

LEACHING CHARACTERISTIC OF RED
GYPSUM UNDER COLUMN AND
OEDOMETRIC CONDITIONS

NOOR HIDAYAH BINTI MOHD HAZARI

B. ENG (HONS.) CIVIL ENGINEERING

UNIVERSITI MALAYSIA PAHANG



SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the Bachelor Degree of Civil Engineering

(Supervisor's Signature)

Full Name : DR. HJ. MOHD YUHYI BIN HJ. MOHD TADZA

Position : SENIOR LECTURER

Date : 14th Jan 2019



STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

(Student's Signature)

Full Name : NOOR HIDAYAH BINTI MOHD HAZARI

ID Number : AA15009

Date : 14th Jan 2019

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ABSTRACT

Red gypsum (RG) is a by-product produced during the extraction of titanium dioxide (TiO_2) from ilmenite ores. Unregulated and rampant disposal activity of RG might be an issued due to waste gypsum arising from chemical industry or power plant annually without being utilize. The leachability of heavy metals of this waste will be leach into the soil groundwater. The main objective of this study is to determine the leaching characteristics of RG under Column and Oedometric conditions and comparing the concentration of elements with Toxicity Characteristic Leaching Procedure (TCLP), Total Threshold Limit Concentration (TTLC) and Drinking Water Quality Standards (NDWQS). The concentrations and types of heavy metals were analyzed using the Inductively Coupled Plasma Mass Spectrometry (ICP-MS) method. Experimental results demonstrated that only a few of the major elements that are traced from both leaching tests carried out by Column leaching test and Oedometer leaching test such as Mg, Ca, Na, Al and K. The highest concentration of major element is Mg (129.5 mg/L). In addition, some of heavy metals produced are Mn, Ni, Cu, Zn and Pb. The highest concentration of these five heavy metals is Zn (0.031 mg/L). However, the value of this concentration still does not exceed the limits provides in TTLC and NDWQS with the value of 5000 mg/L and 3 mg/L respectively. The results recorded element concentration under Column leaching test were higher than Oedometer leaching test. Overall, all element concentrations of RG were found to all relevant consideration standards of TCLP, TTLC and NDWQS as the elements that leach out were found to be less than allowable limits.

ABSTRAK

Gypsum Merah adalah merupakan sisa daripada proses pengekstratan titanium dioksida (TiO_2) dari bijih ilmenit. Di Malaysia, lebih daripada 1000000 ton setahun gipsium merah terhasil. Justeru, aktiviti longgokan RG di tapak pelupusan terkawal semakin berleluasa disebabkan oleh peningkatan sisa gypsum daripada industri kimia dan jana kuasa tanpa dikitar semula. Oleh itu, keterlarutan sisa ini akan diserap masuk ke dalam tanah apabila berlakunya hujan di tapak-tapak pelupusan tersebut. Objektif utama kajian ini dijalankan adalah untuk mengkaji ciri-ciri larutan sisa RG yang mengandungi beberapa logam berat di dalamnya samada menepati piawaian yang ditetapkan oleh Prosedur Penyulingan Ciri Ketoksikan (TCLP), Jumlah Kepekatan Had Ambang (TTLC) dan Piawai Kualiti Air Minuman (NDWQS) melalui ujikaji Larutan Lajur dan ujikaji Larutan Penyatuan. Kepekatan dan jenis logam berat dianalisa menggunakan kaedah Spektrometri Jisim Plasma yang digabungkan secara Induktif (ICP-MS). Keputusan ujikaji menunjukkan hanya beberapa jenis elemen utama yang terhasil daripada kedua-dua kaedah ujikaji yang dijalankan melalui ujikaji Larutan Lajur dan ujikaji Larutan Penyatuan seperti Mg, Ca, Na, Al dan K. Selain itu, antara logam berat yang terhasil adalah seperti Mn, Ni, Cu, Zn dan Pb. Namun, nilai kepekatan yang tertinggi antara kelima-lima logam berat tersebut adalah Zn (0.031 mg/L). Walaubagaimanapun, nilai kepekatan ini masih tidak melebihi had yang ditetapkan di dalam TTLC dan NDWQS iaitu masing-masing dengan nilai 5000 mg/L dan 3 mg/L. Kajian ini juga menunjukkan keputusan kepekatan logam berat dan elemen utama adalah lebih tinggi di dalam ujikaji Larutan Lajur berbanding ujikaji Larutan Penyatuan. Secara keseluruhan, keputusan kepekatan elemen-elemen gipsium merah adalah mematuhi semua piawaian yang dirujuk iaitu TCLP, TTLC dan NDWQS yang mana kesemua nilai kepekataannya adalah tidak melebihi had yang dibenarkan.

