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PASSIVE REMEDIATION FOR METAL-RICH MINE WATER USING STEEL SLAG

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

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PASSIVE REMEDIATION FOR METAL-RICH MINE WATER USING STEEL SLAG

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MOHD SYAKIRIN MD ZAHAR

May 2017

Chair: Faradiella Mohd Kusin, PhD
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Acid mine drainage (AMD) is one of the major environmental pollution that needs to be treated for sustainable environment in the future. AMD formation in the environment has become a great public concern globally due to their effects to human health, flora and fauna. Various efforts have been taken to reduce the metal contamination in AMD through controlling or remediation process. However, the usual conventional method based to treat the AMD problem are costly and required extra procedure to further remove the metal ion from water bodies. This study highlights the potential of steel slag which is industrial by product to remove metal ions from metal-rich acid mine drainage (AMD) based on batch and column system. The removal efficiency, adsorption capacity and behaviors of adsorbents were examined during the metal removal process and incorporated with isotherm and kinetic models. In the batch studies, the removal of metal ions was evaluated by varying the contact time, solution pH, initial metal concentration, adsorbent dosage, size and the effect of competing ions. While for the column study, the factors of adsorbent bed height, flow rate and competing ions were evaluated to obtain the removal performance of steel slag in continuous flow system. Results of batch experiment have indicated >90% metal removal efficiencies when pH of the AMD has reached near-neutral state (6.8-7.5) at 14 hours contact time. Optimum equilibrium time was found at 24 hours whereby all the metals were 99-100% removed. An increased adsorption capacity with a decreased removal efficiency was observed as initial metal concentration increased. In contrast, increasing adsorbent dosage leads to increased removal efficiency but removal tends to be constant after reaching equilibrium amount of 2.0 g. Comparing the effect of competing ions, Fe was not affected despite the presence of other metal ions (100% removal) compared to Mn (59.3% removal) in mixed AMD solution. The adsorption behavior of Fe, Cu, Zn and Mn fits appropriately with the Langmuir isotherm model compared to Freundlich isotherm model indicated that the adsorption process occurred in monolayer surface rather than heterogeneous surface. The adsorption kinetics followed the pseudo-second-order kinetics trend which is consistent with chemisorption and is supported by the intra particle diffusion process. In the column study, the metal ions uptake mechanism is particularly bed depth and flow rate dependent, favoring higher bed depth at 3 cm and lower flow rate at 10 mL/min. The breakthrough curve simulation for metal ions removal were described using BDST and Thomas model. Both models were applied onto fixed bed column experimental data at different bed depths of 1.5, 2 and 3 cm with a constant flow rate of 10 mL/min and influent metal concentration of 27 mg/L. The linear plots at different bed depths indicate that adsorption of all metal fits well with BDST model. The performance of adsorption capacity for Mn is highly affected in mixed solution, by which 81.1% reduction from value in single solution, while Cu was only slightly affected, i.e. 10.1% reduction from value in single solution. Therefore, this study has highlighted the potential of steel slag as an adsorbent for metal-rich AMD with regard to metal removal efficiency, affecting variables, kinetics and models that explain the metal removal behavior.

