# AN INVESTIGATION OF RED GYPSUM AS REPLACEMENT MATERIAL TO IMPROVE THE PERFORMANCE OF GROUNDING SYSTEM

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B. ENG(HONS.) CIVIL ENGINEERING

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# AN INVESTIGATION ON RED GYPSUM AS REPLACING MATERIAL TO IMPROVE THE PERFORMANCE OF GROUNDING SYSTEM

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Thesis submitted in fulfillment of the requirements for the award of the B. Eng (Hons) Civil Engineering

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#### ABSTRAK

Sistem pembumian berfungsi sebagai perlindungan kilat di loji perindustrian dan kuasa. Salah satu parameter utama untuk menentukan prestasi sistem pembumian adalah resistiviti tanah tempatan. Gipsum yang dihasilkan di kilang ataupon berlaku secara semula jadi, merupakan mineral putih atau kelabu yang lembut terdiri daripada kalsium sulfat terhidrat yang digunakan sebagai bahan penambahbaik tanah. Kandungan gipsum didapati meningkatkan kekonduksian bahan elektrik. Sebaliknya, gipsum merah (GM) adalah sisa yang dihasilkan daripada proses sulfat bijih ilmenit untuk memperoleh titanium dioksida. Oleh kerana kandungan gipsum dalam kedua-dua bahan adalah sama, diharapkan gipsum merah boleh menjadi alternatif kepada gipsum putih. Dalam kajian ini, GM diuji dan dibandingkan dengan gipsum putih untuk melaksanakan bahan asas. Ciri-ciri geoteknik seperti sifat fizikal dan kimia diuji terlebih dahulu, diikuti dengan menghasilkan graf soil water retention curve (SWRC) dan menentukan kelakuan resistiviti. Teknik kaedah titik embun cermin dingin dan teknik osmotik digunakan untuk menghasilkan SWRC manakala teknik 2 titik meter resistiviti kotak tanah digunakan untuk mengukur resistiviti kedua-dua gipsum dan GM dalam keadaan yang berbeza. Keputusan ujian menunjukkan bahawa sifat-sifat geoteknik GM adalah berbeza dengan gipsum putih. GM menyerap lebih banyak air dan mempunyai ciri plastisiti yang lebih tinggi berbanding dengan gipsum biasa. Keberintangan gipsum dan GM adalah 8 Ω.m dan 11  $\Omega$ .m, masing-masing. Ini menunjukkan bahawa gipsum putih adalah bahan penambaikan untuk sistem pembumian yang lebih baik daripada GM, namun perbezaannya didapati sangat kecil.

#### ABSTRACT

Earth grounding system serves as lightning-protection in industrial and power plants. One of the major parameters to determine the performance of the grounding system is the resistivity of local soil. Natural occurring or manufactured Gypsum, a soft white or grey mineral consists of hydrated calcium sulphate is used as ground enhancement material. The gypsum content was found to improve the electrical conductivity of materials. On the other hand, red gypsum (RG) is a waste generated from a sulphate process of ilmenite ore to acquire titanium dioxide. Due to the gypsum content in both material are similar it is expected that red gypsum can be an alternative to white gypsum. In this study, RG was tested and compared with white gypsum to perform a grounding material. The geotechnical properties such as physical and chemical properties were tested first, followed by establishing soil-water retention curve and determination of the resistivity behaviour. A chilled-mirror dew-point and osmotic techniques were used to establish SWRC whereas a 2-point resistivity meter and soil box attachment were used to measure the resistivity of both gypsum and RG under different condition. Test results showed that, the geotechnical properties of RG is different to that of white gypsum. The RG absorbed more water and has higher plasticity characteristic as compared to common gypsum. The resistivity of gypsum and RG are 8  $\Omega$ .m and 11  $\Omega$ .m, respectively indicating that gypsum is slightly better grounding material than RG, however the differences was found to be verv small.

# TABLE OF CONTENT

DEC	CLARATION	
TITI	LE PAGE	
ACK	KNOWLEDGEMENTS	ii
ABS	TRAK	iii
ABS	TRACT	iv
TAB	BLE OF CONTENT	v
LIST	Γ OF TABLES	viii
LIST	Γ OF FIGURES	ix
LIST	Γ OF SYMBOLS	X
LIST	Γ OF ABBREVIATIONS	xi
СНА	APTER 1 INTRODUCTION	12
1.1	Background	12
1.2	Problem Statement	15
1.3	Research Objective	15
1.4	Scope of Study	15
1.5	Thesis Overview	16
СНА	APTER 2 LITERATURE REVIEW	18
2.1	Introduction	18
2.2	White Gypsum	18
2.3	Red Gypsum	19
	2.3.1 Titanium Dioxide Industry	19

	2.3.2	RG Waste	20
2.4	Earth	Grounding System	22
	2.4.1	Importance of Earth Grounding System	22
	2.4.2	Earth Grounding Resistance	23
	2.4.3	Grounding Enhancement Material	24
2.5	Soil W	Vater Retention Curve (SWRC)	26
2.6	Soil R	esistivity Behaviour	28
CHA	PTER 3	METHODOLOGY	30
3 1	Introd	uction	30
3.1	Select	ion of Material	30
3.2	Mator	ials Preparation	31
5.5	3 3 1	Powder	31
	332	Shurry	31
34	RG Pł	vsical Properties	31
3.5	RG CI	hemical Properties	32
e le	3.5.1	Cation-Exchange Capacity	32
3.6	Soil W	Vater Retention Curve	33
	3.6.1	Osmotic Technique	33
	3.6.2	Vapour-Equilibrium Technique	35
	3.6.3	Chilled-Mirror Dew-Point Technique	37
CHA	PTER 4	RESULTS AND DISCUSSION	38
4.1	Introd	uction	38
4.2	RG Pł	nysical and Chemical Properties	38
4.3	Soil W	Vater Retention Curve	39

REFE	REFERENCES		46
CHAI	PTER 5	CONCLUSION	45
4.4 Resistivity		43	
	4.3.3	Soil-Water Retention Curve	41
	4.3.2	Vapour Equilibrium Technique (VET)	40
	4.3.1	Osmotic Technique	39

