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Research article

Characterization and reusability suggestions of the sludge generated from a synthetic acid mine drainage treatment using sodium ferrate (VI)

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ABSTRACT

Mining activities are the main cause of generation of the voluminous sludge waste, loaded with metals precipitated from the treatment of acid mine drainage (AMD) and this is always disposed to the landfill. This study aimed at characterizing and suggesting the reusability potential of AMD sludge to reduce the environmental problem caused by its accumulation so that it could become a valuable material. The sludge was obtained after treating a synthetic AMD with a green oxidant sodium ferrate (VI) (Na_2FeO_4) that was prepared by a wet oxidation method. Chemical and physical characterization of a dried sludge generated after treatment was then performed using the Fourier Transform-Infrared and X-Ray powder Diffraction spectroscopy. Scanning Electron Microscopy-Energy Dispersive X-ray Spectroscopy also served to identify the surface morphology of the sludge. The sludge presented a high weight percentage of Fe and O and lower concentrations of other metals such as Al, Mn, Si, and Na. Nitrogen adsorption/desorption isotherms or Brunauer-Emmett-Teller (BET) was used to assess the surface area, pore volume and diameter of the sludge. The BET results showed that the surface area of the sludge obtained after treating the synthetic AMD using Na_2FeO_4 was 31.50 ± 0.03 m^2/g with pore diameter and volume of 52.50 nm and 0.41 cm^3/g, respectively. However, the produced sludge could serve as an adsorbent to remove pollutants from water or to synthesize different magnetic nanocomposites due to its high surface area (>natural zeolite) and high composition of Fe and O.

1. Introduction

The mining sector is the major contributor to the economy of different countries (Rakotonimaro et al., 2017). South Africa is one of those countries whose economy mainly depends on coal and gold mining activities (Kefeni and Mamba, 2020). In 2019, these mining activities contributed almost 44.85% to South African gross domestic product (Plecher, 2020). Unfortunately, around 6000 mines have been abandoned by their owners as indicated by the Department of Mineral Resources while their rehabilitation rate is about 10 mines per year (Olalde, 2016). With newly abandoned mines adding to the massive impact of those abandoned a long time ago, the country is facing now growing health, social and environmental crisis created by inadequate oversight and withering gold industry. However, the most challenging waste generated from these abandoned mines is an acid mine drainage (AMD). AMD is considered as the main environmental pollutant in different countries depending on mining industries (Bruneel et al., 2017). This is principally due to its acidic pH, high sulphate and dissolved metal concentrations (Akinwekomi et al., 2017; Bologo et al., 2009; Kefeni et al., 2017). AMD is mostly generated when mineral sulphides are in contact with oxygen, water and catalytic bacteria (*Thiobacillus ferrooxidans*) after mining activities (Pierre Louis et al., 2015; Kefeni et al., 2017). Therefore, its treatment is highly needed to neutralize the acidity and to reduce or completely remove metals (Sukati et al., 2018). Over the past three decades, the development of cost-effective and sustainable remediation solutions through the metal recovery process for the AMD problem has been the subject of extensive research. In this regard, treatment of AMD by mining industries is a requirement to ensure compliance for discharge. However, the treatment of AMD has been performed using passive and active methods to reduce its environmental impacts (Park et al., 2019). These AMD treatment methods generate sludge with different compositions depending on the treatment process, the quality of water to be treated and the use of neutralizing or oxidizing agents. In mining activities, AMD sludge can be managed in various options where it can be

