



# Efficiency of ball milled South African bentonite clay for remediation of acid mine drainage



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## ABSTRACT

The feasibility of using vibratory ball milled South African bentonite clay for neutralization and attenuation of inorganic contaminants from acidic and metalliferous mine effluents has been evaluated. Treatment of acid mine drainage (AMD) with bentonite clay was done using batch laboratory assays. Parameters optimized included contact time, adsorbent dosage and adsorbate concentration. Ball milled bentonite clay was mixed with simulated AMD at specific solid: liquid (S/L) ratios and equilibrated on a table shaker. Contact of AMD with bentonite clay led to an increase in pH and a significant reduction in concentrations of metal species. At constant agitation time of 30 min, the pH increased with the increase in dosage of bentonite clay. Removal of  $Mn^{2+}$ ,  $Al^{3+}$ , and  $Fe^{3+}$  was greatest after 30 min of agitation. The adsorption affinity obeyed the sequence:  $SO_4^{2-}$  ( $221.8 \text{ mg g}^{-1}$ ) >  $Mn$  ( $30.7 \text{ mg g}^{-1}$ ) >  $Al$  ( $30.5 \text{ mg g}^{-1}$ ) >  $Fe$  ( $30.2 \text{ mg g}^{-1}$ ). The pH of reacted AMD ranged from  $\approx 3$  to 6. Bentonite clay showed high adsorption capacities for Al and Fe at concentration <500 mg/L, while the capacity for Mn was lower. Adsorption capacity for sulphate was >50%. Adsorption kinetics revealed that the suitable kinetic model describing data was pseudo-second-order hence confirming chemisorption. Adsorption isotherms indicated that removal of metals fitted the Langmuir adsorption isotherm for Fe and sulphate and the Freundlich adsorption isotherm for Al and Mn, respectively. Ball-milled bentonite clay showed an excellent capacity in neutralizing acidity and lowering the levels of inorganic contaminants in acidic mine effluents.

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## 1. Introduction

Exposure of sulphide bearing minerals to water and oxygen during and following mining activities accelerates the formation of acid mine drainage (AMD) which is very acidic and metalliferous. The acidity in AMD promotes the leaching of heavy metals from the surrounding geology. Due to its acidic nature and high loads of heavy metals, AMD has high electrical conductivity (EC) and total dissolved solids (TDS) concentration which are considerably above the recommended limits for effluents to be discharged into rivers. Thus mine effluent needs to be contained and managed prior to release

to the environment as an initial step in preventing environmental degradation [1–4].

Discharge of AMD to the environment can reduce the ability of any ecosystem to support life. It lowers water quality rendering it unfit for defined uses such as for domestic and agricultural purposes [5,6]. It also leads to iron sedimentation in aquatic ecosystems due to precipitation of iron hydroxide (yellow boy sludge). The suspended flocs also lead to reduced hunting abilities of fish due to poor visibility in water bodies [7]. Toxic chemical species in AMD pose hazards to terrestrial and aquatic organisms [8]. Acidity in water leads to the migration of certain species less tolerant to acidic conditions affecting the integrity of the ecosystem to support life due to reduced biodiversity [7,9–19]. This has emphasized the need to develop pragmatic solutions to treat AMD by neutralizing the acidity and removing heavy loads of dissolved metals and sulphate.

Due to their high surface areas, cation-exchange capacity (CEC), swelling ability, low cost, abundance and versatility, clays have

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received much attention for treatment of contaminated waters [20]. High surface area enables bentonite clay to efficiently scavenge chemical species from wastewater [21]. High cation-exchange capacity enables the clay to remove low-density chemical species such as Na, K, Ca and Mg from clay matrices through isomorphous substitution by high density poly-cationic species [22]. High swelling ability promotes high retention of polluted water within clay interlayers leading to high exchange capacities [23–35].

South Africa has large deposits of bentonite clay that is projected for extraction in the next 60 years, if the demand does not increase [36]. Gitari [37] has evaluated the ability of pestle-and-mortar milled bentonite clay for the attenuation of metal species concentrations in synthetic AMD and gold-mine tailings leachates. Low surface area, poor adsorption efficiencies and insignificant increases in pH were observed. Consequently, mortar-and-pestle milling was shown to be an inefficient milling procedure for clay minerals [38,39]. Literature reports show that ball-milling of clays leads to fragmentation, distortion, breakage of crystalline networks and cobwebs, and particle size reduction followed by an increase of the surface area, exfoliation of particles and amorphization [40–44]. Thus enhancing the contaminants removal abilities of ball milled bentonite clay [39,45–48]. The present study was designed to demonstrate the ability of ball-milled bentonite clay to neutralize and attenuate heavy metal concentrations in AMD. It also compared the efficiencies of the ball-milled clay to mortar-milled clay for surface area enhancement and metal species removal efficiency. Following a study conducted by Gitari [37], he recommended the testing of bentonite clay using AMD from different sources since he had used gold-mine tailings leachates and synthetic AMD. The present study also investigated some physicochemical properties of ball-milled clay before and after contact with gold-mine AMD.

