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Comprehensive examination of acid leaching behaviour of mineral phases from red mud: Recovery of Fe, Al, Ti, and Si

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ABSTRACT

Red mud represents an environmental and economic liability for the alumina industry in the form of wasted raw material. Although some leaching studies have been performed, there are deficiencies in the current literature regarding the amounts of metals extracted relative to each other, and minimal information regarding silicon contamination of extracts. There is also limited knowledge of extraction efficiencies of different acids (particularly in the case of phosphoric acid) under the same experimental conditions. This study focused on the leaching behaviour of the four most extractable elements present within red mud (iron, titanium, aluminium and silicon). By varying the experimental conditions, acid concentration, and type of acid, a comprehensive dataset of leaching trends was obtained. This allowed for direct comparison of leaching efficiency for the four elements under the same conditions, which was difficult previously due to the variation of experimental conditions and red mud composition between studies. The patterns in recoveries were explained in terms of the reactivities of the mineral phases within red mud and the interaction between the different acids and the reaction surfaces. Out of the four acids studied (nitric, hydrochloric, sulfuric, and phosphoric) phosphoric and hydrochloric acids produced some of the best recoveries for iron (76–78%) and titanium (23–24%), and phosphoric acid also produced the highest recoveries for silicon (49%) and aluminium (50%). The differences observed between the acid types and reaction conditions revealed potential for development of element selective extraction methods. Additionally, explaining leaching behaviour in terms of the mineral phases present allowed easier prediction of expected leaching trends for these four elements, which made this study applicable to red muds with a wide variety of compositions.

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1. Introduction

Metals present in industrial wastes represent significant losses in raw material and are a significant environmental liability (Power et al., 2011). Thus, development of practices to recover and reuse metals from wastes or to treat industrial effluents is potentially of great value. The alumina industry produces one of the largest industrial waste streams in the form of red mud; for every tonne of alumina extracted, between 1 and 1.5 tons of red mud is generated (Liu et al., 2009). Global red mud storage deposits are estimated at around 3 billion tons with an additional 120 million tons produced per annum (Marcel Côté and Wilson, 2012). Due to the highly caustic nature of the red mud slurry and the high proportion of heavy metals within it, disposal has been a major issue (Power et al., 2011). Before 1970, disposal of red mud was achieved through marine discharge, where the slurry was pumped *via* a pipeline into the deep ocean; or lagooning, where the slurry was contained in inland ponds (Power et al., 2011). Recently, emphasis has been placed on dry stacking disposal methods where the residue was thickened to a paste and pumped into pits (Power et al., 2011). The disadvantages of dry stacking and lagooning methods are that they use a lot of land area, which becomes unsuitable for further use. Additionally, disposal of red mud by such methods represents a loss of raw materials in the form of metals.

Red mud is predominantly comprised of oxides of aluminium, iron, titanium, and silicon, with other mixed mineral phases and trace heavy metals present (Evans et al., 2012); the proportion of each species is highly dependent on the original source of bauxite and the refinery conditions used (Evans et al., 2012). However, typically high amounts of aluminium oxide (5–30 wt%), iron oxide (5–60 wt%) and titanium oxide (0.3–15 wt%) make red mud a potential candidate for metal recovery (Evans et al., 2012). In light of this latter fact, considerable research has been undertaken to

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develop methods of extracting these aforementioned metals (Liu et al., 2009, 2012; Piga et al., 1995; Kitajima and Kasai, 1999; Uzun and Gulfen, 2007; Kasliwal and Sai, 1999; Li et al., 2011; Borra et al., 2015; Mishra et al., 2001; Şayan and Bayramoğlu, 2000, 2001; Papadopoulos et al., 2010). Tactics for recovering metals can be divided into two generic approaches, namely chemical (leaching) Uzun and Gulfen, 2007; Borra et al., 2015; Şayan and Bayramoğlu, 2000, 2001; Papadopoulos et al., 2010 and physical (magnetic separation and sintering) (Liu et al., 2009, 2012; Piga et al., 1995; Kitajima and Kasai, 1999; Li et al., 2011; Mishra et al., 2001).

