

Application of Pulverized Kaolin Clay for Remediation of Acid Mine Drainage

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Abstract

This study reviewed the removal of heavy and trace metals by applying kaolin on coal acid mine drainage (AMD). The kaolin clay was characterized using X-ray fluorescence (XRF) and X-ray diffraction (XRD). The effect of pH, contact time, adsorbent dose and initial metal concentration were determined in a batch experiment. The major chemical components of the kaolin clay were discovered to be SiO₂ (49.69%) and Al₂O₃ (39.26%), hence the name aluminosilicate. The most important functional group of kaolin was O–H as an active site for binding positively charged cation. The removal efficiencies of the some of the metal cations increased by raising pH from 2 to 2.62. The most removal efficiency by using kaolin in contact time 45 minutes were 32.53% for SO₄²⁻, 26.10% for Cu, 12.50% for Ni, 84.17% for Zn and 12.38% for Mg. Therefore, this natural native adsorbent is able to remove SO₄²⁻, Cu, Ni, Zn and Mg from the authentic AMD.

Keywords: Heavy Metal Removal, Acid Mine Drainage, Adsorption, Pulverized Kaolin Clay, Process Optimization

Introduction

Previous studies revealed that the acid mine drainage (AMD) has become one of the emerging environmental concern globally (Anawar, 2013; Maree *et al.*, 2013; Simate and Ndlovu, 2014; Esmaili *et al.*, 2019). AMD is formed when the mining activities expose sulphur-bearing minerals (mainly iron sulphides) to air (i.e. atmospheric oxygen), moisture, and acidophilic iron-oxidizing bacteria, resulting in sulphuric acid, dissolved iron, and precipitation of ferric hydroxide (Idaszkin *et al.*, 2017; Masindi, 2017; Park *et al.*, 2019). This resultant sulphuric acid dissolves heavy metals from mined materials to form an acidic pH solution (<4) with elevated concentrations of arsenic, cadmium, lead, copper, and zinc. The study by Feris and Kotzé (2014), further states that the infiltration of AMD to the environment

will pollute both surface and ground water, further affecting the water security. Gaikwad (2010); Simate and Ndlovu (2014) further explained that AMD can originate from abandoned, derelict and defunct mines, and is a continuing by-product of current mining activities.

Several techniques including chemical precipitation (Ramírez-Paredes *et al.*, 2011), ion exchange (Ramírez-Paredes *et al.*, 2011), membrane separation (Park *et al.*, 2019), neutralisation (Sithole *et al.*, 2020), reverse osmosis (Gaikwad, 2010) and adsorption (Esmaili *et al.*, 2019b; Motsi *et al.*, 2009) are mostly used for remediation of heavy metals from AMD. Out of the listed above technologies, adsorption has been discovered to be the preferred technique due to its potential for the removal of metal ions, easy handling, availability of the different

adsorbents and the cost associated with procuring the adsorbent (Motsi *et al.* 2009).

Clay minerals such as bentonite and kaolin are said to have a small particle sizes and porous structure with a high surface area, which have high efficiency for adsorption and cation exchange capacity (Sadasivam and Thomas, 2016; Esmaeili *et al.*, 2019b). In this study, kaolin clay will be studied as a potential adsorbent for the remediation of heavy metal from AMD. Kaolin is characterised as a fine clay mineral cream to dark brown coloured by iron oxide/hydroxides (Sadasivam and Thomas, 2016).

To the authors' knowledge, the investigation of mechano-chemically synthesized kaolin clay as adsorbent for simultaneous neutralization and attenuation of metal species and sulphate has been limitedly reported before. The purpose of this study was to synthesize a kaolin clay adsorbent and evaluate its ability to neutralize acidity and remove metal species and sulphate from metalliferous mine drainage in a single process step.

Materials and methods

Sampling

Kaolinite was obtained from the CSIR. The samples were dry when received and they were in the particle size range of $\leq 32\text{nm}$. There was no additional pre-treatment which was done to the sample. The samples were used as received from the supplier. In addition to the kaolin clay, two 25 litre containers of coal AMD for these experiments were collected from a disused mine shaft near Witbank, Mpumalanga Province, South Africa.

