



Dissolution rates of jarosite-type compounds in H₂SO₄ medium: A kinetic analysis and its importance on the recovery of metal values from hydrometallurgical wastes



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ABSTRACT

This investigation reports the dissolution kinetics of Na-As, K-As and NH₄-As jarosites in a sulfuric acid medium. The effects of the pH, temperature (T), particle size (d₀), and structure substituents (especially in the M and Z positions of the general formula of jarosite-type compounds, MFe₃(ZO₄)(OH)₆) were investigated. The experimental results demonstrate that the temperature is the variable with the most significant effect on the dissolution rate, followed by the pH and particle size. In addition, it was observed that the type of substituent does not affect the dissolution rates in the acid medium. These results are reasonably consistent with the shrinking core model of chemical control for the dissolution of jarosite-type compounds under extreme pH conditions (≤1.0) and control by mass transfer at intermediate pH conditions (>1.0). The results obtained were compared with those of other investigations, and it was observed that the dissolution of jarosite-type compounds could have important implications on the recovery of metal values. Finally, a kinetic model is proposed to describe the dissolution process of jarosite-type compounds in the acid medium.

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1. Introduction

The hydrometallurgical processing of zinc calcine by the conventional roasting-leaching-electrowinning process leads to the formation of leaching residues that consist primarily of sulfates. These residues may contain significant amounts of Zn as well as other metal values, such as Pb, Cu, Ag and Cd, which have to be recovered to obtain an additional economic value (Abkhoshk et al., 2014). There is an iron elimination step in this process because the residues may contain some iron phases in considerable amounts, such as iron(III) hydroxide, jarosite, goethite or hematite (Jackson, 1986). The precipitation of jarosite-type compounds is widely used in the hydrometallurgical industry to control the amount of iron, sulfate, or alkali metals. One of the advantages of this type of precipitation is that the resulting jarosite can be relatively easily filtered and deposited. The optimum removal of iron, sulfate or

alkali metals, with a minor loss of valuable divalent metals, such as Zn, Cu or Mn, can be achieved under highly acidic conditions. Jarosite belongs to the minerals alunite supergroup, with the general formula MY₃(ZO₄)(OH, H₂O)₆: where M = Na⁺, K⁺, Ag⁺, Rb⁺, H₃O⁺, Tl⁺, NH₄⁺, ½ Hg²⁺ or ½ Pb²⁺; Y = Fe³⁺, Al³⁺, Cr³⁺, Cu²⁺ or Zn²⁺; and Z = S(VI), Cr(VI), As(V) or P(V). Therefore, in the specific case of jarosite-type compounds, Y = Fe³⁺ and Z = S(VI), the compounds are represented by the formula MFe₃(SO₄)(OH,H₂O)₆ (Jambor, 1999).

Although alkalis are usually added to initiate the jarosite precipitation, an excess of ferric ions is sometimes added to the sulfate solution to precipitate the excess alkalis (Dutrizac et al., 1996). Arsenic, which is an impurity that is commonly found in several metal concentrates and ores, is solubilized as AsO₄³⁻ during the leaching process, and it can be potentially removed during the precipitation of jarosite-type compounds in hydrometallurgical process solutions (Dutrizac & Jambor, 1986; Dutrizac et al., 1986). The precipitation reaction releases acid that has to be neutralized. Zinc calcine is commonly used as a neutralizing agent, however, it is partially dissolved, and a certain amount of it remains as a solid in the product obtained from this process. The use of calcine results in the loss of Zn, Pb and Ag, which may be present

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in the jarosite structure and/or coprecipitated as sulfates. The precipitation of trace elements (i.e., Ag) in the form of jarosites during leaching, has been associated with the erratic recovery of such values. For instance, May et al. (1973) reported that ores, such as silver jarosite, are also refractory to the more severe treatment sequence that first involves an electrolytic oxidation procedure followed by cyanidation.

