Characterisation of a Rehabilitated Tailings Dam Mine Water as a Potential Acid Mine Drainage and Removal of Heavy Metals Using Locally Produced Activated Carbon*

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Abstract

Mine waters can be acidic or circumneutral and may often contain concentrations of heavy metals, which are produced from mining base and precious metals. In this work, reddish-brown mine water from an old, rehabilitated tailings storage facility (OTSF) of a mine in Ghana was investigated in terms of physical and chemical characteristics. GARD guide and Hill geochemical classification frameworks (GCF) were also used to ascertain the characteristics of the OTSF mine water in terms of acid mine drainage (AMD). The potential for using locally produced Palm Kernel Shell (PKS) activated carbon for heavy metal adsorption in OTSF mine water was also investigated. The kinetics of the adsorption process were tested using pseudo-first-order (PFO) and second-order (PSO) kinetic models. The results showed that the physicochemical parameters of the OTSF mine water measured were all below the Ghana Standard Authority (GSA) effluent discharge limit for general mining except pH = 5.00-5.40. In terms of heavy metals, except As and Cd, Mn ($0.75 > 0.2$ mg/L GSA), Fe ($25.2 > 10$ mg/L GSA), and Pb ($0.31 > 0.1$ mg/L GSA) were all above GSA effluent discharge limit. The OTSF mine water had low chloride but a high sulphate concentration ($395 > 250$ mg/L GSA). The drainage, which measured Eh of 460 mV, showed a moderate oxidising environment of OTSF mine water. The GCF revealed that the OTSF mine water is between partially oxidised to low neutralised AMD with potentially less toxic constituents. Locally produced PKS-activated carbon reduced the concentration of As, Pb, Fe, and Mn of the OTSF mine water below the GSA effluent discharge limit. The adsorption kinetics of potential constituents like Fe and Mn fitted well to PFO and PSO, indicating adsorption as diffusion controlled and chemisorption, respectively. This work has demonstrated the potential of using locally produced PKS-activated carbon as an excellent and cost-effective adsorbent for the treatment of heavy metals from the OTSF mine water.

Keywords: Geochemical Classification, Mine Water, Oxidation, Adsorption, Kinetic Models

1 Introduction

Surface mining and mineral processing generate significant amounts of waste with the potential to pose long-term environmental risks if improperly managed. Major environmental impacts of mining waste include loss of land, followed by its disposal into a tailings storage facility or the waste rock storage area and its generation of acidic waters and other contaminated sediments into the local environment (Fungaro and Izidoro, 2006; Galhardi and Bonotto, 2016). The composition of an ore-bearing rock and the method of processing the ore may contribute to the production of acidic effluent or runoff in a mine (Ziemkiewicz *et al.,* 2003). For instance, a significant concentration of sulphide minerals may be present in waste rock, gangue minerals, and tailings of some gold and base metal extractive industries. Exposure of these sulphide-containing wastes to atmospheric conditions could produce acidic mine waters. Mine waters or drainage may be acidic (pH 0 – 5), circumneutral (pH 6 – 8), or strongly basic (pH 8 – 12) with some concentrations of heavy metals (Nordstrom, 2011).

When iron sulphide-bearing mineral rock, such as pyrite (FeS_2), is exposed to an oxygen-rich environment and water, AMD is created (Warhurst and Noronha, 2000; Akcil and Koldas, 2006; Simate and Ndlovu, 2014). However, mining can accelerate AMD formation by simply increasing the amount of sulphide exposure during the extraction process, even though this process can happen naturally (Akcil and Koldas, 2006). Additionally, oxidising bacteria, particularly acidophiles like acidic *thiobacillus*, contribute to this oxidative weathering process by acting as catalysts to speed up pyrite oxidation at low pH levels (Nordstrom and Southam, 1997; Moodley *et al.*, 2018). Ferrous iron (Fe^{2+}), sulphate ion (SO_4^{2-}) and proton (H^+ acid) are produced in solution when iron sulphide reacts with oxygen and water (Jennings *et al.,* 2008; Simate and Ndlov, 2014). When pH is above 3, through contact with fresh water or neutralising minerals, the SO_4^{2-} oxidation yields sulphuric acid, and the Fe^{2+} is further oxidised to ferric iron (Fe^{3+}), which forms the yellow-orange ferric hydroxide precipitate ($Fe(OH)_3$) (Evangelou, 1995). Eq.1 (Evangelou, 1995; Johnson and Hallberg, 2005) illustrates the

condensed equation frequently used to represent the entire process of AMD:

$$4FeS_2 + 15O_2 + 14H_2O \longrightarrow 4Fe(OH)_3 + 8SO_4^{2-} + 16H^+ \qquad (1)$$
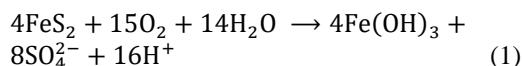
Low pH, high metal concentrations (including iron (Fe), aluminium (Al), manganese (Mn), cadmium (Cd), copper (Cu), lead (Pb), zinc (Zn), mercury (Hg) and metalloid arsenic (As)), high salinity or conductivity, and high SO_4^{2-} ion concentration in mine drainage are features related to AMD formation (Johnson and Hallberg, 2005; Méndez-Ortiz et al., 2007; Moodley et al., 2018). Water with a pH close to neutral has been referred to as "mine drainage" by researchers (Skousen et al., 1998; Mayes et al., 2009; McCarthy, 2011; Parbhakar-Fox and Lottermoser, 2015; Moodely et al., 2018). Poorly built waste rock dumps or tailing storage facilities may release acidic runoff into the environment, contaminating soils, sediments, groundwater, and surface waters (Bain et al., 2000; Armienta et al., 2001; Dong et al., 2018; Skousen et al., 2019). One of the major challenges of mine drainage in the form of AMD is the treatment. If the formation of AMD is improperly managed, environmental deterioration can be hastened (Daraz et al., 2022). According to Nordstrom (2011), mining must plan for the prevention and remediation of these contaminant discharges using hydrogeochemical principles and available technologies. Active and passive treatment technologies are considered the best methods for remediating AMD lately (Johnson and Hallberg, 2005; Akcil and Koldas, 2006; Daraz et al., 2022). Active abiotic treatment involves the use of chemical-neutralising agents like limestone ($CaCO_3$), slaked lime ($Ca(OH)_2$), caustic soda (NaOH), soda ash (Na_2CO_3), quick lime (CaO) and magnesium oxide (MgO) to neutralise or increase pH and precipitate toxic metals from AMD (Coulton et al., 2003; Johnson and Hallberg, 2005; Masindi et al., 2017; Daraz et al., 2022). Passive abiotic treatment involves the use of anoxic limestone drains ("ALD" Kleinmann et al., 1998). The ALD method adds alkali to AMD while keeping Fe in its reduced form to avoid the oxidation of Fe^{2+} and precipitation of $Fe(OH)_3$ on the limestone, which can critically reduce the efficacy of the neutralising agent (Johnson and Hallberg, 2005).