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

PEMULIHAN PASIF BAGI AIR LOMBONG YANG KAYA DENGAN LOGAM MENGGUNAKAN SANGA KELULI

Oleh

MOHD SYAKIRIN MD ZAHAR

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Saliran lombong berasid (AMD) adalah pencemaran alam sekitar yang perlu dirawat untuk alam sekitar yang mampan pada masa hadapan. Pembentukan AMD di alam sekitar telah menjadi kebimbangan umum di seluruh dunia berikutn kesannya kepada kesihatan manusia, flora dan fauna. Pelbagai usaha telah diambil untuk mengurangkan pencemaran logam berat di AMD melalui proses pengawalan atau pemulihan. Walau bagaimanapun, kaedah konvensional yang biasa digunakan untuk merawat masalah AMD adalah mahal dan memerlukan prosedur tambahan untuk mengalihkan lagi ion logam berat daripada air. Kajian ini memfokuskan kepada potensi sanga keluli yang merupakan bahan buangan industri pembuatan keluli sebagai media rawatan untuk air lombong yang kaya dengan logam berat berdasarkan sistem kumpulan dan kolum. Kecekapan penyingkiran, keupayaan penjerapan dan kelakuan penjerap telah dianalisis semasa proses penyingkiran logam dan diuji dengan menggunakan model isoterm dan kinetik. Dalam kajian kumpulan, penyingkiran ion logam dinilai dengan mengubah masa, pH, kepekatan awal logam, dos penjerap, saiz dan kesan ion yang bersaing. Manakala, untuk kajian kolum, faktor-faktor ketinggian katil penjerap, kadar aliran dan ion yang bersaing telah dinilai untuk menilai keupayaan penyingkiran sanga keluli dalam sistem aliran berterusan. Keputusan ujian kumpulan telah menunjukkan> 90% logam disingkirkan apabila nilai pH A pada AMD telah mencapai keadaan hampir neutral (6.8- 7.5) pada masa 14 jam. Masa optimum dicapai pada jam ke 24 di mana semua 99-100% logam berat disingkirkan. Kapasiti penjerapan meningkat dengan penurunan kecekapan penyingkiran telah diperhatikan semasa kepekatan logam awal meningkat. Manakala, peningkatan dos penjerap telah menyebabkan peningkatan kecekapan penyingkiran namun penyingkiran mula menjadi tetap selepas mencapai keseimbangan sebanyak 2.0 g. Membandingkan kesan ion yang bersaing, Fe tidak terjejas walaupun terdapat ion logam lain (100% penyingkiran) berbanding Mn (59.3% penyingkiran) dalam AMD. Tingkah laku penjerapan Fe, Cu, Zn dan Mn sesuai dengan model isoterm Langmuir berbanding dengan model isotherm Freundlich yang menunjukkan proses penjerapan terjadi pada permukaan lapisan mono dan bukannya permukaan heterogen. Kinetik penjerapan pula sesuai dengan kinetik pseudo-second-order yang konsisten dengan penjerapan kimia dan disokong oleh proses penyebaran intra.

Dalam kajian kolum, mekanisme pengambilan ion logam bergantung pada kedalaman katil dan kadar aliran, dimana kedalaman katil tersebut harus setinggi 3 cm dan kadar aliran yang lebih rendah pada 10 mL / min. Simulasi lengkung penerobosa untuk penyingkiran ion logam digambarkan menggunakan model BDST dan model Thomas. Kedua-dua model ini digunakan pada data kolum eksperimen pada kedalaman katil yang berbeza iaitu 1.5, 2 dan 3 cm dengan kadar aliran tetap 10 mL / min dan kepekatan logam yang sebanyak 27 mg / L. Garis linear yang diplotkan kedalaman katil yang berbeza menyatakan bahawa penjerapan logam bersesuaian dengan model BDST. Kapasiti penjerapan Mn amat terjejas pada larutan campuran, yang mana menunjukkan 81.1% penurunan dari nilai pada larutan tunggal. manakala Cu hanya sedikit terjejas, iaitu 10.1% penurunan daripada nilai dalam larutan ion tunggal. Oleh itu, kajian ini telah menekankan potensi sanga keluli sebagai penjerap untuk AMD yang kaya dengan logam, dengan mengambil kira kecekapan penyahan logam, pembolehubah yang memberi kesan dan kinetik dan model yang menerangkan sifat penyahan logam.

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I certify that a Thesis Examination Committee has met on 18 May 2017 to conduct the final examination of Mohd Syakirin bin Md Zahar on her thesis entitled "Passive Remediation for Metal-Rich Mine Water Using Steel Slag" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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LIST OF ABBREVIATIONS

ALD	Anoxic Limestone Drainage
AMD	Acid mine drainage
ARD	Acid Rock Drainage
BET	Brenauer-Emmett-Teller
BJH	Barrett-Joyner-Halenda
Cu	Copper
Cd	Cadmium
EDX	Energy Dispersive X-ray
FAAS	Flame Atomic Absorbtion Spectroscopy
Fe	Iron
FeS2	Iron sulfide
INECAR	Institute of Environmental Conservation and Research
Mn	Manganese
Ni	Nickel
OLC	Open Limestone Channel
Pb	Lead
SAPS	Successive Alkalinity Producing System
SEM	Scanning Electron Microscopy
TDS	Total Dissolved Solids
UNEP/GPA	United Nations Environmental Protection/Global Program of Action
USEPA	United States Environmental and Protection Agency
USGS	United States Geological Survey
WHO	World Health Organization
Zn	Zinc