Characterization of raw and treated acid mine drainage

The CRISON MM40 portable multimeter probe (manufactured in Spain by CRISON instruments) was used to monitor the pH, total dissolved solids (TDS) and electrical conductivity (EC). Anions such as sulphate was analysed using the Ion Chromatograph (850 professional IC Metrohm, South Africa) and photo spectrometer (spectroquad pharo model 100 Merck spectrometer, Johannesburg South Africa). The elemental analysis of the raw and treated AMD samples were analysed

through using the Inductively Coupled Plasma- Optical Emission Spectrometry (ICP-OES) (5800, Agilent, Alpheretta, GA, USA). The accuracy of the analysis was determined by utilizing the certified water standard reference materials (SRM 1643f) purchased from the National Institute of Standards and Technology (NIST), USA. The ICP-OES instrument was set by attaching a 213 nm laser ablation system connected to an Agilent 5800 ICP-OES. Triplicate measurements were taken for each sample analysed. For every 12 runs, the calibration standards were run for quality control. A quality control standard was also analysed at the beginning of the run together with the calibration standards throughout. Both CRMs used for this purpose were basaltic glass types (BCR-2 and BHVO 2G) and were certified reference standards from USGS. To verify the ablation on fused material, a fusion control standard from certified basaltic reference material (BCR-2) was analysed at the beginning of run. The data was processed using Glitter software.

Characterization of kaolin clay material

X-ray diffraction spectroscopy (XRD) analysis

For this study, the powder X-Ray Diffraction patterns (XRD) PANalytical X'Pert through the Cu-K α radiation with an X'Celerator detector (strip detector) was utilized to measure the mineral composition of the kaolin clay material. This X'Pert was capable of measuring both high and lower resolutions as well as enabling a wide range of thin film and powder sample analysis. The XRD patterns were carried out at high angles ($2\theta \approx 10 - 90^\circ$) through a step size and scan speed of 0.02° and 0.03° per second, respectively. A back loading preparatory method were used for the preparation of the feed and product materials. The phases and the relative phase amounts (mass %) were determined using Malvern PANalytical X'Pert Highscore (Plus) version 5.1 software and the Rietveld quantitative analysis, respectively.

X-ray Fluorescence spectroscopy (XRF) analysis

The analysis was done using the Thermo Fisher ARL9400 XP+ Sequential XRF with



WinXRF software. For each batch of samples, the blank and certified references materials were analysed. This was done to observe the accuracy of the instrument via quality assurance and quality control procedures.

Scanning Electron Microscopy analysis

To obtain the morphologies as well as the size distribution of the kaolin material, the high-vacuum scanning electron micrographs (SEM images) were collected through a JEOL JSM7500 microscope. To disperse the kaolin samples, a carbon tape and sputter-coated with a thin conductive layer of gold using an Emitech K950X sputter coater were used. Energy Dispersive X-Ray Analysis (EDX) also known as the EDS was utilized to determine the elemental composition of kaolin clay material. This EDX system utilized was combined to a SEM instrument (FIB-SEM, Auriga from Carl Zeiss, Germany) where the imaging capability of the microscope determined the specimen of interest.

Experimental procedures

To configure the optimum condition for AMD treatment, numerous operational parameters including the effect of contact time, kaolin clay dosage and pollutant concentration were optimized.

Effect of agitation time

An aliquots of 500 mL of acid mine drainage each were pipetted into 1 000 mL flasks to which 5 g of the kaolin clay samples were added. The solutions were equilibrated for 5, 10, 15, 30, 45 and 60 minutes at 300 rpm through a Stuart reciprocating shaker. After equilibration, the solutions were filtered using a 0.45 µm pore nitrate cellulose filter membrane. The filtered solutions were preserved to prevent aging and immediate precipitation on Al, Fe and Mn. This was done by adding two drops of concentrated HNO₃ acid and refrigerating the solution at 4°C before analysing with an ELAN 6000 inductively coupled plasma Optical Emission Spectrometry (ICP-OES) (5800, Agilent, Alpheretta, GA, USA). The pH prior and post agitation was determined using the CRISON multimeter probe (model MM40). A separate solution was left un-acidified for

sulphates analysis by Ion Chromatograph (850 professional IC Metrohm, Herisau, Switzerland).

