degree of linearity suggesting that leaching of the studied ore in NaHS media does not conform to the shrinking core model. Notably, the kinetic behavior in this study deviated from previously reported investigations on As leaching from enargite using NaHS-NaOH media. Tongamp (2010) and Ruiz (2015) reported that this type of leaching was diffusion controlled. The deviation observed in the present study can be attributed primarily to differences in ore mineralogy. The concentrates examined by Tongamp (2010) and Ruiz (2015) consisted primarily of enargite and tennantite, whereas the mineral assemblage of the ore in this investigation also contained scorodite which was inferred to dissolve in alkaline conditions even in the absence of any solvent.

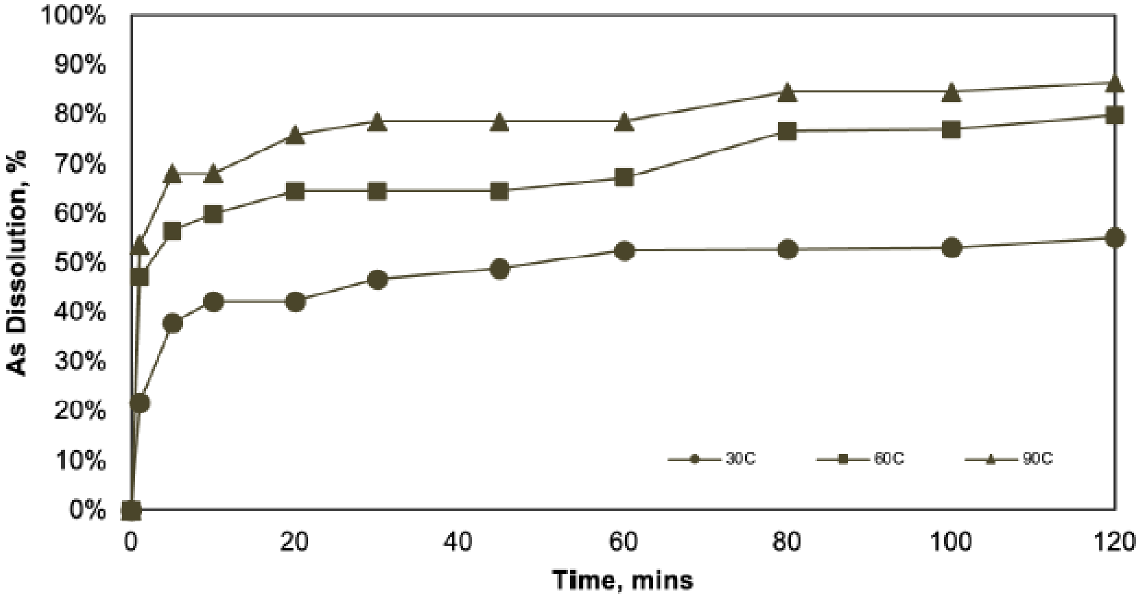

Figure 7. Arsenic dissolution in NaHS-NaOH leaching with varying temperatures at fixed NaHS concentration (1.5M) and P80 of 37 um