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collected into a specific pond or mixed with tailings in a tailing pond. Another option is using the sludge as a tailing cover material, dispose it to mining fields, wells and mine backfill. Furthermore, it is stated that covering the tailings with sludge does not successfully prevent the generation of AMD because the sludge can dry out and crack. However, its alkalinity may not be enough to neutralize the acidity and prevent metal mobilization (Zinck et al., 2010). The main issue associated with the management of AMD is the physical and chemical stability of the sludge and reservoirs. The stable sludge will not affect the environment and may be disposed of in a long-term storage facility. The stability depends on the degree of resilience in the environment, which makes it possible to expressively change the chemical composition of the sludge (Amanda and Moersidik, 2019). Moreover, the next challenge is the storage of sludge which before it is collected, the sludge must be treated to easily regulate and stabilize pollutants in it, but this process is quite expensive (Zinck and Griffith, 2016). Therefore, AMD sludge can be reused rather than being disposed on the landfill. AMD sludge can be reused as coagulant, flocculent and adsorbents to remove contaminants such as rare earth elements and heavy metals from water (Rakotonimaro et al., 2017). At present, a variety of coagulants are widely applied to treat wastewater or drinking water and the most conventional coagulants are ferric chloride, aluminium and ferric sulphates (Chorghe et al., 2017; Talaiekhozani et al., 2017). These coagulants not only reduce sensory indicators (turbidity, chromaticity, etc.) of raw water but also remove various harmful contaminants, especially toxic metals in separate dozing and mixing units (Zhu et al., 2016; Naceradska et al., 2017). However, ferrate (VI), a multipurpose chemical acting as both a green coagulant and oxidizer with high redox potentials ($E^o = 0.72$ V in basic and $E^o = 2.20$ V in acidic conditions) (Eqs. (1) and (2)), has recently been noted as one of the most capable wastewater treatment reagents to replace these conventional oxidants, coagulants and disinfectants (Sun et al., 2014; Yates et al., 2014; Sharma et al., 2016; Matin et al., 2018).

$$FeO_4^{2-} + 8H^+ + 3\acute{e} \rightarrow Fe^{3+} + 4H_2O \qquad E^o = 2.20\ V \tag{1}$$

$$FeO_4^{2-} + H_2O + 3\acute{e} \rightarrow Fe(OH)_3 + 5OH^- \qquad E^o = 0.72\ V \tag{2}$$

Therefore, unique functions make ferrate (VI) as an environmentally-friendly dual-functioning substance that combines oxidation and coagulation and it can reduce the volume to the sludge generated during the treatment of AMD. Ferrate ions could also remove different inorganic pollutants such as manganese (II) and zinc (II) which are barely removed from water by different oxidants, coagulants, adsorbents, and flocculants at $pH < 9.0$ (Sharma, 2011, 2013). For example, during the pre-oxidation process, ferrate (VI) ions can oxidize some substances first to make them easy to settle down, then an *in-situ* formed coagulant or flocculent (ferric hydroxide or ferric ions) with a high surface area is concurrently generated in water treatment processes (Barışçı et al., 2016; Sun et al., 2014; Xie et al., 2016). Besides being used as a coagulant and flocculent, this ferric ion generated from self-decomposition of ferrate (VI) and the oxidation of ferrous ions could perform as an adsorbent in aqueous solution (Goodwill et al., 2016; Liu et al., 2017). For instance, Goodwill et al. (2016) found out that manganese (II) could be removed from water matrix using ferrate (VI) salt in the form of nanoparticles as manganese (IV) at the ratio of 2: 3 of ferrate to manganese, respectively. The effects of *in-* and *ex-situ* generated ferric sludge particles from the removal of trace thallium by potassium ferrate (VI) was fully investigated (Liu et al., 2017). This indicated that the adsorption process has participated in the removal of thallium. However, generated sludge nanoparticles can have a high adsorption capacity for pollutants of interest in water treatment. Among the physical parameters that contribute to the reuse of sludge is the specific surface area and pore size, which affect the retention of pollutants. The higher is the surface area and smaller pore diameter of an adsorbent, the higher is its sorption capacity. The novelty of the study is based on the application of an advanced oxidation process for the treatment AMD using liquid sodium ferrate (VI) (Na_2FeO_4) as an emerging cost-effective, green oxidant, coagulant, and flocculent in a single mixing and dosing unit with a zero-energy input. Thus, this study is devoted to determining chemical composition, pore volume, specific surface area and diameter and further suggesting the reusability potential of the sludge generated through the treatment of a synthetic AMD by Na_2FeO_4.

