



Re-use of waste red mud: Production of a functional iron oxide adsorbent for removal of phosphorous



R.A. Pepper^b, S.J. Couperthwaite^{b,*}, G.J. Millar^{a,b}

^a Institute for Future Environments & School of Chemistry, Physics & Mechanical Engineering, Australia

^b Science and Engineering Faculty, Queensland University of Technology (QUT), GPO Box 2434, Brisbane, Queensland, 4001, Australia

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ABSTRACT

An iron oxide sorbent derived from red mud waste (termed red mud akaganeite (RMA)) was investigated for the treatment of waters containing excess phosphorus (P) based nutrients such as phosphate. The hypothesis was that this innovative sorbent could outperform commercial granular ferric hydroxide (GFH). Sorbent performance was tested by a combination of sorption kinetics and equilibrium studies. Key factors evaluated included influence of phosphorous concentration, solution pH, and presence of competing anions. The kinetics of phosphorous sorption on both RMA and GFH were best described by the pseudo first order model, albeit the RMA material showed a higher rate of sorption. The equilibrium capacity as modelled by the Langmuir equation was 12.9 mg P/g for RMA, which was over 140% greater than that of GFH. X-ray photoelectron spectroscopy suggested that surface hydroxyl species were part of the mechanism for phosphorus uptake. The efficiency of phosphorous uptake by RMA was relatively unaffected by solution pH. In contrast with GFH, a significant loss in performance above pH 8 was found. It was postulated that point of zero charge on the sorbent and ability to release protons and chloride into the water may explain the pH data. Nitrate, sulfate, and chloride had minimal effect on phosphorus uptake by RMA or GFH, but both materials were impacted by the presence of carbonate species. Infrared spectroscopy indicated the presence of carbonate on the sorbent surface. Treatment of stormwater revealed that RMA was potentially suitable as a sorbent for phosphorus from real water samples.

1. Introduction

Phosphorus when present as phosphate in water systems is an essential macronutrient for the growth of both organisms and ecosystems. However, excessive phosphorus levels can be detrimental to these same ecosystems through eutrophication of water resources [1]. Consequently, there is a need to develop effective strategies which remove phosphorus from wastewater. A number of methods have been investigated such as chemical precipitation, biological removal, crystallisation techniques, ion exchange, and adsorption [2]. Sorbent processes are particularly attractive due to their relative ease of use, low cost and efficiency [3].

Iron oxides are a class of minerals that are frequently encountered in the literature with regards to phosphorus remediation. Among these materials is magnetite [4] which had a reported capacity of 15.2 mg P/g. Chitrakar et al. [5] also considered phosphorus adsorption by goethite and akaganeite. The maximum loading for both materials was noted at pH 2, and was 28 and 24 mg P/g for akaganeite and goethite,

respectively. The outlined study also considered the removal of phosphorus from a seawater matrix, in which both materials remained selective for phosphorus. A second study which considered akaganeite materials was by Deliyanni et al. [6] wherein a maximum capacity of 451.20 mg P/g was reported for a surfactant-modified akaganeite (compared to 59.62 mg P/g for unmodified akaganeite). The surfactant bilayer was implicated for the high performance of this material, through facilitation of P sorption through electrostatic interactions. The phosphorus on the un-modified akaganeite was able to be removed with caustic treatment, but the surfactant-akaganeite was unable to be regenerated.

Commercial materials such as granular ferric hydroxide (GFH) have also been considered for phosphorus removal. Genz et al. [7] found that GFH was a suitable sorbent for the post treatment of membrane bioreactor effluents. The maximum capacity was measured as 23.3 mg P/g at pH 5.5, and the authors noted that capacity was reduced to 16.9 mg P/g at pH 8.2. Although competition for adsorption sites between inorganic ions and phosphorus was low, dissolved organic

* Corresponding author at: Science and Engineering Faculty, Queensland University of Technology, P Block, Level 7, P701A-1, Gardens Point Campus, Brisbane, Queensland, 4000, Australia.

E-mail address: sara.couperthwaite@qut.edu.au (S.J. Couperthwaite).

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carbon did compete for adsorption sites. Kartashevsky et al. [8] also considered GFH for phosphorus adsorption from reverse osmosis effluent in order to reduce the impact of calcium phosphate scale. Column experiments revealed that GFH was effective for treating 3300 bed volumes at an inlet concentration of 9.3 mg/L P. Regeneration of GFH was successful with sodium hydroxide, although the capacity of the GFH media was reduced over recurring regeneration cycles. Both these studies demonstrated that GFH was a viable candidate for phosphorus removal in complex or realistic systems; however, the expense of GFH may be prohibitive for larger scale treatment [7].

Despite the demonstrated effectiveness of the previously discussed materials for phosphorus removal from aqueous solution, there are several key aspects which need to be considered. Ideally, suitable phosphorus sorbents should be derived from a waste material if possible. Transforming a waste which inherently may present an economic and potential environmental liability to an industry sector into a saleable product is desirable. In relation to the aforementioned evaluation of materials for phosphorus removal from wastewater, iron can readily be found in waste deposits worldwide. For example, red mud is generated in substantial quantities from the Bayer process used by the alumina refining industry [9]. In harmony with their iron content, modified granular red mud sorbents were found to have a maximum phosphate adsorption capacity of 9.8 mg/g [10]. Another study [11] considered the phosphorus removal ability of a red mud sample that had been modified by hydrochloric acid. Although the acid treatment did improve the capacity of the red mud, the maximum capacity of these materials did not exceed 0.58 mg P/g. Although there has been some attention in the literature to modifying red mud to enhance phosphorus uptake [10–12], there are limited studies that consider the production of higher performing sorbents from this waste material.