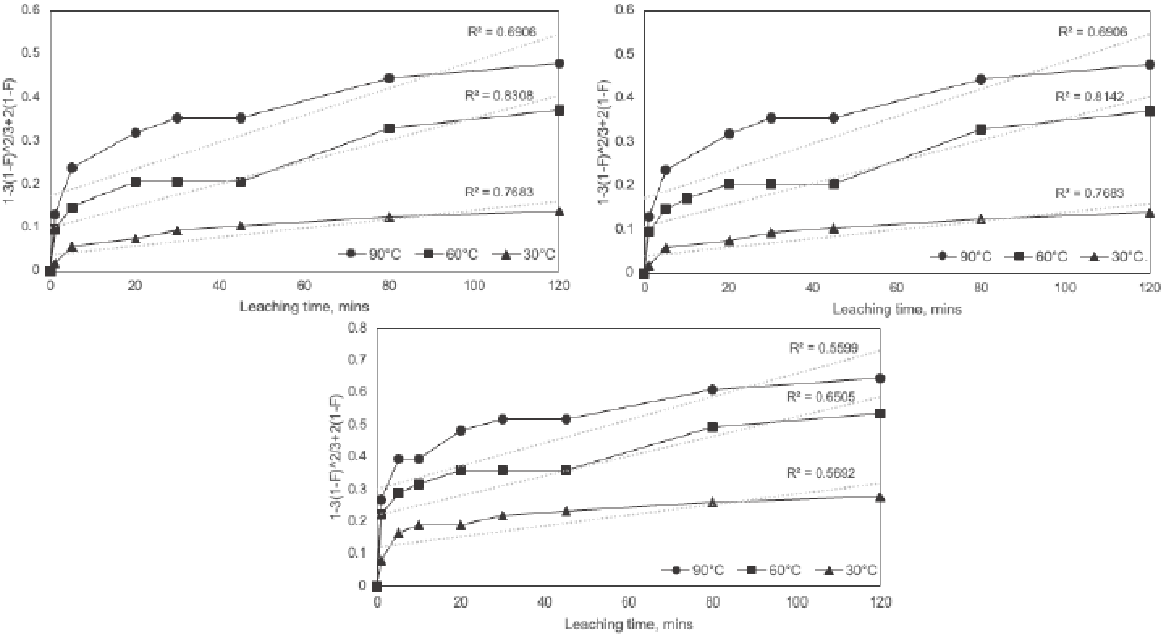

Figure 8. Plots of a) chemical reaction control model b) diffusion control model and c) mixed control for NaHS-NaOH system

The leaching kinetics of this system can be more effectively described using the Avrami model, as represented in Eq 8. This can be converted to a linear expression as shown in Eq 9.

$$-\ln(1-x) = kt^n \qquad \text{Eq 8}$$

$$\ln[-\ln(1-x)] = \ln k + n\ln t \qquad \text{Eq 9}$$

where x is the fraction, k is the rate constant for the reaction, t is the reaction time and n is the Avrami exponent. This model was originally proposed to describe the kinetics of crystallization and solid phase transition but was used successfully to explain the leaching kinetic data of multiple solid-liquid reactions.

The data presented in Figure 7 are replotted in Figure 9 as a function of the natural logarithm of time which resulted in a better linear fit. The model parameter (n) in Eq. 9 was calculated by linear regression analysis. The resulting values of the Avrami exponent were found to be less than one indicating the initial leaching rate is infinite and the rate continually decreases with time [22]. Wen (2024) applied the Avrami model to investigate the leaching behavior of manganese residue. The study reported that leaching systems that follows the Avrami model proceeds randomly and homogeneously over the entire material and leaching rate remains steady under constant temperature conditions. Furthermore, Wen (2024) emphasized that leaching governed by the Avrami model immediately occurs upon contact with the solvent with the overall leaching efficiency increases progressively with time [23]. This behavior was evident in the present study as illustrated in Figure 7. Immediate dissolution was observed at the start of leaching, likely due to the potential dissolution of scorodite.

The rate constants determined from Figure 9 were used to draw an Arrhenius plot, which is shown in Figure 10 where a linear dependence between ln k and 1/T is shown. The activation energy was determined to be 18.1 kJ/mole. This indicates that As leaching using NaHS-NaOH is controlled by a mix of both diffusion and chemical reactions.

