

# Influence of type and amount of organic matters on the iron sorption of acid mine drainage onto reclaimed-mining soils

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Research Article

## Influence of type and amount of organic matters on the iron sorption of acid mine drainage onto reclaimed-mining soils

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### Abstract

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Mining activity may potentially produce acid mine drainage (AMD), which has relatively high acidity and dissolved heavy metal concentrations. Constructed wetland is one of the AMD management methods in which organic matter (OM) plays a very important function in reducing the concentration of heavy metals in AMD through absorption and precipitation processes. Three types of OM (empty fruit bunches of oil palm, chicken manure and water hyacinth) and five levels of OM (0, 10, 20, 30 and 40 Mg ha<sup>-1</sup>) were applied to reclaimed-mining soils (RMS) in an incubation study. A batch experiment was then performed to measure the effect of OM application on the maximum sorption capacity ( $Q_{max}$ ) of iron (Fe) from the AMD onto the mixed soil-OM. The application of OM resulted in increases in soil pH, carboxylic groups, and total functional groups, in which these increases varied based on the types and amounts of OM application. This study also revealed that OM application resulted in increasing Fe sorption. The application of OM increased  $Q_{max}$  values from 2077 to 2348–3259 mg kg<sup>-1</sup> (water hyacinth), to 2607–3635 mg kg<sup>-1</sup> (chicken manure), and to 2219–2992 mg kg<sup>-1</sup> (empty fruit bunches of oil palm). Increasing these  $Q_{max}$  values may ascribe to increasing functional groups of the RMS with OM application. The results prove the importance of OM in controlling the sorption of Fe from AMD onto soils.

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### Introduction

Mining activities may result in acid mine drainage (AMD) that is formed from the reaction between sulphide minerals exposed in mining activities with water and air. The existence of AMD, which has relatively high acidity and heavy metal concentrations, could generate environmental disturbances and ultimately require high costs for the management of these environmental disturbances. The release of AMD to terrestrial ecosystems results in a decrease in soil pH and nutrients such as P, K and Zn (de Klerk et

al., 2016), an increase in metal concentrations such as Al, Fe, Mn and Cu (Miguel et al., 2017), which in turn may result in a 62% reduction in rice production (Choudhury et al., 2017). Thus, an appropriate AMD management is required to prevent and process AMD to comply with the environmental quality standards.

Iron (Fe) is the most common and dominant element in AMD because it is generated from the oxidation process of sulphide minerals (pyrite) in the mining process (Rakotonimaro et al., 2018). Based on the sampling conducted in several studies, the

concentration of Fe in AMD may vary from 10 mg L<sup>-1</sup> (Noor et al., 2021) to 2350 mg L<sup>-1</sup> (Rakotonimaro et al., 2018). Fe is an essential nutrient for several physiological processes and plant growth (Ismail et al., 2020). However, Fe becomes unprofitable and toxic to plants when its concentration is very high in soils (Saaltink et al., 2017). The presence of excessive amounts of Fe in the soil may disrupt the uptake of Ca, Mg, K and P (Penn and Camberato, 2019), which in turn may be significantly reduced tiller number, biomass production, and grain yield of rice (Frei et al., 2016; Turhadi et al., 2019). Low pH of AMD may stimulate the migration of Fe from soils and sediments. Therefore, management is required to prevent the harmful effects of Fe and other metals of AMD on the environment.

One of the methods recommended in AMD management is to drain water from reclaimed-mining soils to constructed wetlands before the AMD is released into open waters. Constructed wetland is AMD management consisting of media, plants, and microorganisms that mimic natural wetlands to drain and process AMD so that it meets environmental quality standards before being released into public waters (Flores et al., 2019). The use of constructed wetland reduces 89%, 91%, and 91% of Pb, Cr, and Cd, respectively, in AMD through sorption mechanism (Mohammed and Babatunde, 2017), and the addition of zeolite and biochar to the constructed wetlands is able for removal of As (35–84%) and Zn (8–24%) (Guo et al., 2020). The high ability of constructed wetlands to reduce heavy metal contents in AMD was also shown by Singh and Chakraborty (2020), who used cow manure and bamboo chips as organic substrates on constructed wetland and reported high efficiency of metal removal (99.7% Cr, 97.8 Ni, 93.7% Co, 91.6% Fe, and 59.7% Al). In addition to having high ability in heavy metal removal, a constructed wetland is also considered as management of AMD, which is low-energy and less-operational requirements compared to the conventional management systems (Wu et al., 2015; Gorgoglione and Torretta, 2018). These research results show that from the technical and economic aspects, the constructed wetland method is a promising method for AMD management.

