



Atmospheric acid leaching mechanisms and kinetics and rheological studies of a low grade saprolitic nickel laterite ore



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ARTICLE INFO

Article history:

Received 28 July 2014

Received in revised form 29 October 2015

Accepted 5 November 2015

Available online 6 November 2015

Keywords:

Saprolitic Ni laterite

Acid leaching

Agitation

Temperature

Pulp rheology

ABSTRACT

Improved atmospheric acid leaching (AL) of complex, low grade nickel (Ni) laterite ores warrants greater knowledge of the exact processes underpinning Ni and pay metal cobalt (Co) extraction rates, acid consumption and pulp handleability. In this study, the influence of agitation rate (600–1000 rpm) and temperature (70 and 95 °C) on the isothermal, batch acid leaching and rheological behaviour of saprolitic Ni laterite slurry (40 wt.% solid) was investigated over 4 h at pH 1. The leaching behaviour was distinctly incongruent and reflected strong temperature-dependent Ni/Co extraction, acid consumption and the proliferation of gangue minerals' constituent elements (e.g., Na, Mg, Al, Fe). Whilst the total mass of acid consumed per ton of dry ore processed was greater at higher temperature, the total kg acid consumed per kg Ni and Co extracted was markedly lower. In all cases, the slurries displayed time-dependent, non-Newtonian, shear thinning rheological behaviour. The pulp viscosities and shear yield stresses were generally greater at lower than at higher temperature, with both increasing dramatically in the course of 4 h leaching. Agitation rate in the range 600–1000 rpm had no noticeable impact on Ni and Co leaching rates, confirming the insignificance of volume diffusion limitation. Although high pulp shear viscosities in the range 37–120 mPa s were observed in the course of leaching, they did not have an impact on Ni and Co leaching mechanisms and kinetics from the saprolitic laterite ore. The mechanism of saprolitic laterite ore leaching appears to follow a chemical reaction controlled, shrinking core model with apparent activation energies of 75.5 ± 3.8 and 81.2 ± 4.1 kJ/mol, respectively, for the release of Ni and Co.

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1. Introduction

Complex, low (<1.5%) grade Ni laterite ores represent ~70% of global reserves but account for only 40% of Ni production. Gradual depletion of high grade Ni sulphide ores which account for ~60% of Ni production and increasing demand for Ni in recent years has prompted the need to process more low grade lateritic ores (McDonald and Whittington, 2008; Quast et al., 2013; Tong et al., 2013; Whittington and Muir, 2000; Xu et al., 2013). Nickel laterites are formed through chemical weathering of rocks which leads to four major zones of mineralization (saprolites, transition, limonites and ferricretes). The saprolitic zone which located at the bottom of the deposit is mostly composed of Ni substituted serpentines or hydrous magnesium silicates (Kyle, 2010; Luo et al., 2009). Saprolites are the most reactive of the laterite ores with mineralogical variations leading to markedly different leaching behaviour. The choice of ore processing route employed is often driven by the grade, mineralogy and chemistry, capital and operating costs as well as environmental requirements (Kyle, 2010).

Atmospheric acid leaching (AL) in agitated vessels is a preferred processing method for low grade Ni laterites (Arroyo and Neudorf, 2004;

Canterford, 1978; Luo et al., 2010; McDonald and Whittington, 2008; Nosrati et al., 2014; Senanayake et al., 2011; Senanayake and Das, 2004). Typical AL conditions (e.g., 1–6 h residence time and 70–95 °C) result in <90% Ni extraction and high acid consumption values (>500 kg/t of dry ore) (Agatzini-Leonardou and Zafiratos, 2004; Kyle, 2010; Liu et al., 2012; Quast et al., 2013; Robertson and van Staden, 2009). For instance, AL studies of serpentinized laterite ore showed that 74% Ni and 51% Co extractions could be achieved after 2 h at 80 °C and acid consumption of 55 kg H₂SO₄/kg Ni (Agatzini-Leonardou and Zafiratos, 2004). It has also been shown that the rheological behaviour of laterite ore slurries plays a vital role in the pre-treatment and leaching processes and performance, impacting on operating pulp density and temperature (Bhattacharya et al., 1998; Klein and Hallbom, 2002; Tong et al., 2013). Conditions conducive to processability, higher throughput and reduced power input at high pulp solid loading are linked to the prevailing particulate fluid rheology (viscosity/yield stress).