2. Materials and methods

2.1. Preparation of the solution

A solution of synthetic AMD was prepared by mixing different amounts of salts in 1L of ultrapure water as presented in Table 1. Fe^{2+}, Al^{3+}, Mn^{2+}, Cu^{2+}, Ni^{2+}, Mg^{2+}, and Zn^{2+} were target metal ions which are always available in real AMD (Johnson and Hallberg, 2005; Diez et al., 2017; Rajak, 2018). The pH of the solution was measured using an OHAUS pH-meter ST320 and maintained at 3 using 2M H_2SO_4 to stop immediate precipitation of ferric hydroxide ($Fe(OH)_3$).

2.2. Generation of the synthetic AMD sludge and its characterization

Liquid Na_2FeO_4 was previously prepared by a wet chemical method and its concentration was determined using UV-Visible spectroscopy. Na_2FeO_4 solution was characterized by its intense purple colour confirming the presence of high valent of iron (Fe (VI)) (Munyengabe and Zvinowanda, 2019). To generate the AMD sludge, an amount of synthetic AMD solution with the composition given in Table 1 was treated using a liquid Na_2FeO_4 (0.05 M) (4:1 v/v) within a contact time of 30 min as an optimum condition obtained in the previous study in which all iron (II) (pale green colour) and Fe (VI) (purple colour) were fully converted into a yellow precipitate (Fe (III)) (Munyengabe et al., 2020). After treatment, the obtained synthetic AMD sludge was dried at 105 °C overnight using the oven, pulverised and then sieved to get a fine powder of the same size (Sapsford et al., 2015; Amanda and Moersidik, 2019). For a reusability suggestion purpose, minerals in dried synthetic AMD sludge were characterized using X-Ray Diffraction (XRD), while functional groups of these minerals were determined using Fourier Transform-Infrared (FT-IR) (Kefeni et al., 2018). The untreated synthetic AMD was not characterized during this study as it did not contain the sludge. Elemental composition and structural morphology were determined using Scanning Electron Microscopy-Energy Dispersive X-ray Spectroscopy (SEM-EDS). Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) methods were used to identify and calculate the pore volume, surface area and pore diameter of the sludge sample (Brunauer et al., 1938; Kefeni et al., 2018). This method was performed by analysing the dry synthetic AMD sludge by nitrogen adsorption isotherm at 120 °C for degassing temperature.

3. Results and discussions

3.1. Sludge characterization

3.1.1. XRD patterns of generated synthetic AMD sludge

Figure 1 shows the XRD pattern for sludge generated through oxidation and coagulation of the synthetic AMD using sodium ferrate.

The sludge sample contained different minerals and salts with a similar chemical composition of halite, hematite or magnetite, vonsenite, and thermonatrite groups. Halite and thermonatrite structures dominated in the sludge sample which is a result of alkali metal salts including potassium and sodium that are characterized with an orthorhombic shape. Vonsenite and hematite or magnetite (oxides of iron) minerals as reference material of similar shape were also present in the sludge, which is highly composed by a group of ferrous, magnesium and ferric ions, and sometimes might contain a small amount of nickel, aluminium, manganese, calcium, potassium, and silicon. The diffraction peak observed at

Table 1. Recipe for preparation of synthetic AMD.

Chemical formula	Molar mass (g/mol)	Masses of ions (mg)	Target ions	Concentrations of ions (mg/L)
$FeSO_4 \cdot 7H_2O$	278.01	1991.30	Fe^{2+}	400.00
$Al_2(SO_4)_3 \cdot 18H_2O$	666.40	500.17	Al^{3+}	40.50
$MnCl_2$	125.84	1.74	Mn^{2+}	0.76
$CuSO_4.5H_2O$	249.68	3.61	Cu^{2+}	0.92
$NiCl_2$	129.60	0.44	Ni^{2+}	0.20
$MgSO_4$	120.37	182.06	Mg^{2+}	36.77
$ZnSO_4$	142.04	61.47	Zn^{2+}	11.43