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Acid mine drainage (AMD) is typically known as a serious environmental problem resulting from active and abandoned mining activities, causing undesirable impacts on the environment, such as on the aquatic life, groundwater, streams, and humans (Akcil and Koldas, 2006; Lottermoser, 2007). Generally, the formation of acid mine drainage (AMD) is caused by oxidation of sulphide minerals such as pyrite (FeS_2), i.e. when exposed to oxygen and water in the presence of sulphide oxidizing bacteria (SOB) (Othman *et al.*, 2016). The process of pyrites decomposition typically produces mine water acidification and also a large number of toxic trace elements that are released during the processes such as Ag, As, Bi, Cd, Co, Cu, Hg, Mo, Ni, Pb, Ru, Sb, Se, Sn, Te, and Zn (Lakovleva *et al.*, 2015).

Also referred to as acid rock drainage (ARD), AMD emanating from mine waste rock, tailing and mine structures such as pits and underground workings, is primarily a function of the mineralogy of local rock material and the availability of water and oxygen. Because mineralogy and other factors affecting AMD formation are highly variable from site-to-site, predicting the potential for AMD can be challenging and costly (Akcil and Koldas, 2006). Characterized by low pH and high concentration of heavy metals and other toxic elements, AMD can severely contaminate surface and ground water as well as soil (Peppas *et al.*, 2000). AMD is thus considered as the worst environmental problem associated with mining activities.

The AMD-related problems had raised so much attention globally because of the environmental impacts caused by the AMD. There are many case studies around the world that had been reported relating to AMD problem. It is apparent that formation of AMD is complicated due to a number of factors that influence its production and it would be highly probable that the constituents vary from region to region (Motsi, 2010). Table 1.1 shows some AMD problems that occur in different study area along with their chemical composition.

Table 1.1: Example of AMD problems in different study area

Study Area	pH	Fe	Cu	Al	Zn	Mn	Ni	As	Reference
Wheal Jane Mine, U.K	2.6-3.1	1720-1900	14-18	170-197	1260-1700	11-25			Motsi, (2010)
California Coast Renge, U.S.A	3.2	368		88.6	1.8	3.8			Jew et al. (2014)
Odiel River, Spain	3.8	4.9	5.4	32.8	11.5	8.1	0.145	0.04	Espana et al. (2005)
Xingren Coalfield, China	5.22	34.1		13.4	4.22		0.5		Xingren et al. (2008)

*Units for all metal concentration are in mg/L

There are several case studies in Malaysia reporting on AMD problem at several ex-mining areas. Some examples of former mining areas in Malaysia include former tin mining sites in Bestari Jaya, Selangor (Ashraf *et al.*, 2012), iron ore mines in Sungai Lembing, Pahang (Alshaebi *et al*., 2009; Yaacob *et al*., 2009), tin mining district in Kinta Valley, Perak (Hamzah *et al.*, 2008), copper ex-mining area in Mamut, Sabah (Jopony and Tongkul, 2009) and Bukit Besi ex-mining area in Terengganu (Yaacob, 2009).

Among these cases, the occurrence of AMD in Mamut, Sabah has been associated with severe environmental degradation affecting several tributaries and rivers in a river catchment in Sabah. Table 1.2 shows some physicochemical characteristics data at Mamut copper ex-mining site that has led to severe AMD phenomenon in Sabah (Jopony and Tongkul, 2009). Among the metals shown in Table 1.2, the concentration of Mn is the highest and is the primary metal of concern in this case. Although there have been reported cases of AMD in Malaysia, this environmental issue receives only little attention in Malaysia, whilst in a broader sense AMD effects may persist for a very long time.

