Iron removal and pH stabilization of synthetic acid mine drainage (AMD) using peat soil and limestone.

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Abstract

A Successive Alkalinity Producing System (SAPS) was evaluated for the remediation of synthetic Acid Mine Drainage (AMD) by using an organic substrate of peat soil and limestone aggregate. The characterization of the AMD was conducted by the testing the AMD effluent originated from active mining site at Pengkalan Hulu, Perak, Malaysia. The characterization of the peat soil and limestone revealed stipulated composition, carbon content in the peat soil and particle size distribution of the limestone. Synthetic AMD was prepared incorporating iron sulphate (FeSO_4) and sulphuric acid (H_2SO_4). The synthetic AMD was acidic ($\text{pH} < 4.0$) and with 50, 75 and 100 ppm Iron (Fe) concentration. A laboratory scale physical model to simulate a tailing pond was developed. The synthetic AMD was introduced and passed through the filtration media. Subsequently, water samples were collected and analyzed to determine pH level. Additionally, the Fe concentration was analyzed using UV-Vis test at 6 to maximum 48 hours' retention time. The residues of the peat soil and limestone from the physical model were further analyzed using SEM-EDX microscopic to determine the remaining iron content absorb or precipitate onto the peat soil and limestone. The analysis revealed that the innovative methodology has successfully reduced more than 85 % iron content and neutralized the pH only after 6 hours of retention time. These results proved the combination of peat soil and limestone potentially can be proposed as the alternate solution for treating the AMD effluent from the mining site.

Introduction

Acid mine drainage (AMD) is an environmental pollutant that impairs water resources due to anthropogenic activities including agriculture plantation, mining and logging. Unfortunately, mining industry often be criticised as a major contributor of the AMD. Exploitation of valuable minerals due to huge demand has unsuspectedly urged uncontrolled mining activities. These uncontrolled and some illegal mining activities escalate serious environmental problems that degrade most of the mining land area, surface water quality (stream and river), and eventually affected the quality of underground water reservoir. Previous study showed mining as a major source of heavy metal contaminations in the water nationwide (Affandi and Ishak 2018). This problem occurred due to formation of the AMD and released into adjacent water bodies from mining sites without proper and effective treatments.

In general, the reactions of the AMD formation are best illustrated by examining the oxidation of pyrite (FeS_2). The oxidation of pyrite occurred when the mineral surface is exposed to an oxidant and water either in oxygenated or anoxic systems (Blowes et al. 2013). The presence of oxygen and water, pyrite oxidizes to form ferrous (Fe^{2+}), sulphate ion (SO_4^{2-}) and hydrogen (H^+) ion (Equation (1)) (RoyChowdhury et al. 2015). The dissolved Fe^{2+} , SO_4^{2-} , and H^+ represent an increase in the total dissolved solids and acidity of the water (Akcil and Koldas 2006).

Then, the Fe^{2+} ion produced reacts with oxygen (O_2) to form ferric ion (Fe^{3+}). If oxygen is low, the chemical reaction in Equation (2) will not occur until the pH reaches 8.5 because the conversion of ferrous to ferric is slow at pH values equal to five and lower under abiotic conditions (Simate and Ndlovu 2014). This reaction is facilitated by the sulphur-oxidizing bacteria (*Thiobacillus thiooxidans*, *Thiobacillus ferrooxidans*) as they utilize the produced energy from this reaction for their metabolism (RoyChowdhury et al. 2015).

Due to the production of H^+ ions, the pH of the whole system drops drastically and instantly becomes highly acidic. If the pH of the system remains over 3.5 to 4.0, Fe^{3+} precipitates in the form of iron hydroxide, $\text{Fe}(\text{OH})_3$ (RoyChowdhury et al. 2015). While, according to Akcil and Koldas 2006, the Fe^{3+} precipitates as $\text{Fe}(\text{OH})_3$ and jarosite at very low pH values between 2.3 and 3.5. The precipitation of insoluble $\text{Fe}(\text{OH})_3$, also release additional acidity as explained in Equation (3).