Improving the quality of AMD with the constructed-wetland method occurs through the precipitation of heavy metals by the mechanism of sedimentation, adsorption, ion exchange and complexation (Sheoran and Sheoran, 2006; Srivastava et al., 2014; Buxton, 2018). Adsorption is the most common mechanism for the removal of heavy metals from AMD (Núñez-Gómez et al., 2019; Rodríguez and Leiva, 2020; Mokgehle and Tavengwa, 2021). The process of sorption occurs through the mechanism of the specific bonding between positive charges of heavy metals from AMD and negative charges of the sorbents (Agha-Beygli et al., 2019; Feng et al., 2019), in which the number of heavy metal removal from

AMD through this mechanism is controlled by the total of negative charges provided by sorbent materials. Thus, the existence of organic matter or waste products from industry and agriculture that can be used as sorbent materials in large quantities has the potential to be used in the management of the AMD through the sorption process.

Problem that often occurs in AMD management using the constructed wetland is the limited sorption capacity for heavy metal removal, in which this sorption capacity is controlled by the number of sorption sites provided by sorbent materials. OM is able for providing sites for the sorption of substance in the AMD. Previous research has shown that increasing pH and decreasing heavy metal concentrations in AMD with OM application (Arce et al., 2017; Idaszkin et al., 2017; Peiravi et al., 2017). In a study using a reactor applied with OM from grass litter, Lefticariu et al. (2015) reported decreases in the concentration of SO<sub>4</sub> (26–35%), Fe (36–62%), Al (78–83%), Mn (2–6%), Ni (64–81%), Zn (88–95%), Cu (72–85%), and Cd (90–92%) compared to the reactor without OM application. In addition, the precipitation of heavy metals Pb and Cu through sorption processes is greater in the OM with high lignin contents compared with OM containing high cellulose (Ge et al., 2014; Li et al., 2018). These studies indicate that the presence of OM is very crucial in determining the rates of sorption of heavy metals on AMD. However, information on the OM variables that control the sorption capacity of substances from AMD is not comprehensively available. This information is very urgent for the success of environmental restoration due to mining activities in Indonesia. Therefore, this study aimed to quantify the effect of the type and amount of OM on the sorption capacity of Fe in AMD management.

## Materials and Methods

### *Sampling and characterization of soils and organic matter*

The soil samples were obtained from the reclaimed-mining area of PT. Galuh Cempaka (3°29'26"–3°29'54" S, 114°45'47"–114°47'19" E) in the Desa Palam, Cempaka district, Banjarbaru city, South Kalimantan province. Subsequently, the sampling process was achieved at a depth between 0–30 cm, using a soil drill at several sampling points. The soil samples were then homogenized after cleaned from the remains of plants, followed by the placement into plastic bags for storage at 4°C. Furthermore, the sub-samples were air-dried to investigate the physical and chemical characteristics of soils (Table 1).

