

LIMESTONE AND GOAT DUNG IN ACID MINE DRAINAGE TREATMENT

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**A thesis submitted in fulfilment of the
requirements for the award of the degree of
Master of Science (Chemistry)**

**Faculty of Science
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This thesis is dedicated to

To my beloved father and mother,

My supervisor and co-supervisor that always help and guide me,

And finally my friends those always assist and support me...

ACKNOWLEDGEMENT

In the name of Allah the Most Merciful and Most Compassionate. Praise to Allah S.W.T for giving His blessing, help and guidance throughout my study.

First and foremost I offer my sincerest gratitude to my supervisor, Associate Prof. Dr. Azli Sulaiman, who has supported me throughout my thesis with his patience and knowledge, and co-supervisor, Dr. Shamsul Kamal Sulaiman.

I would like to thank En. Md Muzayin b. Alimon, Director of Mineral Research Centre Malaysia, Tn Hj Sahar Effendi b. Abdullah Azizi, Ex-Director of Mineral Research Centre Malaysia and Dato' Hj. Zulkifly b. Hj. Abu Bakar, Dr Rohaya Othman, Head of Rock-based Technology Section, Tuan. Hj. Nasharuddin Isa, Ex-Head of Rock-based Technology Section, Mrs Siti Noorzidah Mohd Sabri, Mrs Rogayah Saad, En Hassan Ismail and all of Rock-based Technology Section staff. Mr. Mohd Syahrir Mohd Rozi, Ms. Norinsafrina Mustafa Kamal, Ms. Umahthevy, Mr. Roslan Isa, Mrs. Azlin Zahara Ahmad Zary and all of Mining and Quarrying Technology Section staff.

I would also like to thank the staff of the Mineral Processing Section staff for their helps in the laboratory tests, especially Dr. Nazwin Ahmad, Head of Mineral Processing Section, Dr. Ismail Ibrahim, Mrs. Salmah Baharudin, Mrs. Hjh. Azizah Isa, Mrs. Yeoh Ah Heong and all of Mineral Processing staff; and Mr. Mohd Idham of Silica-based Technology Section. My gratitude also goes to Clay-based Technology Section staff especially Tn Hj. Abd. Rois Abd. Mois. Finally, I wish a special thank you to my parents for their support, encouragement and prayer.

ABSTRACT

Acid mine drainage (AMD) is a major problem nowadays to the environment because it causes acidic water and high heavy metals content. In this study, AMD samples were obtained from a tailing pond of tin mine in Perak. The pond is the recycle water system of the tin ore processing plant and has pH values in the range of 2.5-2.8 and high heavy metals and sulphate contents. This can damage the equipment in the mine such as water pump. The AMD samples from this location were treated with two different materials, namely limestone and goat dung fertiliser. Limestone was used as neutralising material for acidic water and to reduce the heavy metals content. Goat dung fertiliser was used in reducing sulphate in the AMD sample. Four different methods were carried out based on the material used. For the first method, 500 mL of AMD sample was allowed to flow through limestone layered column. The four different limestone sizes used were in the ranges of 2-4 mm, 1-2 mm, 850 μm - 1 mm and 425-850 μm . For the second method, 500 mL of AMD sample was allowed to flow through organic fertiliser layered column. For third method, 500 mL of AMD sample was allowed to flow through organic fertiliser and limestone layers. For fourth method, 500 mL of AMD sample was allowed to flow through limestone and organic fertiliser layers. Single batch column technique was used in all experiments. For each experiment, the permeate was collected every 15 min, 30 min, 45 min, 60 min and 75 min. The results show that pH values obtained in all of the experiments increased after treatment in all methods. The highest pH obtained was 7.17 ± 0.01 by using 300 g of organic fertiliser with 60 min retention time. The lowest sulphate concentration obtained was 666.50 ± 92.63 mg/L by using Method 3 with a ratio of 50 g organic fertiliser to 50 g limestone and retention time of 30 min. The concentrations of heavy metals such as arsenic and chromium were reduced and complied with Standard A and Standard B of the Environmental Quality Act 1974. All methods used were able to increase pH and reduced sulphate concentration. Methods 1, 3 and 4 can reduce heavy metals such arsenic, cadmium, nickel and zinc. Among these methods, Method 3 is the most suitable method in treating AMD.

