



The kinetics of nickel recovery from ferrous containing solutions using an Iminodiacetic acid ion exchange resin

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ABSTRACT

This paper reports on the investigation of the loading rate of nickel onto an iminodiacetic acid ion exchange resin (TP207XL) under the finite solution volume condition in the presence of ferrous in a batch loading system. A hybrid correlation model (McKevitt, 2012) was applied for nickel-ferrous displacement loading data. The experimental fit parameters of the hybrid correlation were used to successfully predict the results of the batch loading experiments by applying the finite difference model. The finite difference model was applied to predict the loading behavior of nickel in the presence of ferrous in the batch system using the modified Helfferich number and the linear isotherm equation. As confirmed, the ion exchange reaction examined in this study was predominantly controlled under some combination of intraparticle diffusion/ion exchange reaction. Regarding the hybrid correlation fit parameters ($D_{app} = 9.95 \times 10^{-12} \text{ m}^2/\text{s}$, $\alpha = 0.65$), these parameters correlated well with the loading behavior of nickel onto resin.

1. Introduction

The use of ion exchange resin and the resin-in-pulp (RIP) process have been considered and proposed as methods of primary or secondary metal recovery from nickel laterite leach operations (Wassink et al., 2006, Mendes and Martins, 2005, Zainol, 2005). Previous studies on the application of several forms of resin for the RIP processes proposed that commercially-available chelating resins with the iminodiacetic acid functional group would be most suitable for nickel recovery (McKevitt et al., 2011; Zainol, 2005). Iminodiacetic acid ion exchange resins have a high affinity for loading nickel. However, analysis of the loaded resin from the RIP process proved that much of the resin capacity will be taken up by impurity elements such as calcium, manganese, magnesium, and ferrous, especially in the last few reactors of an RIP circuit where the fresh resin is contacting low nickel solution. To develop an understanding of nickel impurity displacement in an RIP system, nickel displacement of ferrous iron was chosen for this study. Ferrous was chosen over other impurity elements of interest because it has the closest decomplexing pH to nickel [Ni: 2.1, Fe II: 3.0, Mn: 4.0, Ca: 4.4, Mg: 4.6], (Halle and Rossoni, 2005). Thus, of these impurity elements, ferrous is expected to have the strongest bond to the iminodiacetic resin and is expected to be the most difficult one to displace (Abbasi et al., 2014).

McKevitt developed the hybrid correlation in order to model the loading of base metal ions in an RIP circuit. The detailed information

regarding this model is available in the literature (McKevitt, 2012). McKevitt and Dreisinger evaluated the rate-limiting steps qualitatively by running several loading experiments with copper, cobalt, and nickel onto the TP207 iminodiacetic ion exchange resin (McKevitt and Dreisinger, 2012a). They developed the model first by determining and understanding the rate-limiting step(s) of the ion exchange reaction given in Eq. (1). According to those experimental results, they concluded that the process was controlled by the film diffusion regime in the beginning stage of the process, followed by some combination of intraparticle diffusion and ion exchange reaction (McKevitt and Dreisinger, 2012a). After applying several conventional engineering models to the system during the intraparticle diffusion/ion exchange reaction regime it was concluded that while each conventional model could provide an adequate fit to each individual kinetic tests, none of the models could adequately explain the entire family of curves generated at different solution concentrations with a single intraparticle diffusion coefficient. As a result, they proposed “a new two-parameter hybrid correlation” (McKevitt and Dreisinger, 2012c).



The idea of this new correlation came from the fact that the system dependency on concentration is somewhere between $[\text{Ni}]^0$ according to the Vermeulen model and $[\text{Ni}]^1$ according to Shrinking Core model. Since both models fit the data fairly well for each individual loading curve, McKevitt and Dreisinger combined the two models together by

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introducing a second empirical fit parameter, alpha, to allow for the dependency on solution concentration to vary between zero and one. Since the two models have quite different mathematical expressions, but both are able to describe an individual loading curve adequately, they first approximated the Shrinking Core model by a Vermeulen expression by finding a correlation between the two constants (k_c and k_v). They reported that the difference between the two models for a given curve was minimized when $k_v = 0.677 k_c$.

This empirical fit alpha (α) was found to provide insight into the metal ion loading profile within the resin bead. Regarding the metal loading profile, alpha can be considered as a measurement of sharpness of the boundary present between the loaded and unloaded part of the resin bead. When alpha is equal to zero, the hybrid correlation reduces to Vermeulen's approximation to intraparticle diffusion, whereas for alpha equal to one, the correlation becomes an approximation to the Shrinking Core model, as described in the previous paragraph. The equations describing the hybrid correlation are defined in Eq. (2) where Eq. (3) (F) is the mathematical solution for it. In addition, Eq. (4) also defines a new rate constant, k_h (1/s), introduced by the hybrid correlation (McKevitt and Dreisinger, 2012c). The step-by-step derivation of the hybrid model is explained elsewhere (Abbasi, 2014).

$$\frac{d}{dt} [\bar{M}] = \frac{2k_h ([\bar{M}]_{eqb} - [\bar{M}]^2)}{[\bar{M}]} \quad (2)$$

$$F = \sqrt{1 - \exp(-4k_h t)} \quad (3)$$

$$k_h = \frac{\pi^2 D_{app}}{d_p^2} \left(\frac{16[M]}{\pi^2 [\bar{M}]_{max}} \right)^\alpha \quad (4)$$

The previous work by McKevitt and Dreisinger was examining the loading of individual divalent metal cations onto a resin in the hydrogen form. However, in a resin-in-pulp circuit, the final stages will be depleted in the value metal (for example nickel) and therefore the loading of a non-value metal (for example ferrous iron, if present in solution) may be favored. As the resin advances to the head of the resin-in-pulp circuit, the value metal (nickel) will therefore displace the non-value metal (ferrous) as part of the loading of nickel on the resin. The purpose of this work then was to consider whether the McKevitt hybrid model could be applied to the metal-metal loading process in order to facilitate resin-in-pulp circuit modeling. To achieve this, the application of the modified Helfferich number and the hybrid correlation for nickel displacement of ferrous under finite solution volume condition was examined.

