



**UNIVERSITI PUTRA MALAYSIA**

***PERFORMANCE OF DIFFERENT TREATMENT MEDIA FOR PASSIVE  
REMEDIATION OF CONTAMINANTS ASSOCIATED WITH ACID MINE  
DRAINAGE***

**ZAFIRA BINTI MADZIN**

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DRAINAGE**

By

**ZAFIRA BINTI MADZIN**

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in  
Fulfilment of the Requirements for the degree of Master of Science**

**March 2018**

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

## **PERFORMANCE OF DIFFERENT TREATMENT MEDIA FOR PASSIVE REMEDIATION OF CONTAMINANTS ASSOCIATED WITH ACID MINE DRAINAGE**

By

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**March 2018**

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Mining activities have often been associated with apparent environmental quality degradation and have raised environmental concerns among public. Acid mine drainage (AMD) is typically resulted from mining activities through mineral interactions with atmospheric oxygen and water. This results in the water discharges being undesirably acidic and may contain heavy metals. Hence the need to develop inexpensive and sustainable remedial method to treat contamination is required. This study was carried out to develop a passive treatment method to treat heavy metal release from mining activities. In particular, the purpose of this study is to evaluate the performance of selected treatment media in treating AMD. The treatment media used were spent mushroom compost (SMC), limestone (LS), steel slag (SS) and ochre. The first stage of the experiment was the characterization of proposed treatment media using physico-chemical parameters. Then, a series of batch and column tests were conducted to determine the effectiveness of the treatment media in single and mixed substrates treatment using physico-chemical parameters and heavy metal removal analyses. Synthetic acid mine drainage water was prepared for both batch and column experiments. In the batch test, all single substrates and mixed substrates (in four different ratios namely SM<sub>1</sub>, SM<sub>2</sub>, SM<sub>3</sub> and SM<sub>4</sub>) were tested in anoxic condition for 5 days. Assessment on the removal efficiencies were discovered and for single substrate, both SMC and SS showed promising overall heavy metal removal efficiency of 94.6% and 96.7% ( $r=1.000$ ;  $p<0.05$ ), respectively. Performance of SMC was supported by its high pH value, alkalinity and total organic content, whilst SS has relatively high pH ( $r=0.967$ ,  $p<0.01$ ) compared to other media. As for mixed substrates, SM<sub>3</sub> which composed of 40% SS, 30% SMC, 20% LS and 10% ochre gave the best performance in overall heavy metal removal ( $r=0.999$ ,  $p<0.01$ ). This treatment ratio was then used in column experiment where continuous flow of synthetic AMD water was utilised. The column test using a treatment bioreactor was conducted in lab-scale for 30 days and the results showed noticeable performance in heavy metal removal for Fe, Cu, Pb, Zn, Al, and Mn with the overall removal percentage of 83.70% in column 1 and 99.69% in column 2. The metal

fraction analysis in the column sediment indicated that metal accumulation occurs mainly through adsorption onto organic matter > Fe/Mn oxides > carbonates > exchangeable > residual fractions. The assessment for removal rate in C1 and C2 from highest to lowest metal ascending are Mn>Fe>Zn>Al>Pb. Therefore, at the end of the research, characterization of treatment media has been done to unleash its potential in treating AMD. Then, assessment of batch and column experiment were carried out to measure its physicochemical parameters and heavy metal removal and finally to measure its performance in removal efficiencies, removal rate and first order kinetics.



Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

## **PELAKSANAAN MEDIA RAWATAN BERBEZA UNTUK PEMULIHAN PASIF BAGI PENCEMARAN BERKAITAN SALIRAN LOMBONG BERASID**

Oleh

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Aktiviti perlombongan kebelakangan ini telah dikaitkan dengan degradasi kualiti alam sekitar yang jelas dan telah menimbulkan kerisauan dikalangan orang awam. Saliran lombong berasid (AMD) adalah produk kebiasaan daripada aktiviti perlombongan melalui interaksi antara mineral dengan oksigen di atmosfera dan air. Kejadian ini menyebabkan air yang dikeluarkan menjadi berasid yang tidak dingini dan dan berkemungkinan mengandungi logam berat. Jadi, keperluan untuk membina kaedah pemulihan yang mampan dan tidak memerlukan kos yang tinggi untuk merawat pencemaran ini amat diperlukan. Kajian ini telah dilaksanakan untuk membina kaedah rawatan pasif bagi merawat pengeluaran logam berat daripada perlombongan..Secara terperinci, tujuan kajian ini dilakukan adalah untuk menilai prestasi media rawatan yang terpilih dalam merawat AMD. Media rawatan terpilih yang telah digunakan adalah kompos sisa cendawan (SMC), batu kapur (LS), keluli sanga (SS), dan tanah yang mengandungi mendapan bauksit. Kaedah pertama yang dijalankan dalam eksperimen ini adalah dengan mencirikan media rawatan yang telah dipilih menggunakan parameter fizikal dan kimia. Kemudian, satu siri ujian golongan dan ujian kolum telah dijalankan untuk menentukan keberkesanan rawatan media dalam keadaan substrat individu dan secara substrat campuran menggunakan parameter fizikal-kimia dan juga analisis penyingkiran logam berat. Sintetik air untuk AMD telah disediakan untuk ujian golongan dan ujian kolum. Didalam ujian golongan, kesemua substrat individu dan substrat campuran yang terdiri daripada 4 nisbah berbeza dinamakan SM<sub>1</sub>, SM<sub>2</sub>, SM<sub>3</sub> dan SM<sub>4</sub> telah diuji didalam keadaan anoksia selama 5 hari. Penilaian untuk kecekapan penyingkiran logam berat telah dijalankan dan untuk substrat individu, SMC dan SS keduanya mempunyai penyingkiran kecekapan tertinggi iaitu 94.6% (SMC) dan 96.7% (SS) ( $r=1.000$ ,  $p<0.05$ ). Prestasi SMC disokong dengan mempunyai nilai pH yang tinggi, kealkalian dan jumlah kandungan organik yang tinggi, manakala SS mempunyai nilai pH tertinggi ( $r=0.967$ ,  $p<0.01$ ) berbanding media yang lain. Untuk substrat campuran pula, SM<sub>3</sub> yang mengandungi 40% SS, 30% SMC, 20% LS dan 10% tanah memberikan perlaksanaan terbaik dan penyingkiran logam berat ( $r= 0.999$ ,  $p<0.01$ ). Kandungan karbon organik yang tinggi didalam kedua SMC dan sanga keluli telah meningkatkan

aktiviti mikrob untuk rawatan ini sekaligus meransang mekanisma penyerapan dan pemendapan dalam menyingkirkan logam berat. Nisbah rawatan ini telah digunakan didalam ujian kolum dimana aliran berterusan air AMD sintetik telah dipasang. Ujian kolum telah dijalankan selama 30 hari dan kajian ini menunjukkan kenampakan prestasi didalam penyingkiran logam berat untuk Fe, Cu, Pb, Zn, Al dan Mn iaitu penyingkiran keseluruhan dalam peratusan 83.70% di kolum C1 dan 99.69% didalam C2. Analisis pecahan logam berat menunjukkan berlakunya penggumpalan logam melalui proses penyerapan didalam bentuk; bahan organik > Fe/Mn oksida > karbonat > bahan boleh ditukar > pecahan sisa. Penilaian bagi kadar penyingkiran dalam C1 dan C2 pula adalah  $Mn > Fe > Zn > Al > Pb$ . Kesimpulannya, pada penghujung kajian ini, pencirian media rawatan telah dilakukan bagi melihat potensi media dalam menangani AMD. Kemudian, penilaian bagi kajian golongan dan kolum telah dijalankan untuk mengukur parameter fizikal-kimia dan juga penyingkiran logam dan akhir sekali untuk mengukur pelaksanaan dalam kecekapan penyingkiran, kadar penyingkiran dan juga kinetik tertib pertama.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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## LIST OF ABBREVIATIONS

AMD	Acid Mine Drainage
SRB	Sulphate Reducing Bacteria
RAPS	Reducing Alkalinity Producing System
SLB	Slag Leach Beds
LSB	Limestone Leach Beds
SMC	Spent Mushroom Compose
TVS	Total Volatile Solid
TOC	Total Organic Carbon
DOC	Dissolved Organic Carbon
OLC	Open Limestone Channels
ALD	Anoxic Limestone Drainage
GDP	Gross Domestic Product
SEM	Scanning Electron Microscopy
HRT	Hydraulic Retention Time
LOI	Loss-On-Ignition
TDS	Total Dissolved Solid
USEPA	United States Environmental and Protection Agency
ICP-OES	Inductively Coupled Plasma Atomic Emission Spectroscopy
EDX	Energy Dispersive X-Ray
ZVI	Zero Valent Iron
SAPS	Successive Alkalinity Producing System
SET	Sequential Extraction Technique
MM	Mixed media