Effect of dosage

An aliquots of 500 mL of acid mine drainage each were pipetted into 1 000 mL flasks and different masses (0.5 g, 1.0 g, 2.5 g, 5 g, 10 g, and 15 g) of kaolin clay were added to each flask. The solution was agitated for an optimum time of 45 minutes at 300 rpm through a shaker. After shaking, the pH, metal and sulphates concentrations were analysed as highlighted in the earlier section.

Treatment of coal AMD under optimized conditions

The coal AMD samples were treated at established optimized conditions in order to assess the efficiency of pulverized kaolin clay for the remediation of AMD. The pH and metal concentrations were analyzed as discussed in the earlier section whereas a separate solution was un-acidified for SO₄²⁻ analysis through Ion Chromatography. Metals concentrations were determined through the ICP-OES. Alkalinity and acidity determinations were analyzed as per the procedures described in Standard Methods (APHA, 1989) whereas, pH, EC and TDS were analyzed through the CRISON MM40 multimeter probe. The resultant solid residue post AMD treatment were analyzed to understand the fates of chemical species after treatment.

Calculation of % removal capacity

The Equation (1) below shows the computation of % removal capacity.

$$(\% \text{ removal}) = \left(\frac{C_o - C_e}{C_o} \right) \times 100 \quad (1)$$

Where: C_o = initial concentration, C_e = equilibrium ion concentration and V = volume of solution.

Results and discussion

X-ray fluorescence (XRF) analysis

The elemental composition of kaolin clay and AMD-Kaolin clay are depicted in Table 1.

Table 1 Elemental composition of kaolinite clay and AMD-Kaolinite clay

% Composition	Kaolin clay	AMD-kaolin clay
SiO ₂	49.69	48.19
Al ₂ O ₃	39.26	38
Fe ₂ O ₃	1.32	2.94
MgO	0.715	0.59
P ₂ O ₅	0.0955	0.0798
MnO	0.0121	0.0282
TiO ₂	1.52	1.47
CaO	1.89	1.11
V ₂ O ₅	0.0148	0.0166
K ₂ O	0.413	0.392
SO ₃	0.0229	0.0146
Cr ₂ O ₃	0.065	0.0575
NiO	0.0161	0.0129
ZrO ₂	0.279	0.238
ZnO	0.0125	0.0148
SrO	0.0284	0.0244
Y ₂ O ₃	0.0315	0.0293
LOI	4.4981	6.71391
Total	99.933	99.964

X-ray fluorescence analysis was conducted to determine the chemical composition of both kaolin and AMD treated with kaolin clay samples and subsequent alterations in chemical composition as a result of thermal treatment as shown in Table 1. The kaolin clay is predominantly comprised of Al and Si suggesting that the kaolin is an aluminosilicate. The presence of Fe and sulphur demonstrate the possible adsorption of Fe and S during geological deposition. Kaolin clay material contained 39.26 wt% alumina and 49.69 wt% silica with SiO₂/Al₂O₃ ratio of 1.79. This ratio was slightly decreased to 1.78 for the treated AMD with kaolin sample which confirmed a slight decline in silica content in the AMD kaolin treated samples. Decreasing the silica to alumina ratio will slightly decrease the surface negativity and as a result decreases the adsorption capacity. The presence of the MgO, CaO and K₂O highlight

that they are the main exchangeable cations in the kaolin clay matrices. The presence of base cations on the kaolin clay matrices shows that the kaolin has a potential to neutralise acidic solutions (Equation 4).



The AMD-reacted kaolin clay residues results indicated a raise in the concentrations of Fe and Mn indicating the adsorption and retention/withholding of inorganic contaminants from AMD by kaolin clay. A reduction of Al, Mg, Ti, Ca, K and SO₃ indicate that polycations of Fe and Mn are exchanged by those polycations through isomorphous substitution. This corroborates the ICP-OES results (Table 2) which demonstrated a significant increase in concentrations of Fe, Mn, Cr, Al, Ca partially Mg in the product water.

X-ray diffraction (XRD) analysis

The mineralogical composition of kaolin clay is presented in Figure 1.

Mullite, quartz, hematite, kaolinite, cristobalite, rutile and calcite were discovered to be the mineral phases present in kaolin clay matrices. Mineral phases present in AMD-reacted kaolin clay is further highlighted in Figure 2.

The AMD-reacted kaolin clay was found to contain mullite, quartz, hematite, basanite, cristobalite, rutile and kaolinite. This indicates that there was mineralogical transformation during the treatment of AMD. The introduction of bassanite to resultant solid residue indicates the adsorption of SO₄²⁻ from AMD and an increase in Ca indicate the adsorption of Ca from AMD.