Chemical leaching of metals from red mud is an attractive prospect due to its potential to allow selective recovery through manipulation of experimental conditions. Nevertheless, the current literature is somewhat limited in this regard. Many previous studies have focused on the extraction of a single element, with little regard to others, by varying parameters such as acid concentration, leaching time, leaching temperature, agitation rate and amount of solid in regards to the amount of acid (Uzun and Gulfen, 2007; Kasliwal and Sai, 1999; Borra et al., 2015; Şayan and Bayramoğlu, 2000, 2001; Papadopoulos et al., 2010). Examples of acids used in these leaching studies include sulfuric (Şayan and Bayramoğlu, 2000, 2001), hydrochloric (Kasliwal and Sai, 1999) and nitric (Borra et al., 2015), although phosphoric acid seems to have been overlooked. There exists general agreement that higher concentrations of acid result in greater levels of iron (Fe) and aluminium (Al) leaching Uzun and Gulfen, 2007; Borra et al., 2015; Şayan and Bayramoğlu, 2001, a trend which also extends to other elements such as titanium (Ti) Şayan and Bayramoğlu, 2000, 2001 and rare earth elements (Borra et al., 2015). This latter trend was also observed when elevated leaching temperatures were employed (Uzun and Gulfen, 2007; Kasliwal and Sai, 1999; Şayan and Bayramoğlu, 2000, 2001). Agitation of the sample during digestion also seems to play a role in the amount of dissolution of red mud. For example, Uzun and Gulfen (2007) found that increasing the agitation rate was effective (up to 400 rpm) in accelerating the amount of leached metal, after which no benefit was observed. Other studies (Şayan and Bayramoğlu, 2000) have also demonstrated an enhancement in leaching with higher agitation rates, but the consensus seems to be that it is of lesser importance than temperature or acid concentration. The final variable commonly tested by researchers is the solid to liquid ratio. A higher relative amount of acid compared to red mud has been correlated with higher metal recovery (Borra et al., 2015). Iron extraction as high as 97.46% using 6 M sulfuric acid at 105 °C was reached by Uzun and Gulfen (2007), while aluminium recoveries of 96.82% were reported by Şayan and Bayramoğlu (2001) using 4 M sulfuric acid at 90 °C, with a 4 h reaction time and a solid:solution ratio of 0.04. The same study reported maximum titanium recoveries, using the same conditions, of 96.33%.

The difficulty in evaluating results from these outlined studies is that the reported recoveries are usually with regard to a specific acid concentration and temperature, so it is difficult to compare results. Additionally, the composition of red mud varies between studies, with some experiments using Greek bauxite residue (Borra et al., 2015) that had a higher proportion of iron oxides than Turkish red mud (Şayan and Bayramoğlu, 2000) (44.6 and 37.7%, respectively). Such differences between the starting samples could impact the final composition of the acidic extracts and adds further complexity when comparing specific results from different studies.

Although the studies discussed above have reported good recoveries of their metals of focus, there is little mention with regards to the other elements present in the extract, which is vital information if the extract is intended for further use. Multi-element extraction from red mud has not been studied in detail, particularly in the case of silicon. The hypothesis of this study is that unless side by side extractions are carried out, the leaching patterns for multiple elements relative to each other cannot be determined. To confirm this hypothesis it was necessary to assess the effects that variation of experimental parameters such as reaction time, temperature and solid:solution ratio had on the final amount of each element leached from red mud, what effect acid concentration and type had on the extraction of these four elements, as well as if any elements were favoured over others under certain conditions.

This paper has addressed these questions through a series of leaching experiments, and through analysis of these results some leaching trends have been established for the four elements studied. The results represent valuable insight of leaching trends for four of the most extractable elements found in red mud, particularly in the case of silicon, which has been previously overlooked. The importance of knowing the composition of red mud extracts should not be underestimated, particularly in the context of possible tailored extraction methods or re-use of the extracts for metal scavenging or recovery.

2. Material and methods

2.1. Materials

Red mud used in this study was obtained from an Australian alumina refinery in the form of a slurry. The slurry was subsequently filtered to isolate the solid red mud, washed with deionised water until the filtrate was a neutral pH, dried at 80 °C and finally crushed. The crushed red mud was then sieved to obtain particles smaller than 212 μm. AR grade hydrochloric (32 wt/v%), nitric (70 wt/v%), sulfuric (98 wt/v%) and phosphoric (80 wt/v%) acids were used, and required concentrations for testing purposes were made from these concentrated stock solutions along with MilliQ ultrapure water through serial dilutions.

2.2. Experimental procedure

Acid leaching of red mud was performed using hydrochloric, nitric, sulfuric and phosphoric acids. The general procedure for these experiments involved: adding the desired amount of red mud and acid to 50 mL falcon tubes; allowing the digestion to proceed under specific conditions as described below; and then collecting the solid and liquid phases upon completion of each test. Separation of the solids from the liquid samples was achieved *via* centrifugation (3000 rpm for 10 min, Eppendorf 5702 centrifuge). All liquid samples were syringe filtered prior to storage and solid samples were washed with MilliQ water, dried in an oven at 80 °C and crushed before storage. ORP and pH measurements were taken of the liquid extracts using a Cole-Parmer Ag/AgCl electrode and a TPS pH probe. All testing series explored in this study were conducted at four different acid concentrations (0.01, 0.1, 1.0 and 5.0 M) to compare the leaching and metal recovery profiles. Particulars for each experimental test are as follows:

Temperature leaching trials: A solid:solution ratio of 0.005 and a leaching time of 24 h were used for all tests. The digestions were performed in a pre-heated bead bath using pre-heated acids with manual agitation. Temperatures tested were 25, 40, 55, 70 and 80 °C.

Time leaching trials: Reactions in this series were performed for 1, 4, 24, 48 and 168 h with a solid:solution ratio of 0.005 at room temperature (approximately 25 °C). A rotary stirrer was used to ensure constant, gentle agitation.

Solid:solution ratio leaching trials: Reactions in this series were performed at room temperature on a rotary stirrer to ensure constant, gentle agitation for a period of 24 h. The solid:solution ratios that were tested were 0.00125, 0.0025, 0.005, 0.0125 and 0.025.

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