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## **Research Article**

# Reduction in acidity and heavy metal concentrations of acid mine drainage with organic matter and coal fly ash treatments in two different reclaimedmining soils

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#### Abstract

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Keywords: adsorption AMD remediation functional groups metal removal negative charges Organic matter (OM) has a very crucial role in the management of acid mine drainage (AMD) using a passive treatment system, although information on the use of this system in different reclaimed-mining soils (RMS) is very limited. Therefore, this study aimed to determine the effect of adding OM to RMS with different characteristics. It was carried out by adding only OM or in combination with coal fly ash (CFA) to two RMS with different characteristics (Palam and Cempaka Soils) and quartz sand (control) in a batch reactor experiment. This was followed by the incubation of the mixture of soil/quartz-OM or soil/quartz-OM-CFA at 60% water holding capacity for 15 days. After incubation, AMD slowly flowed into the reactor, and its pH in the reactor was monitored every day for 30 days, while the concentrations of Fe (iron), Al (aluminum), and Mn (manganese) were measured on the 30th day. The results showed that the application of OM on Palam Soil only increased AMD pH by 0.38 units, while Cempaka Soil and quartz sand increased by 4.83 and 5.36 units, respectively. The addition of OM to Cempaka Soil and quartz sand also showed a higher reduction in heavy metals concentration in AMD than those in Palam Soil. It was also discovered that the application of OM combined with CFA led to a higher improvement in AMD quality than only using OM. This study demonstrated that the effect of OM addition on increasing pH and decreasing metal concentration on AAT management with the passive treatment system is controlled by soil characteristics.

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#### Introduction

Acid mine drainage (AMD) resulting from chemical reactions of sulfuric compounds with oxygen is one of problems occurring in mining activities. Due to low pH and high concentration of heavy metals (Núñez-Gómez et al., 2019; Luo et al., 2020), AMD

has become a problem for human health, land, and water resources. Fernández-Caliani et al. (2019) showed high bio-accessibility As, Pb, and Ni on agricultural soils that are occasionally flooded by extremely acid mine waters. The release of AMD into agricultural soils causes a 62% decrease in production (Choudhury et al., 2017), while its flow to open waters

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disrupts benthic invertebrate populations (Mocq and Hare, 2018; Núñez-Gómez et al., 2019) and fish community (Talukdar et al., 2017; Lebepe et al., 2020). This shows the importance of the remediation of AMD to reduce the negative impact of mining activities on the environment.

A highly effective and easy-to-implement method for treating AMD is the application of lime (Kaur et al., 2018; García-Valero et al., 2020), although it is considered expensive and not economical. Another method that is often used is a passive treatment, in which AMD flows through substances that is capable of adsorbing or reducing H<sup>+</sup> ions or heavy metals (Neff et al., 2021). The remediation of AMD using passive treatment is controlled by the natural process of increasing and neutralizing acidity, as well as oxidizing or decreasing and precipitating the metal contents (Skousen et al., 2017). Apart from being cheaper than lime application, passive treatment is also less-operational and requires low energy requirements. Moreover, several studies demonstrated the high effectiveness of passive treatment in AMD remediation (Chen et al., 2020; Singh and Chakraborty, 2020).

The application of passive treatment in AMD management generally employs multiple techniques, frequently in sequences, to neutralize, oxidize, and precipitate ion H<sup>+</sup> and metals in AMD. Several biochemical mechanisms, such as adsorption, precipitation, in exchange, sedimentation, and complexation, play an important role in the remediation process (Sheoran and Sheoran, 2006; Skousen et al., 2017; Buxton, 2018). This method also increases low pH to a value above 6.0 (Clyde et al., 2016; Singh and Chakraborty, 2020), while the contents of total soluble Fe and soluble Fe(II) decreased by 93.7% and 99.0%, respectively (Chen et al., 2020). The application of organic matter (OM) in passive treatment stimulates the adsorption of metals in AMD, which leads to higher metal removal (Pat-Espadas et al., 2018; Chen et al., 2021). In addition, the use of several industrial wastes, such as blast furnace slag, coal fly ash (CFA), as well as paper and steel mills, increases the capability of passive treatment in remediating AMD (Moodley et al., 2018). These results showed that materials capable of enhancing biochemical processes of adsorption, precipitation, ion exchange, sedimentation, and complexation play an important role in controlling the capability of passive treatment for remediating AMD.

