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Evaluation of Physical and Chemical Properties of Red Gypsum from Terengganu, Malaysia

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***Abstract*— Red gypsum waste generated from a local company in Malaysia were investigated in order to determine their physical and chemical properties. Red gypsum is a by-product during the extraction of titanium dioxide from the ilmenite ores 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. 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.**

Keywords—Red Gypsum; Titanium Dioxide; Highly Plasticity; Cation Exchange Capacity; Organic Matter

I. INTRODUCTION

Red gypsum (RG), a reddish brown semi-solid mud is a waste generated from a sulphate process of ilmenite ore which is rich in titanium and iron to acquire titanium dioxide. This process gives RG with a high concentration of sulphur trioxide, calcium oxide and iron hydroxide [1]. The major constituents of RG are hydrated $CaSO_4$ (70 wt %), Fe_2O_3 (30 wt %) and very small amount of [2] - [3]. In this study of RG, [4] and [5] explain detail its characteristic reddish color. Normal gypsum would be white in colour but as for RG, the main contribution factor to its reddish brown appearance is its high composition of FeO_3 consisting of 28.99%. The properties of RG include a high concentration of iron which is important mineral for carbonation reaction, low permeability, water retention ability and high free swell index. RG has many applications to a broad range of industrial and other activities [6]. RG has a very well acceptable strength and stiffness that allows it to be used as an engineering material such as natural soil, cement and slag for use in civil engineering applications [7], replacing natural gypsum in manufacturing of cements and inhibitors in soil erosion [8] for mobilization of heavy metals in soil.

For the controlled low-strength material (CLSM) the main requirements include low permeability and small shrinkage limit. The CLSM is a self-compacting, low strength, cementitious and flowable material used as backfill or void fill as an alternative to compacted fill. Using optimized materials with high specific surface area like RG, it fulfills the condition. Recent investigations of CLSM have shown that adding red RG to soils stiffens and strengthens the soils and more importantly, incorporating materials such as RG in CLSM can make it a cheaper product. Generalized laboratory experiments are usually used for evaluation of the quality which is performance in various applications and behaviour of RG. The contribution of these experiments provides indirect information on the industrial performance of RG and can be used by industry in conjunction with standard tests for more complete information. The previous studies have not evaluated the RG deposits in detail. In this work, new data about the physical, chemical, biological and mineralogical properties of the RG samples from Terengganu deposit, Malaysia are examined and the possibility for correlation between them is assessed.

II. LOCATION AND GEOLOGICAL SETTING

Geological research and industrial utilization of RG in Malaysia were focused for years on the industrial waste from north-eastern Peninsular Malaysia (Terengganu). Current research has been aimed to the utilization of the Fe-rich RG from Terengganu industrial waste which is sits in the northwest by Kelantan, the southwest by Pahang and the east by the South China Sea. In this study, the sample was obtained its origin which was generated by the local company in Malaysia. The manufacturing facility which occupies a 542-acre area is one of the largest industrial sites in Terengganu. The capacity of its plant is about 56 000 metric tons per year [9]. The utilization of RG at Terengganu has been studied by several researchers include [10] and [11]. The area of local RG sampling can be seen in Figure 1.

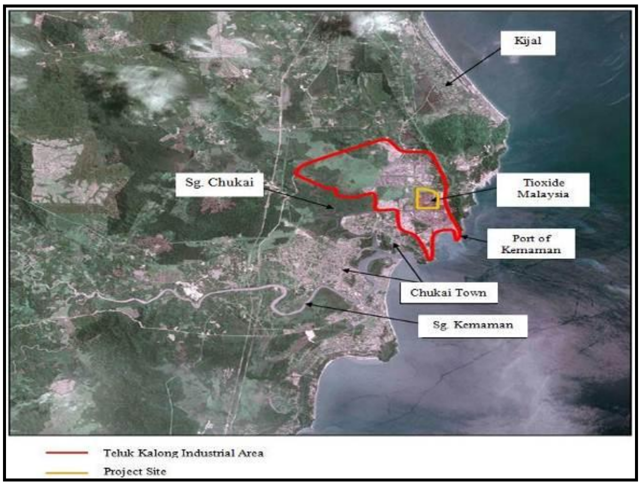

Figure 1: Simplified geological map of the sampling area at Terengganu [12].

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III. SAMPLING AND METHODS

The sample was obtained its origin which was located at Teluk Kalung Industrial Estate, 24007, Kemaman, Terengganu. The samples come with around 10 kilogram of raw RG in around 100 to 300 millimetres diameter lumps of undisturbed soil. The RG sample is the stored in a container since it is not so reactive and sensitive to atmospheric exposure. The sample is then preserved and protected from any unwanted exposure to water spillage and extreme temperature change that may cause inaccuracy during testing. General preparation of RG sample would be breaking down from its lump size to powder form. The RG powder is the sealed in a seal bag according to experimental amount requirement.

The properties testing that have been conducted were physical properties testing and chemical properties testing as shown in Table 3.1:

Table 3.1: Physical and chemical properties testing method

Physical properties	Testing Method
Specific gravity, G_s	Density Bottle [13] (BS 1377: Part 2 1990: 8.3)
Particle size distribution	[13] (BS 1377: Part 2:1990: 9.3&9.5)
Liquid limit, LL	[13] (BS 1377: Part 2:1990: 4.3)
Plastic limit, PL	[13] (BS 1377: Part 2:1990: 5.3)
Shrinkage limit, SL	[14] (ASTM D4943–08)
Water content, *w*	[13] (BS1377: Part 2:1990)
Swell index, C_s	Free swell test [15]
Surface area	(EGME) retention method
Organic matter	[13] (BS 1377: Part 3:1990:4.3)
Chemical properties	Testing method
Cation exchange Capacity	Ammonium acetate method

A. Specific Gravity

Specific Gravity of the test was based on [13] BS 1377-2(1990). The samples around 5-10g were placed in a pycnometer bottle and being weighed. In this test, instead of using distilled water, kerosene was used as a substitute of distilled water because of the high swelling clays properties which it tend to flocculate when it mixed with water [16].