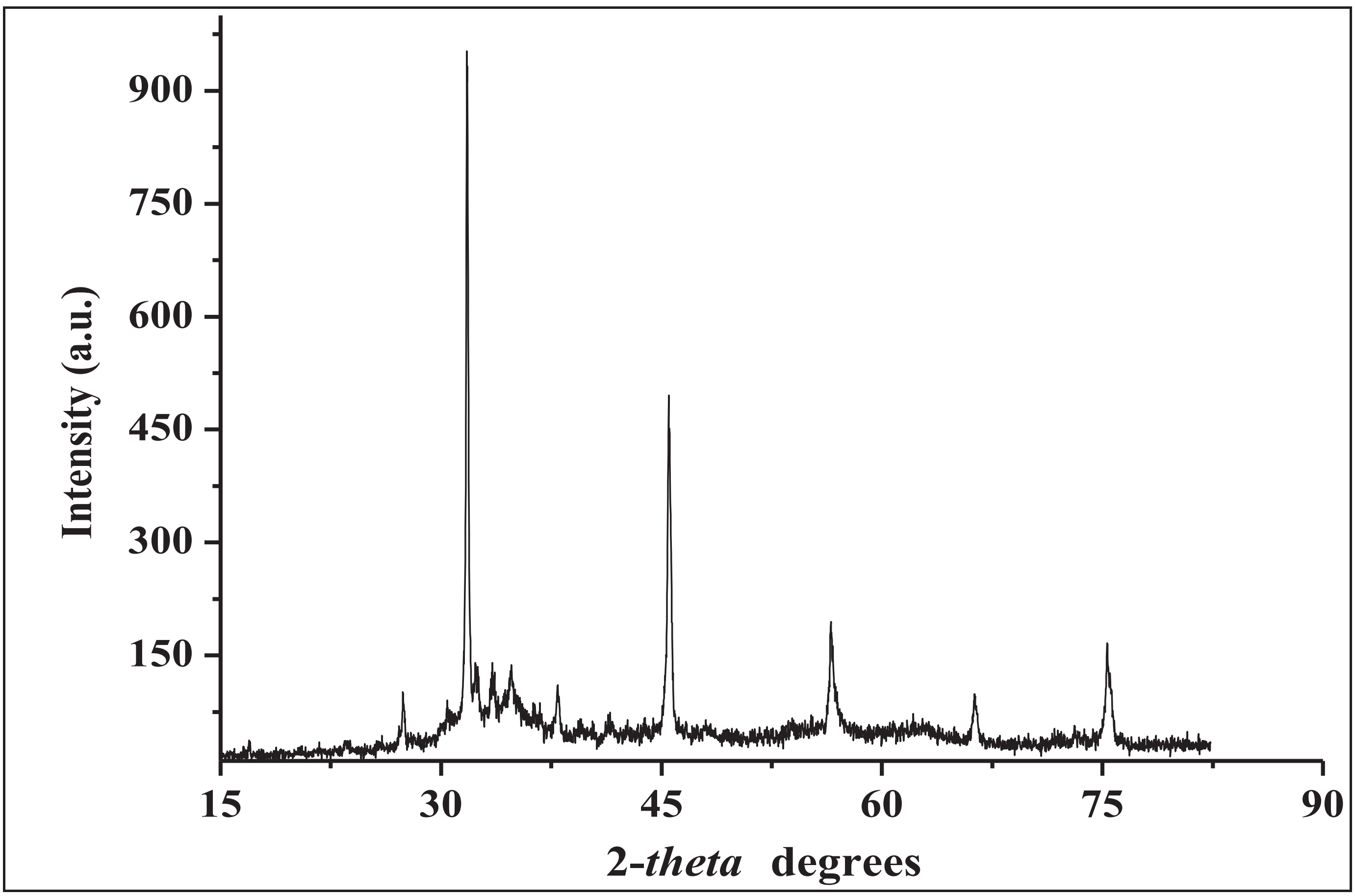

Figure 1. The XRD pattern for synthetic AMD sludge.