Table 1.2: Physicochemical characteristic data of Mamut copper ex-mining

Study Area	pH	TDS	acidity	Sulphate	Fe	Mn	Cu	Zn	Reference
Mamut copper ex mining area, Sabah	2.9-3.75	302-2673	176-1697	292-2808	0.1-7.1	3.8-79.8	2.0-47	0.4-25.4	Jopony and Tongkul, (2009)

*Units for TDS, sulphate and metal concentration are in mg/L. Total acidity in mg $CaCO_3$/L

There are many different technologies of treatment which are available for heavy metal removal. Common methods such as chemical precipitation, coagulation, reverse osmosis, air stripping, adsorption and oxidation have been applied in wastewater treatment (Naiya *et al*., 2009). Adsorption technique has become prominent as it requires low energy and simple design (Wang *et al*., 2009). Researchers have started focusing on the adsorption process as an alternative technique to treat metal contamination in water bodies. According to Feng et al. (2004), adsorption is a technique that is widely used to treat wastewater. The advantages of metal adsorption are utilization of waste materials to remove heavy metals in the environment, the inexpensive cost for treatment procedures, the effective short period and friendly application (Bailey et al., 1999). Wastes from industries have potentially become a low-cost adsorbent for heavy metal removal. It requires little processing to increase its adsorption capacity. Generally, industrial wastes are generated as by-products. They are inexpensive since these materials are available in large quantities (Babel and Kurniawan, 2002).

Industrial by-products/wastes such as fly ash (Srivastava and Mall, 2006), waste iron (Lee *et al*., 2003), metallic iron (Giles *et al*., 2011), hydrous titanium oxide (Chen and Ruckenstein, 1991), electric arc furnace slag (Yusuf *et al*., 2013) and steel slag (Feng *et al*., 2004) are inexpensive and abundantly available. There are various studies that investigated the performance of steel slag as an effective adsorbent to remove heavy metal from water. For example, Yusuf *et al*. (2013) conducted a study that uses electric arc furnace slag to remove nickel from aqueous solution. The findings revealed that adsorption was found to be dependent on the adsorbent dosage, contact time, pH and initial concentration.

In this study, steel slag was selected as the main material to remove heavy metals from acid mine drainage. The study attempts to evaluate the efficiency of steel slag by evaluating some affecting variables such as adsorbent dosage, adsorbent size, pH, initial concentration, column bed height and flow rate. These affecting variables are important to be assessed in order to provide optimum conditions and sufficient information for the potential application in real situation.

1.2 Problem Statement

Acid mine drainage needs to be appropriately treated before being released into the environment as to avoid the contamination due to presence of heavy metals into the surface and/or groundwater. Active treatment is typically used to remediate the AMD. However, initial cost and chemicals used are very expensive thus giving some disadvantages to active treatment. On the other hand, passive treatment that uses industrial by-products or waste materials as treatment media may provide a better choice for treatment selection of AMD cases.

Recently, researchers tend to give attention on adsorption technique that is becoming a popular method for removal of heavy metals from AMD (Omer *et al.*, 2003; Wang *et al*., 2009; Yusuf *et al*., 2013). Natural materials that are available in large quantities or certain waste products from industrial activities may have potential as inexpensive sorbent (Motsi, 2011). However, majority of studies applying the concept of adsorption technique are mainly focusing on the production of adsorbent using low-cost natural materials rather than using readily available materials. Many studies found that low cost natural material required modification steps to further boost the effectiveness of the adsorbent. Modifications are conducted on the adsorbent either physically or chemically. For example, the physical modification such as heating are conducted to improve the specific surface area of adsorbents to increase their removal efficiencies to remove heavy metals (Mohan and Pittman, 2006). In chemical modification process, the adsorbents are pretreated by using chemical reagents such acid and base solution to increase the porosity of the adsorbent for better adsorption capability (Chamarty *et al.*, 2001). All this modification process required high cost to be implemented and at the same time will increase the cost of the natural materials used as adsorbent.

While it is known that production of any kind of natural materials as and adsorbent is not usually economical, use of readily available materials (i.e. industrial by-products or waste materials such as steel slag) provides greater advantages for this treatment method. Steel slag are adsorbent that have ability to remove heavy metals from the synthetic waste water as reported in previous literature (Fang *et al.*, 2004; Filho and Butorina, 2005; Kurniawan *et al.*, 2006). However, the application of steel slag to remove heavy metals in acidic water and AMD is still limited and require further study to evaluate its performance in such extreme condition (e.g highly acidic). Additionally, there are less studies focusing on adsorbent derived from industrial by-product (e.g. steel slag) to remove metals from AMD cases in this country.

1.3 Research Objectives

Generally, the aim of this study is to investigate the potential of steel slag, which is an industrial by-product to remove heavy metals from acid mine drainage. It also provides information regarding steel slag capabilities in terms of its adsorption performance to remove heavy metals from mine water.