Several studies showed that the use of different OM and other materials in passive treatment systems resulted in a varying increase in AMD pH. Research conducted by Clyde et al. (2016) showed that a passive treatment system consisting of rock, peat filter, and limestone is able to improve pH from 4.5 to 6.5. In an investigation using a batch reactor system with a mixture of reclaimed-mining soils (RMS) and empty fruit bunch of oil palm (EFBOP), Noor et al. (2020) reported an increase in pH of AMD from below 4.0 to 7.0 within 15 days. Changes in pH and heavy metals concentrations of AMD in response to the mixture of different OM and admixtures are also observed in remediating AMD using column experiments (Vasquez et al., 2016; Dong et al., 2019). The results showed that the effectiveness of the remediation is controlled by the type of OM and materials, such as soils, CFA, and other ameliorants used in the passive treatment systems. This revealed that information on the effect of different soil characteristics on the capability of passive treatment systems 1 very important in AMD management. Therefore, this study aimed to determine the effect of OM and CFA applications on the improvement of pH and reduction in heavy metal concentrations of AMD in different RMS.

### Materials and Methods

#### Sampling and characterization of soil, organic matter (OM), coal fly ash (CFA), and acid mine drainage (AMD)

The soils used were sampled from two different RMS, namely diamond reclaimed-mining soil in Desa Palam (Palam Soil) and coal reclaimed-mining soil in Desa Tiung (Cempaka Soil), which are administratively located in Cempaka Sub-district, Banjarbaru City, South Kalimantan, Indonesia. Soil sampling on each RMS was carried out at a depth of 0-30 cm using a soil auger at different points. A fter cleaning from soil litter and gravel, the sampled soils were homogenized, put into plastic bags, air-dried, and stored at 4 °C until used for the experiment.

CFA was sampled from the disposal site of the PT Total Power Indonesia (TPI) power plant located in Desa Kasiyau Raya, Murung Pudak District, Tabalong 5 gency, South Kalimantan, Indonesia. Subsequently, the samples were air-dried, filtered through a 2 mm sieve, and stored at 4 °C before use. Quartz was collected from sand mining in Desa Landasan Ulin Selatan, Landasan Ulin Sub-district, Banjarbaru City, South Kalimantan, Indonesia. The collected quartz samples were first washed with 1.0 M HCl, followed by 1.0 M NaOH, and finally washed repeatedly with distilled water and oven-dried at 60 °C.

Before the experiment in the laboratory, the soil physico-chemical characteristics were quantified for each RMS, CFA, and quartz. The texture of each RMS soil was measured using the method described by Gee and Bander (1986), while bulk density (BD) of soils, CFA, and quartz was determined using the procedures of Blake and Hartge (1986). Chemical characterization for soils, CFA, and quartz included the determination of pH (McLean, 1982), organic carbon (C) content (Nelson and Sommers, 1996), total nitrogen (N) (Bremer and Mulvaney, 1982), and total phosphorous (P) (Jackson, 1967). Digestion of soil, CFA, and quartz, and the concentrations of Al (aluminum), Fe (iron),

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Mn (manganese), Ca (calcium), Mg (magnesium), Na (sodium), and K (potassium) in digested solution were quantified using atomic adsorption spectrophotometry (Shimadzu AA6300G). EFBOP was collected from the oil palm plantation of the Perkebunan Nusantara XIII at Ambungan Village, Pelaihari Darict, Tanah Laut Regency, Indonesia. The samples were oven-dried at 60 °C for 72 hours, fine-ground to a 2 mm size, and

quantified for lignin content (Chesson, 1981), carbohydrate (Grandy et al., 2000), organic C (Nelson and Sommers, 1996), and N (Bremer and Mulvaney, 1982). Meanwhile, AMD used for this study was collected from a diamond washing plant in Palam Village, Cempaka District, Banjarbaru City, South Kalimantan, Indonesia. Characteristics of soil, quartz, CFA, and OM used are described in Table 1.

Table 1. Characteristics of soils, quartz sand, CFA, and OM were used for the study. Numbers in the parenthesis represent the standard deviation of the mean (n = 3).