Apart from the chemical usage, activated carbon, clay minerals, different membranes, and other synthetic compounds or adsorbents have been used to remove heavy metals from AMD (Lopez et al., 2018; Daraz et al., 2022). Furthermore, Buah et al. (2016) used locally produced activated carbons to remove metal(loid) like Fe, Mn, Pb, and As from mine wastewater.

An old tailings storage facility (OTSF) has been decommissioned and rehabilitated by a gold mining business in Ghana's Western Region for twenty years now (2002 to date). A conduit around the OTSF, presented in Fig. 1, has been producing reddish-brown mine water for some time now, suspected to be AMD coming from the tailings material that was kept in the TSF over the years. Therefore, this work seeks to: (1) characterise the reddish-brown mine water from the OTSF of the gold mine in terms of physicochemical properties to ascertain its level of compliance with the Ghana Standard Authority (GSA) effluent discharge limit for general mining; (2) characterise the reddish brown mine water in terms of AMD features using established geochemical classification frameworks thus the Global Acid Mine Drainage (GARD) guide by Opitz and Timms (2016) and Hill (1968), and (3) treat the mine water by removing some specific heavy metals using locally produced activated carbon.

2 Materials and Methods

2.1 Sampling and Sample Preparation

Samples of the reddish-brown mine water from the rehabilitated OTSF were collected using cleaned sample bottles from the mine. The sample location of effluent from OTSF drainage from the mine is presented in Fig. 2. During sampling, sample bottles (six 1000 mL plastic bottles each) were submerged and tilted against the flow of the effluent at a depth of 0.25 m and the container was slowly lifted towards the flow following the manual grab and standard sampling procedures.

Fig. 1 Conduit Producing Reddish-Brown Effluent Suspected to be AMD

Fig. 2 Sampling Location of Effluent from OTSF Drainage from the Mine

Each container was filled to about 4/5 full. The sample containers were capped or covered, labelled, and placed in an ice chest containing sufficient ice to prevent contaminants' breakdown during transportation. Samples were transported to the Environmental Monitoring Laboratory (EML) of the University of Mines and Technology (UMaT), Tarkwa-Ghana.

2.2   Sampling Analysis

Prior to the laboratory analysis, in-situ parameters (pH, temperature, dissolved oxygen (DO), oxidation-reduction potential (ORP), and electrical conductivity (EC)) were determined using an Aquaprobe AP-2000 multi-parameter water quality probe and associated aquameter. Samples were well shaken and divided into two portions each at the water and wastewater lab of EML at UMaT. One portion of each sample was used to determine physicochemical parameters following the Ghana Standards Authority (GSA, 2019) environmental protection standard procedures and protocols. As a result, Total Dissolved Solids (TDS), Total Suspended Solids (TSS), EC, DO, temperature, pH, and ORP were measured with calibrated photometers and specific electrodes. For the TSS determination, a sample was poured into a 10 mL vial and placed in the HydroTest (HT 1000) multi-parameter photometer. Also, for the TDS, a sample was poured into a 10 mL vial and then placed in a HydroCheck (HC 1000) photometer. For zero blank, deionised water was put into the 10 mL vial for zero determination in each analysis.

The sample's ORP and pH were measured using combined platinum electrodes and glass electrodes (Aquaprobe AP-2000 meter), respectively. The final values were recorded after the digital panel displayed a final stable value. The ORP electrodes were calibrated using +250 mV ORP calibration standard (Reagecon RS250 Redox Standard). The pH meter was calibrated using a buffer solution (pH 4.2 and 7). The ORP reference electrode used in Aquaread® combination electrodes is a 3M PK1 silver chloride type, which exhibits potential on the hydrogen scale. Therefore, *Eh* (that is, ORP referenced to standard hydrogen potential SHE) was recalculated using Eq. 2.

$$Eh_{SHE} = Eh_{measured} + 207 - 0.7\,(T - 25^oC)$$
$$(2)$$

At 25 ℃, the potential is represented by the value 207, and the correction factor, which is 0.7, is a function of the *Eh* acquisition at temperature *T* (Nordstrom and Wilde, 1998).

The DO of samples was measured using the galvanic DO (Aquaprobe AP-2000 meter) electrode consisting of an electrolyte-filled DO sensor cap with a clear, gas-permeable membrane stretched across a silver electrode. The DO electrode was calibrated at the Zero saturation point. Before the DO analysis on the sample, the 100% saturation point was checked in moist air. For optimum accuracy, the DO100% point was calibrated close to the sample temperature. The EC of samples was also measured using EC Aquaprobe AP-2000. The EC probe was calibrated using the two pre-set calibration standards (using Aquared® RapidCal (EC value 2570 µS/cm)). Both the DO and the EC values were also recorded in a similar fashion, just like the pH and ORP measurements.

The following major anions (SO_4^{2-} and chloride (Cl^-)) concerning mine drainage effluent were also measured from the samples following GSA (2019)

environmental protection standard procedures and protocols. The HydroTest HT1000 multi-parameter photometer was also used for anion analysis. To determine these anions, the water samples were reacted with a standard reagent tablet for SO_4^{2-} and Cl^-. The photometer uses selective wavelengths to measure the colour change produced by the addition of a reagent to the solution of interest. Apart from adding the reagent tablet, the photometer was operated in the same way as the TSS determination.

The concentration of metals and metalloids in OTSF mine water was also determined. In analysing the metal concentrations, samples of the drainage were first acidified with nitric acid to a pH less than or equal to 2 to help preserve the metals in the solution. Metals were analysed in the second portion of the drainage sample. A reagent blank was prepared accordingly, and every experiment was done in three replicates. All metals, excluding As and Hg, were measured using a flame atomic absorption spectrophotometer (FAAS; Shimadzu AA 7000). Hydride vapour generation (HVG-ASS) technique was used to determine As and Hg concentrations in the OTSF mine water.