The specific objectives of this study are:

1- To investigate the characteristics of steel slag as a potential adsorbent for heavy metals removal in acid mine drainage.
2- To evaluate the effect of contact time, solution pH, initial concentration of heavy metal, adsorbent dosage and size, effect of competing ions, effect of bed height and flow rate on adsorption capacity of steel slag for heavy metals removal under batch and column experiment
3- To assess the kinetics of heavy metals adsorption under varying treatment conditions in batch and column experiment using isotherm models, kinetic test and mathematical models.

1.4 Research Questions

Based on the research objectives, the underlying research questions are as follows:
1- What are the characteristics of steel slag that makes it suitable as an adsorbent for heavy metals removal in acid mine drainage?
2- What is the efficiency of heavy metals removal using steel slag as an adsorbent?
3- What are the factors that affect adsorption capacity of steel slag for heavy metals removal?
4- What is the kinetics of heavy metals removal using steel slag?
5- What is/are the mathematical model(s) that best explain the removal of heavy metals using steel slag?

1.5 Scope of Study

This study generally covers the area as follows:

1- This study focuses on the removal of selected heavy metals from acid mine drainage under batch experiment as initial part of the study. Several parameters had been determined to evaluate the performance of steel slag to remove heavy metals from water under different experimental conditions. The variables include contact time, size, dosage, pH and initial concentration of solution. The influencing parameters on metal removals were studied in order to determine and understand the adsorbent and metal behavior during the adsorption process.
2- The study involves the identification of the life-span of steel slag under continuous flow system with effects of some parameters such as column bed depth and flow rate. The data collected from continuous flow system were analyzed with related theories and models, which is important to serve as the basis for the design of full-scale adsorption columns.

1.6 Significance of Study

AMD remains as a toxic legacy of excess contaminants to the accessible waterways affecting the quality of water environments. Therefore, further studies on AMD remediation may provide fundamental understanding on how to deal with such problem, especially on the control of the extent of contamination often resulted by the AMD.

Generally, this study introduces steel slag, which is an industrial by-product locally available in large quantities and they are inexpensive. Recently, researchers are focusing on the production of adsorbents that are low in cost and simple to run or operate. However, the information of using readily available materials or waste products such as steel slag as metal adsorbent to remove heavy metals in AMD is still limited in the literature. The effectiveness of steel slag as a low-cost adsorbent was further investigated and the information gained from the experiments will be useful to the design of potential on-site treatment application for such AMD cases.

In addition, the performance of steel slag was evaluated in batch and continuous flow system to identify the life span of the adsorbents. The data collected were analyzed with some related theories and models that offer some understanding of adsorption mechanism occurring between the adsorbent and adsorbate. It also gives some understanding on the overall performance of steel slag that could be useful to be applied in the future as a low cost and readily available adsorbent.

The expected output from this study is to provide a treatment selection for AMD remediation that is more user-friendly and that does not require the cost of production. Besides that, it also helps to decrease the cost of waste disposal by re-using it back for example, waste product for water treatment purposes, which is seen as an economical approach.

1.7 Thesis Organization

Following this chapter, the remaining chapters of the thesis are outlined below:

Chapter 2 (Literature Review) details on the topics covered throughout the study that consists the fundamentals and occurrence of acid mine drainage (AMD), impact towards environment and related processes, treatment options for AMD, adsorption mechanism and also some adsorbents for AMD remediation.

Chapter 3 (Methodology) presents the research design for this study. The laboratory analysis in this study is divided into batch and column experiments. The varying parameters were determined to evaluate the removal performance of steel slag used in this study. The data collected from both batch and column experiments were analyzed using related theories and mathematical modelling.

Chapter 4 (Results and Discussion) discusses the results of the laboratory batch test and column experiments of the AMD treatment. The findings presented are the characteristics of steel slag along with the efficiency of steel slag to remove heavy metal from acid mine drainage under varying variables in both batch and column experiments. The results of theories and mathematical modelling used were also presented, which serve as some basis for further understanding on adsorption performance.

Chapter 5 (Conclusions) summarizes the major findings of the study in the fulfillment of the research objectives. Recommendations for future studies in this field are also included.

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