Over centuries, researchers dedicated their time to overcome problems created by mining industry. However, some of the treatment methods are too expensive to be adopted in mining operation due to high capital cost and operation cost (Johnson and Hallberg 2005). The conventional method by constructing series of retention pond and adding hydrated lime into the AMD was proven successful to reduce heavy metal concentration and increase pH to the almost neutral level. Unfortunately, the timely maintenance and operation costs are immense. Additionally, this traditional method caused excessive hydrated lime usage; mass production of sludge with low cost-benefit ratio.

Currently, the industries adopted two AMD treatment methods known as active and passive treatments. The latter is the interest of the research. A Successive Alkalinity Producing System (SAPS) is one of the most commonly used passive treatment systems for AMD treatment in recent years. It is a hybrid treatment system incorporating technical merits of anaerobic wetland and anoxic limestone drain (Yim et al. 2015). Three major system elements of this system are the drainage system, a limestone layer and organic layer (Zipper et al. 2011).

Few studies to date have investigate how to improve the effectiveness of SAPS using different organic substrate. Some organic substrate commonly used by researchers are mushroom spent compost, wood shaving and animal manure. Consideration of the organic substrate used in SAPS based on carbon content and absorption ability. Mashalane et al. 2018, used animal manure and sewage sludge in their study manage to remove 90 % of Fe. On the other hand, spent mushroom compost used by Yim et al. 2015 also recorded 90 % Fe removal.

In this research work, passive treatment system of SAPS was adopted and tested on a laboratory scale using peat soil as organic substrate. The objective of this research work is to quantify the effectiveness of the peat soil for AMD effluent treatment. This research looks further into the application of the organic-based material as a treatment media. The peat soil was introduced as an alternative to conventional treatment media such as mushroom spent, wood shaving and animal manure.

Methods

Materials/chemicals

Peat soil (from Simpang Ampat, Penang, Malaysia), limestone aggregate (Lime & Lime Quarry, Baling, Kedah, Malaysia) Iron sulphate (FeSO_4) (Merck, 99 %), sulphuric acid (H_2SO_4) (Merck, 95–97 %) and nitric acid (HNO_3) (Merck, 65 %).

Analytical Methods

AMD characterization

Six main rivers were identified and affected by these profitable mining activities. The AMD effluent samples were collected and analysed and the sampling locations are summarized in Table 1.

Table 1
Sampling point of the river water

Sampling point	River	Coordinate	
		Latitude	Longitude
S1	Kijang River	5°38'44.3"N	101°01'44.4"E
S2	Buluh Reservoir	5°38'44.3"N	101°01'44.5"E
S3	Belimbing River	5°37'36.5"N	101°04'41.6"E
S4	Rui River	5°35'34.1"N	101°03'39.1"E
S5	Kuak River	5°37'44.1"N	101°04'45.4"E
S6	Duri River	5°37'36.5"N	101°04'41.6"E

The samples were successfully tested between March 2019 to April 2019, three sampling for each station. The sampling was conducted during day time between 11 am to 5 pm on 14 March 2019 for the first sampling. Second sampling was carried out on 28 March 2019, while the third sampling happened on 11 April 2019 (after raining event). Both in-situ test and ex-situ test were conducted for analysis purposes. The water samples were kept in 1-L polyethylene bottles, soaked into 10 % of nitric acid overnight and rinsed with deionized water. This method ensures clean and uncontaminated samples. The samples that need to be taken to laboratory have been preserved to make sure the samples are

unchanged from its original state. The samples were immediately acidified by adding HNO_3 to ensure pH lower than two (Affandi and Ishak 2018).

In-situ test was conducted on site to identify pH value, temperature and dissolved oxygen (DO). The reading of pH value and temperature were recorded using YSI Multiparameter. Subsequently, ex-situ tests were conducted in the laboratory for further analysis to determine heavy metal concentrations using Perkin Elmer ICP-OES. The heavy metals that have been detected by ICP-OES are Cadmium (Cd), Copper (Cu), Plumbum (Pb), Zink (Zn), Manganese (Mn), Magnesium (Mg) and Iron (Fe).