Characteristics	Palam Soil	Cempaka Soil	Quartz Sand	Coal Fly Ash	Empty Fruit Bunches of Oil
1		5011			Palm
Texture					
Sand (%)	20.88 (4.74)	23.92 (3.43)	-	-	-
Silt (%)	32.56 (5.76)	43.52 (2.21)	-	-	-
Clay (%)	46.56 (2.53)	32.56 (3.21)	-	-	-
$pH(H_2O)$	2.98 (0.34)	4.42 (0.32)	6.98 (0.45)	7.98 (1.12)	-
Bulk denaty (kg m <sup>-3</sup> )	1.34 (0.08)	1.18 (0.05)	2.23 (0.32)	1.97 (0.34)	-
Organic C (g kg <sup>-1</sup> )	15.74 (0.23)	9.43 (0.76)	0.23 (0.02)	4.56 (0.08)	422.54 (9.56)
N (g kg <sup>-1</sup> )	0.84 (0.08)	0.92 (0.07)	0.11 (0.03)	0.25 (0.10)	12.23 (4.32)
$P(gkg^{-1})$	7.54 (0.11)	4.65 (0.09)	0.14 (0.08)	2.56 (4.32)	-
Ca (mg kg <sup>-1</sup> )	3.67 (0.09)	3.98 (0.34)	0.12 (0.08)	947.21 (9.34)	-
Mg (mg kg <sup>-1</sup> )	4.76 (0.14)	5.76 (0.56)	0.47 (0.09)	1367.88 (7.76)	-
K (mg kg <sup>-1</sup> )	2.11 (0.18)	3.23 (0.23)	0.91 (0.21)	657.65 (6.54)	-
Na (mg kg <sup>-1</sup> )	1.98 (0.09)	1.32 (0.21)	0.12 (0.06)	456.45 (9.56)	-
Al (mg kg <sup>-1</sup> )	49.45 (4.56)	7.76 (0.87)	0.65 (0.06)	532.67 (8.76)	
Fe (mg kg <sup>-1</sup> )	51.34 (1.32)	5.76 (0.08)	0.98 (0.12)	342.45 (7.56)	
Mn (mg kg <sup>-1</sup> )	9.45 (0.34)	4.65 (0.54)	0.00 (0.00)	198.45 (6.57)	
CEC (cmol kg <sup>-1</sup> )	29.87 (4.33)	18.56 (5.43)	3.23 (0.76)	-	-
Carbohydrate (g kg-1)	-	-	-	-	32.43 (4.22)
Lignin (g kg <sup>-1</sup> )	-	-	-	-	96.67 (8.22)

#### Laboratory experiment

The influence of OM and CFA application on the remediation of AMD was quantified through a batch reactor experiment. The treatments consisted of (1) quartz (without treatment), (2) quartz + OM, (3) quartz + OM + CFA, (4) Palam Soil, (5) Palam Soil + OM, (6) Palam Soil + OM + CFA, (7) Cempaka Soil, (8) Cempaka Soil + OM, and (9) Cempaka Soil + OM, in which each treatment had three replicates. A 2,000 g of each soil and quartz was put in a plastic reactor (35 cm x 15 cm x 10 cm), followed by OM and CFA (according to the treatments), each in an amount equivalent to 200 Mg ha-1 in field application was added to the reactor, and mixed homogeneously. Subsequently, free ionized water was added to each reactor to obtain 60% water holding capacity, and the mixture of soil/quartz-OM-CFA was incubated for 15 days. Sub-sampling of approximately 200 g of soil was carried out for each reactor at the end of incubation to measure pH (McLean, 1982), specific surface areas (Sepaskhah et al., 2010), cation exchange capacity (CEC) (Rhoades, 1982), and total function groups (Kim and Park, 2016). After incubation, AMD flowed slowly into each reactor until the surface reached a height of 3 cm from the soil/quartz level in the reactor. The pH in the reactor was measured on the first and second days, followed by every 2 days after AMD flowed into the reactor for 30 days using a portable pH meter (Hanna HI98190). The measurements of metal concentrations were carried out after 30 days by collecting 50 mL of treated AMD from each reactor, which was filtered using a 0.45 m syringe filter (MF-Millipore® Membrane Filter). The concentrations of Fe, Al, and Mn in treated AMD were quantified using atomic absorption spectrophotometry (Shimadzu AA6300G).