Scanning Electron Microscopy (SEM) – Electron Dispersion Spectrometry (EDS)

The SEM-EDS of raw and reacted kaolin clay is highlighted in Figure 3 and 4 respectively.

The SEM micrographs highlighted the occurrence of spherical agglomerates on the kaolin clay matrices at 25 μm magnification. The SEM-EDS results are concurring with the XRF results indicating Al and Si as the major elements. High concentrations of base cations such as Mg, Ca and K were also detected in the raw kaolin sample. Fe, Ti and Cr was also

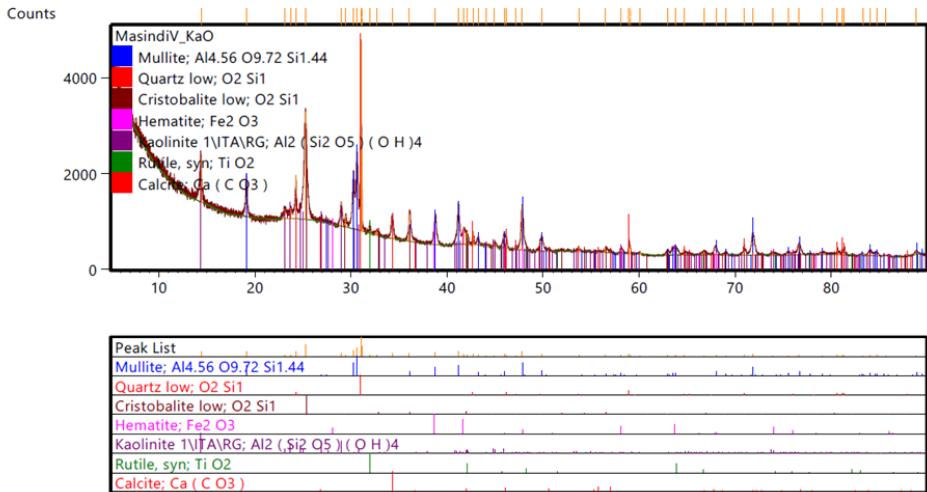


Figure 1 Mineralogical composition of kaolin clay

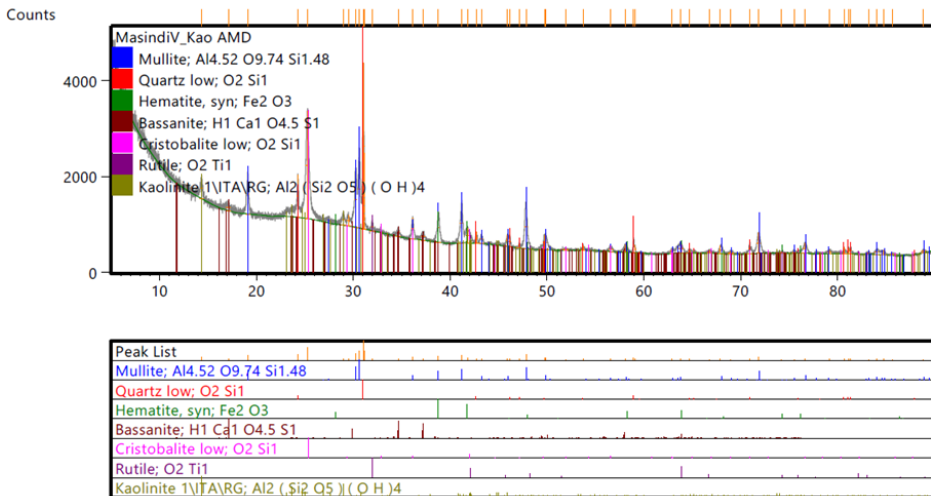


Figure 2 Mineralogical composition of AMD-reacted kaolin clay

observed to be present on the feedstock; this might have been incorporated to clay during depositions and weathering processes. S and O were noted to be present on the kaolin matrices and this may be introduced to clay surfaces over hydrolysis and decomposition of organic compounds.