The production of zinc involves the generation of large amounts of jarosite-type compounds (namely ammonium jarosite and sodium jarosite), which represents a potential environmental problem considering the massive increase in iron residues and the presence of toxic elements (i.e., As, Cd, Pb). On the other hand, it is noteworthy that these residues may have considerable economic value and therefore, the recovery of metals from the waste of zinc production represents an attractive research area to reduce the environmental harm and produce new resources (Rashchi et al., 2005; Lutandala & Maloba, 2013). Efforts have been carried out on the development of waste treatment processes in the hydrometallurgical processing of zinc to re-use jarosite-type compounds. Malenga et al. (2015) proposed a selective process to leach nickel from ammonium jarosite in an alkaline medium (KOH, NaOH, NH_4OH) in the presence of EDTA (the complexing agent) and in synergy with Na_2S (the reducing agent). Şahin and Erdem (2015) recovered Pb from zinc processing residues by an alkaline leaching process with NaOH. Kul and Topkaya (2008), Ruşen et al. (2008), and Turan et al. (2004) recovered Zn, Pb and Ge from residues obtained from a zinc plant by leaching with H_2SO_4 . Han et al. (2014) reported a way to recover Zn, Ag and anglesite from jarosite residues by a calcination process, which was combined with a sulfuration-flotation process. Ju et al. (2011) presented a combined process of roasting-leaching/ NH_4Cl with cementation-leaching/NaOH for the recovery of Zn, Ag, Pb, Cu, Cd and Fe from jarosite residues produced in the hydrometallurgical processing of zinc.

According to Gasparrini (1981), the main factors that have to be considered in the selection of any technique for the recovery of an economically attractive element are the following: the mineral species in which the metal of interest is concentrated, its association with other species, the chemical structure of the other minerals and the grain size. Leaching with different agents is a simple technique and therefore, much work has been performed to investigate the effects of the chemical composition, temperature, pH and particle size of jarosite-type compounds on the dissolution rates in several reaction media; thus, the kinetic analysis of the dissolution of Ag-jarosites in alkaline media has been widely investigated (Roca et al., 1993; Patiño et al., 1994; Patiño et al., 1998; Cruells et al., 2000; Salinas et al., 2001; Patiño et al., 2003; Roca et al., 2006).

However, due to the increasing interest in recovering valuable metals from industrial residues, several leaching agents (e.g., hydrochloric acid, nitric acid, lye, brine, ammonia, ammonium carbonate, ammonium chloride and some carboxylic acids) have been used as attractive alternatives in the recovery process. Among them, sulfuric acid has attracted the most attention (Şahin and Erdem, 2015); however, its effects on the dissolution kinetics of jarosite-type compounds have not yet been reported.

Because most of the studies on the dissolution of jarosite-type compounds have been conducted under alkaline media and the effects of sulfuric acid on the dissolution kinetics of Na-As, K-As and NH_4 -As jarosites (which are common phases in hydrometallurgical wastes) have not been reported, the aim of this investigation is to study the kinetics of the dissolution of the abovementioned jarosites under an acidic medium (H_2SO_4) and different temperature conditions. The objective of this kinetic study is to determine the factors that affect the recovery of metal values, under acid conditions, from residues obtained from hydrometallurgical activity. The variables investigated here are: the pH temperature (T), particle size (d_0) and type of substituent in the M and Z positions in jarosite-type compounds. Although Na, K, NH_4 , Fe and As do not have any economic value, their concentration in jarosite-type compounds is always higher than that of the metal values

(i.e., silver and lead); therefore, the global dissolution rate of the jarosite-type compounds can be determined by the dissolution rates of these species. To conduct this study, it was necessary to perform the following steps: i) synthesize and characterize the jarosite-type compounds; ii) select the kinetic model that best fits the experimental results, thus identifying the controlling stage that describes the dissolution process of these compounds in a H_2SO_4 medium; iii) investigate the effects of the experimental variables on the dissolution rates to determine the kinetic parameters and the kinetic equations; iv) compare the results obtained with other results reported in the literature for the dissolution of jarosite-type compounds, and v) establish the behavior exhibited by the economically valued species (Ag and Pb) during the dissolution process in the acid medium.