2.3 Geochemical Classification of OTSF Mine Water in Terms of AMD

Different jurisdictions have different legal frameworks for managing mine water, including requirements for discharge licenses, tailings storage, and post-mine closure obligations (Thomashausen *et al.,* 2018; Thisani *et al.,* 2020). Researchers have proposed classification framework for AMD characteristics to highlight pollutant levels and geochemistry of AMD streams to avoid confusion among stakeholders and negative environmental effects (Starke, 2002; Kaur *et al.,* 2018; Thisani *et al.,* 2020). Environmental authorities and site rehabilitation professionals have found these classifications to be useful decision-making tools (Jarvis and Younger, 2000; Thisani *et al.,* 2020). The most widely used classification tools are the Global Acid Mine Drainage (GARD) Guide (Opitz and Timms, 2016), as shown in Table 1. The GARD guide usilises TDS and pH as primary indicators for AMD characterisation (INAP, 2009; Opitz and Timms, 2016). Also, the framework by Hill (1968), as shown in Table 2, remains a broad but simple AMD geochemical classification method that is still in use today. To determine whether the OTSF mine water produced is AMD, both the frameworks by GARD guide (INAP, 2009) and Hill (1968) adopted from Opitz and Timms (2016) and Thisani *et al.* (2020) were used to ascertain the characteristics of OTSF mine water following Tables 1 and 2. The acidity term found in the framework of Hill (1968) was estimated for OTSF mine water using an aqueous acidity estimator from the ABATES v.1.4 software (a spreadsheet-based tool). This tool estimates the acidity of the mine drainage water using pH and soluble metal data and the acidity was expressed as mg $CaCO_3$/L.

Table 1 The GARD Guide Mine Water Classification (Source: INAP, 2009)

GARD Guide Classification	Class description	Thresholds
Acid Rock Drainage/Acid Metalliferous Mine Drainage and	• Acidic pH • Moderate to elevated metals • Elevated sulphate	pH < 6
Neutral Mine Drainage (NMD)	• Near neutral to alkaline pH • Low to moderate metals • Low to moderate sulphate	• pH > 6 • Sulphate < 1000 mg/L • TDS < 1000 mg/L
Saline Drainage (SD)	• Neutral to alkaline pH • Low metals (only moderate Fe) • Moderate sulphate, Mg, and Ca	• pH > 6 • Sulphate > 1000 mg/L • TDS > 1,000 mg/L

Table 2 Hill Framework for AMD Geochemical Classification (Hill, 1968)

Class	Class Description	Thresholds (Ac = Acidity [mg/LCaCO₃])		
Class I	Acid mine drainage	pH = 2.0-4.5 Ac = 1000 -15000	Fe^{2+} = 500-10,00 mg/L Fe^{3+} = 0 mg/L	SO42- = 1,000-20,000 mg/L Al = 0-2,000 mg/L
Class II	Partially oxidised and/or neutralised	pH = 3.5-6.6 Ac = 0–1000	Fe^{2+} =0-500 mg/L Fe^{3+} = 0-1,000 mg/L	SO42- = 500-10,000 mg/L Al = 0-20 mg/L
Class III	Neutral and not oxidised	pH = 6.5-8.5 Ac = 0	Fe^{2+} = 0 mg/L Fe^{3+} = 0 mg/L	SO42- = 500-10,000 mg/L Al = 0 mg/L
Class IV	Oxidised and neutralised/alkaline	pH = 6.5-8.5 Ac = 0	Fe^{2+} = 0 - 500 mg/L Fe^{3+} = 0 mg/L	SO42- = 500-10,000 mg/L Al = 0-2,000 mg/L

2.4 Production of Activated Carbon from Palm Kernel Shells

According to Bakar *et al.* (2016) and Nizamuddin *et al.* (2016), the palm kernel shell is known for its exceptional physicochemical properties, including its high carbon, low ash, low sulphur compound content, high mechanical strength, porous surface, high chemical stability, various functional groups and insolubility in water. For these reasons, it was chosen as the precursor to produce activated carbon in this study. Crushed palm kernel shells (PKS) were collected from an oil palm processing mill in Tarkwa, Ghana. After sieving to acquire a particle size range (- 5.60 + 4.00 mm), the crushed material was cleaned with water to remove any dirt and then dried at 105 ℃ in an oven. In a locally produced Gas Fired Static Bed Pyrolysis-Activation Reactor designed by Buah *et al.* (2015), a 4 kg sample of the PKS (-5.60 + 4.00 mm) was carbonised at a temperature of 900 ℃. The carbonisation was maintained at this temperature for 1 hour to produce char. After combining the char, a representative sample of 300 g was taken for activation. The char was activated in the reactor for 240 min at 900 ℃ using steam as the activating agent at a flow rate of 4.25 mL/min/g char. The yield, surface area, pore volume, and surface shape of PKS-activated carbons were all evaluated. The weight of the final products was divided by the initial weight of the dried palm kernel shells. This was done to determine the weight per cent yield of the activated carbons.

2.5 Characterisation Measurement

The surface area and the micropore volume of the produced activated carbons were measured by Brunauer, Emmett, and Teller (BET) surface area device (Micrometrics, ASAP 2060) at 77 K by means of standard BET procedure using nitrogen adsorption isotherm. Before the measurement, the samples were degassed at 120 ℃ for 2 hours. A relative pressure range of 0.05 to 0.25 was used to calculate the BET surface area. The activated carbons were also examined for the development of pores using a Zeiss EVO MA 15 scanning electron microscope (SEM) equipped with an energy dispersive x-ray spectrometer (EDS; Bruker). Gold/palladium was sputtered onto the produced activated carbons, serving as a conductive layer for effective imaging at 4.0 kV. Also, samples of the OTSF mine water were filtered to obtain a solid residue that was air-dried for SEM/EDS studies to understand the composition of the precipitate in the mine drainage.

2.6 Neutralisation of the Mine Drainage and Heavy Metal Adsorption Studies of the Locally Produced Activated Carbon

The OTSF mine water sample was treated using simple neutralisation and adsorption treatment methods. Samples were poured into a 1 L bevelled glass bottle and pH of 5.40 was measured. Calcium oxide (CaO) dose of 0.00011 g was measured using Ohaus Pioneer Semi-Micro analytical balance and then added to the sample in the bevelled glass bottle, after which it was manually shaken for roughly 60 seconds. The dose amount was determined using simple stoichiometric calculations at a pH of 5.40. A reading of 7.19 was recorded as the pH after the addition of CaO.

Five 1 L bevelled glass bottles of mine drainage, each containing 12 g of locally made activated carbon with a surface area and porous structure with a size range of (-5 +2) mm, were used to conduct the heavy metal adsorption experiment (labelled A, B, C, D, and E). The bottles A, B, C, D and E were mounted on a roller and rotated at 150 rpm for 5, 15, 30, 45, and 60 min, respectively, at 27 ℃ room temperature. The Whatman filter paper (0.45 μm pore size) was used to filter the solution samples. The concentration of heavy metals of interest in the filtrate was then determined using FAAS and HVG-AAS. The amount of adsorbate (metals) adsorbed by activated carbon at equilibrium and the percentage removal was determined using Eqs. 3 and 4, respectively.

$$q_e = \frac{(C_o - C_e)V}{m} \qquad (3)$$

$$\%R = \frac{(C_o - C_e)}{C_o} \times 100 \qquad (4)$$

where q_e (mg/g) is the amount of adsorbate adsorbed at equilibrium, C_o (mg/L) and C_e (mg/L) are the

initial and equilibrium adsorbate concentration in the mine drainage, respectively; V (L) is the mine drainage volume used, and m (g) is the mass of the adsorbent (produced activated carbon).

