



Research article

Metal removal from solution using readily available inorganic reactive materials



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ABSTRACT

This research evaluated using inorganic adsorbents for removal of metals from solution. Batch experiments were carried out to determine the effects of adsorbent dosage, initial metal concentration and agitation time on metal removal. The results showed increasing metal ion removal with increasing adsorbent dosage, metal concentration and agitation time. Removal efficiencies of Al^{3+} and Mn^{2+} were reported at above 99% and 98%. Kinetic studies showed that the three adsorbents data on both Al^{3+} and Mn^{2+} removal were pertaining to pseudo-first-order and second-order kinetics and had a good fit to Elovich indicating that the adsorption rate-limiting step could be inferred as chemical sorption.

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1. Introduction

Mining in South Africa has been a driving force behind one of Africa's biggest and richest economy. Gold mining in South Africa commenced in 1886 in the Johannesburg area and it continued until the early 1960s (Naicker et al., 2003). South Africa's gold export amounted to \$3.8 billion making it the world's leading producer of gold in 2005 which accounted for 12% of global gold production (Yager, 2004). The mining operations left the Witwatersrand mine voids as the deepest in the world with underground final depths of 2500 m below surface. The conglomerates contain pebbles of quartz which comprises about 3% of pyrite and lesser amount of other sulphides bearing rocks inclusive of gold (Naicker et al., 2003). After the extraction of valuable commodities, mine tailings were dumped nearby and exposed for oxidation by oxygenated rain water. As of September 2002, untreated acidic mine water has been uncontrollably decanting from Black Reef Incline Shaft and the No.17 and 18 Winze Shafts into Tweelopiespruit which is a tributary of Bloubank which discharges into Crocodile River Naicker et al., (2003) and Ochieng et al. (2010). Acid mine drainage (AMD) which was formed through oxidation of sulphide rocks on the mine tailings, has been seeping into the mine voids causing level rise until it reached the surface.

AMD is acidic water which forms when sulphide-bearing materials predominantly pyrite are exposed to oxygen and water and

sometimes the reaction is catalysed by the presents of Acidithiobacillus bacteria (Akcil and Koldas, 2006). First, sulphuric acid and ferrous sulphate are produced followed by production of orange-red ferric hydroxide and more sulphuric acid during a two-stage oxidation process of pyrite (McCarthy, 2011). The formation of acid mine drainage is a natural process which could take about 15 years for ferric iron to produce acid but presence of bacteria shortens the reaction time down to 8 min (Metesh et al., 1998). Drainage from coal mines is less acidic (in terms of proton acidity as opposed to mineral acidity) due to moderately high carbonate content of the host rock which provide buffering capacity. In contrast, drainage from metal mines and spoils are more acidic and contains high concentration of metals (Hallberg, 2010). Formation of AMD varies from site to site because of different mineralogy and other factors affecting formation of AMD and this makes predicting the potential for AMD to be more exceptionally challenging and very costly. The nature and size of associated risks and feasibility of mitigation options vary from site to site (Akcil and Koldas, 2006).

Most areas find it impossible to prevent formation or migration of AMD from its source and in such cases, it is only required to collect, treat, and discharge mine water (Johnson and Hallberg, 2005). The cost benefits of utilising inorganic materials has seen increased application of passively treating acid mine drainage (Gusek and Clarke-Whistler, 2005). Passive treatment system relies on the biological, geochemical and gravitational activities. They do not require frequent maintenance or continuous addition of chemicals. Reactive materials are positioned in the path of contaminated plume and move through the material as it flows,

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typically under its natural gradient (creating a passive treatment system) and treated effluent comes out the other side (Puls et al., 1999). In this research, batch tests were conducted to assess the neutralizing and metal removal capacity of coal ash (fly and bottom ash), bentonite clay and charcoal.

2. Experimental

2.1. Materials and methods

Coal fly ash was collected from Eskom power station in Ermelo Mpumalanga, commercial bentonite clay and charcoal were purchased from Sigma Aldrich. Aluminium and Manganese sulphates to prepare synthetic AMD were purchased from Rochelle Chemicals. Sulphuric acid and Sodium hydroxide to manipulate the pH were also purchased from Rochelle Chemicals. All chemicals were used as received without purification. The reactive materials were sieved and dried in a 105 °C oven to get rid of any moisture.