Peat soil characterization

The peat soil sample is collected from Simpang Ampat, Penang, Malaysia. The peat soil contains nutrient-rich humus compound, red burnt soil, charcoal and river sand. The Carbon, Hydrogen, Nitrogen and Sulphur (CHNS) analysis (Perkin Almer Series II CHNS/O Analyzer 2400) and SEM-EDX (Gemini-Zeiss SUPRA 35VP and EDAX AMETEK-Z2) analysis were conducted to characterize basic compositions of the peat soil especially the carbon content.

The samples were grounded using a ring mill and sieved into five different sizes at 425 μm , 300 μm , 225 μm , 150 μm and 75 μm (Hwidi et al. 2018). The particulate sample at 75 μm was taken for the CHNS and SEM-EDX analysis.

Limestone characterization

Limestone characteristic is one of the main factors that determine the effectiveness of passive treatment. Therefore, the limestone characterization must be conducted comprehensively. In this research, the limestone originated from a limestone quarry in Baling, Kedah, Malaysia was assessed in detail. Limestone composition analysis reveals the percentage of CaCO_3 . Ideally, the limestone should be at least 90 % CaCO_3 and should have maximum size of 1.3–1.9 cm (Bhattacharya et al. 2008). On the other hand, Zipper et al. 2011 suggested that the systems are built with high-calcium (> 90 %) limestone in the 10 to 15 cm size range. However, other researchers, Mashalane et al. 2018 used limestone with 85 % of CaCO_3 in their study of passive treatment system in Carolina, Mpumalanga.

The limestone sample has been collected at quarry production stockpile via grab sampling method. About 1200 gram of limestone aggregate was used for size distribution analysis. Eight different size fractions of sieve were selected which are 20, 14, 10, 6.3, 4.75, 2.8, 2.3 mm and pan. Sieve results are reported as the differential weight percentage retained on each sieve as well as percentage of cumulative weight less than the sieve size.

Synthetic AMD composition

The system consists of peat soil as organic substrate and limestone aggregate to treat the synthetic AMD with low pH level (below 4) and high iron concentration (50 to 100 ppm). The simulated AMD used in this study containing high acidity (pH 3 to 4), high concentration of Fe and sulphate. Distilled water was used

due to its purity and less contaminant compared to tap water. The distilled water was collected in a 60 L container. The H_2SO_4 was pipetted approximately 2.3 ml and dissolved into 60 L container.

Then, to prepare concentration of Fe as heavy metal in the synthetic AMD, FeSO_4 powder was incorporated accordingly. The consumption of FeSO_4 is shown in Table 2. The FeSO_4 was weighed and quantitatively transferred into the 60 L acidic solution and stirred thoroughly to make up the solution with respectively contained 50, 75 and 100 ppm of Fe concentration.

Table 2
Weight of the FeSO_4 for 60 L

Fe conc., ppm	FeSO_4 , g
50	8.160
75	12.240
100	16.321

Lab scale configuration

The experiment was setup using transparent polypropylene tanks. The synthetic AMD was prepared in 60 L solution tank to supply the synthetic AMD solution to SAPS tank (Tank A and Tank B) by gravitational flow as shown in Fig. 1. The dimension of the SAPS tanks are 45 cm length, 32 cm width and 27 cm depth. The limestone aggregate was packed at the bottom of the SAPS tanks and covered the effluent pipe (Fig. 2). Subsequently, peat soil was placed over the limestone. As suggested by Zipper et al. 2011, no compact and physical disturbance on the organic layer to prevent creation of zones of preferential vertical flow, that will ‘short circuit’ the system and decrease treatment effectiveness. Total thicknesses for both media is 15 cm height. The synthetic AMD height was set constant at 5 cm throughout this study and was gravitational supplied from solution tank to SAPS tanks through influent pipe at the top of the tank. Then the synthetic AMD was flown downward through the peat soil before contact with limestone before being discharged thru effluent pipe.