# LIST OF TABLES

Table 2.1	Properties of WG against RG	20
Table 2.3	Soil resistivity and earthing resistance for different type of soils	25
Table 2.3	Soil resistivity and earthing resistance for different type of soils	25
Table 3.1	Standards used for characteristics testing	29
Table 3.2	Saturated salt solutions and corresponding suction	31
Table 4.1	Physical and chemical properties result of RG from the experiment	33

# LIST OF FIGURES

Figure 2.1	Diagram of suplhate process	19
Figure 2.3	Schematic diagram of a dew-point Water Potentia Meter, WP4	24
Figure 3.1	RG stored in container	28
Figure 3.2	Set-up of osmotic technique	31
Figure 3.3	Procedure of vapour equilibrium technique	32
Figure 3.4	WP4C Potentia dew meter for soil suction reading	34
Figure 4.1	Osmotic technique on change in water content as time elapse	35
Figure 4.2	Vapour Equilibrium on change in water content as time elapse	36
Figure 4.3	SWRC drying curve of RG	37

# LIST OF SYMBOLS

Ω	ohm
%	percentage
Ts	Sample temperature
Tb	Chamber temperature
$\mu$	micron

# LIST OF ABBREVIATIONS

RG	Red Gypsum
WG	White Gypsum
SWRC	Soil-Water Retention Curve
VET	Vapour Equilibrium Technique
IEEE	Institutes of Electrical and Electronics Engineers
GEM	Ground Enhancement Material
PL	Plastic Limit
LL	Liquid Limit
SL	Shrinkage Limit
CEC	Cation Exchange Capacity
Gs	Specific Gravity
S	Applied suction
c	Concentration of PEG solution in gram
PEG	Polyethylene glycol
MPa	Mega Pascal

#### **CHAPTER 1**

#### **INTRODUCTION**

#### 1.1 Background

Lightning strikes affect the building structures by producing high electric field on the surface and around the building structure (Omar *et al.*, 2016). Up to the present, several methods have been proposed to improve the lightning performance of transmission lines, including utilizing unbalanced insulation, adding extra insulation, adding coupling overhead ground wires, reducing protective angles of overhead shield wires, decreasing grounding resistance of tower grounding devices, and installing line surge arresters on transmission lines (Youping *et al.*, 2006).

Earth grounding system serves as lightning-protection in industrial and power plants (Liu *et al.*, 2019). When grounding system is under fault, the system will separate the fault current to the earth by providing low resistivity path to decrease the earth potential rise at the local grounding system (Nazar *et al.*, 2018). A grounding system refers to metallic wire of various geometric shapes and sizes, acting as electrodes and buried in the soil (Nazar *et al.*, 2018)

Usually, the impulse grounding resistance is used to measure the performance of transmission tower grounding devices, which is a ratio of the peak value of the impulse voltage generated on the grounding device and the peak value of the impulse current flowing through the device (Youping *et al.*, 2006). The grounding impedance is reported

to be the most important criteria for the grounding system so its impedance should be maintained in low levels for long duration (Azmi *et al.*, 2019). Soil type is a primary factor to determine the grounding resistance value but the problem is different soil possesses different behaviour and characteristic such as resistivity, ionization, and the level of corrosive environment (Azmi *et al.*, 2019). Electrical resistivity of the soil can be considered as a proxy for the spatial and temporal variability of many other soil physical properties (i.e. structure, water content, or fluid composition) (Samouëlian *et al.*, 2005).

Soil resistivity is the key factor that determines what the resistance of a grounding electrode will be, and to what depth it must be driven to obtain low ground resistance (Igboama and Ugwu, 2011). In fact, to obtain the low level of impedance in the rocky and sandy soil is somewhat impossible, therefore, many researchers have introduced several techniques to reduce and maintain the grounding resistant in low level (Azmi *et al.*, 2019). . In these cases, ground enhancement materials or backfill material are used to enhance the grounding system to attain the required ground electrode resistance (Boling, 2006; Lim *et al.*, 2015).

A good ground enhancement material should provide low earth resistance over a long period with little variation of resistivity value (Lim *et al.*, 2015). Bentonite is one of the suitable example of backfill material in decreasing and maintaining the low grounding resistance of electrodes for a long time due to its high water absorption and retention tendency (Lim *et al.*, 2015). According to research by Fukue *et al.*, (1999), the resistivity for bentonite is high when water content is low, however, when the water content in bentonite is more than 40%, the resistivity is as low as  $3\Omega m$ .

Recently, researchers have taken an initiative to study the possibility to use the waste product in a grounding system (Azmi *et al.*, 2019). Several types of waste products from the industry have been identified in reducing grounding impedance such as fly ash, rice straw ash and bagasse ashes (Nazar *et al.*, 2018).

On the other hand, white gypsum (WG) which occurs in several forms, most common among which are the dihydrate (CaSO<sub>4</sub>2H<sub>2</sub>O) and anhydrite (CaSO<sub>4</sub>) are used widely in industry is also a water absorber, which may improve electrical conductivity of material is commonly used as backfill material (Karni and Karni, 1995).

In addition, red gypsum (RG) is a kind of waste product that is produced during the production of titanium dioxide in the industry (Fauziah, *et al.*, 1996). The total accumulation of RG in Malaysia is at least 340,000 tons per year (Kamarudin and Zakaria, 2007). One of the co-product which is RG which contain mainly iron hydroxide and gypsum (Fe(OH)<sub>2</sub>+CaSO<sub>4</sub>) (Fauziah, *et al.*, 1996). RG is named due to its reddish brown like colour due to a high presence of Fe<sup>3+</sup> (Iron III) but with a properties of gypsum. Due to the smaller particles of iron in contrast to the red gypsum particles, it will coat around the gypsum particles and thus suspected to be the main concern to its unexpected behaviour (August *et al.*, 2003).