## CHAPTER 1

### INTRODUCTION

#### 1.1 Background of study

Drainages from active and abandoned metal mines are acute and pervasive form of aquatic pollution, (e.g. primarily iron, zinc, lead, cadmium) to both surface and groundwaters. In Malaysia, some surface water bodies in Lembah Kinta Perak, Bestari Jaya Selangor, Sungai Lembing Pahang and Mamut, Sabah ex-mining areas are impacted by such discharges (Ashraf *et al.*, 2011; Ashraf *et al.*, 2012; Gazzaz *et al.*, 2012; Ali *et al.*, 2006). Recently, an overexploitation of bauxite mining activity has been identified in Kuantan, Pahang. Red discharges have been released to the surface waters and produced acidic, heavy metal-containing water which are characterised as acid mine drainage (AMD) (Madzin *et al.*, 2015)

Bioremediation can remove pollutants in both active and passive methods however active methods require addition of chemicals and regular maintenance (Kusin *et al.*, 2016). Active methods involve regular chemicals as reagents and labor input for controlled operation as active treatment systems can be engineered according to its design parameters (Taylor and Murphy, 2005) hence due to its high cost and intensive maintenance, passive treatments are widely used as a treatment (Roychowdhury *et al.*, 2015). Much research in recent years have focused on the identification of low-cost, low maintenance i.e. passive treatment options for the remediation of these polluting discharges (Obiri-Nyarko *et al.*, 2014; Liu *et al.*, 2015; Vasquez *et al.*, 2016; Kusin *et al.*, 2016; He *et al.*, 2016).

For instance, the remediation of net-alkaline, iron-rich mine drainage has been regarded as proven technology especially in many European and American applications of passive treatment systems (e.g. wetland-type systems and bioreactors) (Younger *et al.*, 2002). However, the removal of zinc and manganese, which are prevalent in discharges from metal mines (and also is the case in Malaysian mine-impacted discharges), are more difficult to nearly impossible.

Whilst iron is readily removed by the generation of oxyhydroxides in passive treatment, the hydroxide solubility products of zinc and other divalent metals are higher than that of iron, so that a higher target pH is required to remove these metals as hydroxides (Diaz *et al.*, 1997). In contrast, the solubility product of sulphides of these metals are lower than that of iron, so organic substrates that contain sulphate-reducing bacteria (SRB) in treatment bioreactor may offer a more feasible approach to remove such metals at the pH values typically achievable in passive treatment systems (Mayes *et al.*, 2011), and in timescales that result in reasonably sized systems.

The development of bioreactors which harness bacterially mediated heavy metals reduction have been widely tested since the observations of Tuttle *et al.* (1969) on the effectiveness of acid-tolerant sulphate reducing bacteria in removing metals as solid monosulphides from acid mine drainage. SRB perform the key terminal reductive step for metal removal which is rate-limited by the supply of carbonaceous organic matter to the SRB. For instance, a laboratory-scale sulphate reducing bioreactor or reducing and alkalinity producing systems (RAPS) have been developed to field-scale treating coal mine drainage where principal contaminants of concern include Fe, Al and  $\text{SO}_4^{2-}$ , i.e. some compost-based units around the UK (Jarvis and Younger, 1999; Matthies *et al.*, 2012; Mayes *et al.*, 2011).

However, passive systems in which sulphate reduction will occur vigorously and consistently enough to effect the removal of metals to the low level sufficient for compliance with regulatory regulations for mining-related metal pollutants have not yet been developed. The removal of zinc is of interest as it is the most commonly encountered pollutant in rivers draining former metal mining districts in Malaysia (e.g. Ashraf *et al.*, 2011; Gazzaz *et al.*, 2012). The use of bioreactor such as the RAPS were initially developed to overcome challenges and restrictions for anoxic limestone drainage (ALD) for low dissolved oxygen and heavy metals (Ordonez *et al.*, 2012) and very successful in treating heavy metals (Taylor and Murphy, 2005; Wang, 2010). However, studies have shown that the restriction of this bioreactor treatment system is in terms of long term performance and the replenishment of the organic substrates (Wilkin *et al.*, 2014; Obiri-Nyarko *et al.*, 2014; Taylor *et al.* 2015).