2. Materials and methods

2.1. Reagents and solvents

The jarosite-type compounds were synthesized by using the following reagents: iron(III) sulfate n-hydrate, anhydrous sodium sulfate, anhydrous potassium sulfate, anhydrous ammonium sulfate, arsenic pentoxide and sulfuric acid (97.9%), which were purchased as reagent-grade chemicals from J.T. Baker. The chemical analysis and monitoring of reactions was performed by using standards (PerkinElmer Pure) of Fe, Na, K and As. All of the experiments were performed with ultrapure deionized water with a resistivity of $\approx 18.0 \text{ M}\Omega \text{ cm}$.

2.2. Synthesis and characterization of the jarosite-type compounds

Samples of the K-As, Na-As and NH_4 -As jarosites were synthesized according to the procedure described by Patiño et al. (2013). Syntheses were performed by controlling a mixture of $\text{Fe}_2(\text{SO}_4)_3 \cdot n\text{H}_2\text{O}/\text{M}_2\text{SO}_4/\text{As}_2\text{O}_5/\text{H}_2\text{SO}_4$, where M represents the monovalent alkaline species (Na^+ , K^+ , NH_4^+). The initial content of Fe^{3+} , the alkali concentration and the pH were the variables used for the syntheses of these jarosite-type compounds. Since the increase in the reaction yield is directly proportional to the increase in the concentrations of Fe^{3+} and M in the initial solution, a stoichiometric excess of the reagent concentrations used in this work was added. Then, for a 1 L volume of the initial solution, the pH was varied from 1.2 to 1.6 with 20% H_2SO_4 (v/v) solution, which was used to avoid low yields and prevent the formation of unwanted phases, such as scorodite (Dutrizac and Jambor, 1986). Table 1 summarizes the compositions of the different solutions. Additionally, to compare the dissolution rates of a jarosite-type compound without arsenic (the non-arsenic jarosite), a K-jarosite obtained in a previous work with the following approximate formula, $[\text{K}_{0.67}(\text{H}_3\text{O})_{0.33}]\text{Fe}_{2.43}(\text{SO}_4)_{2.11}[(\text{OH})_{4.10}(\text{H}_2\text{O})_{3.53}]$, was included (Reyes et al., 2016).

The chemical composition of the compounds was determined by atomic absorption spectrometry (AAS). Here, 1 g of each of the obtained precipitates was dissolved in a 50% (v/v) solution of concentrated hydrochloric acid. Analyses were carried out in an atomic absorption spectrometer (AAS, PerkinElmer Analyst 200, Waltham, MA USA) to determine the K, Na, Fe and As concentrations. The SO_4^{2-} content was determined from the BaSO_4 precipitate by gravimetric analysis. For the NH_4 -As-jarosite, the nitrogen content was determined with an elemental analyzer (PerkinElmer 2400 Series II CHNS/O, Waltham, MA USA), and the aqueous NH_4^+ was determined with an ion meter (Oakton Benchtop Ion 700, Vernon Hills, IL USA) coupled to a double-junction ammonium ion-selective electrode (Oakton) with a measurement range from 0.1 to 18,000 ppm. To determine the mineralogical composition, the obtained solids were also analyzed by X-ray Diffraction diffractometer (XRD, SIEMENS D-500 AG, Munich, Germany) using $\text{Cu-K}\alpha$ radiation ($\lambda = 1.54056 \text{ \AA}$). The morphology of the solids was examined with a scanning electron microscope (SEM, JEOL JSM-5900LV, JEOL USA, Inc., Peabody, MA, USA) equipped with an energy dispersive X-ray spectrometer detector (EDXS, INCA X-Sight Model, Oxford Instruments

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