In this study, RG is tested for its characteristic that will be useful information for unsaturated soil and highly plasticity clay that are current scarce. Highly plasticity clay is often used in creating a barrier or backfill material for grounding system. General properties such as physical and chemical properties are tested using standard laboratory procedure. Testing included the measurement of particle size analysis, cation-exchange capacity and some important properties including atterberg limit, surface area and organic matter

#### **1.2 Problem Statement**

Red gypsum (RG) is a by-product during the extraction of titanium (IV) oxide from the ilmenite ores and it causes storing problems. Due to its large amount of coproduct, RG is always dealt with landfill technique of disposal but it will cost up a lot of land. It cannot be used in cement factory due to its reddish colour. Hence, RG cannot be sold in market. Red gypsum is a material that can improve the conductivity of soil. When conductivity is higher, the electrical resistivity will be lower. Thus, in order to solve the enormous amount of red gypsum being wasted in landfill, red gypsum can be replaced as grounding material to replace the unavailable bentonite due to its properties and characteristics.

#### **1.3** Research Objective

The primary objective of this research were as follows:

- (i) to determine the properties of red gypsum
- (ii) to establish soil-water characteristics retention curve (SWRC) for red gypsum
- (iii) to compare the resistivity of red gypsum and white gypsum

#### 1.4 Scope of Study

In this study, RG from Venator Materials Corporation was considered and experiment are conducted on RG. The resistivity of RG can be compared with common WG following to Standards Institute of Electrical and Electronics Engineers (Standard IEEE) using a 2 point resistivity meter.. The performance of red gypsum is being analysed in laboratory with varied water content whether dried or wet. The limitations of the study are in term of resistivity, grounding and red gypsum.

#### **1.5** Thesis Overview

**Chapter 2** presents the overview of other research related to the study which include earth grounding system, white gypsum, red gypsum, soil water characteristic curve and soil resistivity behaviour. Earth grounding system is explained on its importance and how the factors affecting soil resistivity influence the grounding resistivity. This chapter also overview about gypsum which function as backfill materials in earth grounding system. Waste gypsum that mention in this chapter is red gypsum. Origin, structure, properties and applications of RG are discussed. Furthermore, mechanisms of water absorption of RG in modifying the plasticity and resistivity is explained. Lastly, soil water retention curve (SWRC) is explained in this chapter to illustrate the relation between soil water content and soil suction.

**Chapter 3** presents the experimental methodologies of this study which include the selection of materials such as WG and RG. The preparation of RG and WG samples, physical properties, chemical property and resistivity of materials whether dried or slurry. Physical properties of RG that to be determined include liquid limit (BS 1337: Part 2 1990: 4.3), plastic limit (BS 1377: Part 2: 1990: 5.3), shrinkage limit (ASTM D4943-08), specific gravity (BS 1377: Part2: 1990:8.3), swelling index (IS: 2720 (Part 40) 1977), specific surface area (ISO 9277:2010, DIN ISO 9227: 2013), loss of ignition (BS1377: Part 3: 1990: 4.3) and soil water characteristics curve (ASTM C387-99) while cation exchange capacity (ASTM D6836-07) is the chemical property to be determined. Lastly, resistivity test will be conducted by using 2-pin soil box method (ASTM G57) to determine and compare the resistance of RG and WG.

**Chapter 4** presents the results obtained from the laboratory tests. The results are compared and discussed by using RG and WG samples. Besides, all the physical

properties such as liquid limit, plastic limit, shrinkage limit, specific gravity, swelling index, specific surface area, loss of ignition and soil water retention curve, chemical property such as cation exchange capacity and resistivity behaviour are compared and explained with or without adding of water.

**Chapter 5** presents the conclusion of this study which include results and discussions, any important findings and recommendations should be made for next research.

### **CHAPTER 2**

#### LITERATURE REVIEW

#### 2.1 Introduction

This chapter presents relevant information pertaining to the study undertaken. This chapter is presented into two subsections which will be discussed in this chapter. The focus of the chapter will be on the white gypsum (WG) and red gypsum (RG) and the technique that are going to be use in this research.

### 2.2 White Gypsum

Gypsum is a sulphate mineral made up of hydrated calcium sulphate, CaSO<sub>4</sub>.2H<sub>2</sub>O. It is a very soft mineral with a Mohr's scale hardness of 1.5-2.0 and can be easily scratched with a finger nail (Bhamidipati, 2016). Gypsum consists of about 21% water by weight and 50% water by volume as cited by Bhamidipati (2016). According to Louie *et al.*, (2012), heating of gypsum causes it to lose three-fourths of its water and form calcium-sulphate hemihydrate (2CaSO<sub>4</sub>.H<sub>2</sub>O) which is commonly known as plaster of Paris. This material is mixed with water to form a paste that dries and sets to form a hard material.

Due to its abundance and physical and chemical properties, gypsum is widely used as a construction material in many parts of the world (Afsharian *et al.*, 2016). In engineering, gypsum is used to produce wallboard and plaster of Paris. It is added to Portland cement and to calcium sulfoaluminate cements. Gypsum also used to prevent premature hardening or flash setting, and it is used in agriculture as a soil conditioning agent as cited by Tang *et al.*, (2018).

#### 2.3 Red Gypsum

#### 2.3.1 Titanium Dioxide Industry

One of the TiO<sub>2</sub> industry in Malaysia would be Venator Materials Corporation which is located at Teluk Kalong Kemaman Terengganu Malaysia. Compared to ordinary belief, titanium is not widely used in metal and alloys but rather is TiO<sub>2</sub> pigment that have a greater demand due to its uses in providing whiteness and opacity in a vast range of products such as coating, cosmetics and food (Zhang *et al.*, 2018). There is an annual global production of 4.5 million metric tonnes of TiO<sub>2</sub> and only 4%-5% is used in metallic titanium (Rosenbaum, 1982)

