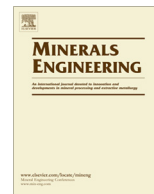




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An impedance spectroscopy study on the mitigation of clay slime coatings on chalcocite by electrolytes

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ABSTRACT

The flotation practice and the previous studies have demonstrated that chalcocite flotation can be depressed by the slime coating of clay minerals (Peng and Zhao, 2011; Zhao and Peng, 2012). In this study, how kaolinite coats chalcocite surface and how electrolytes mitigate this coating were studied using electrochemical impedance spectroscopy (EIS) for the first time. It was found that the formation of kaolinite coating on chalcocite surface decreased the dielectric constant and increased the impedance of chalcocite. Therefore, a reduction in impedance of a clay-coated mineral surface reflects the mitigation of the slime coating. It was also found that kaolinite coated the oxidized chalcocite surface corresponding to the depressed chalcocite flotation in the presence of kaolinite. Electrolytes reduced the electrostatic attraction between kaolinite and chalcocite, resulting in the mitigation of kaolinite coating in saline water and improved chalcocite flotation. For the cations (Li^+ , Na^+ and K^+) and anions (F^- , Cl^- and I^-) examined in this study, the larger ions reduced the slime coating more than the smaller ions presumably due to the greater decrease of electrostatic attraction between chalcocite and kaolinite.

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1. Introduction

The deleterious effect of clay minerals on copper flotation is well known in the mineral processing industry. Clay minerals such as kaolinite, bentonite and smectite are often associated with valuable minerals and pose a widespread problem in mineral flotation (Cruz et al., 2013). Currently, the ways to treat high clay ores include blending them at a small proportion with normal ores, processing them at low solid concentrations, using viscosity modifiers and removing clay slimes from the flotation feed (Connelly, 2011; Peng and Zhao, 2011; Quast et al., 2008; Zhang and Peng, 2015). The difficulty in treating high clay ores may be related to the unique properties of clay minerals.

Clay minerals are anisotropic and hydrated phyllosilicates with unit cells comprising a layer of one alumina octahedral sheet and either one (such as kaolinite) or two (such as smectite) silica tetrahedral sheets (Brigatti et al., 2006). A single particle may comprise several layers stacked on top of one another. Due to isomorphous substitution (e.g., Al (III) replacing Si (IV) in the SiO_2 layer), the

basal faces of clay minerals carry a constant negative charge which is pH independent (Luckham and Rossi, 1999; Zhao et al., 2008). At the edges of the layers, the tetrahedral silica sheets and the octahedral alumina sheets have broken primary bonds. The electrical charge of the edge, arising from hydrolysis reactions from broken Al–O and Si–O bonds, is pH dependent. The edges of clay particles are positively charged in neutral and acid pH ranges depending on the type of clay minerals (Swartzen-Allen and Egon, 1974). The anisotropic charges on edges and basal faces allow clay slime coating on the surface of a range of minerals.

It has been well documented that clay slime coating occurs on galena, coal and bitumen surfaces through the electrostatic attraction, reduces surface hydrophobicity and then depresses mineral flotation significantly (Gaudin et al., 1960; Arnold and Aplan, 1986; Liu et al., 2005). In the previous study, Peng and Zhao (2011) investigated the effect of bentonite on the flotation of chalcocite in terms of slime coating. They found that, as a result of surface oxidation, chalcocite became positively charged after grinding and was electrostatically attracted to bentonite which was positively charged, resulting in bentonite slime coating and the depressed flotation of chalcocite. Zhao and Peng (2012) also confirmed the occurrence of bentonite slime coating on chalcocite in flotation by Cryo-SEM measurements.

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On the other hand, in industrial practice, bore water and hyper-saline water with a high content of electrolytes have been used in mineral flotation. The effect of electrolytes on mineral flotation has been studied by a number of researchers from a different perspective (Paulson and Pugh, 1996; Craig et al., 1993; Hancer et al., 2001). Peng and Bradshaw (2012) demonstrated that pentlandite flotation in the presence of lizardite was improved in saline water through the mitigation of lizardite slime coating on pentlandite surface. They found that saline water minimized the magnitude of the zeta potential on both lizardite and pentlandite and therefore the electrostatic attraction between them. Zhao and Peng (2014) reported that with the addition of NaCl, chalcocite flotation in the presence of kaolinite was improved significantly and the high NaCl concentration was favourable for the flotation. It was found that the tested cations and anions mitigated the negative effect of kaolinite by the order of $K^+ > Na^+ > Li^+$ and $I^- > Cl^- > F^-$. It is interesting that this order follows the Hofmeister series from the least hydrated to the most hydrated ions. In that study by Zhao and Peng (2014), the mechanism responsible for the beneficial effect of cations and anions on chalcocite flotation in the presence of kaolinite was not identified. It seems that this effect was associated with the mitigation of kaolinite slime coating on chalcocite surface.