Determination of physical properties of soil samples included soil texture (Gee and Bander, 1986) and bulk density (Blake and Hartge, 1986). The characterization of soil chemical properties consisted of soil pH H<sub>2</sub>O (1:5.0) (McLe, 1982), organic C using the Walkley and Black method (Nelson and

Sommers, 1996), total nitrogen using the Kjeldahl method (Bremer and Mulvaney, 1982), available P (Jackson, 1967), P extracted using HCl 25% (Sudjadi et al., 1971), exchangeable K, Na, Ca and Mg extracted using  $\text{NH}_4\text{OAc}$  1.0 N pH 7.0 (Knudsen and Peterson, 1982; Lanyon and Heald, 1982), water-extractable iron by the orthophenanthroline method (Krishna-Murti et al., 1970), and cation exchangeable capacity (CEC) by the method described by Rhoades (1982). The OM used in this study were empty fruit bunches of oil palm (EFBOP), water hyacinth and chicken manure. Empty bunches of oil palm and chicken manure were sampled respectively from oil palm plantations and small-holder chicken farms in the Desa Batu Mulia, Kintap

Sub-District, Tanah Laut District, South Kalimantan Province. Water hyacinth (*Eichhornia crassipes*) was obtained from rivers in the Desa Palam, Cempaka Sub-District, Banjarbaru City, South Kalimantan Province. After sampling, those OMs were oven-dried at 60 °C for 72 hours and then grind to a-2 mm size. The three OMs were then characterized to determine organic C content using the Walkley-Black method (Nelson and Sommers, 1996) and total nitrogen content using the Kjeldahl method (Bremer and Mulvaney, 1982). The contents of carbohydrate (hot water-soluble C) and lignin for each organic matter were quantified by the methods of Grandy et al. (2000) and Chesson (1981), respectively (Table 1).

Table 1. Characteristics of soil and organic matters.

Characteristics	Soil	Chicken Manure	Water Hyacinth	Empty Fruit Bunches of Oil Palm
Texture				
Sand (%)	33.03 (5.79)*	-	-	-
Silt (%)	23.45 (7.98)	-	-	-
Clay (%)	43.52 (2.56)	-	-	-
pH ( $\text{H}_2\text{O}$ )	3.73 (0.11)	-	-	-
Bulk density ( $\text{g cm}^{-3}$ )	1.38 (0.02)	-	-	-
Organic C ( $\text{g kg}^{-1}$ )	10.87 (0.16)	391.03 (0.54)	298.70 (4.17)	442.24 (5.06)
N ( $\text{g kg}^{-1}$ )	2.76 (0.04)	51.91 (0.78)	20.05 (3.34)	17.19 (2.13)
P ( $\text{g kg}^{-1}$ )	5.78 (0.10)	-	-	-
Exchangeable Ca ( $\text{cmol kg}^{-1}$ )	3.67 (0.12)	-	-	-
Exchangeable Mg ( $\text{cmol kg}^{-1}$ )	1.32 (0.11)	-	-	-
Exchangeable K ( $\text{cmol kg}^{-1}$ )	1.32 (0.09)	-	-	-
Exchangeable Na ( $\text{cmol kg}^{-1}$ )	2.12 (0.10)	-	-	-
Water-extractable Fe ( $\text{mg kg}^{-1}$ )	43.45 (2.30)	-	-	-
CEC ( $\text{cmol kg}^{-1}$ )	28.76 (1.03)	-	-	-
Hot water soluble C ( $\text{g kg}^{-1}$ )	-	36.21 (1.21)	24.50 (0.09)	21.36 (1.12)
Lignin ( $\text{g kg}^{-1}$ )	-	12.56 (2.12)	16.14 (1.17)	32.56 (2.12)

\* Numbers in the parenthesis are the standard deviation of mean (n=3).

#### Incubation experiment

The incubation experiment in the laboratory was carried out by adding OM according to the treatments to 2000 g of reclaimed-mining soils and then the mixed soil-OM were incubated at 60% soil water-filled pore space (WFPS) for 15 days. The amount of added OM consisted of 5 (five) levels: 0, 10, 20, 30 and 40 Mg  $\text{ha}^{-1}$ . Throughout the incubation period, the water contents of the mixed soil-OM was retained at 60% WFPS. Subsequently, soil samples were characterized for soil pH (McLean, 1982), carboxyl groups (Schafer, 1984) and total functional groups (Kim and Park, 2016) at the end of the incubation period.