The industry extract TiO<sub>2</sub> from an ore named ilmenite by sulphate process. Sulphate process which use concentrate sulphuric acid could produce about 40% TiO<sub>2</sub> pigment out of total production (Gazquez *et al.*, 2013). In sulphate process, ilmenite (40% - 60% TiO<sub>2</sub>) or titanium slag (72% - 85% TiO<sub>2</sub>) or even a carefully controlled blend, is digested with concentrated sulphuric acid (98%). A highly-exothermic reaction is initiated by the addition of measured quantities of steam, water and diluted sulphuric acid.(Hughes *et al.*, 2011). Figure 2.1 showing the process of titanium dioxide production. From the sulphate process, one of the co-product formed would be RG resulting from the final stage of TiO<sub>2</sub> washing and added with lime or limestone for neutralisation. The RG formed mainly of iron hydroxides and gypsum according to the reaction below:

$$Ca(OH)_2 + H_2SO_4 \rightarrow CaSO_4 \cdot 2H_2O \tag{2.1}$$

$$FeSO_4 + Ca(OH)_2 \rightarrow Fe(OH)_2 + CaSO_4$$
(2.2)

The magnitude of co-products generated from an annual process of 142,000 metric tonnes from Huntsman industry, there will be a generation of 70,000 metric tonnes of RG (Gazquez *et al.*, 2013)



Figure 2.1 Diagram of suplhate process Source: Gazquez *et al.*, (2013)

#### 2.3.2 RG Waste

RG or secondary gypsum is basically calcium sulphate in varying states of hydration with iron oxide varying from 3-35% and some other trace elements (August *et al.*, 2003). RG is produced globally with the same raw material and same processes. They might be some minor difference in composition due to the impurities or attached other element at the ilmenite ore. RG is currently an industrial waste material which is currently disposed. Once the waste is generated, it is likely to be dump through landfill rather than reprocessing it to be utilized or commercialized (Gazquez *et al.*, 2013) since reprocessing

it is not economical friendly due to the reprocessing fee to purified gypsum is greater than final product value (August *et al.*, 2003)

One of the common difference would be the colour of the gypsum which red gypsum is practically reddish brown and gypsum is white. Normal gypsum would be white in colour but as for red gypsum, the main contribution factor to its reddish brown appearance is its high composition of FeO<sub>3</sub> consisting of 28.99% (Azdarpour *et al.*, 2018). RG has a very well acceptable strength, stiffness and a very low permeability that allows it to be used as an engineering material such as cement, natural soil and slag for uses in civil engineering application. Table 2.1 is a comparison between the properties of WG and RG. RG may not be applicable in gypsum board production due to its reddish brown colour, but it has a very interesting properties of its own that are applicable in other field such as soil conditioner, replacing natural gypsum in manufacturing of cements (Gazquez *et al.*, 2013) and inhibitors in soil erosion for mobilization of heavy metals in soil (Fauziah *et al.*, 1996)

Properties	White Gypsum	<b>Red Gypsum</b>
рН	7.4	7.4
Free Moisture (%)	10-17	10-50
Particle Density (mg/m <sup>3</sup> )	3.05	2.71
Dry Density (mg/m <sup>3</sup> )	1.21	1.21
Erodability (Dispersiveness)	non-dispersive 2	dispersive
		4
Liquid Limit (%)	58	105
Plastic Limit (%)	non-plastic	non-
		plastic
Californian Bearing Ratio (CBR %)	not obtainable	23.05
Optimum Water content (%)	39	41
Consolidation Testing (50-400 kN/m <sup>2</sup> )	-	-
Coefficient of Volume Change (Mv m <sup>2</sup> /year)	3.643-0.32	0.907-
		0.179
Coefficient of Consolidation (C <sub>v</sub> m <sup>2</sup> /year)	1.19-0.102	0.855-
		0.232
Coefficient of Secondary Compression ( $C_{\alpha}$ m <sup>2</sup> /year)	0.05-0.02	0.004-
		0.002
Compression Index C <sub>c</sub>	0.218	0.242
Swell Index C <sub>s</sub>	0.08	0.131
Peameability ( $kv_x 10^{-9}$ m/s)	95-104	194-355
Strain to Failure (%)	5	10

Table 2.1 Properties of WG against RG

Source: August et al., (2003)

### 2.4 Earth Grounding System

### 2.4.1 Importance of Earth Grounding System

Grounding system is one of the important elements in electricity, where it is defined as a zero voltage point in electrical system in which the point is normally connected to ground or earth as cited by (Shuhada *et al.*, 2016). It is a system that has form of grid of horizontally buried conductors, supplemented by a number of vertical rods connected to the grid (Hamzah, 2009). Electrical grounding systems are designed to dissipate unwanted electrical charges as efficiently as possible (Lim *et al.*, 2015) In grounding systems designed to cater for lightning protection, considerably large amount

of lightning-brought charge has to be dispersed by the grounding system within microsecond time scale (Weng Choun *et al.*, 2012; Lim *et al.*, 2015).

Dallas (2008) in his article mentioned that there are three main importance of the grounding system. The first one is for overvoltage protection. Lightning, line surges or unintentional contact with higher voltage lines can induce dangerous high voltages to the electrical distribution system wires. Grounding provides an alternative path around the electrical system of the building, thus minimizes damage from such occurrences. Secondly, grounding system is used for voltage stabilization. It is known that there are several voltage sources in electrical system. If there are no common reference point for all these voltage sources, hence it would be extremely difficult to calculate their relationships to each other. Further, earth is the most omnipresent conductive surface, and so it was adopted in the very beginnings of electrical distribution system is for current path in order to facilitate the operation of overcurrent devices. This is the most important purpose of grounding system because it provides certain level of safety to humans and properties in case of equipment damages

#### 2.4.2 Earth Grounding Resistance

The grounding resistance is reported to be the most important criteria for the grounding system (Lim *et al.*, 2015). A lower grounding resistance corresponds to superior grounding so its impedance should be maintained in low levels for long duration (Chen *et al.*, 2006). Practically, the back-strike trip could happen when the grounding resistance is high. Soil type is a primary factor to determine the grounding resistance value. Different soil possesses different behaviour and characteristic such as resistivity, ionization, and the level of corrosive environment as cited by (Teh, 2017)

Soil ionization increases the soil conductivity and decrease the soil resistivity. Meanwhile, corrosion could degrade the electrode conductivity performance. Besides, the weather conditions in which the location of a system is about to be installed, also compose a complex factor on grounding system, due to the variation in the soil resistivity value. The grounding resistance is reported to be vary frequently because of the weather condition and surrounding environment. In fact, to obtain the low level of resistance in the rocky and sandy soil is somewhat impossible. Therefore, many researchers have introduced several techniques to reduce and maintain the grounding resistant in low level (Azmi *et al.*, 2019). Desired grounding resistance can be achieved by connecting a number of individual electrodes instead of using single low grounding resistance electrodes (IEEE Std. 142, 2007). However, it is very difficult to get the expected grounding resistance by increasing the grid conductors as it may cost high (Azmi *et al.*, 2019).