Adsorption kinetics were used to study the effect of contact time on mass transfer during adsorption process and to identify the equilibrium time and adsorption capacity (Ricordel *et al.*, 2001; Mailler *et al.*, 2016; Tran *et al.*, 2017). The feasible adsorption experimental data was studied and tested using the pseudo-first order (PFO) and pseudo-second order (PSO) kinetic models, following Eqs. 5 and 6, respectively.

$$q_t = q_e(1 - e^{-k_1 t}) \qquad (5)$$

$$q_t = \frac{q_{ek_2}^2 t}{1 + (k_2 q_e t)} \qquad (6)$$

where q_e and q_t are the amounts of adsorbate uptake per mass of adsorbent at equilibrium and at any time t (min), respectively; and k_1 (1/min) is the rate constant of the PFO equation. For PSO, q_t (mg/g) is the amount of adsorbate adsorbed at time t (min); and k_2 (g/(mg × min)) is the rate constant of the pseudo-second order equation.

3 Results and Discussion

3.1 Physicochemical Parameters, Metals, and Anions of the OTSF Mine Drainage

The physicochemical, metals, and anions components of the rehabilitated old tailings storage facility (OTSF) mine water are presented in Tables 3 and 4, respectively. Each criterion is compared to the environmental protection standard established by the Ghana Standard Authority (GSA, 2019) for effluent discharge for general mining. The OTSF mine water is mildly acidic with a pH range of 5.00 – 5.40, and it also has a low acidity content (Table 3). The low acidity content in mg CaCO$_3$/L could be attributed to the low solubility of metals in the mine drainage with the corresponding increased pH of 5.40. The mine water from the OTSF contained very high concentrations of TDS (approx. 659 mg/L) and EC (999 µS/cm) but were below the GSA standard for solids of 1000 mg/L and 1500 µS/cm, respectively. The TSS recorded was 28.3 mg/L. The heavy metals/metalloid(s), Cu, Zn, As, Ni, Cd, Co, Cr, and Hg, identified in the mine drainage were all below GSA (2019) effluent discharge standard. The concentrations of Fe, Pb, and Mn were higher than the permissible limit of the GSA (2019). Both Fe and Pb were about three times higher than the

permissible limit of GSA standard and Mn was about four times higher than the permissible limit of GSA standard set for general mining effluent (Table 2). Regarding the GSA effluent requirement, SO$_4^{2-}$ concentration was likewise high. High concentrations of Fe and SO$_4^{2-}$, as well as some elevated levels of heavy metals, may be caused by the oxidation and dissolution of an iron sulfide mineral such as pyrite, which produces an acid mine drainage-like effluent with a low pH. The natural water quality may suffer if this effluent is allowed to flow into the environment, particularly if it joins a natural water stream within the mining village.

3.2 Characterisation of Locally Produced PKS-Activated Carbon and OTSF Precipitate

The SEM micrograph of locally produced PKS-activated carbon after pyrolysis and activation at 900 ℃ is shown in Fig. 3. There is a complete attack on the outer surface and inner pore wall at a high burn-off of 900 ℃ resulting from a high activation hold time of 240 min. New pores are effactually formed. This leads to greater adsorption capacity due to an increase in surface areas. From Table 5, the BET surface area and micropore area of the PKS-activated carbon were approximately 632 m^2/g and 555 m^2/g, respectively, at an activation time of 240 min. The yield of the activated carbon produced relative to the time of activation decreases from 100 wt % to 70 wt%. The decrease in the yield of the activated carbons could be attributed to more of the primary decomposition and removal of the volatile material to the secondary decomposition of the char residue in the gas-fired static bed reactor at an activation time of 900 ℃. Again, numerous micropore volumes were generated as against the mesopore volumes on the structure of the PKS-activated carbon. This is an important property of activated carbon that plays a critical role in the physical adsorption of contaminants from aqueous processes.

The SEM image of the precipitated residue from OTSF mine water is shown in Fig.4. The morphology of the precipitate looks like gelatinous strands, as shown in the white strands in the SEM micrographs. This indicates the presence of oxide or hydroxide formed in the mine drainage. The EDS spectrum revealed that the chemical composition of the precipitated residue from the mine drainage is Fe, O, Si, and Cl. Table 6 presents the EDS chemical composition of the precipitated residue of the mine drainage.
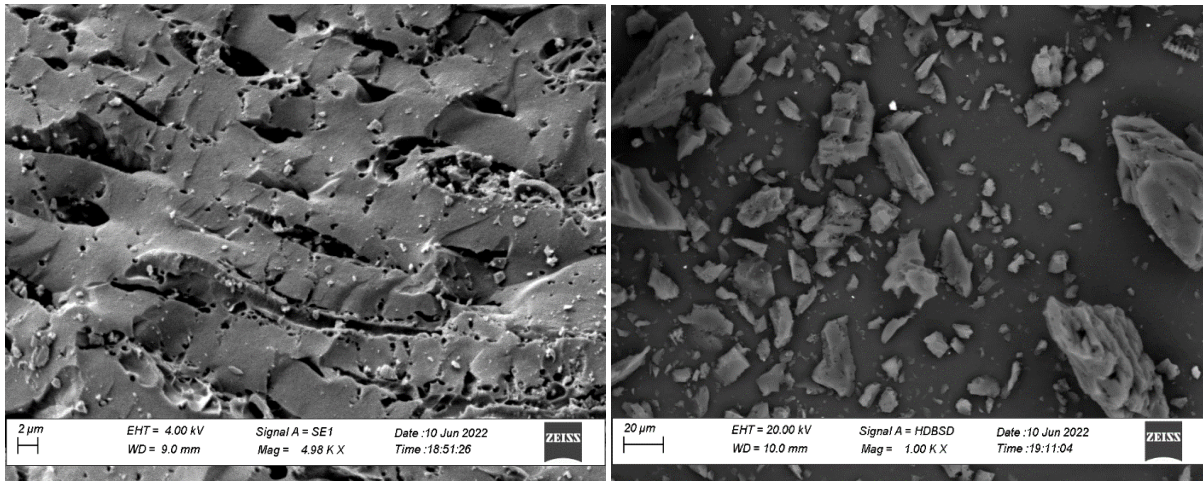
Fig. 3 SEM Images of Palm Kernel-Based Activated Carbon