#### Sorption experiment

After completion of the incubation period, a batch experiment was carried out by reacting soils with AMD (ratio soil : AMD = 1 : 100), in which the AMD being used has been diluted in order to obtain different

levels of Fe concentration in the AMD (0, 20, 50, 75, 100, 125, 150 and 175 mg  $\text{Fe L}^{-1}$ ). The dilution of AMD was performed using a solution containing 24 mg of  $\text{K}_2\text{SO}_4 \text{ L}^{-1}$ , 10 mg  $\text{NaCl L}^{-1}$ , and 20 mg of  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O L}^{-1}$ , then the pH of the diluted AMD was adjusted to pH 6.0 by addition of HCl or NaOH. The batch experiment was performed by reacting the soils with diluted AMD in a 50 mL centrifuge tube, followed by mechanical agitation for 8 hours in a dark space. Subsequently, the suspension was centrifuged for 30 minutes at 2000 g and the supernatant was filtered using a Whatman 42 filter paper. In addition, each treatment was prepared for three replications. The Fe concentration in the supernatant was evaluated spectrophotometrically with the orthophenanthroline method (Krishna-Murti et al., 1970). Moreover, the quantity of adsorbed Fe was obtained by the difference between the Fe concentration in the solution before and after equilibrium. Furthermore, the Fe sorption

data were then fitted to the Langmuir equation using the least square curve fitting with Microsoft Excel® (de Levie, 2001).

### Statistical analysis

The effect of the type and amount of OM application on changes in soil properties and variables in the Langmuir equation was quantified through analysis of variance (Anova). Before the Anova was carried out, the Shapiro-Wilk and Bartlett tests were carried out to ensure that the data to be analyzed had a normal distribution and a homogeneous variety, respectively. If the treatment has a significant effect, the mean difference test was conducted to differentiate the effect among all treatments. All of these statistical tests were carried out using the GenStat 12<sup>th</sup> Edition (Payne, 2008).

## Results and Discussion

### Effect of organic matter addition on changes in soil pH and functional groups

The variance analysis results showed the application of various types and amounts of OM significantly influenced the soil pH. Figure 1 shows the reaction of reclaimed-mining soils and the OM components. Different OMs had the different capability in raising soil pH. The addition of EFBOP up to an amount of 20 Mg ha<sup>-1</sup> was not able to increase soil pH (Figure 1). Increasing soil pH was observed when 30–40 Mg ha<sup>-1</sup> EFBOP were applied to the soils. A similar trend was shown when water hyacinth applied to soils, only the addition of water hyacinth with an amount exceeding 20 Mg ha<sup>-1</sup> was able to increase soil pH (Figure 1). Different patterns were shown in the treatment of chicken manure, in which the application of this OM at any amounts was able to increase the soil

pH (Figure 1). Therefore, the results of the present study were known to correspond with previous investigations. For instance, Clark et al. (2007) showed the variable effects on soil pH was due to the separate OM addition, where the easily decomposable OM components, including chicken manure, generated a substantial increase in the soil pH, compared to other OMs. Several researchers also reported an improvement in soil pH in the presence of OM (Wang et al., 2019; Laurent et al., 2020; Shen et al., 2020). The influence of OM addition on soil pH occurs through several mechanisms. Aluminium (Al) and iron (Fe) may be a source of soil acidity through the hydrolysis of Al and Fe which releases H<sup>+</sup> ions into soils (Bohn et al., 2001). The application of OM to the soil stabilizes Al and Fe in the soils through the binding reaction of Fe and Al in soils (Wang et al., 2009) so that the hydrolysis of Al and Fe which releases H<sup>+</sup> ions to soils could be suppressed. Previous studies showed a decrease in the amount of exchangeable aluminium in the soils with the OM application (Wang et al., 2009; Jiang et al., 2018).