One of the important factors in designing a successful bioreactor is the selection of reactive media. Throughout years, studies have come out with a wide range of reactive materials in order to remove AMD and increase pH for example spent mushroom compost (Sheoran *et al.*, 2010; Wu *et al.*, 2016; Zagury *et al.*, 2006), limestone (Aziz *et al.*, 2008; Molahid *et al.*, 2018; Cheong *et al.*, 1998), steel slag (Duan and Su, 2014; Name and Sheridan, 2014; Zahar *et al.*, 2015) and ochre (Olimah *et al.*, 2015; Sahoo *et al.*, 2014; Sapsford *et al.*, 2015). Each media have the potential in treating heavy metals such as Fe, Mn, Al, Pb and Zn.

In many bioreactors and/or wetland-type system, the carbon source is initially present as labile cellulose-rich materials. This leaves more recalcitrant lignin-dominated material for later breakdown and limits supply of low-molecular weight carbonaceous material to the SRB communities (Pulles *et al.*, 2004). Reactive treatment media were used in this study and each media were determined of their performance in treating AMD,

individually and in mixed substrates. The necessity for replenishment of the carbon source in wetland-type system has long been acknowledged but it is something that can often be neglected in full-scale bioreactor operation. Thus, the addition of multiple organic substrates to assist the remediation process and sources of carbon are something to be discovered and taken into account in this study.

The remediation process is based on the reduction of sulphate, under anoxic condition which consumes protons and generates alkalinity, whilst simultaneously releasing sulphide to form a precipitate with divalent metal ions (Walton-Day, 1999, Nuttall and Younger, 2000; Cheong *et al.*, 2010). Other mechanisms that may immobilise metals in the treatment systems such as adsorption and co-precipitation may simultaneously take place alongside the bacterial sulphate reduction (e.g. Wang and Reardon, 2001). As an example, adsorption and co-precipitation mechanism especially in reducing manganese from mine water were done effectively using treatment media such as steel slag (Name and Sheridan 2014; Zahar *et al.*, 2015). However, the effect of mixing the steel slag with other reactive media will be discovered in this study.

The overall aim for the remediation of heavy metals in AMD is therefore to perform an effective passive treatment technique. Therefore, this study is focused on identifying suitable carbon sources as the treatment substrate to maximise the rate of metal reduction. Due to constraints such as available costs, high loading rates of some environmentally significant discharges, organic and inorganic wastes are being considered as the potential carbon substrates in this study. In simulating the passive treatment technique, identical hydraulic, chemical, biological and environmental conditions must be assumed in the laboratory-scale bioreactor to that in the field.

## **1.2 Problem statement**

Mining activities and overexploitation of the mining resources have been associated with various environmental quality degradations and have raised environmental concerns among public. For instance, the most notable cases are the recent bauxite mining in Kuantan, Pahang and copper mining in Mamut, Sabah. The impacts of mining have left the environment a dreadful effect of water quality degradation visibly seen in the surroundings of the affected areas. The deterioration of surface water and ground water quality is a widespread concern mainly originating by accidental discharges and leaching from the mining operations. Leaching of discharges can cause various environmental impacts to the environment and one of the examples is the heavy metal contamination. Acid mine drainage (AMD) is known as a serious pollution arising from active and abandoned mines (Akcil and Koldas, 2006) that can adversely impact the overall ecosystems (Choudhary and Sheoran, 2012). AMD is characterised by low pH, high in heavy metals and sulphates (Zagury *et al.*, 2007; Evans and Ko, 2013). AMD can result in various environmental disasters such as acidification and metal pollution to water discharges (Choudhary and Sheoran, 2012). As AMD contain variety of dissolved heavy metals, it can easily enter to human body system hence cause heavy metal contamination to human. For example, high amount of manganese in bauxite mining is harmful to human as Mn is a common toxic metal that can cause diseases related to kidney, lung

and also intestinal damage (Mayes *et al.*, 2011). Long term exposure of heavy metals can accumulated in vital organs and glands and also interfere human biological functions (Singh *et al.*, 2011).

There are various approaches that have been done in treating AMD (Cheong *et al.*, 1998; Johnson and Hallberg, 2005; Taylor and Murphy, 2005) however some are too expensive for full-scale treatments (Neculita *et al.*, 2008) and can only be reduced effectively using biodegradable organic substrates (Zagury *et al.*, 2007). Selection of organic substrates as reactive media in passive treatments have been conducted by previous studies where different media have been used to remove metals however the readily media proposed were easily exhausted hence inhibit its availability for long term performance (Regmi, 2009; Puls, 2006; Roychowdhury *et al.*, 2015). Hence there is an urge to come out with a combination of potential individual substrates as a mixed reactive media in order to promote better efficiency and prolonged treatment performance in treating heavy metals.