Table 3 Physicochemical Parameters of the OTSF Mine Drainage

Parameters	OTSF Mine Drainage*	GSA
Temperature (°C)	26.77 (±0.67)	≤ 3 above ambient
DO (mg/L)	7.39 (±0.16)	-
pH	5.00 – 5.40	6.0 – 9.0
EC (µS/cm)	999.2 (±1.18)	1500
TDS (mg/L)	658.67 (±1.53)	1000
TSS (mg/L)	28.3 (±1.57)	50
Eh (mV)	460.17 (±2.25)	-
Acidity (mgCaCO$_3$/L)	68 (± 2.00)	150

Mean and standard deviation

Table 4 Metals and Anions of OTSF Mine Drainage

Parameters	OTSF mine drainage*	GSA
Al (mg/L)	0.06 (± 0.015)	5.0
Ca (mg/L)	69.7 (± 0.64)	250
Na (mg/L)	57.7 (±2.09)	200
K (mg/L)	9.5 (±0.40)	5
Mg (mg/L)	15.8 (±0.35)	2
Fe (mg/L)	25.2 (±0.4)	Total Fe (10)
Mn (mg/L)	0.95 (±0.15)	0.2
Cu (mg/L)	0.002	5.0
Pb (mg/L)	0.33 (±0.02)	0.1
Zn (mg/L)	0.081 ± (0.013)	10
As (mg/L)	0.025	1.0
Ni (mg/L)	0.003	0.02
Co (mg/L)	0.036	0.1
Cr (mg/L)	<0.001	0.5
Cd (mg/L)	0.032	0.1
Hg (mg/L)	<0.005	0.005
SO$_4^{2-}$ (mg/L)	395 (±5)	300
Cl$^-$ (mg/L)	8.3 (±0.26)	250

Mean and standard deviation

Table 5 Properties and Characteristics of PKS Activated Carbon

Properties	Mean ± SD	%RSD
Yield(wt.%)	70.3 ± 2.06	2.94
BET surface area (m^2/g)	632.31 ± 10.5	1.66
Micropore area (m^2/g)	555.63 ± 4.85	0.873
Total pore volume (cm^3/g)	0.408 ± 0.020	4.94
Micropore volume (cm^3/g)	0.333 ± 0.018	5.47
Mesopore volume (cm^3/g)	0.073 ± 0.0015	2.08
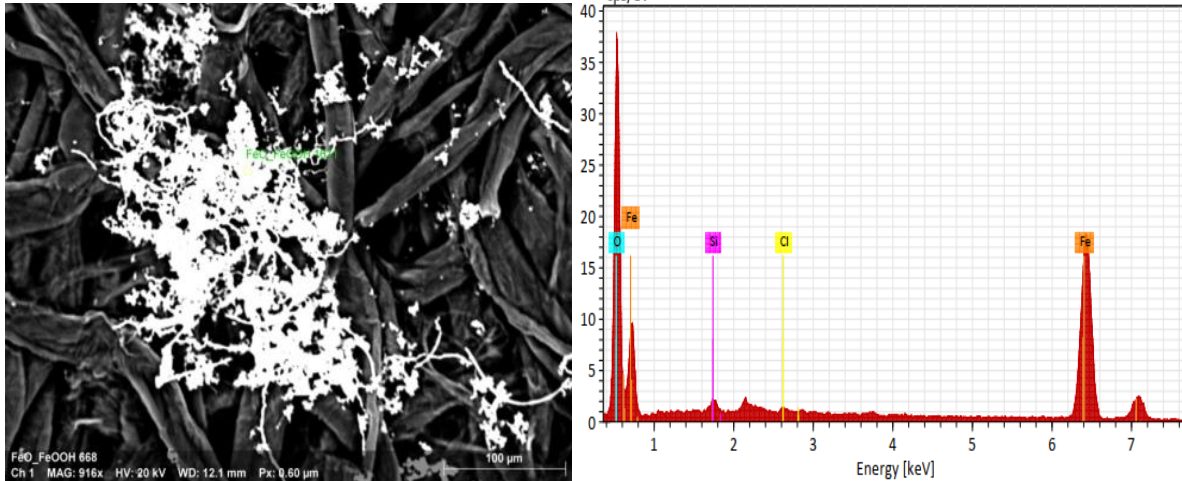
Fig. 4 SEM Morphology (left) and EDS Spectrum (right) of OTSF Mine Drainage

Table 6 Energy Dispersive X-Ray Spectroscopy (EDS) Chemical Composition of the Mine Drainage Precipitate

Element	%Atm Wt	Absolute Error
Carbon	24.92	2.00
Oxygen	56.73	3.00
Silicon	10.84	0.31
Chlorine	0.80	0.06
Iron	6.72	0.19
Sum	100.00	

The EDS revealed that the precipitate was made up of 6.72% Fe, 47.40% O, 15.90% Si, and 1% Cl in atomic weight per cent. The presence of iron oxide or oxyhydroxide gives the drainage the reddish-brown precipitate colour. The chemical composition of the mine water residue could be attributed to Fe from iron sulphide oxidation, which further precipitates as iron oxide and will act in water as either (iron hydroxide or goethite). The presence of Si could come from quartz and other silicate minerals.

3.3 Geochemical classification of OTSF mine drainage in terms of AMD

From Table 3, since the pH of the OTSF mine water is less than 6, the water may be classified as AMD according to the GARD guide (INAP, 2009) (Table 1). Even though the pH of the effluent is less than 6,

the SO_4^{2-} and TDS concentrations are less than 1000 mg/L. This clearly shows that the oxidation process producing low to moderate SO_4^{2-} and TDS concentrations may be partial to warrant serious AMD characteristics of the OTSF main water. To confirm this, Hill's (1968) framework for AMD geochemical classification (Table 2) was also used. From Tables 3 and 4, following Hill's framework, the OTSF mine water determinant characteristics are as follows: pH of the mine water is in the range of 3.5 – 6.6, Fe is in the range of 0 – 500 mg/L in the case of Fe^{2+} or 0 -1000 g/L in the case of Fe^{3+} and SO_4^{2-} is outside the range of 500 - 10, 000 mg/L but closer to the lower range of 500 mg/L; therefore the OTSF mine water can be classified as partially oxidised mine drainage and/or neutralised mine drainage according to Hill (1968), thus class II. This partial oxidation could indicate the early stages of AMD within the conduit of OTSF. The partial

oxidation is affirmed by the Eh value of 460.17 mV (0.46017 V), as shown in Table 3.