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LIST OF SYMBOLS

°C	Degree Celsius
%	Percent
M _v	Coefficient of volume change
C _v	Coefficient of consolidation
C _α	Coefficient of secondary compression
C _c	Compression index
C _s	Swell index
C _u	Undrained Shear Strength
Na	Sodium
Mg	Magnesium
Al	Aluminium
Ca	Calcium
K	Potassium
Cr	Chromium
Mn	Manganese
Fe	Iron
Co	Cobalt
Ni	Nickel
Cu	Copper
Zn	Zinc
As	Arsenic
Mo	Molybdenum
Cd	Cadmium
Ba	Barium
B	Boron
P	Phosphorus
Pb	Lead
Si	Silicon
Al ₂ O ₃	Aluminium(III) Oxide
SiO ₂	Silicon Dioxide
SO ₃	Sulfur Trioxide

CaO	Calcium Oxide
TiO ₂	Titanium Dioxide
V ₂ O ₅	Vanadium(V) Oxide
Cr ₂ O ₃	Chromium(III) Oxide
MnO	Manganese Oxide
Fe ₂ O ₃	Iron(III) Oxide / Ferric Oxide
CuO	Copper(II) Oxide
ZnO	Zinc Oxide
SrO	Strontium Oxide
ZrO ₂	Zirconium(II) Oxide
RuO ₂	Ruthenium(II) Oxide
Re ₂ O ₇	Rhenium(VII) Oxide
HgO	Mercury(II) Oxide
CaCO ₃	Calcium Carbonate
FeCO ₃	Iron Carbonate
CO ₂	Carbon Dioxide
CaSO ₄	Calcium Sulphate
TiOSO ₄	Titanyl Sulphate
FeSO ₄	Iron Sulphate

LIST OF ABBREVIATIONS

ASTM	American Society for Testing Material
BS	British Standard
CBR	Californian Bearing Ratio
CEC	Cation Exchange Capacity
DOE	Department of Environment
GGBS	Ground Granulated Blast furnace Slag
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
LL	Liquid Limit
MPW	Mineral Processing Waste
NDWQS	National Drinking Water Quality Standard
PDF	Permanent Disposal Facility
PL	Plastic Limit
SL	Shrinkage Limit
SW	Schedule Waste
TCLP	Toxicity Characteristic Leaching Procedure
TTLC	Total Threshold Limit Concentration
XRD	X-Ray diffraction
XRF	X-Ray fluorescence

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Red gypsum (RG) is a by-product during the extraction of titanium dioxide (TiO_2) from the ilmenite ores (Kamarudin and Zakaria, 2007; Azdarpour et al., 2014; Azdarpour et al., 2015a; Azdarpour et al., 2015b; Azdarpour et al., 2015c; Azdarpour et al., 2017; Mahazam and Azmi, 2016; and Fauziah et al., 1996). The red colour of this waste product is due the presence of iron (Fauziah et al., 1996). In Malaysia, RG produced is more than 1000000 tonnes annually and generate a large amount of volume (Sidek et al., 2017). Most of this waste is dumped in secured landfills without being utilized (Sidek et al., 2017). With the current trend of production, eventually there will be a new application from RG waste generator to build an additional landfill to cater the growing demands of its RG waste handling.

Malaysia is considered one of the major RG producers in the world (Azdapour et al., 2015b). In recent years, a lot of research have been come out to determine the applicability of RG. By using RG in an effective way, landfill problem can be reduced as well as make benefit on it. For example, utilizing RG in cement brick manufacturing can reduce the piling of RG at landfills significantly (Sidek et al., 2007). According to Ahmad et al., (2012), gypseous soils are usually stiff in their dry state due to the cementing action provided by gypsum, but great loss of strength and pronounced increase in compressibility occur when gypsum is dissolved by partial or full saturation. This problem becomes more severe when the water flows through such soils causing loss of mass due to the leaching of gypsum.