2.2. Adsorption experiments

The experiments were conducted in an orbital shaker to provide continuous motion at 150 rpm. 500 ml bottles were used as reaction bottles. Synthetic feed solution containing 54 mg Al³⁺/l and 321 mg Mn²⁺/l was prepared as per the characterized data of mine water collected from Randfontein (Black Reef Incline, 17 and 18 Winzes) Johannesburg, South Africa by Tutu et al., (2008). Sulphuric acid was used to adjust the pH of the solution to 3.2. The adsorbents and metal solutions were mixed at g adsorbent/400 ml solution and agitated. Volumetric pipette was used to draw samples into 30 ml sample bottles. The samples were centrifuged at 4000 rpm for 10 min to facilitate solid/liquid separation before filtration using 47 mm diameter filter paper with 0.45 µm pore size. The samples were preserved with nitric acid and stored in a refrigerator awaiting analysis. pH meter was used to measure pH and ICP-OES was used for metal content analysis.

2.2.1. Effect of adsorbent dose on pH and metal removal efficiency

Table 1, shows experimental recipe which was followed. Adsorbent dosage was varied from 1 g, 3.5 g and 10 g/400 ml of feed solution having fixed concentration of 54 mg Al³⁺/l and 321 mg Mn²⁺/l and pH of 3.2. The study by Mohan and Gandhimathi (2009), concluded that an optimum dose of adsorbent and constant pH was observed at 240 min. The reaction time for this study was set at 240 min (4 h). After the reaction, has reached completion, samples were taken and analysed for metal content and pH was measured.

2.2.2. Effect of initial metal concentration

Initial metal concentration study was conducted by varying Al³⁺ concentration from 20, 35–54 mg/l and Mn²⁺ concentration from

100, 200–321 mg/l for 4 h. Aluminium and Manganese sulphates were dissolved in deionized water on magnetic stirrer to get a homogenous solution.

2.2.3. Effect of time

Optimum values from the previous tests (effect of dosage and initial concentration) were used for kinetic study in which known amount of adsorbent and initial concentration were fed to the reaction bottles and the reaction/agitation time was varied from 10 min to 70 min to investigate the effect of time on metal removal and pH.

2.3. Theory

2.3.1. Adsorption kinetics

To study the adsorption kinetics, the experimental data obtained was subjected to pseudo first (Eq. (2)) and second order (Eq. (3)) plots and Elovich equation plot (Eq. (4)).

The first order:

$$\log(q_e - q_t) = \log q_e - \frac{K_1}{2.303} t \tag{2}$$

where q_t (mg/g) is the amount of adsorbate adsorbed at time t (min), q_e (mg/g) adsorption capacity at equilibrium and K₁ is the rate constant of pseudo-first-order sorption. The slope of plot log (q_e - q_t) versus t will be used to calculate pseudo-first-order kinetic rate constant k₁.

The second-order kinetic model can be expressed as follows:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \tag{3}$$

where k₂ is the rate constant of pseudo-second-order adsorption (g/mg.min). The slope and intercept of plot t/q_t versus t will be used to calculate the pseudo-order rate constants k₂ and q_e.

Elovich equation is expressed as follows:

$$q_t = \beta \ln \alpha \beta + \beta \ln t \tag{4}$$

where β and α are Elovich constant.

3. Results and discussion

3.1. Effect of adsorbent on pH

To investigate the effect of adsorbent dosage on pH, different adsorbent doses (1 g, 3.5 g and 10 g/400 ml feed solution) were used with a maximum concentration of 54 mg Al³⁺/l-Al and 321 mg Mn²⁺/l while the reaction time was set at 4 h. The pH was measured using a pH meter. Table 2, present the obtained results. Addition of fly ash to both aluminium and manganese samples saw an increase in pH because typical fly ash contains compounds such as MgO and

Table 1

Adsorbent dosage experimental recipe.

$$\text{Optimum dose interpolation : } d = d_1 + \frac{f_2 - f_1}{f_2 - f_1} (d_2 - d_1) \tag{1}$$

d represent the desired value (Optimum dose in g_400 ml)
d₁ and d₂ are high and lower value with desired value in between
f₁ and f₂ are the closest approximation data points

Bottle	Fly Ash	Natural Clay	Activated Charcoal
1	1 g	1 g	1 g
2	3.5 g replicate	3.5 g replicate	3.5 g replicate
3	10 g	10 g	10 g

Table 2

pH of treated solution by the adsorbents.

Dosage (g)	Fly Ash	Bottom Ash	Bentonite Clay	Activated Charcoal
a. Aluminium samples				
1	4.7	4.9	6	7.0
3.5	7.3	6.2	7.0	7.3
10	8	6.2	7.4	7.8
b. Manganese samples				
1	4.6	4.1	3.8	4
3.5	7.5	7.6	5.7	4.8
10	11.5	5.6	5.9	5.6

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