ABSTRAK

Saliran asid lombong (AMD) merupakan satu masalah yang utama pada masakini kepada alam sekitar kerana ia menyebabkan air berasid dan kandungan logam berat yang tinggi. Dalam kajian ini sampel AMD telah diperolehi dari sebuah kolam hampas lombong bijih timah di Perak. Kolam ini merupakan sebuah kolam sistem kitar semula air dari kilang pemprosesan bijih timah dan mempunyai nilai pH dalam julat 2.5-2.8 serta kandungan logam berat dan sulfat yang tinggi. Ini boleh menyebabkan kerosakan kepada peralatan perlombongan misalnya pam air. Sampel AMD dari lokasi ini telah dirawat dengan menggunakan dua bahan yang berbeza iaitu batu kapur dan baja najis kambing. Batu kapur telah digunakan sebagai bahan untuk meneutralkan air berasid dan mengurangkan kandungan logam berat. Baja najis kambing telah digunakan untuk mengurangkan kandungan sulfat di dalam sampel AMD. Empat kaedah berbeza telah dijalankan berdasarkan bahan yang digunakan. Bagi kaedah pertama, 500 mL sampel AMD telah dialirkan menerusi turus yang mengandungi lapisan batu kapur. Empat saiz batu kapur yang berbeza yang telah digunakan adalah dalam julat 2-4 mm, 1-2 mm, 850 μm - 1 mm dan 425-850 μm . Bagi kaedah kedua, 500 mL sampel AMD telah dialirkan menerusi turus yang mengandungi lapisan baja organik. Bagi kaedah ketiga, 500 mL sampel AMD telah dialirkan menerusi lapisan baja organik dan lapisan batu kapur. Bagi kaedah keempat, 500 mL sampel AMD telah dialirkan menerusi lapisan batu kapur dan lapisan baja organik. Teknik turus kumpulan tunggal telah digunakan dalam semua eksperimen. Bagi setiap eksperimen, resapan diambil pada setiap 15 min, 30 min, 45 min, 60 min dan 75 min. Keputusan menunjukkan bahawa nilai pH yang diperolehi dalam semua eksperimen telah meningkat selepas rawatan dalam semua kaedah. Nilai pH yang paling tinggi diperolehi adalah 7.17 ± 0.01 dengan menggunakan 300 g baja organik dan masa penahanan 60 minit. Kepekatan sulfat yang paling rendah diperolehi adalah 666.50 ± 92.63 mg/L dengan menggunakan Kaedah 3 dengan nisbah 50 g baja organik kepada 50 g batu kapur dan masa penahanan 30 min. Kepekatan logam berat misalnya arsenik dan kromium telah berkurangan dan mematuhi Piawai A dan Piawai B Akta Kualiti Alam Sekitar 1974. Semua kaedah digunakan mempunyai keupayaan untuk menaikkan pH dan mengurangkan kepekatan sulfat. Kaedah-Kaedah 1, 3 dan 4 boleh menurunkan kepekatan logam berat misalnya arsenik, kadmium, nikel dan zink. Antara kaedah-kaedah ini, Kaedah 3 merupakan kaedah yang paling sesuai untuk merawat AMD.

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LIST OF ABBREVIATION

Al	-	Aluminium
ADMI	-	American Dye Manufacturers Institute
AMD	-	Acid mine drainage
ARD	-	Acid rock drainage
BOD	-	Biochemical oxygen demand
°C	-	Degree celcius
Ca ²⁺	-	Calcium ion
CaO	-	Calcium oxide (quicklime)
CaCO ₃	-	Calcium carbonate (limestone)
Ca(OH) ₂	-	Calcium hydroxide (hydrated lime)
CaSO ₄	-	Calcium sulphate (gypsum)
Cd	-	Cadmium
CO ₂	-	Carbon dioxide
COD	-	Chemical oxygen demand
Cr	-	Chromium
Cu	-	Copper
DNA	-	Deoxyribonucleic acid
EDX	-	Energy dispersive X-ray spectrometry
Fe(OH) ₃	-	Iron hydroxide
FeS ₂	-	Pyrite
FESEM	-	Field emission scanning electron microscopy
HCl	-	Hydrochloric acid
HClO ₄	-	Perchloric acid
HF	-	Hydrofluoric acid
HNO ₃	-	Nitric acid
ICP-OES	-	Inductively coupled plasma optical emission spectrometry

kg	-	Kilogram
LOI	-	Loss on ignition
μm	-	Micrometer
MIW	-	Mining influenced water
mg/L	-	Milligram per litre
Mt	-	Metric tonne
NH_3	-	Ammonia
NH_4Cl	-	Ammonium chloride
SiO_2	-	Silica
SO_2	-	Sulphur dioxide
SOB	-	Sulphide oxidising bacteria
Sn	-	Tin
SnO_2	-	Cassiterite
SRB	-	Sulphate reducing bacteria
XRF	-	X-ray Fluorescence