SAPS operation

Six experimental set up were conducted. Tank A and Tank B were running simultaneously for each run with same influent Fe concentration and pH level. The treated solution was collected at effluent pipe within 6 hours’ interval of retention time. During effluent water sample collection, pH and temperature were measured in-situ and 15 ml of the sample was collected to measure the Fe concentration by using Ultraviolet-Visible (UV-Vis) spectrophotometry. The actual pH level and Fe concentration during experiment is shown Table 3.

Table 3
Actual Fe concentration and pH level during experiment.

Experiment	Tank	Peat soil depth, cm	Fe conc., ppm	pH
Experiment 1	A	5.0	57.35	3.40
	B	10.0		
Experiment 2	A	5.0	53.62	3.77
	B	10.0		
Experiment 3	A	7.5	79.82	3.83
	B	7.5		
Experiment 4	A	7.5	73.30	3.83
	B	7.5		
Experiment 5	A	5.0	95.36	3.84
	B	10.0		
Experiment 6	A	5.0	94.20	3.64
	B	10.0		

Result And Discussion

AMD characterization

Generally, the pH levels for the collected sample were between 6 to 8 except for pH reading for first sampling at Station 5 with pH equal to 4.70 due to discharge of AMD from several mine sites into the streams which eventually connected to Kuak River. Another reason that related to this finding is s due to logging and agriculture activities along the river. The ground that contains sulphide mineral was exposed, oxidised and formed AMD.

Most of the heavy metal concentration were found exceeding the allowable limit by the Mineral Development (Effluent) Regulations 2016 except for the Mg element. Based on previous geological study at this area conducted by Sapari et al. 2016, Mount Paku located at Pengkalan Hulu was traced with major elements such as As, Fe, Cu and Ti. Other elements such as Pb, Bi, Sb, Ba, Cr and Co were observed as well. Normally, the traced elements found as complex Fe-Cu-As-Bi-Sb-Pb bearing minerals. Other study also found that Fe, Mn, As, Al and Ni concentrations have exceeded the recommended Ministry of Health (MOH) guidelines (Affandi and Ishak 2018).

Heavy metal concentration for the third sampling is higher and mostly exceeded the limit. The highest Fe concentration reading was recorded is 188.5870 ppm (S3-3; Station 3, third sampling), As is 1.13 ppm (S6-3), Zn is 22.668ppm (S2-3), Pb is 114.871 ppm (S4-3), Cu is 6.107ppm (S5-3) and Cd is 2.492ppm (S5-3).

During the third sampling day, it was raining the night before, which caused higher heavy metal concentration as compared to the first and second sampling. This proved that conventional method of the AMD treatment via retention pond and hydrated lime dosing are not effective during raining day. Overflow of the AMD at the discharge point or lack of efficiency of the retention pond in term of design volume to trap heavy metal caused the problems. By adopting this conventional method, the AMD is treated by the settlement of heavy metal inside the retention pond. In this case, during the raining day, the flowrate of the AMD to be treated will increase and the heavy metals are not having enough time to settle hence, it will discharge out as untreated effluent with high concentration of heavy metals.

Peat Soil Characterization

The carbon content of the peat soil from CHNS analysis is show in Table 4. The peat soil sample can be classified as low carbon content compare to other organic substrate. Previous study was reported with 25.6 % carbon content by using mushroom compost (Yim et al. 2015).

Table 4
Peat soil composition
by CHNS analysis.

Element	Wt %
Carbon	10.693
Hydrogen	1.2393
Nitrogen	0.173

The carbon content recorded from EDX analysis is 6.24 % (Fig. 3). Major element in the peat soil is oxygen (O) 50.01 % followed by silica (Si) 25.18 %.