The generic Eh-pH diagram for water showing several features is presented in Fig. 5. Fig. 5(a), which describes the locus of the measured pH and Eh values in natural waters and the types of waters in which the measurements have been made, was used to describe the position of OTSF mine drainage in the natural environment. At the measured Eh of 0.460 V and pH 5.40, the OTSF mine drainage is just above the transitional zone in a moderate oxidising environment, and this confirms that the drainage produced could be a result of partial oxidation of OTSF material with low acid-generating iron sulphide and large neutralising capacity minerals. This is because the sample location has hydrothermal gold mineralisation occurring in pyrite and pyrrhotite alteration selvedges and is also associated with vein minerals such as carbonate, muscovite, tourmaline, ilmenite, and apatite according to the geology of the area. The Fe Eh-pH diagram is shown in Fig. 5(b). It can be seen that the OTSF mine water plot within the $Fe^{2+}/Fe(OH)_3$ field indicates that Fe could be removed as an iron-hydroxide precipitate or as FeO.OH (goethite). Even though Fe and S are not of concern, they impact the fate and transport of trace metals. However, within the mildly acidic drainage, there is a significant quantity of dissolved Mn (0.95 mg/L Mn), which exceeded the GSA standard (0.2 Mn mg/L) for "mine" effluent. As Fe could precipitate out as iron-hydroxide or goethite, Mn, on the other hand, is soluble in the Eh-pH range of OTSF mine drainage and will remain in solution (Fig. 5C). The source of Mn could be a result of exchangeable Mn in clay. These may also come from ilmenite and tourmaline as solid solution minerals in the hydrothermal mineralisation or Mn in siderite concretions, similar mineralisation as seen in the work of Larsen and Mann (2005). According to Larsen and Mann (2005), if siderite was the main source of the dissolved Mn, the acid formed by oxidation of pyrite in a mine waste could be reacted with siderite, with a concomitant release of Mn and Fe to the solution percolating through the mine waste. This explains the partially oxidised and neutralised drainage of the OTSF mine water.

3.4 Neutralisation and Removal of Heavy Metals and Sulphate from OTSF Mine Drainage

The neutralisation and adsorption process of heavy metals and SO_4^{2-} at different reaction times with PKS-activated carbons are shown in Table 7. For 1 L of OTSF mine drainage with 12 g of the activated carbon at a contact time of 5, 15, 30, 45, and 60 min,

the adsorbent (PKS-activated carbon) greatly reduced the concentration of the heavy metals. For instance, As and Pb were reduced to threshold values below the detection limit at almost all neutral pH of the mine drainage. This could be due to the very low concentration of As and Pb, which give the carbons enough room to adsorb almost everything from the OTSF mine drainage with less insignificant amount to be detected. The PKS-activated carbon showed a sharp metal and SO_4^{2-} reduction for the first 5 min of contact time for both Fe, Mn, and SO_4^{2-}, respectively. Fe was significantly reduced from 25.2 mg/L to 6.0984 mg/L, and Mn was reduced from 0.95 mg/L to 0.4313 mg/L and these represent 76% and 55% removal of Fe and Mn, respectively (Fig. 6).

At a contact time of 5 min, Fe and SO_4^{2-} were reduced below the GSA effluent discharge requirement, but Mn was not. Therefore, a further increase in the adsorption time increased the removal efficiency of both metals and hence reduced Mn concentration below the GSA effluent discharge requirement of 0.2 mg/L at 30 min (Table 7). The equilibrium of metal removal (97.8%) was achieved at 15 min for Fe and almost everything was removed at 60 min (99.1%) and 30 min for Mn (87.9% and approximately 89% at 60 min) (Fig. 6). This means that maximum removal of Fe can be achieved within 15 min of contact between PKS-activated carbon and OTSF mine water, but for Mn, a contact time of 30 min becomes appreciable for the highest removal of 88%, which is consistent with Buah *et al.* (2016). The higher adsorption rate within the first 5 min is due to the higher number of available adsorption sites (Zhou *et al.*, 2011; Zhou and Xue, 2013). The Eh of the OTSF mine drainage was also reduced from 460 mV to approximately 131 mV during the adsorption process (low-reducing environment), where the activity of the mixed redox couple is reduced. At almost neutral pH and reduced Eh, both Fe and Mn exist as $Fe^{2+}/Fe(OH)_3$ and Mn^{2+} (Fig .5).

The adsorption capacity of the PKS-activated carbon for Fe was higher than Mn (Table 8). This indicates the affinity of surface-active groups of the carbons for both metals. The differences in the trend of adsorption for Fe and Mn metal ions may depend on their initial concentrations or the adsorption principle and phenomena, which depends on the charge density of the cation and the diameter of the hydrated cation where the bigger diameter, Mn^{2+} ionic radius = 80 pm, has minimum adsorption than Fe^{2+} = 76 pm (Goher *et al.*, 2015). The higher the charge, the smaller the metal ion and the stronger the metal cation hydration, leading to maximum adsorption and faster equilibrium for some metals (Erdem *et al.*, 2004; Goher *et al*., 2015; Buah *et al.*, 2016).
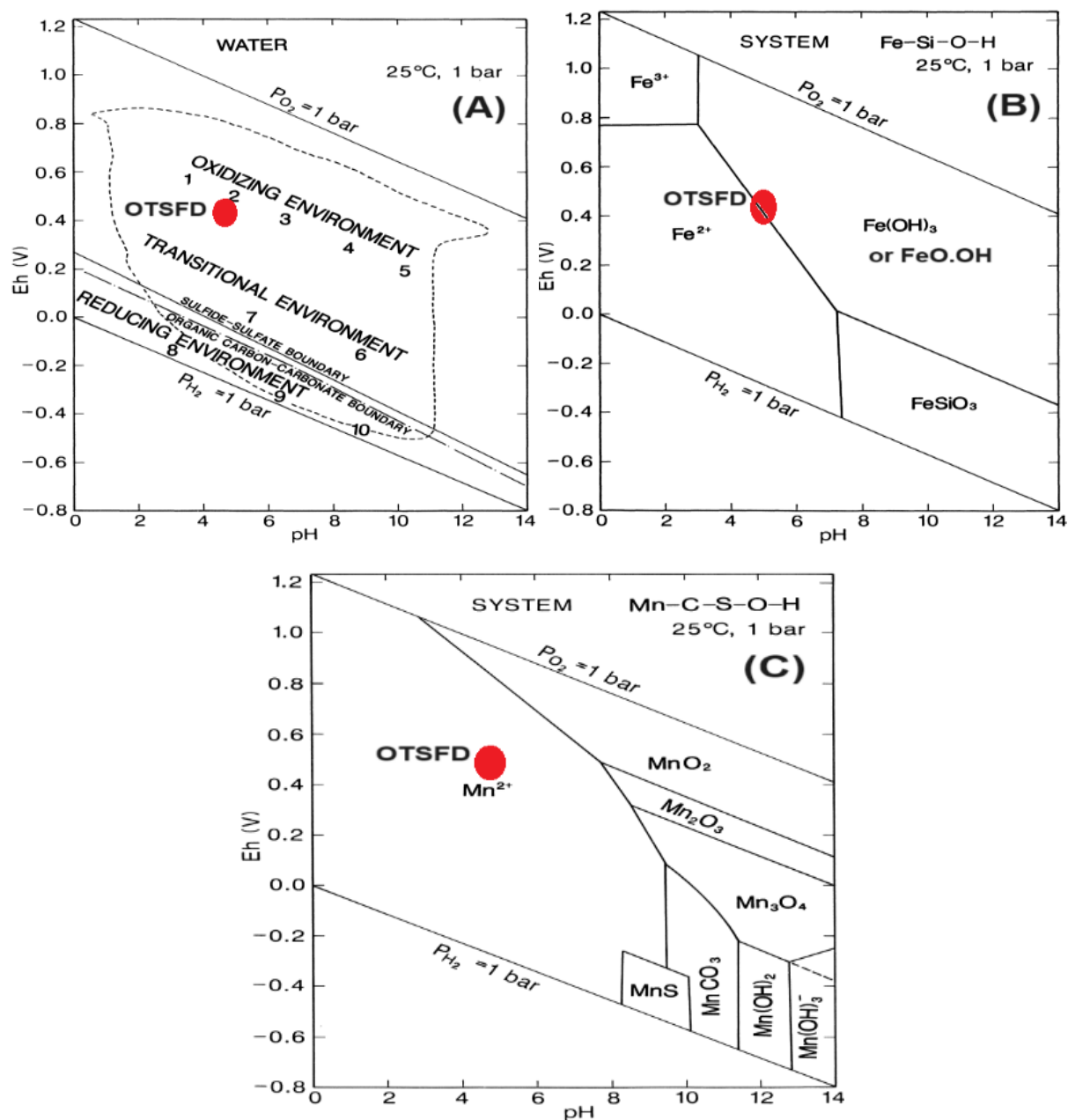
Fig. 5 Generic Eh-pH Diagram for Water Showing Several Features (A); Specific environments are (numbers on figure): *1 mine waters; 2 rain; 3 streams; 4 normal ocean water; 5 aerated saline water residues; 6 ground waters; 7 bog waters; 8 water-logged soils; 9 euxinic marine waters; 10 organic-rich, saline water*; **Eh-pH diagram for part of the system Fe-Si-O-H (B); Eh-pH diagram for part of the system Mn-C-S-O-H (C)** (source: Brookins, 2012)