Recent studies of the leaching behaviour of Ni laterite ores in sulphuric acid have revealed a strong mineralogy and chemistry dependent behaviour (Agatzini-Leonardou and Zafiratos, 2004; Das and de Lange, 2011; Griffin et al., 2002; Liu et al., 2012; Luo et al., 2010; MacCarthy et al., 2014; McDonald and Whittington, 2008; Nosrati et al., 2014; Quast et al., 2013; Reid and Barnett, 2002; Senanayake

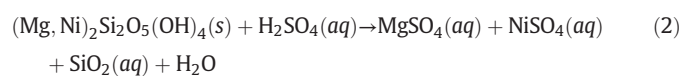
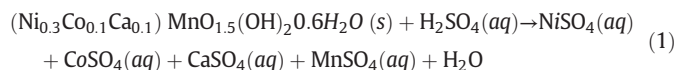
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et al., 2011; Xu et al., 2013). A study by McDonald and Whittington (2008) revealed that Ni leaching rate may be improved through ore pre-treatment (e.g., sulphurization, roasting) and/or prudent selection of process variables (e.g., temperature elevation, particle size reduction, salt addition). Recent studies by Nosrati et al. (2014) and MacCarthy et al. (2015a) have shown that during AL of a siliceous goethitic ore at pH 1, increasing temperature from 70 to 90 and 95 °C lead to dramatic improvement in Ni and Co extraction rates. Their findings suggested that at higher slurry solid loading and fixed pH (1), resulted in reduced Ni and Co extraction rates.

The effect of ore mineralogy on the column leaching performance of three low grade Ni laterites (saprolitic, siliceous goethitic and goethitic) has been investigated at ambient temperature (Xu et al., 2013). The results showed that whilst the saprolitic ore displayed the highest leaching rate, that of the goethitic ore was the least. Another column leaching study of agglomerated siliceous goethitic Ni laterite ore showed that after 100 d of acid irrigation, ~90% Ni and 80% Co extractions were obtained for finer (–2 mm) ore feed whilst this decreased noticeably for ore feeds with coarser (2–15 mm) particles (Quast et al., 2013). The mineralogical/chemical complexities and variability of laterite ores necessitates the development of ore-specific processing strategies for optimal recoveries via AL method (Griffin et al., 2002; Kyle, 2010; McDonald and Whittington, 2008; Reid and Barnett, 2002).

Unravelling the exact leaching mechanisms and kinetics of low grade laterites is challenging due to the complex nature of the ore and the presence of different Ni/Co-host gangue minerals and associations (Agatzini-Leonardou and Zafiratos, 2004; Hirasawa and Horita, 1987; Liu et al., 2007; Luo et al., 2009, 2010; McDonald and Whittington, 2008). The leaching mechanism involves the proton attack of Ni/Co mineralized particles and the host gangue mineral phases to release value Ni and Co from their lattices alongside other gangue metals. For instance, three important chemical reactions that occur during H₂SO₄-based leaching of asbolane and Ni-substituted serpentinites and goethite particles in laterite ores are summarized in Eqs. (1), (2) and (3), respectively (Rubisov et al., 2000; Senanayake et al., 2011).



On the mechanism of AL of saprolitic ores, it has been shown to involve partial decomposition of the Ni host Mg-silicate structure with very little silica release (McDonald and Whittington, 2008). Rice and Strong (1974) proposed a mechanism which involved the removal of the hydroxyl bonds that hold the octahedral brucite layer and tetrahedral silica layers. This causes the weak Mg–O bonds to breakdown, leading to the release of Ni and Mg whilst the silicate structure remains in place. The acid leaching mechanism is also shown to involve volume diffusion of protons through a porous residual layer to react with the unreacted saprolite ore particle core (Liu et al., 2007; Luo et al., 2010; Rinn and Fetting, 1982). Overall, the controlling mechanism for the leaching process is not dependent only on the mineralogy of the major serpentinitized phase but also on the minor host minerals such as nontronite, goethite, magnetite and quartz (Madejová et al., 1998; McDonald and Whittington, 2008; Sidhu et al., 1981).