Recently, red mud has been used as a starting point for the synthesis of nanocrystalline and high surface area akaganeite through a selective leaching method [13]. The synthesised red mud akaganeite (RMA) proved to be a highly selective sorbent for fluoride compared to a commercial akaganeite-based material, granular ferric hydroxide (GFH). However, the performance of this material as a sorbent for other species (such as phosphorus) is yet to be assessed. As such, the aim of the current study was to determine the suitability of RMA as a sorbent for phosphorus and compare its performance to commercial GFH. The hypothesis was that the red mud derived sorbent had phosphorus adsorption capacities that were comparable, if not superior to, commercial equivalents. In order to test this hypothesis, the following research questions were addressed: (1) what is the phosphorus equilibrium and kinetic sorption behaviour of RMA; (2) is RMA selective for phosphorus in the presence of common competing ions; (3) what is the mechanism of phosphorus removal on RMA; (4) how does RMA perform with regards to phosphorus in the complex matrix of a real wastewater sample; (5) how does the overall performance of the red mud derived material compare to commercial GFH. Consequently, the synthesised RMA sorbent was tested across comprehensive adsorption experiments were completed which included kinetic, equilibrium, pH, and competitive studies. The results of the current work demonstrated the value of the synthesised RMA material as a possible solution to the issues of bauxite residue utilisation and phosphorus contamination of water bodies.

2. Materials and methods

2.1. Chemicals and materials

The red mud used in this study was sourced from an alumina refinery located in Australia. Prior to use, the residue was rinsed to remove remaining caustic impurities which remained after the Bayer process. The washed residue was placed in an oven (90 °C) until dry. The dried residue was subsequently crushed, and a size fraction of 200–400 microns was obtained for further experimentation. The chemical and mineralogical composition of the red mud used in this study

Table 1
Composition of stormwater.

Parameter	Value	Units
pH	6.12	
Turbidity	0.74	NTU
Suspended solids	39.0	mg/L
Total Inorganic Carbon	0.40	mg/L
Ammonia as N	5.03	mg/L
Ba	0.04	mg/L
Ca	23.4	mg/L
K	4.19	mg/L
Li	0.01	mg/L
Mg	6.04	mg/L
Na	33.5	mg/L
P	10.0	mg/L
S	22.1	mg/L
Si	0.90	mg/L
Ni	11.3	mg/L
Cu	0.01	mg/L
Zn	1.61	mg/L

has been detailed in earlier work [13]. In summary, the major components present were iron (29.8%), aluminium (21.7%), silicon (12.3%), titanium (6.9%) sodium (5.2%), calcium (1.8%), magnesium (1.5%), sulphur (0.2%), potassium (0.2%), and manganese (0.04%). GFH was supplied by GEH Wasserchemie as a granular solid. Reagents used were obtained from ChemSupply and of analytical grade. Chemicals used included: hydrochloric acid (HCl) (32%); sodium hydroxide (NaOH); sodium chloride (NaCl); sodium hydrogen phosphate (NaH_2PO_4); sodium sulfate (Na_2SO_4); sodium nitrate (NaNO_3); and, sodium bicarbonate (NaHCO_3). A stormwater sample from Queensland, Australia was obtained and the water quality displayed in Table 1.

2.2. Extraction of iron from red mud and synthesis of iron oxides

The sorbent material used in this study was prepared from an extract of red mud through a sequential leaching process [14]. An initial wash of the red mud was performed for 60 min at room temperature using HCl (1 M), followed by digestion of the washed residue with 5 M HCl at 80 °C for a period of 24 h. The sequential leaching process allowed a final extract to be obtained that was both high in iron and low in other elements which may interfere with later stages of the synthesis [15]. The final sorbent was prepared from this extract by adjustment of solution pH to a value between 2.5 and 3 by application of a solution of NaCl/NaOH. The reaction was aged for 3 days at a temperature of 70 °C and then the sorbent was filtered from the reaction liquor and washed to remove residual NaCl. The washed sorbent was oven dried at 70 °C before characterisation and adsorption experiments. The prepared sample was determined to be comprised of the akaganeite phase only, with some aluminium and titanium inclusion (0.9 and 2.9 wt%, respectively) [14].

2.3. Phosphorus sorption methodology

Test solutions were prepared by dissolution of appropriate amounts of NaHPO_4 in deionised water. After completion of sorption tests, samples were immediately passed through 45-micron syringe filters and residual P (see Section 2.4.4) was analysed to determine phosphorus loading according to Eq. (1):

$$q = \frac{V}{m}(C_o - C_e) \quad (1)$$

Where: q = phosphorus loading (mg/g); V = volume of solution (L); m = mass of sorbent (g); C_o = initial P concentration (mg/L); and C_e = concentration of P after completion of test (mg/L).

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