An assessment of the leachability of toxic substances from contaminated soils is crucial for evaluating their environmental impact on soil and groundwater. Leaching test

are commonly used to assess the leaching potential of toxic substances and understand the leaching behaviour. Several standards of Column Leaching tests (ISO/CD 21268-3, 2017; DIN 19528, 2009; USEPA Method 1314, 2013) enable contact between contaminated soil and flowing leachant and used to indicate the leaching behaviour of target substance under simulated field conditions. Column tests are well-suited to determining the concentrations of constituents that can be released over a relatively small number of pore volumes, which corresponds to shorter time periods and desorption or dissolution rates of contaminants from various materials (Manoj et al., 2015).

In this study, the leaching characteristic of RG will be tests under Column and Oedometer conditions and compare with the Toxicity Characteristic Leaching Procedure (TCLP), Total Threshold Limit Concentration (TTLC) and National Drinking Water Quality Standard (NDWQS). The TCLP is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphase wastes (TCLP Method 1311, 1992). While, TTLC is an analysis to determine the total concentration of each target analyte in a sample (Micro Analytical Laboratories, Inc., 2018). The NDWQS describes the quality parameters set for drinking water. This guidelines are intended to support the development and implementation of risk management strategies that will ensure the safety of drinking water supplies through the control of hazardous constituents of water (World Health Organization, 2006). All the concentration of elements need to be comply to all these three allowable limits to assess the potential for land application.

1.2 Problem Statement

Red gypsum (RG) is known as waste gypsum produced from chemical industry or power plant such as Hunstman Tioxide, Kemaman, Terengganu, Malaysia which process a production and manufacturing. This waste has been categorized as schedule waste as comply in Environmental Quality Act 1974, under the Environmental Quality (Schedule Waste, SW 205); waste gypsum arising from chemical industry or power plant (DOE, 2010). Meanwhile, the schedule waste need to be disposed in hazardous landfill or permanent disposal facility (PDF). In fact, there are some heavy metals found in RG which may cause pollution (Fauziah et al., 1996).

Anyhow, there is no minor or major case reported yet regarding this hazardous, but still it has to be investigated as this thing labelled as perilous waste. In this study,

this waste going to be tests under some leaching condition in the laboratory to conclude if there is no harmful substances at all then it could be good to reuse it to benefit others, but leaching maybe an issue. So, leaching characteristic of RG is crucial for determination assessment of environmental hazard.

1.3 Research Objectives

The purpose of this study is to test either the waste of RG known as scheduled waste as listing in SW 205 by DOE is classified as stable or unstable waste characteristic by conducting all the testing in laboratory. This study also covers the following objectives:

- (i) to determine the leaching characteristic of red gypsum under Column and Oedometer conditions.
- (ii) to compare leaching characteristic with Toxicity Characteristic Leaching Procedure (TCLP), Total Threshold Limit Concentration (TTLC) and Drinking Water Quality Standard (NDWQS).

1.4 Scope of Study

In this study, Red Gypsum from Huntsman Tioxide Sdn. Bhd. in Kemaman, Terengganu, Malaysia was considered. Samples were transported from the site to the laboratory. Several laboratory test were carried out to obtain the geotechnical waste properties and the parameters required to determine the leaching characteristic of RG. Two major laboratory tests need to consider to obtain the leaching water of sample by conducted column test while oedometer test should applied some different loading for the sample used. The leaching water sample, then will be tested in Central Laboratory of University Malaysia Pahang using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) analyser to determine the parameter of the sample. From the results of sample parameters by ICP-MS testing, then compared the leaching characteristic of RG under Column and Oedometer conditions with the Toxicity Characteristic Leaching Procedure (TCLP), Total Threshold Leaching Concentration (TTLC) and National Drinking Water Quality Standard (NDWQS) to consider the leaching contamination environment hazard.

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