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Mining industry operations can be classified into three sections: mining, mineral processing, and metallurgical extraction. Mining is the initial activity in the profitable exploitation of mineral source extraction. It is defined as the extraction of material from the ground to obtain one or more component parts of the mined material. Mineral processing or beneficiation aims to physically separate and concentrate the ore minerals, whereas metallurgical extraction aims to destroy the crystallographic bonds in the ore mineral in order to recover the sought after element or compound. At mine sites, mining always goes along with mineral processing of specialising forms, for example crushing, grinding, separation and flotation. It is sometimes accompanied by the metallurgical extraction of commodities such as gold, copper, nickel, uranium or phosphate, for example heap leaching, vat leaching and *in situ* leaching. These three principal activities of the mining industry can produce wastes, known as mine wastes. Mine wastes can be categorised as solid, liquid or gaseous and they are by-products of mining, mineral processing and metallurgical extraction. Mine wastes are undesirable, economically valueless and pile up at mine sites (Lottermoser, 2010). Many terms are used by researchers or environmentalist when referring to liquid mine waste such as acid mine drainage (AMD), mining influence water (MIW) (McLemore *et al.*, 2009) or acidic and metalliferous drainage (McCullough and Lund, 2011).

The present study aims at evaluating the use of limestone and organic materials in cleaning-up AMD generated from a tin mine and to reduce the content of heavy metals and sulphate, by increasing the pH value of AMD. AMD occurs when sulphide minerals are exposed to oxygen in environment and releases to ecosystem. AMD occurs at active mine or abandoned mine. Passive treatment by using column technique was applied in this study.

Nowadays many active or abandoned mines have encountered AMD problem. Table 1.1 shows the name list of active and abandoned mines that caused AMD problem in Malaysia. There are several factors that can affect the amount and toxicity of AMD, among which are the mineralogy of the rock material, the surface area, the oxygen concentration and the quantity of water that react with the sulphide minerals. High content of heavy metals and the high concentration of acid can cause bad effects to the surface water, ground water and soil (Luptakova *et al.*, 2012). The AMD contains high concentration of inorganic acid, dissolved Fe(II), sulphates and a lot of toxic metals such as copper, cadmium and arsenic (Flores *et al.*, 2012). For example, the impact of mining effluent water takes place in the abandoned Mamut Copper Mine. Mamut Copper Mine was the only copper mine operated in Malaysia. The mine is located in Sabah, East Malaysia. The mine began operation in 1975 and ceased operation circa 1999. Generally, seepage water from mine pit area has a low pH value of 2.90 to 3.75, high sulphate content of 292 to 2808 mg/L and high concentration of toxic metals such as aluminium, manganese, iron, copper and zinc (Jopony and Tongkul, 2009).

Table 1.1: Active and abandoned mines involved in AMD

No.	Name or Place of Mines	Status of Mine	Type of Ores
1.	Bukit Ibam, Rompin, Pahang	Active	Iron
2.	Bukit Botol, Pahang	Abandoned	Iron
3.	Padang Piol, Pahang	Active	Iron
4.	Sungai Lembing, Kuantan, Pahang	Abandoned	Tin
5.	Penjom, Kuala Lipis	Active	Gold
6.	Bukit Koman, Raub, Pahang	Active	Gold
7.	Bukit Besi, Dungun, Terengganu	Active	Iron
8.	Mukim Bandi, Kemaman, Terengganu	Active	Iron
9.	Mamut Copper Mine, Ranau Sabah	Abandoned	Copper

Source: (Wan Yaacob *et al.*, 2009; Mohd Rozi and Khor, 2012; Panahi *et al.*, 2012; Isidore *et al.*, 2012)