In the above backdrop, researchers during the past several decades have started to explore the suitability of using backfill materials to minimise grounding resistance and impedance of electrical grounding systems (Lim *et al.*, 2013). Such techniques are implementation of reducing agent/backfill materials (Jasni *et al.*, 2017) or performed the chemical treatment in the grounding system (Gouda *et al.*, 2010)

#### 2.4.3 Grounding Enhancement Material

In the past several decades, researchers have started to explore the suitability of using enhancement materials (Azmi *et al.*, 2019). Nowadays, ground enhancing material or conductance-enhancement material is practice widely to decrease earth resistance due to high materials cost of earth conductors and limited space (Lim *et al.*, 2015). Ground enhancing material is usually termed as backfill materials. These materials are placed

inside the trench, where the grounding electrode is installed and mixed with the natural soil (Chen *et al.*, 2006). These materials are reported to have the ability to reduce the resistance between the contact areas. Hence, the total soil resistivity will be reduced (Azmi *et al.*, 2019).

A good backfill material provide and maintain lower earth resistance than background soil for a long time and non-reactive with electrode (Nazar *et al.*, 2018). One of the most common backfill material is bentonite. Bentonite has well conductive property, high water absorption and retention as well as sticky property to most of the types of surface that it touches (Lim *et al.*, 2013).

Ground Enhancement Material (GEM) from ERICO is another alternative for backfill material (Switzer, 2018). Switzer states that GEM has more advantages over bentonite. GEM is maintenance free as it is chemically stable and does not corrode ground electrode due to its low content of sulphate and chloride. According to ERICO, GEM powder provide resistivity less than 0.02  $\Omega$ m and this resistance is constant throughout the life of grounding system once it is set.

Other material such as metal oxide, a waste product of steel industry is also proven as good backfill material. It possesses criteria of good backfill material (Lim *et al.*, 2013) and less than 1% of corrosion on Galvanized Iron electrodes after more than two years in contact (Lim *et al.*, 2015). Another research by Jasni *et al.*, (2017) shows that coconut coir peat and paddy dust do decrease the grounding resistance values but comparing to planting-clay soil and bentonite, their grounding resistance is higher.

According to Nazar *et al.*, (2018), rice straw ash and bagasse ash is also capable as backfill material to obtain a low resistance reading. However, this material still cannot reduce the grounding resistance in accordance to Std IEEE.

#### 2.5 Soil Water Retention Curve (SWRC)

Water is retained in the soil by Van der Waals forces, electrostatic forces between molecules of different solutes and the solid phase, and interfacial tension in the capillaries. At low water contents, the forces of molecular attraction are predominant (Pozdnyakov *et al.*, 2006).Water content and its energy state characterized the water status in soil. The two principles form of energy are kinetic energy and potential energy (Caron and Boudreau, 2006). However, the kinetic energy is negligible as the water movement in soils is relatively slow. Difference in potential energy of water in soil tend to move from higher potential energy to lower potential energy until equilibrium (Mohd Tadza, 2011). When water flow, larger pores in soil will drain first and the remaining pores will hold water more tenaciously. In this condition, the water in pores is under suction or negative hydrostatic pressure (Lu *et al.*, 2017)

Soil-water characteristic curve (SWRC) shows the relationship between water content and soil suction, where the water content is the water quantity contained in the pores of the soil (Ramli, 2015) and the soil suction or partial vapour pressure of soil water is the free energy state of soil water (Caron and Boudreau, 2006). Caron and Boudreau (2006), also state that water content in soil can be express in gravimetric water content, volumetric water content or degree of saturation while soil suction can be presented in matric suction or osmotic suction.

There are several methods to measure soil suction such as equilibration of small soil samples over salt solutions of known osmotic suction until water content are fixed, thermocouple psychrometry and measurement of water activity (Mohd Tadza, 2011).

However, the first method may consume weeks to months to get final result while the second method is limited to suction of range 0.2MPa to 8.0 MPa (Gee *et al.*, 1992). Water activity meter is the device used to measure the water activity in the range of 0.1 to 1.0 (Gee *et. al.*, 1992). Later, water activity meter is modified to dew-point Water Potentia Meter (WP4), chilled-mirror dew-point psychrometer or chilled-mirror hygrometer to avoid the consequence of the temperature fluctuation of the surrounding on the suction (Li *et al.*, 2018). Figure 2.3 shows a schematic diagram of a dew-point Water PotentiaMeter, WP4. The device consists of a mirror and photodetector cell, a temperature sensor, a fan and a sealed chamber.