46° is attributed to the characteristic low crystalline metallic iron (Yavuz et al., 2006; Alexandrescu et al., 2010) while the characteristic peaks observed at 28°, 33°, 57°,67°, and 75° of the sludge might be ascribed to the α-Fe(OOH) (Varanda et al., 2002).

3.1.2. BET or nitrogen adsorption/desorption isotherm analysis

Nitrogen adsorption/desorption isotherm results such the pore diameter, surface area and pore volume of the dried synthetic AMD sludge obtained in this study and similar BET results of sludge available in the literature are both presented in Table 2.

According to International Union of Pure and Applied Chemistry (IUPAC) classification, the pores are divided into three groups due to their diameter (i) macropores (>50 nm), (ii) mesopores (2 < pore size<50 nm) and (iii) microspores (<2 nm). However, the obtained porous material with diameters ranging between 23 and 52 nm observed from the nitrogen adsorption/desorption isotherms at 77K with an

Table 2. Comparison of BET results obtained in this study with the literature.

Material	Pore volume (cm^3/g)	Surface area (m^2/g)	Pore diameter (nm)	References
Sludge	-	23–114	-	Rakotonimaro et al. (2017)
Sludge	0.06	22.60 ± 0.20	10.70	Amanda and Moersidik (2019)
Synthetic AMD sludge	0.41	31.50 ± 0.03	23–52	This study

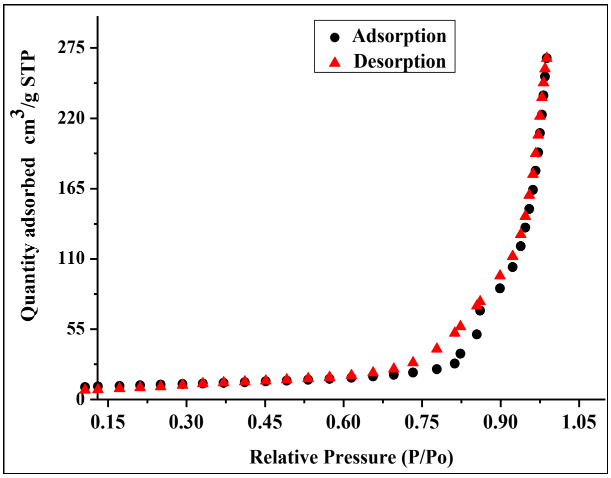

Figure 2. Nitrogen adsorption/desorption isotherm curves of synthetic AMD sludge.

average of 37.50 nm was classified between mesopore and macropore. Comparing with the literature, this obtained pore diameter of the sludge generated through the treatment of a synthetic AMD using Na_2FeO_4 was smaller than the magnetic zeolite nanocomposite size (90–100 nm) synthesized by Ahmadi et al. (2016) through a chemical co-precipitation method. This result was further confirmed by Figure 2, which shows the characteristic feature of the type-II isotherms according to IUPAC nomenclature that is convex to P/P_o axis at a high relative pressure ($P/P_o > 0.40$) (Po and P denote the saturation and equilibrium pressures of nitrogen, respectively) (Ramesh et al., 2014; Muttakin et al., 2018).

The adsorbed molecules in type-II isotherms are normally gathered around the most favourable sites on the surface area of the porous material (Thommes et al., 2015). However, among the physical features that contribute to the reuse of sludge is the surface area, which effects the retention of pollutants. The higher is the surface area and smaller pore diameter of a composite, the higher is its sorption capacity.

3.1.3. Characterization of the sludge using SEM-EDS

Figure 3 shows SEM micrographs of the synthetic AMD sludge at three different resolutions (61: top-left, 1000: top-right and 6250: bottom-left) and the EDS spectrum (bottom-right in blue colour) showing the weight per cent of the analyzed elements.