In 2010, there were 12 active tin mines and 25 among retreatment plants operated in Malaysia. These mines were nine open cast mines, one gravel pump and two dredges. These operating mines were five in Perak, three in Pahang, two in Terengganu and one in Johor. The open cast mines had contributed the highest production at 2,052 tonnes, followed by panning at 283 tonnes, among retreatment plants at 274 tonnes, dredging at 54 tonnes and gravel pump mines at six tonnes. Perak is the highest producer of tin in Malaysia and in 2010, an amount of 2,398 tonnes was produced from this state (Ismail *et al.*, 2011). The oldest and currently the largest open cast tin mine in Malaysia is located in Perak state. The average production of this mine from year 1907 to 2006 was about 735 tonnes tin metal per year or estimated to be 61 tonnes per month (Ismail *et al.*, 2010).

The main mineral of tin is cassiterite (SnO_2). Theoretically, tin (Sn) content in cassiterite is 78%. However, in the majority of cases, cassiterite contains impurities and the tin content may vary from 65% to 78%. The major impurities of cassiterite include tantalum, niobium, titanium and other elements, usually in the form of solid solutions. Solid solutions also known as mixed crystal was mentioned by Charles, 2001. The impurities in the cassiterite often have a pronounced effect on flotation properties of

cassiterite. Based on the study from a number of tin ore bodies, tin can be classified into three major groups: group 1: Cassiterite in pegmatitic veins contains significant quantities of $(\text{Nb,Ta})_2\text{O}_5$ with traces of wolframite and manganese. This type of cassiterite is fragile and tends to slime during grinding; group 2: Cassiterite from quartz veins. Cassiterite from this group contains about 1% $(\text{Nb,Ta})_2\text{O}_5$ and about 0.3–0.4% wolframite; group 3: Cassiterite from sulphide veins usually contains vanadium, sulphur and wolframite (Pryor and Vrobel, 1951; Bulatovic, 2010). Cassiterite is one of the inert minerals and very hard to disintegrate, that is why it forms sand deposits by decomposition of pegmatic and quartz-cassiterite deposits. These deposits are common in Asia (Bulatovic, 2010). Figure 1.1 shows the tin ore or cassiterite mineral (Sulaiman, 2008).



Figure 1.1: Tin ore

Tin is not a toxic metal, but organotin chemical is toxic such as tributyltin (TBF). Tributyltin was introduced in 1970s as additive in antifouling paints. Tributyltin becomes toxic to crustaceans at concentrations of less than $1 \mu\text{g/L}$ (Hill, 2010).

Tin can be used in many fields, for example lead free electronics, which use tin as substitutes for lead as a solder material (Pearce, 2013). In other words, lead free solder contains more than 95% tin. Nowadays, solder industry monopolies half of all tin produced in the world. Lithium ion batteries, which used tin can make lithium ion batteries

last more than three time longer. Tin also being used as a new generation of stainless steel, for example Nippon Steel Japan announced in November 2011 an entirely new generation product of stainless steel (NSSC FW series) based on tin (MSC, 2011). Tin which is used in fuel catalyts, can save up to 10% of fuel consumption in vehicles, ships and generators. Tin is replacing antimony as fire retardants used in most plastics. Tin fire retardants are available in the form of zinc hydroxystannate and zinc stannate. Tin also replaces antimony in brake pads and replaces lead in lead free-bearings in car industry. Tin is ahead in the race for the next generation of cheaper solar cell materials for example kesterite contains 30% tin, is the first to cross the 10% efficiency barrier in an IBM research laboratory. New formulation product comprising aqueous preparations of tin (II) fluoride and zinc sulphate can be used to prevent and treats skin diseases in bovine, equine, canine and other animal sectors. In toothpaste, tin is very suitable to be used as ingredient in toothpaste for example stannous fluoride can kill bacteria and fills microscopic holes in teeth compared to normal fluoride (MSC, 2011).