From Figure 4, the SEM micrographs presented minor modification in the kaolin clay morphology since the spherical agglomerates are still unbroken suggesting that the reaction of kaolin clay and AMD did not affect the mechanical structure of kaolin clay. Elemental composition indicate that

the resultant residues are predominantly characterised by Si, O and Al hence the name alumino-silicate. Fe was also observed to be present, this may be due to low ion exchange for kaolin and adsorption as a result of slight pH increases. This indicates that kaolin clay increased the Fe concentration and can be further substantiated by a substantial Fe increase in the treated water, proving that Fe has been precipitated by kaolin clay. The drastic decrease in concentration of Mg, Cr and K on the secondary residue (Figure 4) and a slight decrease in the treated AMD (Table 2) demonstrated a relative a low

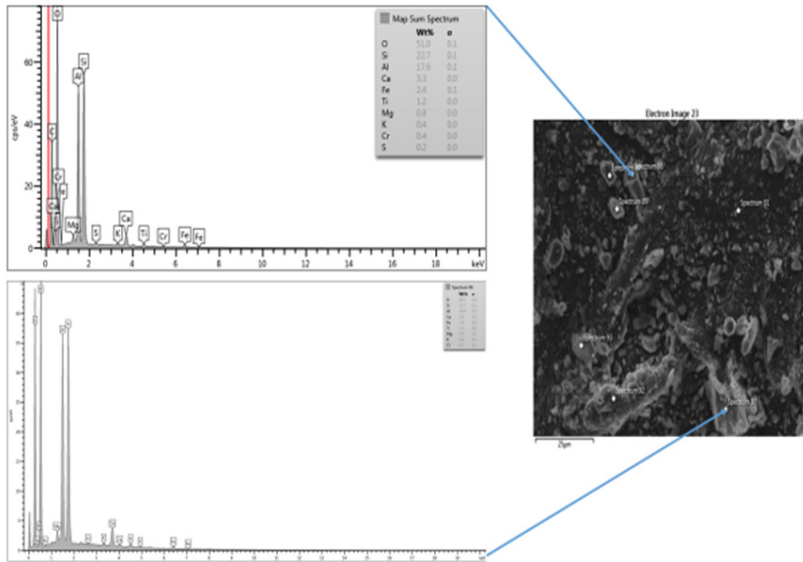


Figure 3 Morphology and elemental composition of raw kaolin clay

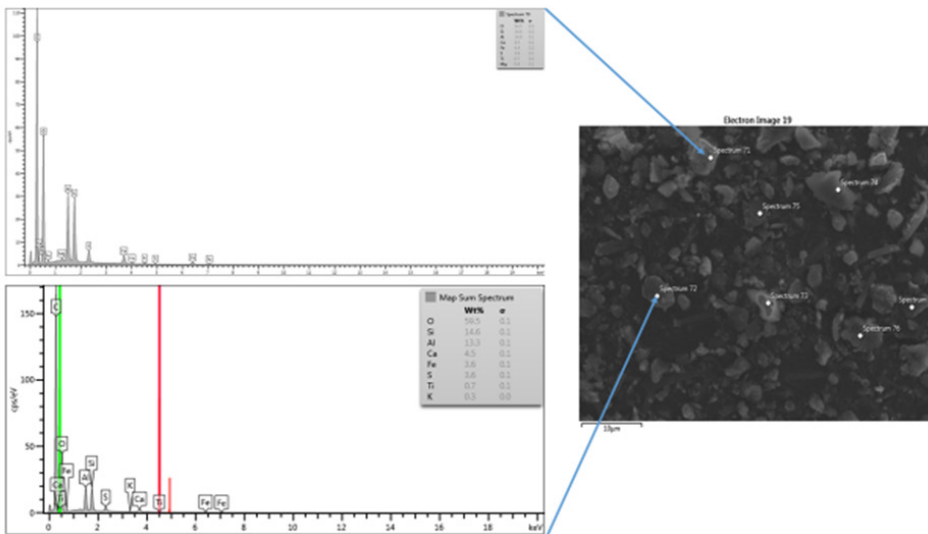


Figure 4 Morphology and elemental composition of reacted kaolin clay

exchanged cation. Ca and Ti was found to raise in the resultant residues indicating the presence of calcite. Sulphur was also found to have risen in the resultant residues suggesting that the kaolin clay mineral is a sink of sulphur from AMD.

Batch experiments

Results for optimization of adsorption and metals attenuation conditions are presented in Figure 5 – 8 respectively.