Increasing soil pH with OM application also appears through the oxidation process of organic acid anions. Inorganic cation content in plants is generally higher than inorganic anions, and plants synthesize organic acid anions such as oxalate, malate and citrate to maintain the balance between cations and anions. The anion oxidation of these organic acids through decomposition processes of plant residues consume H<sup>+</sup> ions and release OH<sup>-</sup> ions, so that the soil pH will increase (Yan et al., 1996; Butterly et al., 2011; Butterly et al., 2013). Other mechanisms for increasing soil pH due to OM application include the ammonification of organic nitrogen that is contributed ion OH<sup>-</sup> in the soils (Yan et al., 1996; Butterly et al., 2013; Fujii et al., 2020;) and specific adsorption of organic molecules (Haynes and Mokolobate, 2001).

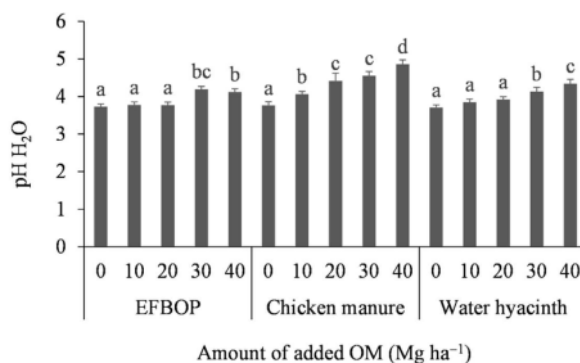


Figure 1. Influence of OM addition on soil pH. Vertical lines above the bar represent the standard deviation of mean (n=3). Similar letters above the vertical line indicate similar effects of the treatment based on the least significant difference (LSD) test at  $\alpha$  5%.

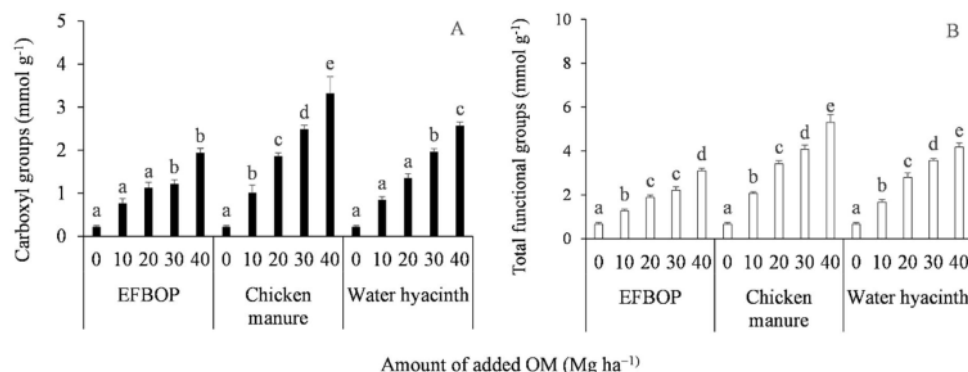


Figure 2. Influence of OM addition on carboxyl groups (A) and total functional groups soil pH (B). Vertical lines above the bar represent the standard deviation of mean ( $n=3$ ). Similar letters above the vertical line indicate similar effects of the treatment based on the least significant differences (LSD) test at  $\alpha$  5%.

The results also showed that OM application affected the number of carboxyl groups and total functional groups of soils. The effects of OM application on carboxyl groups and on functional groups are presented in Figure 2. The effect of OM application on carboxyl groups is similar to the effect of OM application on soil pH. The application of EFBOP and water hyacinth in low amounts ( $\leq 20$  Mg ha<sup>-1</sup>) was not able to increase the number of carboxyl groups. On the other hand, chicken manure application at all amount was able to increase the number of carboxyl groups (Figure 2A). In contrast, the total functional groups of soils increased with the OM addition at all types and amounts (Figure 2.B).

The increasing number of carboxyl groups that varies based on the type of OM is related to the different characteristic of OM. Chicken manure had low organic C and high total N contents (low C/N ratio), while EFBOP had high organic C and low total N contents (high C/N ratio) (Table 1). In addition, chicken manure contained high easily-decomposable organic C (hot water-soluble C) and relatively low lignin content, while EFBOP contained high lignin and low easily-decomposable organic C (Table 1). This causes chicken manure to decompose more easily than other OMs (EFBOP and water hyacinth). The easier OM to be decomposed, the more functional groups are contributed to the soils. The results of the study are in accordance with several previous studies that reported increasing functional groups of soils with OM addition (Chen et al., 2002; Rivero et al., 2004; Hamoud, 2010).

