



Synthesis of magnetite from iron-rich mine water using sodium carbonate



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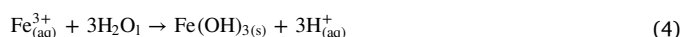
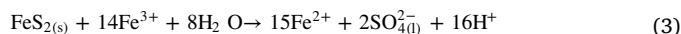
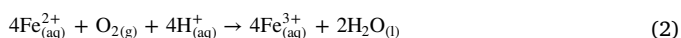
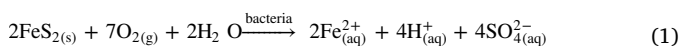
Magnetite nanoparticles
Acid mine drainage
Magnetite synthesis
Co-precipitation
Sodium carbonate
Fe-rich mine water

ABSTRACT

In recent decades, much research has been paying attention on the treatment and desalination of acid mine drainage (AMD) with minimal focus on the recovery of minerals from the resultant residues. Solid sludge produced after the treatment process is highly mineralised and technologies are required for the processing of the final sludge for possible industrial application. Conventionally, magnetite is synthesized using iron-rich, industrial grade chemical reagents making magnetite expensive to produce. This has urged the need to come-up with pragmatic and sustainable technologies of recovering magnetite from waste materials. This has led to the development of the present study which aimed at pre-treating AMD in such a way that magnetite could be generated from the recovered sludge. Synthesis of magnetite nanoparticles was evaluated at varying temperature gradients. The principal mechanism governing the metals recovery was selective precipitation. This was achieved by manipulating the pH and aeration rate of the reaction mixture. Experimental results revealed that optimum conditions that are suitable for the recovery of magnetite nanoparticles from AMD were 2:1 mol ratio of Fe(II)/Fe(III), pH ≥ 10 and temperature ranging from 25 to 100 °C. The purity of synthesized magnetite was 24 (Wt.%) for Al-removed magnetite and 28 (Wt.%) for magnetite synthesized with no Al-removal. Particle size analysis indicated the presence of magnetite nanoparticles having diameters of 0.5 nm. SEM-EDS and mapping revealed the presence of Fe and O on the matrices of synthesized material hence confirming that the recovered material is magnetite. This study successfully proved that magnetite nanoparticles can be synthesized from iron-rich mine drainage.

1. Introduction

Depending on the hydrogeology of the mine voids, there are four types of mine drainages, namely: acid, neutral, saline and basic mine drainage [1,2]. Out of those, Acid Mine Drainage (AMD) has been identified as an issue of prime concern due to the nature and magnitudes of its environmental impacts and footprints [3]. This has made AMD a paramount subject that needs urgent attention in international scientific communities [4]. Acid mine drainage is formed from hydro-weathering and oxidation of sulphide bearing minerals [5–7]. The formation of AMD can be explained by the following reactions [3,8,9]:



These reactions are also mediated by sulphate oxidising bacteria (SOB) (Eq.(1).) [2]. They lead to the formation of acidic mine water [10]. The acidic nature further solubilises chemical species in the host rocks adding them to the resultant effluent [11]. Acid mine drainage is primarily composed of H^{+} (aq), SO_4^{2-} (aq), Fe(II), Al(III) and Mn(II) as the major components [12]. The composition of AMD also includes heavy metal ions such as Cu, Ni, Zn, Co and Cr in addition to As [13] and alkaline earth metals such as Mg and Ca [11]. The presence of the heavy metal ions has prompted a need to treat AMD prior to discharge to protect aquatic ecosystems [14].

The treatment of AMD can either be active or passive. Active treatment involves addition of alkalis such as lime and limestone [15–17], magnesite [11], magnesite tailings [3], brucite [12], periclase

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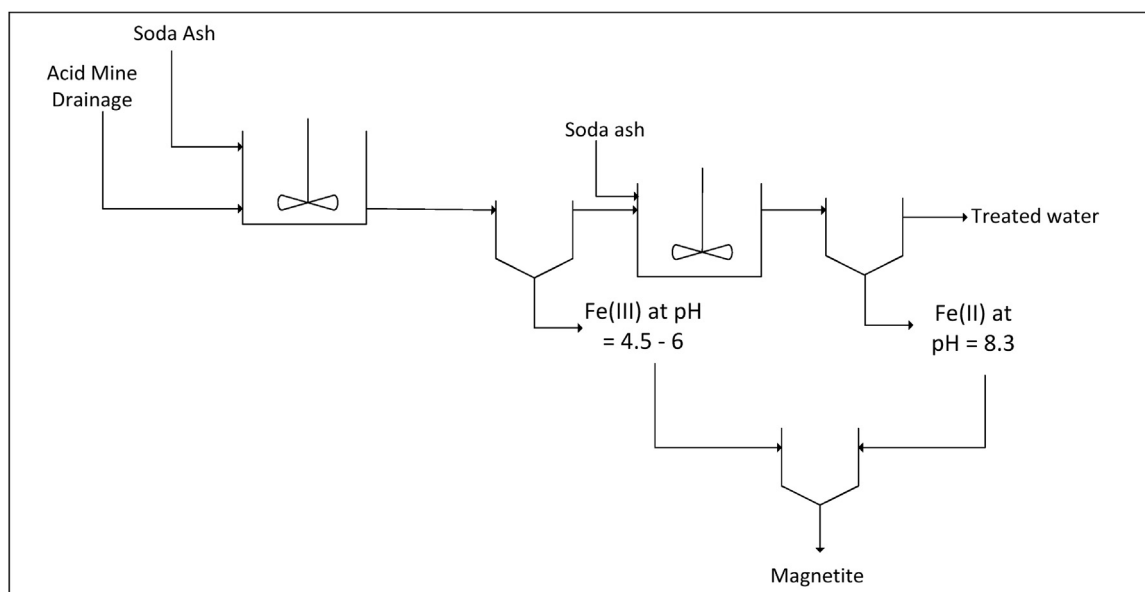
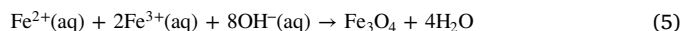