B. Particle Size Analysis

Particle Size Analysis of the samples was determined according to [13], hydrometer method will be used to test the particle size distribution. The oven dried sample is sieves using different sieve sizes (5mm, 3.35mm, 1.18mm,0.6mm, 0.3mm, 0.15mm, 0.063mm) and sieved using mechanical shaker.

C. Atterberg Limit

Atterberg limit include of liquid limit, plastic limit and shrinkage limit values were determined by [13] and [14].

i. Liquid Limit

According to [13], the sample passed 425µm sieve test was weighed about 300g and were placed on the glass plate. The result will be recorded for at least four sets of values of penetration range 14 to 28 mm using cone penetration.

ii. Plastic Limit

[21] stated that the sample will be placed in the metal cup about 20g. The sample was divided into 3 sub samples for about 10g each and it was divided again into three more and was moulded using fingers to equalize the distribution of moisture content. The soil and container was weighed and oven-dried about 24 hours for determination of standard moisture content.

iii. Shrinkage Limit

For Shrinkage limit test used Standard Test Method for Shrinkage Factors of Soils by the Wax Method [15]. The sample used is the same as from the liquid limit test where the soil is oven-dried and pass 2mm sieve. The sample was placed in a mould and the wetness was used at 1.2 times of 20mm penetration and the measurement was continuously recorded until until it stabilized.

D. Water Content

Water Content was obtained using [13] as the standard references. The mass of an empty container with its lid before and after placed the sample was recorded.

E. Swell Index

Swelling potential test was measured by using free swell test [15] and it can be describe as the percentage increase in the volume from original loose from clay to its suspension form in water. When there was no change in the volume, free swell result can be recorded.

F. Surface Area

Specific Surface Area for this study was carried out using ethylene glycol monoethyl ether (EGME) retention used the wet technique. In this test, about 1 g of sample passing 63 µm sieved, silica gel, calcium chloride ($CaCl_2$) and EGME was used.

G. Organic Matter

Organic matter of samples was determined by loss on ignition test based on [13]. The crucible was used as the main apparatus in this will heated to 440± 25°C and maintained for a period not less than 3 hours until the constant mass was obtained for ignition. The result recorded when the sample was cooled to room temperature in the desiccator and then was weighed as m_3 to nearest 0.001g

Chemical properties analyses of this study focused on two parts, which is chemical properties and chemical composition.

H. Cation Exchange Capacity

Chemical properties were obtained by using Cation Exchange Capacity (CEC). The exchanging process means that when there was readily available exchangeable cation, the higher reactivity cations from the solution will replace the adsorbed cation in the soil [17] - [18]. CEC is the amount of positive charge attained from 100g of dry soil. Therefore, it is usually expressed in meq/100g. In this study, a simplified method suggested by [19] was used where 5 gram of RG was mixed with ammonium acetate solution in a 50ml Whatman Vectaspin 20 centrifuge tube and ammonium hydroxide solution was used to increase the pH value to 7. A Jenway 3450 pH and conductivity meter was used as an indicator that the pH is at a constant level of 7.

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IIII. RESULT AND DISCUSSION

The physical and chemical properties of RG are tabulated as shown in Table 4.1.

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

Properties	Result
Physical properties	
Specific gravity, Gs	3.16
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 (m2/g)	814.72
Swell index, Cs (%)	180
Chemical properties	
Cation Exchange Capacity (CEC),meq/100g	1.243

The result for this study will be compared with the properties of ordinary gypsum (white gypsum) and red gypsum as shown in Table 4.2.

Table 4.2: Property of White Gypsum against Red Gypsum [20]

Properties	White Gypsum	Red Gypsum
pH	7.4	7.4
Free Moisture (%)	10-17	10-50
Particle Density (mg/m^3)	3.05	2.71
Dry Density (mg/m^3)	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 Moisture Content (%)	39	41
Consolidation Testing (50-400 kN/m^2)	-	-
Coefficient of Volume Change (M_v m^2/year)	3.643-0.32	0.907-0.179
Coefficient of Consolidation (C_v m^2/year)	1.19-0.102	0.855-0.232
Coefficient of Secondary Compression (C_α m^2/year)	0.05-0.02	0.004-0.002
Compression Index C_c	0.218	0.242
Swell Index C_s	0.08	0.131
Peameability ($kv_x 10^{-9}$ m/s)	95-104	194-355
Strain to Failure (%)	5	10

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 [21] (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 determinations 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.

For the chemical properties analyses by Cation Exchange Capacity (CEC) Method was found to be only 1.24332 meq/100g which indicates the lower negative charge, less cations that can be held and less organic matter presence in soil [17]..

IV. CONCLUSION

From the result and discussion following conclusions are being obtained:

1) The objective of this research has been achieved well which is to determine the physical and chemical properties of Red Gypsum.
2) The properties of RG obtained from the experiment were all appropriate and match to the expected result from this type of soil.
3) Data obtained a relevant to the finding from literature that is related to Red Gypsum’s properties.

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