Effect of agitation time

Variations in the concentrations of Fe, Al, Mn, SO_4^{2-} and Zn with an increased agitation time is depicted in Figure 5a, whereas the variations in concentrations of Ca, Mg and conductivity levels with an increased agitation time is depicted in Figure 5b.

As depicted in Figure 5a and 5b, it can be seen that there was a raise in pH level with an increase in agitation time. From 1 – 45 minutes of agitation, the pH of the

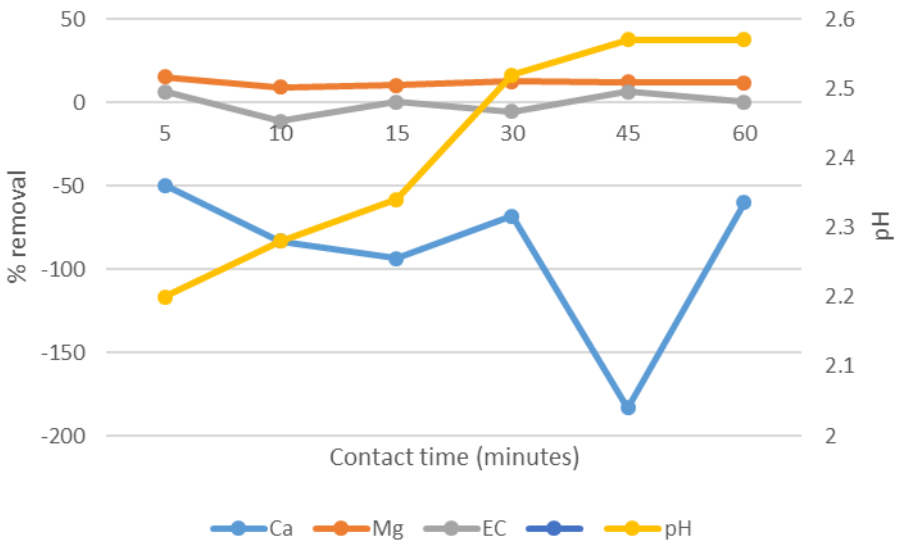


Figure 5a Effect of agitation time on adsorption and removal of Al, Fe, Mn, Zn and Sulphate (5g kaolin clay, 32 μm , 300 rpm shaking, 25 $^{\circ}\text{C}$).

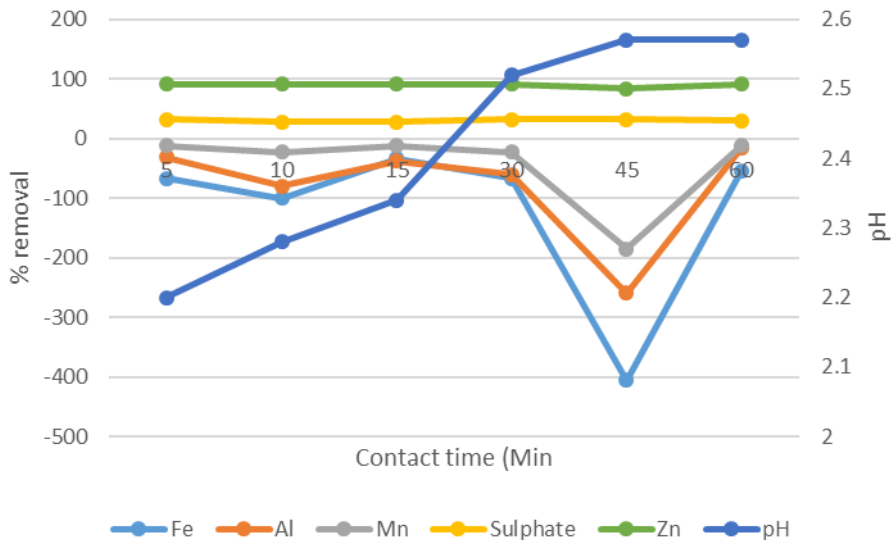


Figure 5b Effect of agitation time on adsorption and removal of Ca, Mg and EC (5g kaolin clay, 32 μm , 300 rpm shaking, 25 $^{\circ}\text{C}$).

solution was found to have raised from 2.2 to > 2.57. Subsequently, no substantial pH change was observed. The concentration of Zn was noticed to decline with an increase in agitation time. After 45 minutes, > 84% of Zn was removed from the ADM solution. This result is in agreement with the study by Ali *et al.*, (2020); which demonstrated that kaolin

can be utilised as an alternative adsorbent to remove Zn from AMD. With regards to SO_4^{2-} removal, it was also noticed that its concentration decrease with an increase in agitation time. More than 28% of SO_4^{2-} was removed prior to 15 minutes of agitation and subsequently, no substantial removal in SO_4^{2-} was noticed. Ca, Mn, Fe, and Al removals

were noticed to decrease with an increase in agitation time. Above 100% concentration of Ca, Mn, Fe and Al was introduced with an increase in agitation time.