Figure 2.3 Schematic diagram of a dew-point Water Potentia Meter, WP4 Sources:Tian (2014)

Headspace of the sealed block chamber equilibrate the water potential of air in the chamber and the water potential or suction of the sample while chamber fan helps to accelerate the equilibration. The headspace vapour pressure is measured and the saturation vapour pressure is computed when water potential stable. In-built software will calculate the total suction of the soil specimen (in MPa and pF units) and show on the LCD panel of the WP4 along with the specimen temperature detected by the temperature sensor (Thakur *et al.*, 2006)

SWRC is affected by initial water content and stress state but the effects decrease when suction increases (Tian, 2014). Other factor such as soil mineral composition, pore structure, soil body of contractility, and consolidation pressure also affected the characteristic curve directly or indirectly (Ramli, 2015). According to research by Li *et al.*, (2018), SWRC using chilled- mirror dew-point method is not affected by compaction factor. Compacted or uncompacted soil does not influence the relationship between water content and soil suction plot. Besides, dry unit weight of soil has not much influence on SWRC (Thakur *et al.*, 2006)

#### 2.6 Soil Resistivity Behaviour

Soil resistivity is the key factor that determines what the resistance of a grounding electrode will be, and to what depth it must be driven to obtain low ground resistance (Samouëlian *et al.*, 2005). All soils conduct electrical current, with some soils having good electrical conductivity while the majority has poor electrical conductivity. The resistivity or inverse of conductivity of the soil is obtained using resistivity meter (Igboama and Ugwu, 2011). Table 2.3 shows the soil resistivity and earthing resistance required for different type of soil. Stoney soil has the highest soil resistivity which is  $0.001 \Omega$ .m.

Type of soil	Soil resistivity (Ω.m)	
Farming soil, loamy and clay soil	100	
Sandy clay soil	150	
Moist sandy soil	300	
Concrete 1:5	400	
Moist gravel	500	
Dry sandy soil	1000	
Dry gravel	1000	
Stoney soil	30000	
Rock	107	
Bentonite	3	
Marconite	0.001	

Table 2.3: Soil resistivity required for different type of soils

Source: Igboama and Ugwu (2011)

Soil resistivity has most influence by water content of soil. The soil's water content is important because it helps chemicals in the soil that surround ground conductors carry the electrical current. (Klairuang *et al.*, 2008; Igboama and Ugwu, 2011). Flow of electricity in the soil is greatly electrolytic as it is affected by the transport of ions dissolved in moisture (Youping *et al.*, 2006). The soil resistivity should be low to avoid back flashover of lighting transmission line and to keep the ground potential rise in safety tolerance limits (Harid, 2012). Table 2.4 shows the effect of water content on soil resistivity. From the table, it is clearly shows that increasing of water content decrease the soil resistivity for top soil, sandy loam and silica based sand.

	Water content by	Soi	l resistivity (	<b>Ω.m</b> )
	weight, %	Top soil	Sandy	Silica
	-	_	loam	based sand
0		1000000	1000000	-
2.5		2500	150	3000000
5		1650	430	50 000
10		530	185	2100
15		210	105	630
20		120	63	290
30		100	42	-

Table 2.4: Effect of water content on soil resistivity

Source: Harid (2012)

#### **CHAPTER 3**

#### METHODOLOGY

#### 3.1 Introduction

This chapter presents the methods that is adapted in this study. The characteristics of RG were investigated through standard laboratory practices which are physical and chemical. The physical characteristics considered include liquid limit, plastic limit, shrinkage limit, specific gravity, swelling index, specific surface area and loss of ignition while cation-exchange capacity (CEC) is the chemical characteristic of soil to be investigated. In addition, soil-water retention curve (SWRC) of RG is established by many techniques which included osmotic and vapour-equilibrium technique to show the relation of water content and soil suction. To complete this research, resistivity of RG and WG is tested using 2-pin soil box method to find out whether the value comply to be used as ground enhancement material

### **3.2** Selection of Material

Two different gypsum were considered in this study. RG, was obtained from Venator Materials Corporation which located at Teluk Kalung Industrial Estate, Kemaman, Terengganu, whereby WG was obtained from Gyproc Malaysia located at Port Klang, Selangor. The samples were brought back to the laboratory in seal plastic containers as shown in Figure 3.1.



Figure 3.1 RG stored in container

# 3.3 Materials Preparation

#### 3.3.1 Powder

Samples were crushed and sieved and only the samples which passed through 425  $\mu$ m were considered in this study. These soil samples are kept in sealed plastic bag and to be used for all the experiments

#### 3.3.2 Slurry

The samples were added with water at different water content to form slurry materials to test for resistivity behaviour.

#### 3.4 RG Physical Properties

The physical properties of soil indicates that the soil colour, soil texture, soil structure bulk density, horizonation, and soil consistence. The physical properties of RG were tested before proceed to the soil suction measurement. The laboratory testing included particle size distribution test, specific gravity test, atterberg limit test, shrinkage limit test, free swell test, loss on ignition test, and specific surface were tested. All the tests were follow different standards as shown in Table 3.1.

Physical properties	Testing Method
Specific gravity, G <sub>s</sub>	Density Bottle (Small pyknometer) method (BS 1377:
	Part 2 1990: 8.3)
Particle size distribution	Simple dry sieving and hydrometer analysis (BS 1377:
	Part 2: 1990: 9.3 and 9.5)
Liquid limit, LL	(BS 1377: Part 2: 1990: 4.3)
Plastic limit, PL	(BS 1377: Part 2: 1990: 5.3)
Shrinkage limit, SL	Standard Test Method for Shrinkage Factors of Soils by
	the Wax Method (ASTM D4943 – 08)
Water content, w	Oven drying at 105 °C (BS1377: Part 2: 1990)
Specific surface area	Wet technique EGME (BS 4359-1:1984)
Swell index, Cs	Free swell test (Gibbs and holtz, 1956)
Surface area	Ethylene glycol mono-ethyl ether (EGME) retention
	method
Loss on ignition	(BS 1377: Part 3: 1990: 4.3)
Chemical properties	Testing method
Cation exchange Capacity	Ammonium acetate method (Chapman, 1965; Lavkulich,
	1981)

 Table 3.1
 Standards used for characteristics testing

#### 3.5 RG Chemical Properties

-

#### 3.5.1 Cation-Exchange Capacity

CEC is a measure of quantity for the cations which able to be absorbed and held by soil. It shows the capacity of soil to hold the cations. CEC was determined by using ammonium acetate method. 5 g of RG mixed with ammonium acetate solution in a 50 ml of Whatman Vectaspin 20 centrifuge tube and ammonium hydroxide solution which used to increase the pH value. The indicators used in this test were Jenway 3450 pH and conductivity meter to shows that the pH is 7. Soil samples were placed in a centrifuge before filtered with Whatman 42 filter paper. 1 M ammonium acetate solution poured 4 times in order to make all the soil samples filtered completely before poured the next soil samples. The extracted liquid was collected and analysed by using Inductively Coupled. The concentration of cations was expressed as positive charge centimoles per kilogram