# Data analysis

Analysis of variance was performed to quantify the effect of OM and CFA application on changes in pH and metal concentrations in treated AMD. The Bartlett and Shapiro-Wilk tests were carried out to ensure all collected data had homogeneous variance arg normal distribution, respectively. Subsequently, the least significant difference (LSD) test at p<0.05 was carried out for the treatments that had a significant effect on the observed variables. All statistical analyses were conducted using GenStat 12<sup>th</sup> Edition.

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#### **Results and Discussion**

# Characteristics of soils, quartz sand, coal fly ash (CFA), and organic matter (OM)

The soils used in this study had different textures, where Palam and Cempaka Soils had clay and clay loam texture, respectively (Table 1). The organic C content in Palam Soil was almost twice that of Cempaka Soil. However, Cempaka Soil had a higher pH than Palam Soil. The content of Al, Fe, and Mn as well as CEC in Palam Soil were also high (Table 1). These results indicated that both soils have different characteristics.

Quartz sand had a neutral pH of 6.98 with a high BD of 2.23 g cm<sup>-3</sup>. The contents of elements such as N, P, K, Ca, Mg, Na, K, Al, Fe, and Mn in the quartz sand used were very low (<1.0 mg kg-1) (Table 1). The EFBOP used had a very high organic C content of 423 g kg<sup>-1</sup> and a low N content of 12 g kg<sup>-1</sup>; therefore, the C/N ratio was relatively high (35). The results of the analysis showed that EFBOP had a higher lignin content than carbohydrates (Table 1). CFA showed an alkaline pH of 6.98 and had a high density of 1.97 kg m-3. The organic C content, total N and P in CFA were also very low, reaching only 4.56 g kg<sup>-1</sup>, 0.25 g kg<sup>-1</sup>, and 2.56 g kg-1, respectively. The favorable characteristic of this coal ash was the relatively high contents of Ca, Mg, Na, and K cations, in which the Ca and Mg contents reached 947.21 mg kg-1 and 1367.88 mg kg-1, respectively. High Al and Fe contents were also observed, which were 532.67 mg kg-1 and 342.67 mg kg-1, respectively. The characteristics of CFA used in this study are in line with the one used as a soil ameliorant for RMS improvement (Oklima and Suryaningtyas, 2015).

## Effect of organic matter (OM) and cash (CFA) application on changes in pH of acid mine drainage (AMD)

The results showed that the pH of AMD in untreated Palam Soil was within the range of 2.27-2.78 (Figure 1). The addition of OM to Palam Soil led to a small increase in AMD pH by 0.38 units, from 2.78 to 3.16. However, the AMD showed a very significant increase from 2.78 to pH 8.19 when OM in combination with coal ash was applied to Palam Soil (Figure 1 and Figure 2A). These results indicate the dominant effect of CFA application on the increase in AMD pH. Alkali elements such as CaO and MgO in CFA play an important role in AMD remediation. The hydroxide ion formed from the dissolution reaction of CaO and MgO neutralizes H+ ions from AMD (Gitari et al., 2008; Jones and Cetin, 2017). The results of this study are in accordance with previous studies that showed increasing pH of AMD with CFA application (Kalombe et al., 2020; Mahedi et al., 2020).

In contrast to Palam Soil, the OM application to Cempaka Soil and quartz sand was able to suppress AMD acidity. The addition of OM increased AMD pH

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from 2.78 to 7.61 and from 2.78 to 9.15 when the OM was combined with CFA (Figures 1 and 2A). In quartz sand, the pH increased to 8.14 with the addition of OM and 9.07 when OM was applied with CFA. These results are in line with previous studies that the application of OM in a passive treatment system of AMD management increased the pH of AMD (Noor et al., 2020; Thisani et al., 2021). The presence of OM can suppress the acidity of AMD thing gh the decomposition process of OM, releasing functional groups such as carboxylic and phenolic groups, which are negatively charged and react with positively charged hydrogen ions from AMD. In this study, the total functional groups increased from 0.09-0.74 mmol g<sup>-1</sup> to 6.94-12.55 mmol g<sup>-1</sup> with OM application (Table 2). Saidy et al. (2021) reported that the addition of OM with different amounts and types in the RMS increased the total functional group of OM by 365-696%. The neutralization reaction of H+ ions by functional groups of OM led to a decrease in H<sup>+</sup> ions in AMD, which in turn increased the pH. This indicated the important role of functional groups resulting from the decomposition of OM in improving the pH of AMD.