### **1.3 Significance of study**

As the impacts of acid mine drainage can persist for decades and centuries, and even after the cessation of the mining activities, there is a need to develop inexpensive and sustainable remedial method to treat the contamination. Therefore, in this study a passive treatment approach was adopted as to develop promising treatment technology for mine-impacted waters. A treatment bioreactor which is one of the passive AMD treatments was suggested in this study as it is low in cost, requires less maintenance and the substrates are abundantly available. The bioreactor has high potential in reducing heavy metals such as Mn, Fe, Al, Zn and Pb, and most importantly to produce alkalinity to neutralise the acidic water. Four types of reactive media have been chosen and tested individually and in mixed ratio to prove its treatment performance in treating AMD. These reactive media were relatively chosen as each of the substrate has the ability to increase pH and to treat heavy metals (namely Mn, Fe, Zn, Pb). This study provides a detailed investigation in the performance assessment of the passive treatment in removing heavy metals alongside with evaluation of each substrate chosen in this study, i.e. physicochemical characterisation of each substrate and the removal mechanisms in treating the heavy metals. Therefore, the findings provided can be useful for on-site treatment if intended especially for application in such cases in Malaysia.

## **1.4 Research objectives**

The main objective of this study is to develop a passive remediation technique using a treatment bioreactor to treat heavy metal release from mining activities. The specific aims for this research are;

1. To characterise different reactive media for potential treatment of acid mine drainage (AMD) waters
2. To evaluate physicochemical parameter improvement and heavy metal removal for AMD treatment performance
3. To assess the removal efficiency, pollutant removal rate and removal kinetics for AMD treatment performance.

## **1.5 Research questions**

The underlying research questions are:

1. What would be physicochemical characterisations of the treatment media suitable for remediation of AMD waters?
2. How is the performance of the treatment media in single and mixed substrate treatment in terms of physicochemical parameter improvement and heavy metal removal?
3. What are the removal efficiencies of heavy metals in the batch and column experiments?

## **1.6 Scope of work**

This study focuses on the development of a laboratory-scale passive treatment bioreactor for remediation of mine-contaminated water. A series of batch and column (upward continuous flow) experiments were conducted to evaluate the performance of the proposed treatment media in terms of physicochemical parameter improvement, heavy metal removal efficiencies, pollutant removal rate and mechanisms of heavy metal removal in column sediment. The reactive media used (spent mushroom compost, steel slag, ochre and limestone) were analysed for their physicochemical parameter characterisation suitable for the treatment of the AMD. The removal performances of heavy metals were tested in both single and mixed substrates treatment. The substrate used for the column bioreactors were chosen from the most effective ratio of reactive media in the batch test. Synthetic AMD was used in the experiments based on the actual characteristics of mine water from the real case studies.

Limitations were inevitable when conducting the research. For example the availability of organic substrates as the reactive media which were a challenge as different media were located in different places and pre treatment of media took time and labour. Other than that, designing and building the appropriate bioreactor to accommodate an efficient bioreactor to function well was one of the challenges that raised.



## **1.7 Thesis organization**

Chapter 1 provides the introduction of the research, which consists of the background of the study, problem statement, research objectives, and the significance of the study.

Chapter 2 describes the literature reviews relating to the research topic, and provides the overview of the topics that are covered throughout the study. This mainly consists of the overview of mining industry and the environmental impacts, background of AMD formation and consequences, and the treatment technologies for AMD.

Chapter 3 focuses on the research design of the study, which includes the parameter characterisation technique for the treatment media, and the methods of mine water analysis performed in this study. Two main approaches used in this study are the batch experiment and column experiment for analysis of contaminant removals. This mainly includes the experimental set up of the batch and column experiments for physicochemical parameter improvement and heavy metal removals. The technique for sediment column analysis is also included.

Chapter 4 discusses the results of the batch and column experiments for the AMD treatment. The discussion begins with the characterisation of the potential treatment media for AMD, followed by their performance as single media and in the mixed substrates. Performance of the AMD treatments are evaluated from both batch and column experiments. Evaluation of the most suitable ratio of mixed substrates is discussed for the overall potential remediation of the AMD. In the column experiment, the findings explain on the overall performance of the bioreactor including the treatment performance, removal efficiency, kinetic orders, and also sequestration of heavy metals in column sediment.

Chapter 5 summarises the major findings of this study in fulfilling the research objectives. Recommendations for future studies are also included in this study.

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