#### **3.6** Soil Water Retention Curve

#### 3.6.1 Osmotic Technique

For osmotic technique, air dried powder will be used for the wetting osmotic testing. Spectra/Por® Dialysis Membrane molecular weight cut-off (MWCO) value of 3500 semipermeable membrane was used for my applied suction range. The membrane is first immersed in distilled water for 10 minutes to remove the glycerine coating before using (Delage and Cui 2008). Around 5 gram of RG sample prepared will be inserted into a membrane and clipped to prevent leakage or unwanted osmosis from happening. The sample used will be as discussed in the sample preparation part and the sample will be mixed will to obtain a homogeneity for a better result. The pressure is altered by the concentration of PEG used. The applied suction is generated by mixing the PEG with distilled water with different ratio to create different concentration as shown in Eq. (3.1)

$$s = 11 c^2 \tag{3.1}$$

Where, s, is the applied suction and c, is concentration of PEG in gram of water

Once a set of PEG in gram has been weigh required for desire suction is prepared, the PEG will be mixed in the distilled water in a beaker. The process is fasten by using magnetic stirrer to speed up the dilution until a good homogeneity is reached. The top of the beaker are to be sealed to prevent the distilled water to evaporate to the atmosphere and thus increasing its concentration. Once the preparation of the solution is complete, the solution is extracted for chilled mirror hygrometer testing. The WP4C measures water potential by determining the relative humidity of the air above a sample in a closed chamber (an AOAC-approved method; also conforms to ASTM 6836). Once the sample comes into equilibrium with the vapor in the WP4C's sealed chamber, the instrument finds relative humidity using the chilled mirror method. Once the applied suction of the PEG solution had been determined, a refractometer was used to determine its brix's index.

The sample is the immersed into the solution for the osmotic wetting process. Magnetic stirred is used to ensure the homogeneity of the solution so that the wetting process is not interfered by PEG molecule surrounding the membrane. The sample will be taken out from the solution for measurement once per day until it archive stable or equilibrium state. Evaporation is a major effect of the slightest change of weight if the measurement process is prolonged. Accuracy would be in sense of precision in handling where all the material and apparatus are to be handled with care to prevent accidents from occurring. The arrangement of this technique will be arranged as illustrated in Figure 3.2



Figure 3.2 Set-up of osmotic technique

Once it had reached equilibrium stage, the sample is taken out from the membrane to be oven dried to seek for its current water content at the specified applied suction. Once again the PEG solution is extracted at the end of the experiment to check for its current applied suction with chilled mirror hygrometer

#### 3.6.2 Vapour-Equilibrium Technique

The SWRC wetting curve will be continued with the extension of higher pressure with vapour equilibrium technique. This technique is very time consuming where materials like clay may take up to a few month to archive desired equilibrium state. Therefore, with our university's laboratory available vacuum desiccator flask of five, five sample will be prepare for five different type of pressure created ranging from 10-300MPa with different salt solution. Salt solution used in the vacuum flask can creates pressure and it is depending of the salt solution type used. Table 3.2 refers to the saturated salt solutions

Salt Solution	Suction, kPa
Potassium carbonate	112.547
Sodium chloride	38,738
Potassium chloride	23,187
Potassium nitrate	8,635
Potassium sulphate	3,544

 Table 3.2
 Saturated salt solutions and corresponding suction

The salt solution selected for this study were stated in Table 3.2. The salt is prepared accordingly and mix well with distilled water in the desiccator flask. The process was fasten again with magnetic stirrer. Once the salt solution had archived homogeneity, the salt solution is extracted for chilled mirror hygrometer testing for its current applied suction. Once the solution is ready, the wire mesh was put into the desiccator as a porous barrier from contacting the sample and the salt solution. The desiccator is then closed with its lid and tighten to prevent evaporation and to be pressurized.

RG is prepared in air dried powder form in around 5 gram. The sample is then put onto an inert container and weight for the mass. Next, all five samples along with the container were put into the desiccator flask, each in different desiccator flask. At first, the sample is monitored daily for its change corresponding to the pressurized system. When the sample is going to be stable, the measurement frequency is lowed in order to not interrupt the system. The sample is carefully monitor until no further change is observed. Finally the sample was taken out of the desiccator and oven dried for its current water content corresponding to each of the applied suction. The weighing of the sample when it reached equilibrium are to be done with haste and accuracy since removing it from its pressurised environment will result in reverting the atmospheric state of the RG soil if the measurement prolonged for a long time. Figure 3.3 illustrates the procedure of vapour equilibrium technique (VET).



Figure 3.3 Procedure of vapour equilibrium technique

#### 3.6.3 Chilled-Mirror Dew-Point Technique

This techniques is for determining the soil suction of the samples, WPC4 model were used for this research and the result could be gain based from the data given from the model. The samples of RG were prepared with various water volume from 0.1mL until 1.0mL, then it is kept in plastic sealed bags so the water content does not evaporates. The samples retain in secure box for a week before it is ready for testing.

Next, the model need to be open and wait until the value of *Ts-Tb* become 25.0. The samples from the first preparation were taken out from plastic sealed bags and an amount of it will be placed onto the plastic holder. It should be covered half of the holder and then put into the sample drawer, the reading range for verify calibration have to be around -0.01 to -0.19. Turn the knob to the left for confirmation and data can be read. Figure 3.4 shows the WPC4 Potentia Dew Meter to read the suction reading.



Figure 3.4 WP4C Potentia dew meter for soil suction reading

#### **CHAPTER 4**

#### **RESULTS AND DISCUSSION**

# 4.1 Introduction

This chapter discuss about two segment which is result from conducted experiments and a discussion on the corresponding result on the testing conducted on RG sample. Final result from physical and chemical properties testing, soil suction behaviour of RG from osmotic technique and vapour equilibrium technique and combination of SWRC curve from both technique

#### 4.2 **RG Physical and Chemical Properties**

The properties of RG are summarized in Table 4.1.