Table 7 Neutralisation and Adsorption Equilibrium Concentration of Heavy Metals

SID	Contact Time (min)	pH	Eh (mV)	As (mg/L)	Pb (mg/L)	Fe (mg/L)	Mn (mg/L)
O*	0	5.40	460.17	0.025	0.33	25.2	0.95
A	5	7.20	141.20	<0.001	<0.003	6.0984	0.4313
B	15	7.32	138.75	<0.001	<0.003	0.5544	0.3762
C	30	7.20	136.70	<0.001	<0.003	0.2772	0.1149
D	45	7.21	131.85	<0.001	<0.003	0.2520	0.1102
E	60	7.22	130.86	<0.001	<0.003	0.1764	0.1054

O mean initial sample condition without neutralisation and activated carbon for adsorption.*

Table 8 Maximum adsorption capacity of Fe, Mn and Sulphate.

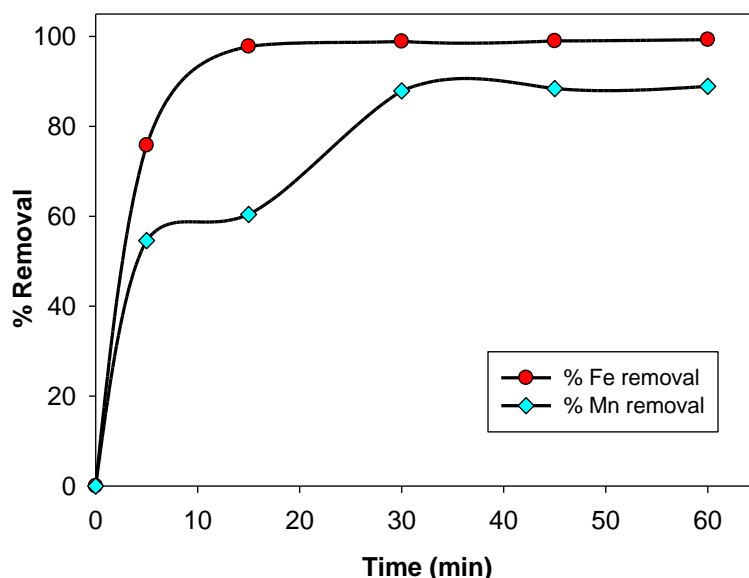| Time (min)	q_e (expt., mg/g) Fe	q_e (expt., mg/g) Mn
5	1.5918	0.0432
15	2.0538	0.0478
30	2.0769	0.06958
45	2.0790	0.06998
60	2.0853	0.07038

Fig.6 Effect of Contact Time on Adsorption of Fe and Mn from OTSF Mine Water using 12 g of PKS-Activated Carbons

3.5 Adsorption Kinetics

Adsorption is a physicochemical process that mainly depends on the ability of an adsorbent like PKS-activated carbons to accumulate heavy metals from aqueous solution. Therefore, to understand the reaction rate and adsorption mechanism, an evaluation of the experimental data was carried out using the PFO and PSO kinetic models following Eqs 5 and 6. The validity of experimental data to both kinetic models was deduced by comparing the coefficients of determination (R^2) values and normalised standard deviation NSD (%) computed using Eq. 7. The NSD (%) was utilised to substantiate the kinetic model used to describe the adsorption rate mechanism.

$$NSD\% = 100\sqrt{\frac{\Sigma[(q_{exp}-q_{cal})/q_{exp}]^2}{n-1}} \qquad (7)$$

where n refers to the number of data points, and q_{exp}, and q_{cal} (mg/g) refer to experimental and calculated adsorption capacities, respectively. A lower value of

NSD (%) signifies a good fit between the experimental and the calculated adsorption capacities. Plots of the PFO and PSO kinetic models are presented in Fig. 7 for Fe and Mn as the heavy metals of concern in the OTSF mine water. For Fe, the experimental data fitted all the two kinetic models; however, there was a better fit for the PFO than PSO because the PFO model provided the best R^2 value of 0.9999 and NSD of 0.144% with q_e and k_1 of 2.08 mg/g and 0.2810 min$^{-1,}$ respectively as shown in Fig. 7A.