#### **Influence of organic matter addition on the Fe sorption of acid mine drainage**

The results showed that the Fe sorption from AMD onto reclaimed-mining soils applied with different types and amounts of OM fitted reasonably to the

Langmuir isotherm with a value of  $r^2$  varied between 0.98 and 0.99 (Figure 3 and Table 2). The addition of OM increased the maximum sorption capacity ( $Q_{max}$ ) of Fe from AMD onto soils based on the order of EFBOP < water hyacinth < chicken manure (Table 2). Increasing the amount of OM applied to soils also resulted in increasing the values of  $Q_{max}$ . The  $Q_{max}$  values increased from 2078 mg kg<sup>-1</sup> in the soils without OM addition to 2348–3259 mg kg<sup>-1</sup> with the addition of water hyacinth, 2607–3635 mg kg<sup>-1</sup> with the addition of chicken manure, and 2219–2992 mg kg<sup>-1</sup> with the addition of EFBOP (Table 2).

The effect of OM addition on Fe sorption from AMD onto reclaimed-mining soils is related to increasing the number of negative charges resulted from the OM addition. Fe in the AMD that has a positive charge reacts with a negative charge of soils (Chi and Amy, 2004; Xu et al., 2020). The results showed that the OM addition onto reclaimed-mining soils increased the total functional groups and carboxyl groups (Figure 2). The increase in the amount of added OM will be followed by increases in the total number of functional groups and carboxyl groups of soils, which in turn will be followed by increasing the  $Q_{max}$  of Fe (Table 2). Results of this imply that increasing the amount of OM addition results in increasing the amount of heavy metal removal from AMD. Several studies have shown the effect of OM addition on increasing the amount of heavy metal removal of AMD through the adsorption process. OM has a high capability (> 94%) in the removal of Fe, Zn and Mn of AAT through the mechanism of sorption and metal precipitation in batch experiments using synthetic AMD (Zagury et al., 2006). The important role of OM in the sorption of heavy metals from AMD is related to the increasing sorption sites provided by added OM (Skousen et al., 2017).

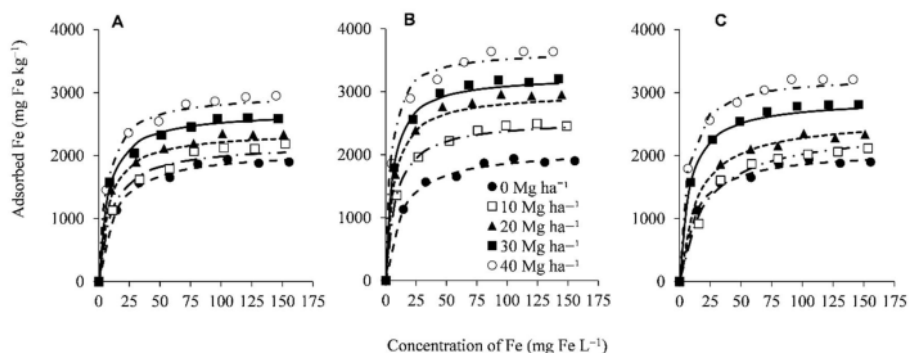


Figure 3. Adsorption of Fe onto reclaimed-mining soils applied with empty fruit bunches of oil palm (A), chicken manure (B), and water hyacinth (C) based on the Langmuir isotherm. Lines denoted the experimental data fitted to the Langmuir isotherm, while symbols represented experimental data.

Table 2. Langmuir isotherm for Fe sorption onto reclaimed-mining soils applied with different types and amounts of OM matter.