The higher carbon content of organic substrate, the higher the effectiveness of sulphate reducing medium to oxidize sulphide minerals via precipitation reaction. Carbon exist to provide bacteria to utilize dissolved oxygen in the AMD as energy source.

Limestone characterization

Figure 4 shows the image of the limestone at 100 and 500 times of magnification via SEM, respectively. The close up indicates the existing of the porous structures on the limestone surface. The porosity of the limestone is vital where precipitation of heavy metal such as iron hydroxide (Fe(OH)₃) expected to occur with aid of the porous structure.

The EDX analysis identified multiple mineral elements in limestone sample as the result shows in Fig. 5. Ca is the most abundant element (70.51 %) followed by O (26.31 %). Chemical composition of the limestone from XRF analysis is summarised in Table 5. The highest compound contained in the limestone is a CaO which is 98.55 %. Other compounds are too marginal less than 1 %.

Table 5
Composition of the limestone via XRF analysis.

Compound	Wt. %
SiO ₂	0.61
TiO ₂	< 0.1
Al ₂ O ₃	0.14
Fe ₂ O ₃	0.18
MgO	0.45
CaO	98.55
K ₂ O	0.54
P ₂ O ₅	< 0.10
Total	100

Figure 6 shows cumulative weight passing vs size from the particle size distribution analysis. It was noted that D80 was at size 18 mm and D50 was at size 14 mm, respectively. Only 20 % from the limestone sample is more than 18–20 mm and 50 % of the total limestone sample is more than 14 mm. Therefore, most of the limestone aggregates are within 10 to 20 mm. Limestone size range in a passive treatment study is not specific, it is totally depending on tailing pond size or pilot column set up. Previous studies reported various size ranges. For instance, Zipper et al. 2011, in his paper stated that limestone size in actual design of SAPS that has been used was 100 mm to 152 mm. In contrast, Yim et al. 2015 via column set up that almost similar to this study adopted coarse gravel limestone particle size between 20 to 30 mm.

Temporal changes of pH level and Fe concentration

In all experiments, regardless of Tank A or Tank B, the pH level and Fe concentration of effluents transformed from strong acidic and high Fe concentration to neutral and low Fe concentration solution, respectively. There are two major factors contributed to the changes; alkalinity generation by sulphate reduction bacteria (SRB) activity in organic substrate and dissolution of limestone (CaCO₃). According to

Kastyuchik et al. 2016, Ca is the most important acid neutralizing element. Meanwhile, Fe removal mechanism could be via adsorption on to the surface of organic substrate, precipitate as iron sulphide (FeS) and precipitate as iron hydroxide (Fe(OH)₃). Both organic substrate and limestone layer play an important role in this system to increase pH level and to remove Fe concentration of the synthetic AMD.

After 6 hours of retention time, pH level in all experiments increased drastically as shown in Fig. 7. Considering during this period, it is likely that alkalinity is generated by SRB activity. Most of the SRB grow optimally between pH levels of 6 to 8. Beyond this pH range, there is a decline in microbial sulphate reduction rate (Ayangbenro et al. 2018). Reaction between SO₄²⁻ and organic matter produces bicarbonate (HCO₃⁻). However, the pH level for Experiment 1, 2 and 6 were dropped at 12 hours onwards while the pH level in Tank B for the experiments were maintained at higher level than. The result proved that sufficient amount of the organic compound could allowed SRB activity to generate alkalinity and maintain the pH at neutral level for long period. According to the study conducted by Yim et al. 2015, alkalinity generation rate progressively increase with the height of the organic substrate layer not the volume of the substrate. The researcher also suggests that acid neutralizing capacity must be evenly distributed along the height of the organic substrate material.