The Fe following PFO kinetic model means that the adsorption occurs as diffusion control through the interface, and this assumes that the rate of change of Fe uptake with time is directly proportional to the difference in saturation concentration and the amount of PKS-activated carbon uptake with time, which is applicable over the initial stages of an adsorption process (Lagergren, 1898). For Mn, the experimental data fitted better to the PSO model than the PFO with R^2 value of 0.9644 and NSD value of 11.2%, as shown in Fig.7D, producing q_e and k_2 values of 0.07595 mg/g and 2.7232 (g/mg × min).
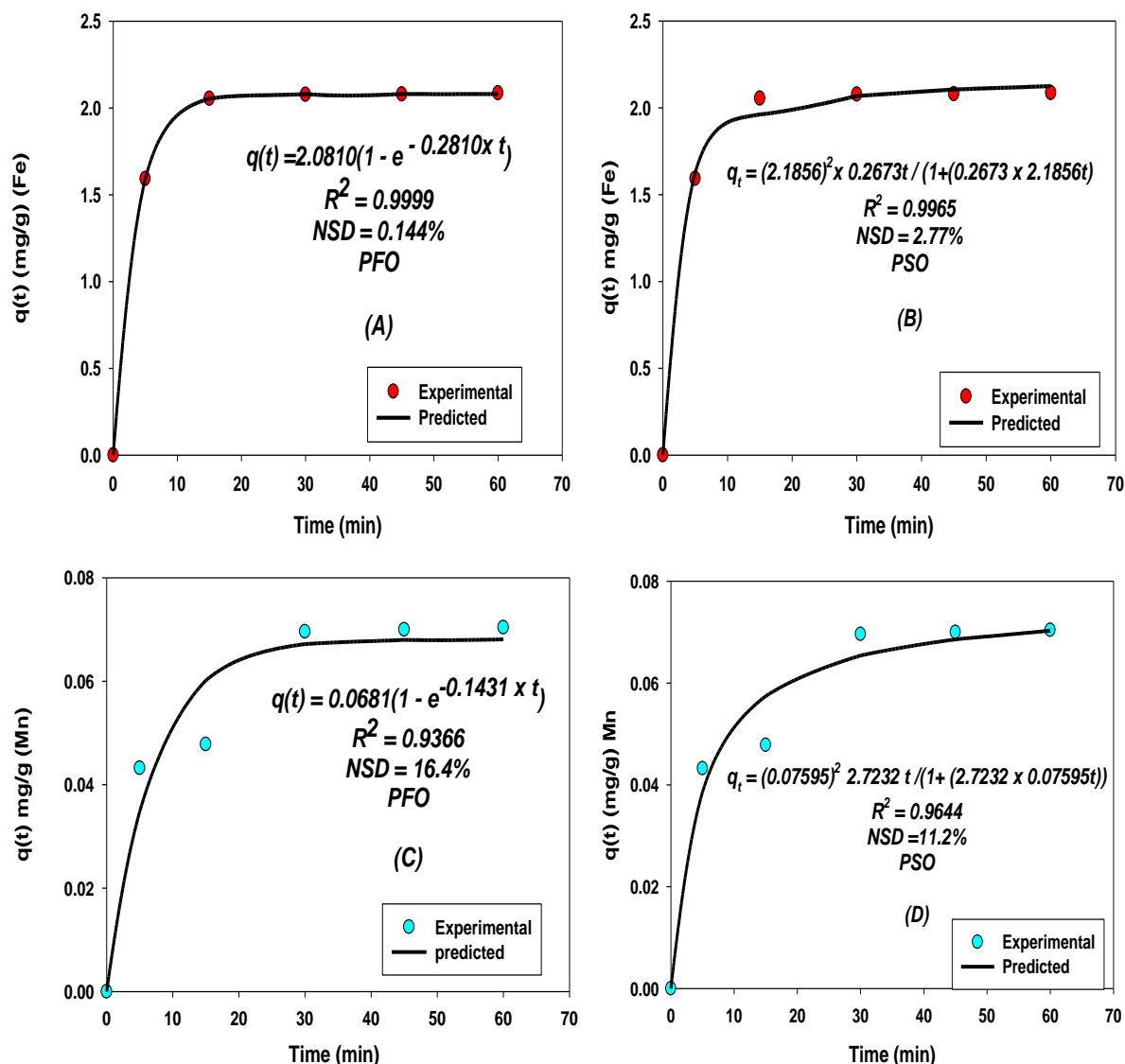
Fig. 7 Pseudo-First Order and Second Order Kinetic Models for Fe (A and B) and Mn (C and D), respectively.

The PSO model assumes that the rate-limiting step between Mn ion and the PKS-activated carbons is chemical adsorption or chemisorption throughout the whole range of adsorption and is dependent on adsorption capacity, not the concentration of the adsorbate (Ho and McKay, 1998; 1999).

4 Conclusions

Reddish brown mine water from an old, rehabilitated tailings storage facility (OTSF) has been characterised in terms of physical characteristics and chemical constituents. The physiochemical parameters of OTSF mine water were below the GSA effluent discharge limit for general mining except for mildly acidic pH (pH of 5.00-5.40), which was outside the GSA effluent discharge requirement range. Heavy metals such As, Cd, Fe, Pb, and Mn were determined as part of the chemical compositions of OTSF mine water. Except

for As and Cd, all other heavy metals had concentration values above the GSA limit. Again, OTSF drainage had a low Cl^- concentration but high SO_4^{2-} concentration above the GSA limit. A value of 460 mV for Eh showed a moderate oxidising environment of OTSF mine water.

Global Acid Mine Drainage (GARD) guide and Hill (1968) geochemical classification frameworks were used to ascertain the OTSF mine water characteristics in terms of AMD. It was revealed from both frameworks that the OTSF mine water was partially oxidised to low neutralised AMD with less toxic constituents like low Fe, SO_4^{2-}, and other metal ion concentrations.

The concentration of heavy metals like As, Pb, Fe, and Mn were successfully reduced from OTSF mine drainage by using activated carbons that were produced from waste palm kernel shells (PKS)

utilising a locally designed gas-fired static bed pyrolysis and activation reactor. SEM and BET analysis revealed the presence of pore spaces with high surface area, which indicates the presence of active adsorptive sites of the derived activated carbon.

The major heavy metal contaminants Fe and Mn fitted well to PFO and PSO adsorption kinetic models, indicating adsorption as diffusion controlled and chemisorption, respectively. The removal of heavy metals from the OTSF mine water can be accomplished effectively and affordably with the help of an excellent absorbent produced locally.

Further investigations in the study area are undoubtedly warranted, especially carrying out the comprehensive geochemical characterisation of the OTSF mine water to identify the possible sources of mildly acidic mine drainage with high SO_4^{2-}, Fe, Mn, and Pb constituents, which were above the GSA limit. Also, having a cost-effective treatment process designed to treat the high SO_4^{2-} concentration from the OTSF mine drainage to an acceptable level before discharge into the environment is recommended.

Acknowledgements

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