Perak has the largest deposit of limestone in Peninsular and second state with the highest limestone resources in Malaysia. Analysis showed that 65% of limestone hills are located in Kinta Valley with 30% is located at Simpang Pulai and Keramat Pulai. Limestone reserves in these two areas are 219.6 million tonnes, or 60% of total reserves available in the State (Aliman *et al.*, 2009). In 2010, about 32.4 million tonnes of limestone was produced from 81 quarries. This production decreased by 10% from 35.8 million tonnes recorded in 2009. Most of the limestone produced was from the state of Perak. In that year, Perak was a leading producer of limestone in Malaysia which amounts to 4.4 million tonnes or 44 percent of total national output. The other states that involved high limestone production in 2010 were Sarawak, Kedah, Perlis, Negeri Sembilan, Pahang and Selangor (Ismail *et al.*, 2011). Malaysia lime and limestone powder industry are still in small scale compared to world market. In 2010, there were 49 manufacturers of lime and limestone powder manufacturers recorded in Malaysia. They were 20 lime manufacturers, 26 limestone manufacturers and 3 dolomite manufacturers (Hussein *et al.*, 2011). The lime and limestone product are very suitable to be applied as acid mine drainage treatment.

There are four industries in the agricultural sector that are potential to be developed namely flower, poultry, livestock and other agricultures (Ariff *et al.*, 2012). The waste from these agriculture industries can be turned into organic compost or fertilizers which in turn can be used to treat acid mine drainage (AMD) especially in case of sulphate content in the effluent. For example, enhancement of agriculture products has indirectly contributed to the high demand for the use of organic fertilizers. If chicken farm waste is directly use as an organic fertilizer without any particular processes, it will cause environmental problem (Arifin *et al.*, 2006). Organic compost or organic materials can be used in the remediation of mining effluent water pollution especially in reducing the sulphate content (Zagury *et al.*, 2006; Riefler *et al.*, 2008).

Previous studies showed that agriculture solid waste for example palm oil fuel ash, coconut shell, rice husk, mangosteen peel, corn cobs and durian shell also can be used as materials to treat heavy metals in waste industrial water as biosorbents. The heavy metals that can be treated by these agricultural solid wastes are copper, lead, zinc, nickel and chromium (Mohammed *et al.*, 2014).

1.2 Statement of Problem

The study is focussed on the AMD problem that occurred in a tin tailing pond. The tin tailing pond is in a tin mine located in northern Perak. The tin tailing pond is a recycled pond which involves the activities related to tin ores. The tin ores are extracted from hard rock materials that have been processed in the processing plant. Consequently, these activities can cause AMD. This mine collects all the material from its processing plants and then discharged back into the tailing pond. The processing plants used crusher, milling and gravity concentration techniques such as palong, jig and shaking tables in extracting tin ore. The tailing pond is the detention reservoir which is the overflow water or run off that occurs with heavy rain or over flow from the nearest ponds. The pond contains heavy

metals, high sulphate content and its pH value is below 4. The damaging of equipment such as water pump can be avoided if the water in the tailing pond is less acidic. Therefore, a suitable remedial technique needs to be carried out to arrest this problem.

1.3 Significance of Findings

The study has proven that passive treatment by using single vertical column can be used as a technique to treat AMD generated from an active tin mine. The study has shown that limestone can be used as neutralisation agent and can reduce heavy metals content in AMD. The study also has determined that organic fertiliser (goat dung) can reduce the content of sulphate in AMD and the combination of limestone and organic fertiliser with two different arrangements; organic fertiliser/limestone and limestone/organic fertiliser can be used to treat AMD.

1.4 Objectives of Study

The objectives of this research are:

- 1) To show that the combination of limestone and organic fertiliser is suitable to be used in acid mine drainage treatment.
- 2) To determine the ability of organic fertiliser to reduce the content of sulphate.
- 3) To optimise the parameter of mass of limestone and organic fertiliser in a vertical column in neutralising acidic water and reducing heavy metals.

1.5 Scope of Study

Sample of high grade limestone that was used in this study is from a quarry located at Simpang Pulai, Perak. Characterisation of the limestone was done by using X-ray Fluorescence (XRF). The aim of the characterisation is to determine the content of the major mineral phases and impurities. Other material that was used in this study was organic material (goat dung). This organic material was in form of ready mix fertilisers that can be bought from nurseries around Ipoh area. The combination of two materials were also used by two different arrangements. First arrangement, organic fertiliser was placed on the top layer and limestone was second layer in the column. For second arrangement, limestone was placed on the top and organic fertiliser as second layer in the column. The water sample was collected from a tin tailing pond in the area of the mine. ICP-OES equipment, DR2800 portable spectrophotometer and pH meter were used to analyse the water samples before and after treatment.

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