Other than that, primary factor governing alkalinity generation is the rate at which limestone dissolved, which is effected by solution chemistry (Zipper et al. 2011). Considering this reaction happen at final stage of the SAPS prior discharged out the synthetic AMD solution as effluent. Limestone is expected to dissolve most rapidly during the first few hours of AMD contact as CaCO₃ dissolved more rapidly at lower pH and happened within first 6 hours of retention time. The pH level of Tank A in Experiment 1, Experiment 2 and Experiment 6 gradually declined after 6 hours suggesting that the synthetic AMD in contact with the limestone became saturated with dissolved Ca²⁺ and HCO₃⁻ and the rate of limestone dissolution reduced considerably. During this period, the SRB activity in Tank A do not take place to generate alkalinity where the organic functionality is expected to degrade over time, due to microbial biodegradation (Zipper et al. 2011). Besides, decreases of pH level in Tank A is due to precipitation of Fe(OH)₃ by reacting with oxygen in limestone layer where the reaction produced H⁺ ion.

Removal of Fe concentration associated with alkalinity generation from the SRB activity and limestone dissolution. The main Fe removal mechanism via adsorption on the surface of organic substrate, precipitation of Fe sulphides (FeS) due to sulphate reduction reaction and Fe(OH)₃ where the formation process is occurs rapidly when pH equal to 4 or higher (Ji et al. 2008). From the results of all experiments, Fe removal were exceptional even some experiments recorded different Fe concentration reading between Tank A and Tank B. Influent Fe concentration and peat soil depth influences system performance toward sustainability of the Fe removal.

Based on Experiment 2, the Fe concentration of Tank A suddenly increased at 24 hours' retention time but then declined again. This trend suggests that additional Fe element was leached out from the limestone

that contains Fe_2O_3 compound (0.18 %). There might be more supply of Fe to outcompete the Fe removal process such as absorption or precipitation.

Differing to temporal changes of Fe concentration for Tank A in Experiment 6, where the Fe concentration at 36 hours of retention time was significantly increased until at the end of experiment. This can be considered to be a result of ferric (Fe^{3+}) precipitate as $\text{Fe}(\text{OH})_3$ as such Fe^{3+} underwent reduction to Fe^{2+} and was released into aqueous solution (Clyde et al. 2016). When AMD introduced and in contact with an organic substrate (peat soil), dissolved metals precipitate as metal hydroxides by alkalinity produced from the organics matter. The reaction of $\text{Fe}(\text{OH})_3$ reduction to Fe^{2+} process is shown in Equation (5) below where electron (e^-) represents as electron donor, which is generally supplied by an organic substrate (Johnson and Hallberg 2005).

Other reason of Fe concentration increment is due to availability of the organic surface for absorption mechanism of the Fe. Since the peat soil depth for Tank A in Experiment 6 was only 5 cm and entertained high Fe concentration of synthetic AMD influent, the Fe removal efficiency would gradually decrease over time because of the available surface for absorption on the peat soil becomes smaller and limited. Besides, increased of Fe concentration and fail to perform to remove Fe over long-term due to exhaustion of the substrate required for sustaining the SRB.

Conclusion

The characteristic of peat soil and limestone play an important role on pH neutralization and Fe removal. The porosity structures of peat soil give an advantage for Fe absorption. Besides, presence of SRB in the peat soil give an aid on pH neutralization other than limestone dissolution.

The peat soil thickness was identified as one of the major parameter for the SAPS sustainability. Sufficient volume of the peat soil could increase longevity of SRB activity during SAPS treatment.

The lab scale study of SAPS for the treatment of synthetic AMD using peat soil and limestone successfully demonstrated that the system was capable to renovated the pH level to neutral level and reduce Fe concentration. However, the study of retention time need to be explore more in detail for future study to determine the optimum hydraulic retention time for the SAPS.

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Figures

Figure 1

Plan view schematic diagram of the experiment setup

Figure 2

Actual configuration of the SAPS tank

Figure 3

EDX analysis result of the peat soil

Figure 4

Image of the limestone under (a) 100 times magnification and (b) 500 times magnification

Figure 5

Composition of the limestone via EDX analysis

Figure 6

Size distribution analysis of the limestone

Figure 7

Temporal changes of the pH value and Fe concentration