Physical properties	Result
Specific gravity, Gs	3.163
Particle size distribution	Clay
Liquid limit, LL	91.97%
Plastic limit, PL	58.28%
Shrinkage limit, SL	17.50%
Water content, w	17.25%
Specific surface area	814.72 m <sup>2</sup> /g
Swell index, C <sub>s</sub>	180%
Chemical property	Result
Cation exchange capacity	87.63 meq/100g

 Table 4.1
 Physical and chemical properties result of RG from the experiment

Based on the results, RG has a high specific gravity which may be due to the presence of iron which usually iron-rich soil would have high specific gravity in range of 2.75 to 3.0 but could be higher (ASTM D 854-92). Normally, calcium carbonate which have specific gravity of 2.7 and iron that have specific gravity of 3 to 7 depending on its state of being cast, ore, slag or compound like iron carbonate may alter the RG specific gravity to a higher value if compare to ordinary gypsum.

For particle size determination where it may require that RG to be pastel carefully without really breaking down its crystal but just to separated off the lump of soil. Although oven drying tends to remove the cohesion force when water is dried off, RG is a type of clay that absorb back moisture from the air or surrounding easily and thus causing the soil to be wet and hard to be sieve. When crushed, RG shows a satisfactory amount of passing through 2mm sieve and its liquid limit and plastic limit shows that RG is a type of clay from AASHTO soil classification chart.

Specific surface area of RG for every gram is high which also proves that RG particle is fine and its water retention ability. The free swell index of RG is also extremely high which shows that RG is an expansive soil and from shrinkage limit what shows RG will retracts significantly when dried out.

# 4.3 Soil Water Retention Curve

#### 4.3.1 Osmotic Technique

Figure 4.2 shows the wetting process over time elapse for RG sample using osmotic technique. In a total of 6 RG sample with different applied suction, the trend of the wetting process or water content gain as the time elapse can be seen from this graph. The suction applied for each RG sample from the PEG solution are measured with chilled mirror and shown below the graph.

From this pattern, it can be noticed that elapsed time versus water content increase for the RG studied at an applied suction between 3.20 MPa to 10.40 MPa. From this curve, it is indicated that the changes in the water content of the clay specimens were significant during the first 7 days of testing until it started to increase in water content after that. The changes in the water content is lower if the suction induced at the specimens was high whereas the changes in water content is higher if the suction induced at the specimens was low



Figure 4.1 Osmotic technique on change in water content as time elapse

#### **4.3.2** Vapour Equilibrium Technique (VET)

Figure 4.2 shows the wetting process over time elapse for RG sample using vapour equilibrium technique. In a total of 5 RG sample with different applied suction, the trend of the wetting process or water content gain as the time elapse can be seen from this graph. The range of suctions were between 3.6 MPa to 111.77 MPa. The time taken to complete this experiment was about 51 days. The changes in mass of the specimens

were recorded every three days until no further changes occurred and then the water content left in the specimens were obtained. The suction applied for each RG sample from the salt solution are measured with chilled-mirror and shown below the graph.

The changes in the water content is bigger if the suction induced at the specimens was low whereas the changes in water content is higher if the suction induced at the specimens was high.



Figure 4.2 Vapour equilibrium technique on change in water content as time elapse

#### 4.3.3 Soil-Water Retention Curve

Figure 4.3 shows the combination of the drying curves from both osmotic technique and vapour equilibrium technique. In can be clearly seen that the difference between these two methods is their range of suction. Osmotic technique gives a lower range suction where it varies from 0.13 MPa to 1.6 MPa. The higher range suction value can be observed from the VET where it varies from 3.6 MPa to 111.77 MPa. The suction

applied for each of the RG sample from osmotic and vapour equilibrium technique were combined to form this graph. At lower range of suction, the resulted water content in the RG is higher and at higher range of suction, the resulted water content is smaller.

RG water adsorption behaviour from wetting test which around 10 MPa is the starting amount of applied suction where water will enter the soil accordingly. When the water enter the soil until the air pore in the soil is at residual stage, the adsorption of water will reduce. Even at the lowest applied pressure, the soil could only revert back to a certain wet state and not be able to return entirely to its initial the wet sample prepared according to it liquid limit for drying test. Even under a high suction of 111.77 MPa, RG is still able to adsorb water. A very high suction is required to fully remove the water from the soil water retention ability.



Figure 4.3 SWRC drying curve of RG

#### 4.4 **Resistivity**

Figure 4.4 below shows the resistivity behaviour and water content. Based on the graph, both RG and WG resistivity is decreasing when the water content increasing. The resistivity of WG is lowest when water content is highest compared to resistivity of RG when the water content at the highest. Supposedly the highest water content of RG should obtained the lowest resistivity of water content instead of WG.

Louie *et al.*, (2012) in his research states that gypsum has poor electrical conductivity due to ionic bonding between its cationic and anionic radicals of which it is made, is a kind of salt and the absence of free electrons. According to Boling (2006) soil with higher conductivity will provide the lower soil resistivity. Navarete (2018) in his study proved that RG is comprises of several chemical elements since it is a by-product of titanium dioxide. Since pure gypsum has lower conductivity, RG which is impure should have higher conductivity. The higher the conductivity of soil, the lower the resistivity of soil.

The error in resistivity behaviour may happened because of soil box which is not covered properly with RG that tends to mislead the value. Besides, the error is due to incorrect water content measurement.



Figure 4.4 Water content versus resistivity relationship for RG and WG

# **CHAPTER 5**

### CONCLUSION

Based on the findings of this study, the following conclusion were drawn:

- The properties of RG and WG has been obtained. The geotechnical properties of RG and WG showed that they are different materials based on their physical and chemical properties.
- 2. SWRC of RG for adsorption (wetting) has been established. RG able to absorb and retain water higher than WG, hence RG has higher plasticity than WG.
- 3. The resistivity value of RG and WG has been determined. From the result it indicates RG is able to be replaced as ground enhancement material in solving grounding system problems.

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