The results showed that changes in AMD pH of Palam and Cempaka Soils responded differently to the addition of OM. The application of OM to Palam Soil increased the AMD pH from 2.25 to 3.16, while that of Cempaka Soil increased from 3.66 to 7.61 with the OM addition (Figure 2A). The differences in the change in pH due to the application of OM are related to the variations in soil pH, Al, and Fe contents in both soils. The pH of Cempaka Soil was higher than that of Palam Soil, while Al and Fe contents were higher in Palam Soil (Table 1). The elements Al and Fe function as a source of soil acidity by producing H+ ions through hydrolysis reactions (Cook et al., 2000; Strawn et al., 2015). The number of H+ ions in Palam Soil-AMD increased with the hydrolysis reaction of these elements from the soil. This means only a small part of H<sup>+</sup> ions could be neutralized by functional groups of OM, which cause a little change in the pH of AMD in Palam Soil. Meanwhile, Cempaka Soil with low Al and Fe contents contributed small addition of H+ ions in Cempaka Soil-AMD and was largely neutralized by functional groups of OM, which in turn resulted in a significant increase in AMD pH.

#### Effect of organic matter (OM) and coal fly ash (CFA) application on the removal of heavy metals in acid mine drainage (AMD)

The application of only OM or in combination with CFA caused a decrease in Al, Mn, and Fe concentrations in AMD. It reduced Al concentration by 34-57% in quartz/control, Palam Soil, and Cempaka Soil, which increased to 72-90% when OM was combined with CFA (Figure 2B). The application of OM and CFA led to a decrease in the concentration of Fe in AMD from 2.25-5.25 mg L<sup>-1</sup> to 0.32-3.62 mg L<sup>-1</sup>, while OM reduced the Fe to 0.65-4.37 mg L<sup>-1</sup> (Figure 2C).



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Figure 1. Changes in pH of acid mine drainage in response to OM and CFA application and without OM and CFA application (S) on Palam Soil, Cempaka Soil, and quartz (control).

Similarly, the concentration of Mn in AMD reduced by 22-83% from the initial value with the OM in quartz/control, Palam Soil, and Cempaka Soil, while Mn concentrations were reduced by 85-92% due to the addition of combined OM and CFA (Figure 2D). These results indicated that the reduction in heavy metal concentrations was optimal when OM was applied together with CFA in remediating AMD.

The decreasing concentrations of Fe, Al, and Mn in AMD with the addition of OM are related to the reaction (adsorption process) between heavy metals, which had a positive and a negative charge on the soil minerals. The negative charge of the soils increases with the presence of functional groups derived from added OM to the soils. A previous report showed that the adsorption of heavy metal onto soil mineral surfaces is determined by the number of negative soil charges (Choppala et al., 2018; Kalombe et al., 2020). Increasing negative charges of the soil in this study were indicated by the increase in CEC with the addition of OM. The addition of OM increased the soil CEC in Palam and Cempaka Soil from 17.33-29.92 cmol kg<sup>-1</sup> to 13.49-35.39 cmol kg<sup>-1</sup>, respectively (Table 2). OM 6 ays an important role in increasing the negative charge of the soil through the production of carboxyl and phenolic groups from the microbial decomposition of OM (Huang et al., 2019). Increasing the negative charge of the soil also stimulates the adsorption of positively charged metal onto soil minerals, which reduces the amount of metal in the AMD.

Changes in the concentration of heavy metals in Palam and Cempaka Soils showed different responses to the addition of OM. The application of OM to Palam Soil led to a decrease in Fe, Al, and Mn concentrations in AMD by 1%, 34%, and 22%, respectively, while the concentrations of these metals in AAT in Cempaka Soil decreased to 71%, 57%, and 83% (Figures 2B, 2C, and 2D). This difference is due to the variations in the soil characteristics of both samples. The contents of

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Fe, Al, and Mn in Palam Soil are higher than those in Cempaka Soil (Table 1); therefore, more OM is required for the sorption process of the metals. This leads to the OM addition to Palam Soil causing a smaller decrease in metal concentration than that of Cempaka Soil. The results also indicated that the effect of OM addition on the reduction in metal concentrations of AMD is determined by the characteristics of the soil used in the passive treatment system.