Fig. 1. Schematic presentation of magnetite synthesis.

[18], hydrated lime [7], sodium hydroxide [19] or sodium carbonate [20] to raise the pH of the AMD and precipitate majority of dissolved transition metal ions. From the documented literature, AMD contains elevated concentrations of Fe ions that are worth recovering [21]. Few researchers have recovered Fe from mine water for the synthesis of pigments and other industrial products [22,23]. Considering the quantity of AMD generated annually, the potential to recover iron products from AMD is very significant and feasible [24]. In South Africa, close to 360 ML/day of metalliferous mine water are discharged to the environment [3]. Research studies have used commercially available salts to synthesize magnetite [25–28] or a combination of commercial grade Fe-salts with AMD recovered Fe-salts [24].

Magnetite has numerous industrial applications that include: ferro-fluid technology [29], information storage [29], photo-degradation [30], photo-anode [30], catalyst [31], biomedicine [32], controlled drug delivery [33], wastewater treatment [25,34] and magnetic formation [35]. Also, there are many ways of synthesizing magnetite from iron-rich salts and these include: chemical precipitation, thermal decomposition, and sonochemical synthesis [24,25,30,35]. Amongst these methods, chemical precipitation is the most and widely used approach due to its versatility [25].

Chemical precipitation involves: (1) co-precipitation of Fe^{3+} and Fe^{2+} ; (2) oxidation of Fe^{2+} to Fe^{3+} and precipitation of Fe^{3+} ; (3) unaerated precipitation of Fe^{2+} , followed by (4) co-precipitation of the Fe(II) and Fe(III) species. This process happens under unaerated conditions. The most common salts for magnetite synthesis are ferrous and ferric chlorides or sulphates [25,30,35]. Kefeni, et al. [25] synthesized magnetite from $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, Valenzuela, et al. [33], Gnanaprakash, et al. [31], Mahadevan, et al. [32], and Nagarjuna, et al. [30] synthesized magnetite from $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, and $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$. Faiyas, et al. [36] and Stefan, et al. [35] synthesized magnetite from $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$, and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$. To date, there has been no published study that has reported the syntheses of magnetite solely from AMD, except a study that reported the use of ferric iron from AMD and ferrous iron from commercial grade salts [24]. In addition, most of the magnetite synthesis processes takes place at elevated pH and temperature (60–100 °C), where nitrates functioned as electron acceptors to form NO [24]. As anticipated in the present study, the formation of magnetite using co-precipitation of selectively recovered iron-salts can be represented by the following equation [33]:



This study was therefore designed with the aim of recovering Fe(II) and Fe(III) solely from Fe^{2+} rich mine water produced during coal mining and washing processes, and explore the potential feasibility of synthesizing magnetite.

2. Materials and methods

2.1. Sampling

Raw AMD generated from coal washing and mining processes in Mpumalanga Province, South Africa, was collected and sealed in high-density polyethylene (HDPE) plastic bottles. The solids and debris in the water samples were removed by settling and the remaining suspended solids were removed by filtration (0.45 μm perforated membrane filters). Samples were stored at 4 °C until use for magnetite recovery experiments. Sodium carbonate (AR Na_2CO_3 , > 99% purity, Lab Consumables) was used for fractional and sequential recovery of Fe(III) and Fe(II) from acid mine drainage.

2.2. Pre-treatment of AMD samples for magnetite synthesis

Chemical species were fractionally and selectively precipitated from mine water using soda ash. This was done to minimise interferences during formation of magnetite. The following approach was adopted to ensure a selective precipitation of Fe-species from AMD. Trivalent species of Fe(III) and Al(III) were precipitated in the first reactor at $\text{pH} > 4.5$. The sludge was recovered and stored safely for magnetite synthesis. The supernatant solution was stored in an air free environment and taken over the second reactor to recover Fe(II) at $\text{pH} > 8.3$. The sludge was also store in air free environment until utilization for magnetite synthesis.

2.2.1. Removal of Fe(III) and Al(III) by selective precipitation

A volume (180 L) of AMD was transferred into a reactor tank and the pH was adjusted from 2.8 to 4.5 by adding incremental portions 10% sodium carbonate solution to precipitate Al(III) and Fe(III) on the desired pH. The Fe(III)/Al(III) rich precipitate was separated from the pre-treated AMD by centrifugation. Thereafter, the Al(III) and Fe(III) free water was then used as feedstock for ferrous sludge production. The resultant water was analysed using ICP-MS (7500ce, Agilent,

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