The SEM micrographs indicated that synthetic AMD sludge has an uneven shape mesh-like structure with inner surface looking spongy and highly porous whereas outer edges of the mesh have an uneven surface with mostly flake-like appearance. The flake-like part of the sludge might be composed by silica oxide while the spongy porous one assumed to be composed of metal oxide particles (Hallam et al., 2012; Bose and Tiwari, 2019). The EDS elemental mapping confirmed the presence of 8 elements in a solid synthetic AMD sludge with high weight percentages of Fe (45.0 wt. %), O (38.3 wt. %) and Na (7.6 wt. %) (from the breakdown of Na_2FeO_4), also lower concentration levels (<3.0 wt. %) for Si, Mg, Mn, Zn, and Al. The synthetic AMD sludge showed significant presence of silica, known as a common impurity in different salts and this is therefore expected to be present in the sludge. Comparing with the literature, the weight percentage of Fe in the sludge was 6 times to the concentration of Fe while weight percentage of O was quite similar to the ones found by Amanda and Moersidik (2019). However, both BET and SEM-EDS results for the produced synthetic AMD sludge suggest its reusability as an adsorbent due to high surface area and metal content such as iron, sodium, magnesium, silicon, and aluminium. Previous studies showed that the metal content such as Fe and Al in the sludge could make it a suitable adsorbent to remove pollutants (Amanda and Moersidik, 2019). The sludge containing a big amount of iron sulphate oxy/hydroxides and iron oxides can sequester through adsorption and coprecipitation of cations and oxyanions (Kirby et al., 1999; Sibrell and Tucker, 2012). For example, the combination of Si and Al has been used to synthesize

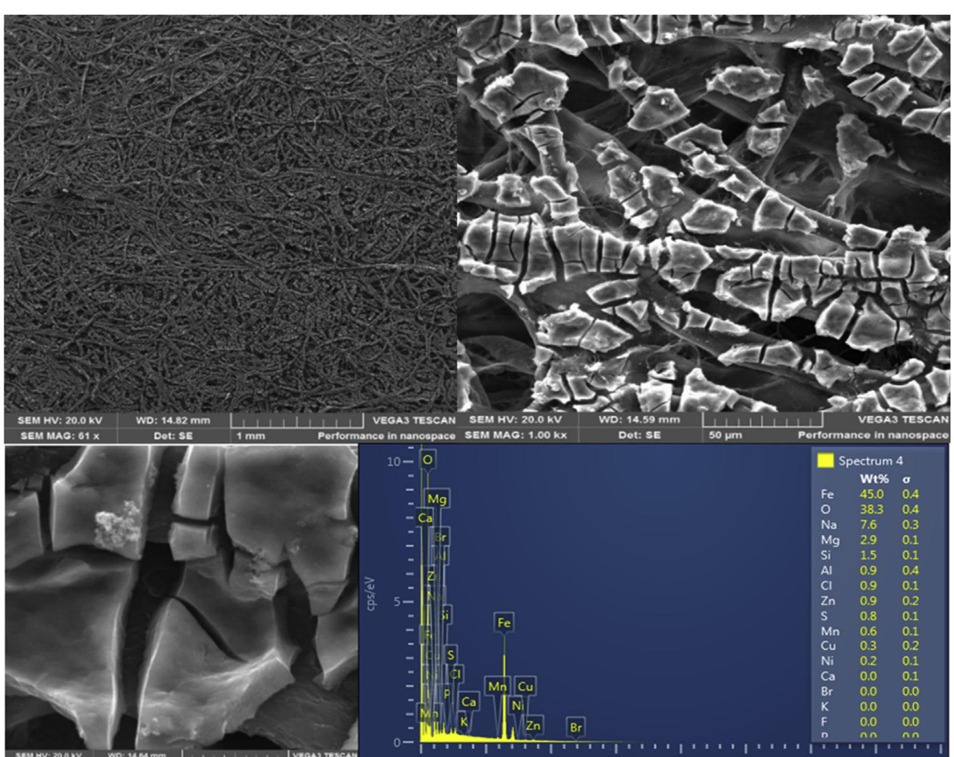

Figure 3. SEM micrographs and EDS spectrum results of the synthetic AMD sludge.