Previous studies also showed a decrease in metal concentrations of AMD with the OM addition. Lazareva et al. (2019) reported that the metal adsorption reaction of AMD with natural OM decreased metal concentration in AMD. Arce et al. (2017) showed an increasing formation of organicmetal aggregate complexes in metal neutralization in AMD with the OM addition. The results indicated the important role of OM in reducing metal concentrations in AMD by releasing functional groups. In this study, it was discovered that the CFA and OM addition gave a greater reduction in heavy metal concentrations in AMD than those using only OM, as presented in Figures 2B, 2C, and 2D. This indicated the significant contribution of CFA in reducing AMD metal concentrations using a passive treatment system. The contribution of CFA in increasing the sorption of heavy metals from AMD is related to the presence of FeO, AIO, and other oxides that increase the specific surface areas.

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Treatments	Soil pH	Soil CEC	<b>Total Functional</b>	Surface Areas
		(cmol kg <sup>-1</sup> )	Groups (mmol g <sup>-1</sup> )	(m <sup>2</sup> g <sup>-1</sup> )
Control				
Quartz	6.25 (0.32) a*	1.99 (0.62) a	0.09 (0.01) a	0.19 (0.04) a
Quartz+OM	8.32 (0.54) b	14.11 (3.06) b	12.55 (1.75) b	0.16 (0.05) a
Quartz+OM+CFA	9.24 (0.47) c	18.45 (1.42) c	14.76 (1.33) c	9.00 (1.18) b
Palam Soil				
Soil	2.37 (0.45) a	17.33 (0.85) a	0.74 (0.21) a	12.83 (1.31) a
Soil+OM	3.38 (0.07) b	23.49 (1.80) b	6.94 (1.25) b	11.83 (1.95) a
Soil+OM+CFA	8.65 (0.24) c	28.84 (0.54) c	8.69 (1.73) c	18.42 (0.94) b
Cempaka Soil				
Soil	3.66 (0.65) a	29.92 (0.21) a	0.25 (0.07) a	9.07 (0.86) a
Soil+OM	7.73 (0.24) b	35.39 (1.45) b	8.31 (0.65) b	9.44 (0.70) a
Soil+OM+CFA	9.40 (0.98) c	41.08 (0.54) c	10.75 (1.46) c	16.37 (0.72) b

Table 2. Changes in selected soil characteristics in response to OM and CFA application. Numbers in the parenthesis represent the standard deviation of the mean (n = 4).

\* Similar letters in each column indicate an identical effect of the treatments for each soil/control based on the LSD test at p<0.05.

The application of OM and CFA to RMS also increased the specific surface area by 44-81% (Table 2). This is in line with Saidy et al. (2020), which stated that the specific surface area is increased by applying CFA to RMS. Similarly, investigations showed that the application of CFA increases the adsorption of metals or other elements onto soil minerals (Mujtaba-Munir et al., 2020; Shirin et al., 2021). Kalombe et al. (2020) revealed the high effectiveness of CFA in reducing metal contents such as nickel(II), copper(II), zinc(II), Al, and Fe from the solution. The results showed that an increase in reactive sites in soil minerals with the CFA addition improves the metal sorption process onto soil minerals, which significantly reduces the metal concentration in AMD.

#### Conclusion

The results showed that the application of OM to RMS, which was then drained by AMD increased the pH and decreased the concentration of Fe, Al, and Mn in AMD. The effect of adding OM on increasing pH and decreasing metal concentrations in AMD is controlled by the characteristics of the soil used in AMD management with a passive treatment system. The functional groups resulting from the decomposition of OM neutralize H<sup>+</sup> ions and adsorb positively charged metals in AMD, leading to an increase in pH and a decrease in metal concentration. The results also showed that the addition of OM and CFA resulted in a significant decrease in acidity and metal concentrations in AMD, compared to the addition of only OM. The contribution of CFA to the reduced acidity occurs through the dissolution of Mg and Ca oxides, which produce hydroxyl ions and neutralize the acidity in AMD. Furthermore, the addition of CFA to RMS increased the surface area of the CFA-soil complex, which in turn increased the metal sorption process of AMD onto soil minerals. The results of this study indicate that remediating AMD with the addition

of OM produces optimal results when applied in combination with CFA.

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