Depending upon the controlling acid leaching mechanism, several kinetic models have been developed (Gbor et al., 2000; Levenspiel, 1999). In general for saprolitic ores, the shrinking core model has been shown to apply (Apostolidis and Distin, 1978; Luo et al., 2010). This model assumes that the solid particle retains its bulk size whilst its unreacted core shrinks progressively with reaction time leading to the

formation of a peripheral porous layer (Gbor et al., 2000; Levenspiel, 1999; Luo et al., 2010). The overall acid leaching reaction involves proton diffusion through the liquid film and the reacted porous layer, chemical reaction at the surface of the unreacted core and volume diffusion of reaction products through the porous layer and into the liquid film back into the bulk solution. The leaching rate is controlled by the slowest of these processes. Eqs. (4)–(6) summarize the shrinking core models.

$$x = kt \quad (4)$$

$$1 - 3(1-x)^{2/3} + 2(1-x) = kt \quad (5)$$

$$1 - (1-x)^{1/3} = kt \quad (6)$$

where x is the fraction of leached/dissolved metals (e.g., Ni, Co), k (min^{-1}) is the apparent rate constant and t (min) is time. Eq. (4) assumes the controlling step is volume diffusion, Eq. (5) assumes it is controlled by porous layer diffusion and Eq. (6) assumes the controlling step is the chemical reaction on the surface of the unreacted core. Based on the rate constants, the activation energy (E_a) for the reaction can be determined by using the Arrhenius relation (Eq. (7)):

$$k = k_0 e^{-E_a/RT} \quad (7)$$

where E_a (kJ mol^{-1}) is the activation energy, R ($8.314 \text{ J K}^{-1} \text{ mol}^{-1}$) is the ideal gas constant, k_0 (min^{-1}) is the pre-exponential factor and T (K) is the absolute temperature. For volume diffusion controlled processes, the E_a is generally low ($<20 \text{ kJ mol}^{-1}$) whilst for chemical reaction controlled processes it is high ($>40 \text{ kJ mol}^{-1}$) (Habashi, 1969).

Despite the advancement in knowledge and understanding of acid leaching behaviour of saprolitic (SAP) laterite ores by reported studies, there is persistence of a dearth of fundamental knowledge vital for marked improvement. In this work, isothermal, atmospheric acid leaching mechanism and kinetics of Ni/Co and gangue mineral elements from a Western Australian saprolitic ore, as well as the concomitant pulp rheology, are investigated. Specifically, the effect of batch leach time (up to 4 h), temperature (70 and 95 °C) and agitation rate (600–1000 rpm) on the leaching and rheological behaviour of 40% (w/w) solid dispersions was studied at pH 1. The mineralogy and chemical compositions of the solid feed and leach products, pulp chemistry and rheology data were characterized.

2. Experimental methods

2.1. Materials and methods

Polydispersed, –2 mm low grade, saprolitic Ni laterite ore from Western Australia was used in this study. The 10th, 50th and 90th percentile particle sizes, D_{10} , D_{50} and D_{90} , respectively, determined by laser diffraction (Malvern Mastersizer X, Malvern UK), were 8, 53 and 630 μm , respectively. The particles' specific surface area measured by the BET method was 46.3 m^2/g . 40% (w/w) solid dispersions were used for all experiments. The pH and temperature of 10^{-3} M KNO_3 (0.2 dm^3) solution used as background electrolyte were set to 1 and 70 or 95 ± 2 °C, respectively, before dry solids was added. Sulphuric acid (98 wt.%) was used to adjust and control the dispersion pH whilst high purity Milli-Q water (specific conductivity $<0.5 \mu\text{S cm}^{-1}$, surface tension at 20 °C of 72.8 mN m^{-1} and pH of 5.6) was used in preparing all solutions.

2.2. Batch leaching tests

Isothermal, batch leaching tests were conducted for 40% (w/w) solid dispersions in a well-sealed, acid resistant 0.5 dm^3 , cylindrical borosilicate glass vessel stirred by an overhead impeller powered by a variable

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