2. Materials and method

2.1. Material

In this study, all the experiments were carried out using one of the recently manufactured large bead iminodiacetic ion exchange resins (Lanxess Lewatit MonoPlus TP207XL) to investigate the loading behavior of ferrous and nickel ions. This polymeric resin is used for ion exchange experiments in either the hydrogen or sodium form. For this work, the as-received resin was converted to the hydrogen form following the manufacturer's recommended procedure. The hydrogen form and chelate structure of this iminodiacetic ion exchange resin are illustrated in Fig. 1. Prior to use, the resin was wet-screened to remove the -710 and $+850$ μm fractions to give resin in the size range of 710 – 850 μm . This resin was selected for the purpose of this research, since according to the previous study it has a narrow size distribution for a large bead resin, which is desired in the RIP application by providing for easy screening of the resin from solids (McKevitt, 2012). In addition, this resin showed the fastest loading rate for this size fraction compared to other commercially available resins (McKevitt, 2012).

Table 1 summarizes the resin (TP207XL) operational capacity reported by McKevitt (McKevitt, 2012) and Littlejohn and Vaughan

(Littlejohn and Vaughan, 2012) for the same resin bead particle size of 710 – 850 μm at pH 4. As described in this table, the capacity for this resin is rated between 2.0 and 2.36 (eq/L). The resin capacity unit of eq/L is the number of equivalents of exchangeable ions per unit volume of resin. Note that in this work, all resin volumes are in terms of the wet-settled bulk volume of the resin in the hydrogen form.

A synthetic solution was prepared for each experiment from ferrous and nickel sulphate salts with various target concentration ranging from 100 to 10,000 mg/L using American Chemical Society (ACS) grade reagents.

2.2. Method development for batch system

The original methodology was developed previously by McKevitt to study the loading behavior of metal ions such as copper nickel and cobalt (McKevitt, 2012). The hybrid correlation model was validated at a constant solution pH of 4 under finite solution volume conditions (FSV), where the composition of the solution varied with time. In addition, pH 4 was maintained for the entire time of the experiment by means of the pH-stat method modified by McKevitt (McKevitt, 2012). A constant pH in the test solutions is obtained in the standard pH-stat method that utilizes a pH electrode and controller for adding alkali hydroxide (base). The rate of base addition can be used to calculate rate of metal ion loading onto the resin.

2.3. Nickel-ferrous displacement loading

In order to perform the $\text{Ni}^{2+}/\text{Fe}^{2+}$ displacement experiments, it was necessary to have the resin loaded as much as possible with ferrous iron initially. As a result, the 5 mL resin samples for displacement tests were loaded from solutions with very high concentrations of ferrous ($\sim 10,000$ mg/L) under infinite solution volume conditions. In these experiments, the test solution was deaerated for approximately 20 to 30 min with 1 L/min nitrogen gas sparging. This results in limiting the ferrous oxidation reaction of the ferrous sulphate added to the solution to make up the specific desired concentration.

Once the resin was loaded with ferrous ions, it was introduced to the test solution containing the desired nickel sulphate concentration with a background concentration of ferrous close to 10,000 mg/L. The target of 10,000 mg/L ferrous concentration was used based on the previous study of ferrous loading onto iminodiacetic resin. Fig. 2 illustrates the equilibrium isotherm from that work for ferrous loading onto TP207XL iminodiacetic ion exchange resin at pH 4, 30 °C, and constant iron concentration. This figure shows that the strip and load data are reasonably represented by the application of the mass action law and that a change of ± 0.5 g/L Fe from an initial concentration of 10 g/L Fe will have very little impact on the equilibrium resin loading (Abbasi et al., 2014).

Similar to the ferrous loading experiments of the previous study, in displacement tests, it was desired to maintain a constant pH of 4 in the test vessel. However, since these displacement tests were performed under finite solution volume conditions (i.e. the concentration of ferrous and nickel sulphate in solution were not kept constant) there was no need of nickel or ferrous sulphate stock addition to the test vessel. As illustrated in Fig. 3, for an experiment starting with nearly 9000 mg/L iron as ferrous sulphate and target concentration of 2500 mg/L nickel as nickel sulphate, the concentration of nickel ion in solution decreased from 2400 to 1985 mg/L, while the ferrous ion concentration slightly increased from 9230 to 9510 mg/L due to removal of nickel ions from solution with concurrent displacement of ferrous ions from the pre-loaded resin. According to the isotherm model of the previous study, the equilibrium loading values of ferrous ions increases from approximately 49.0 to 49.1 g Fe/L_{Resin} when the solution concentration varies between 9230 and 9510 mg/L ferrous under infinite solution volume condition. Therefore, it can be concluded that the change in equilibrium driving force caused by the ferrous iron displacement from the resin is

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