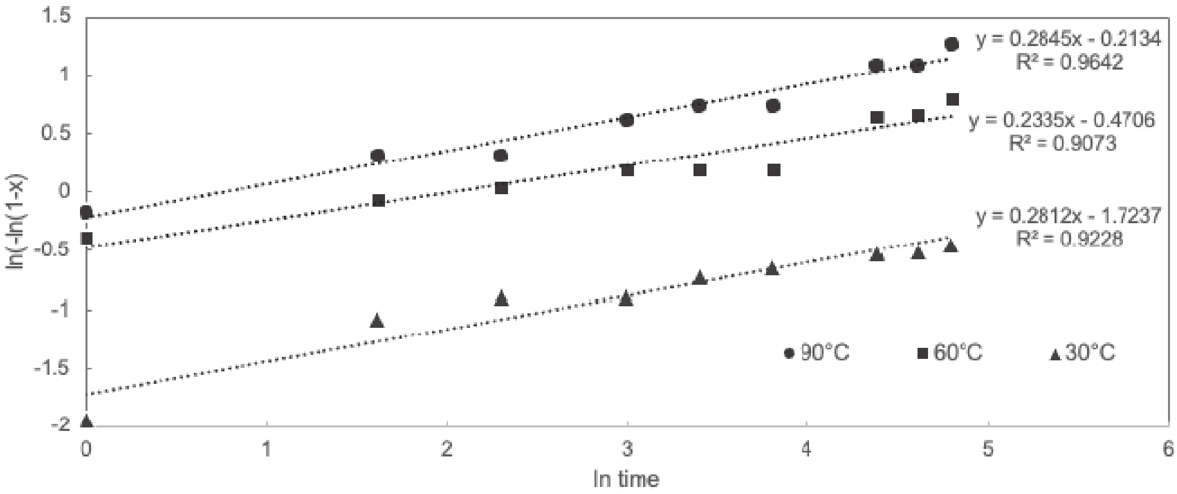

Figure 9. Kinetic modeling of As leaching in NaHS-NaOH using the Avrami equation

The leaching rate can also be influenced by the physical properties of the ore. The arsenic minerals occur in various particle size fractions, but a greater proportion is in the finer size fraction based on the MLA data. Furthermore, enargite exhibits a high degree of liberation but still has a substantial proportion that is associated with other minerals. The highly liberated and fine particles are expected to dissolve rapidly due to increased surface area and direct exposure to the leaching medium. On the other hand, arsenic particles associated with other minerals could contribute to slower dissolution kinetics.

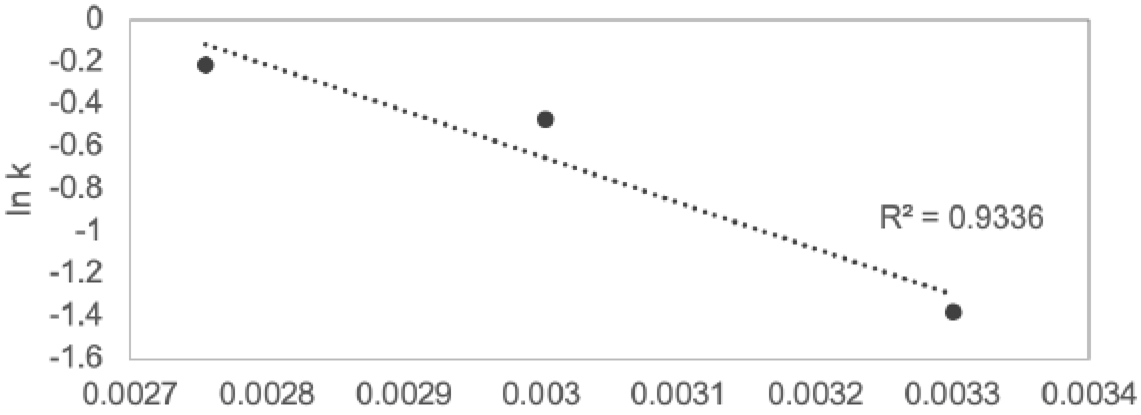

Figure 10. Arrhenius curve for As dissolution in NaHS-NaOH

IV. CONCLUSION AND RECOMMENDATION

This study investigated the selective removal of arsenic from enargite and scorodite ore via alkaline leaching using sodium hydrosulfide. The Cu-As complex ore contains 1.06% As and 1.9% Cu wherein arsenic occurs as 4% enargite (Cu_3AsS_4) and 1% scorodite (FeAsO4.2H2O). The presence of scorodite indicates that the enargite has undergone oxidation. Results also show that 97% of the copper is from enargite with no other copper sulfide minerals observed.

The arsenic dissolution in the absence of the NaHS solvent is attributed to the dissolution of scorodite at alkaline conditions which contributes to at most 33% As. The leaching kinetics deviate from the shrinking core models. The initial leaching rate is fast as more soluble forms of arsenic are dissolved. However, as the reaction progresses, the rate significantly decreases due to the consumption of more refractory forms of arsenic. The Avrami model typifies this behavior and produces a more linear fit for the plot of ln(-ln(1-x) against the natural logarithm of time. From

this model, the calculated activation energy is 18.1 kJ/mol. This indicates that the As leaching using these solvents is controlled by a mix of diffusion and chemical reactions.

V. ACKNOWLEDGEMENT

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