Effect of pulverized kaolin clay dosage

Variations of Fe, Al, Mn, SO_4^{2-} and Zn concentrations in relation to an increase in kaolin dosage is depicted in Figure 6a, whereas the variations of Ca, Mg and conductivity levels with an increase in kaolin dosage is depicted in Figure 6b.

The effect of kaolin dosage for the attenuation of inorganic contaminants in AMD was determined at 45 minutes of agitation. Figure 6a and 6b depicted a slight increase in pH of the AMD in relation to an increase in kaolin clay dosage. The pH increased from 2.2 – 2.62 making it appropriate for attenuation of some contaminants in AMD. The insignificant pH increase may be due to the calcite dissolution in kaolin clay matrices as revealed by XRD. The release of base earth alkali metals owing to isomorphous

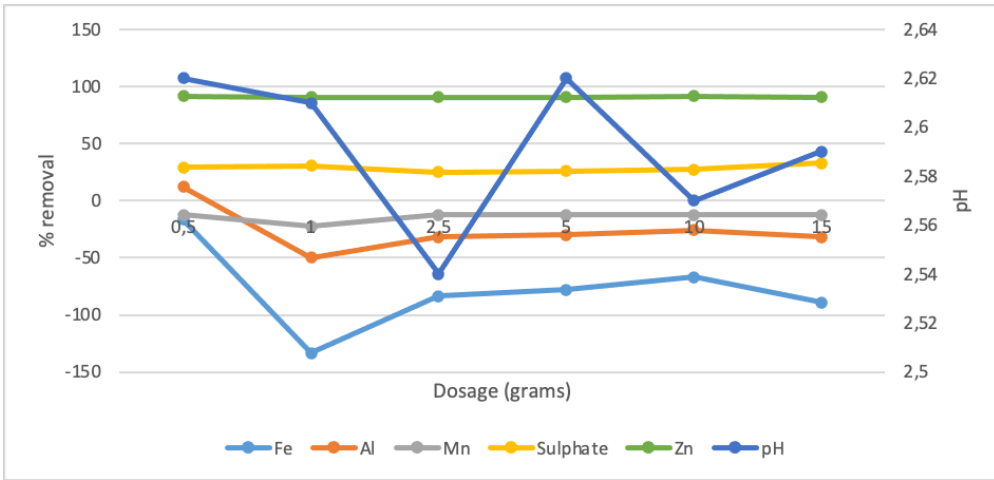


Figure 6a Effect of kaolin dosage on adsorption and removal of Al, Fe, Mn, Zn and Sulphate (AMD, 32 μm , 45 mins, 300 rpm shaking, 25 °C).