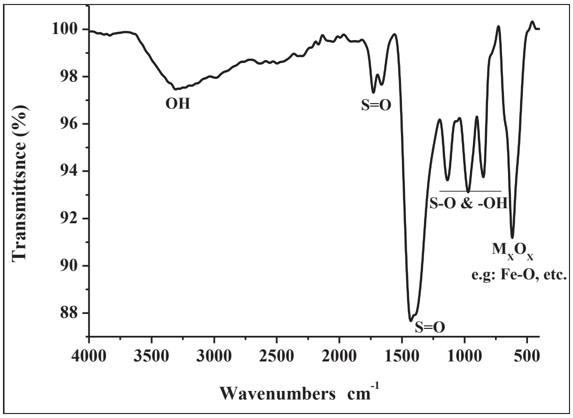

Figure 4. FT-IR spectrum of synthetic AMD sludge.

magnetic cancrinite as an adsorbent through some synthesis processes (Bian et al., 2019). However, the produced synthetic AMD sludge could be reused to treat drinking water, wastewater and real AMD and to prepare magnetic adsorbent (e.g.: magnetic cancrinite), magnetized multiwall carbon nanotubes, nano-zero valent iron (Fe^0), and iron/iron oxide-based nanocomposites (e.g.: α-Fe_2O_3–TiO_2 nanocomposite) due to its high composition especially in Fe and O (Alexandrescu et al., 2010; Badi et al., 2015, 2018; Yousefzadeh et al., 2018; Bian et al., 2019).

3.1.4. FT-IR spectroscopic characterization

Different transmittances and absorptions of sludge collected after treatment of synthetic AMD using Na_2FeO_4 and the peaks of different functional groups of precipitated chemicals are shown in Figure 4.

The FT-IR spectrum results confirm the formation and presence of different minerals in the sludge as indicated by the XRD data. The peaks found at 619.96 cm^{-1} are attributable to the metal oxides (such as iron oxide), which usually appear between 400 and 650 cm^{-1} (Alexandrescu et al., 2010; Mahmoud, 2017; Ahmadi et al., 2020), the peaks 1426.24, 1639.39 and 1730.92 cm^{-1} might correspond to S=O vibration stretching of different sulphate minerals (Kefeni and Mamba, 2020). The peak observed at 3315.94 cm^{-1} is ascribed to ν-OH due to water in the sludge (Flores et al., 2019). The three peaks also observed between 1140 and 750 cm^{-1} are attributable to OH and S–O vibrations of mineral compounds such as $[Fe^{3+}{}_3(OH)_6(SO_4)_2]^-$ and $[Fe^{3+}O(OH, Cl)]$ (Francioso et al., 2010). This can be supported by the literature where Mejía et al. (2015) identified these minerals at 1190, 1085 and 1008 cm^{-1}. These results promised that synthetic AMD sludge from the treatment of AMD by ferrate salts can be reused as adsorbent of good quality for metal removal from industrial wastewater treatment due to high content in iron (ferrihydrite) normally characterized by a high surface area.

4. Conclusion

Sludge sample generated from synthetic AMD treated with a green oxidant Na_2FeO_4 was mainly composed of amorphous iron oxides (Fe–O) and a small amount of non-toxic elements such as Mg, Si, Al and Zn as indicated by the results of FT-IR, XRD and SEM-EDS analyses. The self-decomposition of Na_2FeO_4 generated a useful and non-toxic substance, ferric ions which are normally used as a coagulant in water treatment. The nitrogen adsorption/desorption isotherm results show that the surface area of the synthetic AMD sludge is 31.50 $\pm$ 0.03 m^2/g while pore diameter and volume are 52 nm and 0.41 cm^3/g, respectively. The adsorption pore diameter sizes clearly show that synthetic AMD sludge is meso/macro-porous material. The higher weight percentage of O, Fe, Si, Mg, Al and Zn and high surface area could be benefited to synthesize different adsorbents and metal oxides for water treatment purpose.

Declarations

Author contribution statement

Alexis Munyengabe, Caliphs Zvinowanda, John Ngoni Zvimba, James Ramontja: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Competing interest statement

The authors declare that there is no conflict of interest.

Additional information

No additional information is available for this paper.

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