Despite the widespread problem of slime coating in mineral flotation, a direct detection of slime coating is not available. Traditionally, zeta potentials on two single minerals are measured to infer the electrostatic attraction which may cause one mineral to coat the other mineral. Recently, Cryo-SEM has been used to detect slime coating. The advantage of Cryo-SEM is that samples taken from slurries are snap-frozen to preserve mineral surfaces in the vitrified water without crystallisation as ice (Battersby et al., 1994). Coupled with energy-dispersive X-ray spectroscopy (EDS) to identify the elemental composition of chosen mineral areas, Cryo-SEM can confidently detect slime coating. However, strictly speaking, Cryo-SEM is not an in-situ technique involving a sampling process. It is also sophisticated with limited accessibility.

The electrochemical impedance spectroscopy (EIS) is a sensitive analytical technique with a high accuracy, allowing an in-situ detection of the formation of surface layers on the electrode which is well correlated with the electrode capacitance. The measured impedance data can also reflect the kinetics of surface electrochemical reactions in-situ with a minimum of surface modification (De Wet et al., 1997; Niu et al., 2014). Ekmekçi et al. (2010) used EIS to study the adsorption of $CuSO_4$ and SIBX on pyrrhotite surface and correlated pyrrhotite impedance with its flotation behaviour. Guo et al. (2015) demonstrated that a change in the impedance of pyrite electrode was consistent with the adsorbed xanthate. Mu et al. (2015) reported that the presence of lignosulfonate-based biopolymers on pyrite surface was reflected by a change in impedance of pyrite electrode. In this study, for the first time, the EIS was applied to investigate the slime coating and its mitigation on mineral surface. It is expected that the impedance of a mineral electrode in the absence and presence of slime coating is different.

2. Experimental

2.1. Materials and reagents

A kaolinite sample was purchased from Sibelco Australia Limited Company to study the interaction between clay minerals and chalcocite in the flotation context. It contains 70.3% kaolinite, 18.8% muscovite and 6.8% quartz based on quantitative XRD analysis.

Sodium chloride (NaCl), lithium chloride (LiCl), sodium fluorite (NaF), potassium chloride (KCl) and sodium iodide (NaI) of analytical grade were used to make different ionic strengths of solution. These electrolytes vary in both anion and cation sizes. All solutions were made with AR grade chemicals and de-ionized (DI) water.

2.2. Electrochemical measurements

A hand-picked natural massive chalcocite specimen of a high purity was used to make a working electrode. XRD analysis showed little impurities of the chalcocite specimen. The electrode was connected with a copper wire using silver-loaded conducting epoxy, and then mounted into a non-conducting epoxy resin, exposing only one side with a geometric surface of approximately 0.1 cm^2 and this value was used to calculate the current density.

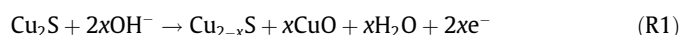
A fresh electrode surface was prepared by abrading with a silicon carbide abrasive paper (1200 grits) and then rinsed with deionized water prior to each experiment (Velasquez et al., 2001; Smith et al., 2009). Chalcocite electrode was immersed in clay and/or electrolyte-containing solutions for 2 min under stirring and then rinsed with DI water. The electrode was then immediately transferred into the electrochemical cell as a stationary working electrode.

Conventional three-electrode system was employed for the electrochemical measurements. A platinum plate with a surface area of 1 cm^2 was utilised as an auxiliary electrode (counter electrode). Potentials were measured against a Ag/AgCl reference electrode filled with 3 M KCl which has a potential of +0.204 V against standard hydrogen electrode (SHE). A Radiometer PGZ100 potentiostat was used in combination with a Frequency Response Analyser (FRA). The background electrolyte performed in all electrochemical experiments was pH 7 buffer solution (prepared by 0.1 M potassium dihydrogen phosphate). EIS was performed in the above buffer solution with a volume of 200 mL at room temperature in the absence and presence of electrolytes and kaolinite individually and in combination. Deionized water ($18 \text{ M}\Omega \text{ cm}$) was used in all electrochemical experiments. Open circuit potential was taken as the direct current (DC) voltage (starting potential) of EIS measurements. The alternating current (AC) voltage (amplitude) was 10 mV. Typically, the working electrode surface was allowed to react in the solution for 5 min to enhance the stabilisation at the open circuit potential after which the EIS was obtained. The potential scan rate was 20 mV s^{-1} for all cyclic voltammetry measurements. The variation in the current was recorded as a function of scan potential and reported as current density. Good reproducibility of the electrode pre-treatment and electrolyte preparation was confirmed by carrying out measurements in separate solutions and with freshly abraded electrode.

3. Results and discussion

3.1. Cyclic voltammetry

The voltammogram of chalcocite electrode in background solution is shown in Fig. 1. Chalcocite voltammograms with surface reactions in aqueous solutions have been frequently reported (Sato, 1960; Velasquez et al., 2001). The peak from approximately 0.05–0.25 V on the positive-going sweep was attributed to the oxidation of chalcocite resulting in the formation of cupric oxide and a sulfur-rich sub-layer (covellite) as shown in Reaction (1).



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