## 2. Materials and methods

### 2.1. Sampling

Bentonite clay was supplied by ECCA Holdings (Pty) Ltd., Cape Bentonite mine (Cape Town, South Africa). Raw AMD was collected from a disused gold-mine shaft near Krugersdorp, Gauteng Province, South Africa.

### 2.2. Preparation of bentonite clay

The raw bentonite was washed by soaking in ultra-pure water and draining after 10 min. The ultrapure water used was such that it covered the entire sample in the beaker and was allowed to overflow. The procedure was repeated four times. The washed bentonite was dried at 105 °C for 24 h. The dried samples were milled into a fine powder (Retsch RS 200 ball-mill, Fritsch, Germany) and sieved (<32 µm particle size). The ball-mill was cleaned with uncontaminated quartz after milling each sample to avoid cross-contamination.

### 2.3. Simulated AMD

Synthetic acid mine drainage (SAMD) was used for experimentation as real AMD is extremely difficult to work with in optimization due to oxidation and hydrolysis on exposure to the open leading rapid changes in chemistry. A simplified solution containing the major ions found in AMD was prepared as described by Tutu, et al. [49].

The composition of SAMD used in this study is shown in Table 1. AMD was simulated by dissolving the following quantities of salts in 1000 mL of Milli-Q ultra-pure water (18 MΩ), 7.48 g Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>·H<sub>2</sub>O, 2.46 g Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>·18H<sub>2</sub>O, and 0.22 g MnCl<sub>2</sub> to give a solution of 2000 mg/L Fe<sup>3+</sup>, 200 mg/L Al<sup>3+</sup> and 100 mg/L Mn<sup>2+</sup> and 5 mL of

0.05 M H<sub>2</sub>SO<sub>4</sub> was added to make up the SO<sub>4</sub><sup>2-</sup> concentration to 6000 mg/L. The salts were dissolved in 1000 mL volumetric flasks. Prior to the addition of ferric sulphate, 5 mL of 0.05 M of H<sub>2</sub>SO<sub>4</sub> was added to ensure a pH <3, in order to prevent immediate precipitation of ferric hydroxide. For the batch experiments, the working solutions were prepared from these stock solutions by appropriate dilutions.

### 2.4. Characterization of aqueous solution

Total dissolved solids (TDS), pH, and electrical conductivity (EC) were monitored using a CRISON MM40 portable pH/EC/TDS/Temperature multimeter probe. Aqueous samples were analyzed using ICP-MS (7500ce, Agilent, Alpharetta, GA, USA) for metal cations and sulphate concentration was determined using ion-chromatography (IC; 850 Professional IC, Metrohm, Herisau, Switzerland). The accuracy of the analysis was checked by simultaneous analysis of water standards [National Institute of Standards and Technology, (NIST)].

### 2.5. Mineralogical, chemical and microstructural characterization

Mineralogical composition of bentonite clay and resulting solid residues was determined using XRD (Philips PW 1710 Diffractometer; graphite secondary monochromatic source). Elemental composition was determined using XRF (Thermo Fisher ARL-9400 XP+ Sequential XRF equipped with WinXRF software). Morphology was determined using SEM-EDS (JOEL JSM-840, Hitachi, Tokyo, Japan). Functional groups were determined using Perkin-Elmer Spectrum 100 Fourier Transform Infrared Spectrometer (FTIR) equipped with a Perkin-Elmer Precisely Universal Attenuated Total Reflectance (ATR) sampling accessory equipped with a diamond crystal. Surface area and porosity were determined using BET analysis (Micromeritics Tristar II, Norcross, GA, USA). The cation-exchange capacity of bentonite clay and AMD-bentonite clay complex was determined using the ammonium acetate method [36]. Point of zero charge was determined by solid addition method [37].

### 2.6. Experimental procedures

To determine the optimum conditions for AMD treatment with milled bentonite, the following operational parameters were optimized: shaking time, adsorbent dosage and adsorbate concentration. All experiments were performed in triplicate and the data averaged.

#### 2.6.1. Effect of time

Portions (100 mL) each of simulated AMD were pipetted into 250 mL flasks into which 1 g portions of bentonite clay were added. The mixtures were equilibrated for 1, 5, 10, 20, 30, 60, 180, and 360 min (Stuart reciprocating shaker, 250 rpm). After shaking, the mixtures were filtered (0.45 µm pore nitrocellulose filter membrane). The filtrates were preserved by adding two drops of concentrated HNO<sub>3</sub> to prevent aging and immediate precipitation of Al, Fe and Mn, and refrigerated at 4 °C prior to analysis by ICP-MS.

**Table 1**  
SAMD used in this study.

Salt dissolved	Species	Concentration (mg/L)
Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> ·18H <sub>2</sub> O	Al <sup>3+</sup>	200
Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> ·H <sub>2</sub> O	Fe <sup>3+</sup>	2000
MnCl <sub>2</sub>	Mn <sup>2+</sup>	100
H <sub>2</sub> SO <sub>4</sub> and Al and Fe salts	SO <sub>4</sub> <sup>2-</sup>	6000

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