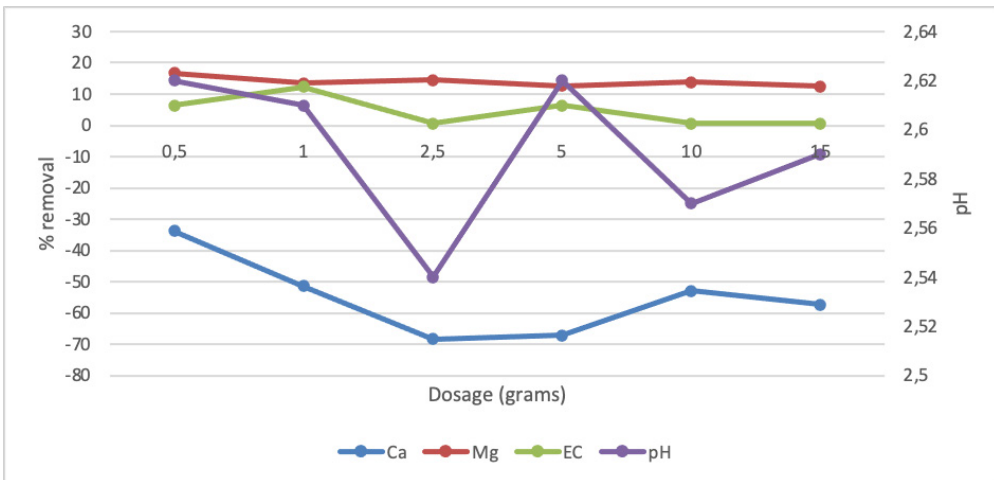


Figure 6a Effect of kaolin dosage on adsorption and removal of Ca, Mg and EC (AMD, 32 μm , 45 mins, 300 rpm shaking, 25 °C).



substitution by polymeric species of Al^{3+} and Fe^{3+} may also add to an increase in pH of the supernatants. The removal effectiveness of metals species was observed to increase with an increase in kaolin clay dosage. The adsorption affinity at 5 g was found to be $\text{Zn} > \text{SO}_4^{2-} > \text{Mg} > \text{Mn} > \text{Al} > \text{Ca} > \text{Fe}$. Therefore, 5 grams was considered the optimum dosage for the attenuation of contaminants in AMD.

Treatment of AMD under optimised conditions

The physicochemical composition of raw and kaolin clay treated acid mine drainage is shown in Table 2.

Table 2 highlights that the AMD under the study is predominately characterised by a very acidic pH (<2) together with high loads of electrical conductivity (EC). This is mainly as a result of high concentrations of Fe, Al, Mn, Ca, Mg and SO_4^{2-} . Cu, Zn, Pb, Ni, and Cr are also available traces. The existence of Fe suggests that the mine effluent was formed from weathering of pyrite. The concentrations of SO_4^{2-} , Cu, Ni, Pb and Zn were noticed to have slightly decreased post AMD treatment. Base cations were also available in solution at elevated concentration suggesting that as the metal species are adsorbed onto clay surfaces, base cations are replaced through cation exchange. Ca was observed to have

increased after the AMD treatment suggesting the possible adsorption of calcite (Figure 1) as shown by XRD. Trace metals of Cu, Cr, Pb, Zn and Ni were also observed to decrease slightly. This may be defined by exchange of low density cations from clay matrices in exchange of those chemical species. Even though some of the chemical have been slightly reduced using the kaolin clay, it can be seen that further improvement on the material is still required to meet the water quality requirements to ensure compliance to stringent regulations that governs water quality.

Conclusions

The utilization of pulverized kaolin clay for neutralisation and removal of heavy metals from coal AMD has shown good performances on certain water quality parameters (Zn, Cu, Pb, Cr and SO_4^{2-}) in reclaiming polluted water. XRF showed the material used in the study is alumina-silicate due to the predominant of Al and Si minerals. SEM micrograph further revealed no alteration to the morphology of kaolin clay after reacting with AMD. The reaction of AMD with the pulverized kaolin clay resulted in a slight raise in pH and slight reduction in major metals concentrations. EC was observed to increase post AMD treatment with kaolin clay hence indication possible

Table 2 Physicochemical composition of raw and kaolin clay treated acid mine drainage

Parameter	Units	Feed water		Kaolin treated AMD
SO_4^{2-}	mg/L	15000	0 – 500	10656
Fe	mg/L	1800		3500
Mn	mg/L	98	0 – 0.05	122
Cr	mg/L	0.09	0.01	0.22
Cu	mg/L	0.59	0 – 1	0.3
Ni	mg/L	1.6	0 - 0.07	1
Pb	mg/L	0.2	0 – 0.01	0.09
pH @ 25°C	pH units	2	6 – 10	2.6
Al	mg/L	500	0 – 0.9	1800
Electrical Conductivity @ 25°C	mS/m	1709	0 – 700	1999
Zn	mg/L	120	0 – 0.5	10
Ca	mg/L	495	0 – 32	1200
Mg	mg/L	622	0 - 30	501

release of base cations to aqueous solution through ion exchange processes. Optimum condition of using pulverized kaolin clay was observed to be 5 g of adsorbent dosage and 45 minutes of equilibration. Notwithstanding that some major cations were slightly removed from AMD, a further refinement on the material is required to further remove the major cations to achieve drinkable standards.

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