Type/Amount of Organic Matter (Mg ha <sup>-1</sup> )	Q <sub>max</sub> (mg kg <sup>-1</sup> )	k (L mg <sup>-1</sup> )	r <sup>2</sup>
Water Hyacinth			
0	2077.54 (56.36)*a**	0.07066	0.98
10	2438.49 (75.32) b	0.06198	0.99
20	2570.33 (45.95) c	0.08767	0.99
30	2886.03 (69.36) d	0.13258	0.99
40	3258.67 (56.32) e	0.18962	0.99
Chicken Manure			
0	2077.54 (56.32) a	0.07066	0.98
10	2608.69 (45.39) b	0.12383	0.99
20	3005.13 (53.67) c	0.15612	0.98
30	3233.35 (78.32) d	0.19220	0.99
40	3634.68 (65.32) e	0.25077	0.99
Empty Fruit Bunches of Oil Palm			
0	2077.54 (65.36) a	0.07066	0.98
10	2219.36 (54.84) b	0.09787	0.98
20	2404.32 (56.36) c	0.11993	0.99
30	2710.36 (86.36) d	0.13683	0.98
40	2992.36 (45.32) e	0.16010	0.99

\* Numbers in the parenthesis are the standard deviation of mean (n = 3).

\*\* Similar letters indicate similar effects of the treatment based on the least significant differences (LSD) test at  $\alpha$  5.

Richard et al. (2020) compared several types of OM: peat moss, compost, HCl extracted algae, wood ash, algae and sawdust in their ability to absorb Ni from AMD and reported that peat moss has the highest maximum sorption capacity of Ni based on the Langmuir isotherm. The highest capability of peat moss to adsorb Ni is highly related to the lowest point of zero charge (ZPC) compared to other OMs, implying that peat moss possess high negative charge at neutral pH (batch experiments conducted at pH 7.0). The high negative charges of peat moss are also supported by the fact that peat moss possess high

cation exchangeable capacity (CEC) (Richard et al., 2020). Strosnider and Nairn (2010) examined the ability of three treatments: limestone, municipal wastewater, and mixed limestone–municipal wastewater to reduce heavy metal concentrations in AMD and reported that the application of municipal wastewater was able to reduce As concentrations from 19.4 to 0.34 mg L<sup>-1</sup> through the adsorption process. Treatment of AMD with materials containing organic matter (municipal wastewater) is considered to be the best treatment technology for the AMD neutralization and metal removal, in which this technology is able to

remove 52–84% of Al and 74–86% of Fe from the AMD (Hughes and Gray, 2013). Compost resulted from household waste processing may also be utilized to reduce the heavy metal contents in AMD, in which the mechanism for reducing heavy metal content occurs through heavy metal absorption on the surface of the compost (Gibert et al., 2005). Gustafsson et al. (2011) reported strong sorption of  $Pb^{2+}$  onto the peat layer, and only a small part of the sorbed  $Pb^{2+}$  could be released back into the solution. The results of this study confirm the results of previous studies which show that the sorption process of Fe onto reactive sites provided by OM plays a very crucial role in the management of AMD.

### Conclusion

Results of this study revealed that the application of organic matter to reclaimed-mining soils was able to improve several soil characteristics that affect the adsorption process (soil pH, number of carboxylate groups and number of total functional groups). The ability of organic matter to improve soil characteristics is controlled by the characteristics of organic matter. Organic matter which is relatively easy to decompose, respond more quickly in increasing soil pH, the numbers of carboxyl groups and total functional groups of soils. The results also showed that the data of Fe sorption from acid mine drainage onto reclaimed-mining soils added with organic matter followed the Langmuir equation. The application of OM increases the maximum adsorption capacity ( $Q_{max}$ ) of Fe onto soils, where the increasing value of  $Q_{max}$  was based on the order of empty fruit bunches of oil palm < water hyacinth < chicken manure. Increasing the amount of organic matter applied to reclaimed-mining soils also results in an increase in the value of  $Q_{max}$ . The effect of the type and amount of organic matter on the maximum adsorption capacity of reclaimed-mining soils to absorb Fe from acid mine drainage is related to increasing the number of functional groups in soils due to organic matter application. This study proves the addition of organic matter is required as substrates for increasing sorption sites in the management of acid mine drainage.

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