PROCESSING OF RED GYPSUM USING HYDDROCYCLONE

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SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING
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DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled ‘Processing of Red Gypsum Using hydrocyclone’. I also declare that it has not been previously submitted for the award of any degree and diploma or other similar title of this for any other examining body or University.

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Witnessed by

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ABSTRACT

Red Gypsum (RG) is another potential waste material that can replace cement in cement production. Red gypsum (RG), also 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 oxide. High iron content on gypsum inhibits the decomposition of the red gypsum (RG). The addition of iron can increase the quantity of the liquid phase and effect the decomposition of red gypsum. However, the excess liquid phase caused by the high iron content will wrap the gypsum and thus inhibits its decomposition as a binder in cement production. The characterization of RG such as physical and chemical properties are tested using standard laboratory standard. Testing including the measurement of particle size analysis and X-Ray Fluorescence (XRF) and some important properties including moisture content and specific gravity. So, ad addition of different type of acid on that physical processing can upgrade the Red Gypsum (RG) content and can produce information on the possibility in using of Red Gypsum (RG) on cement industry in conjunction with standard test for more complete information.

ABSTRAK

. Gipsum Merah (RG) adalah satu lagi bahan buangan berpotensi yang boleh menggantikan simen dalam pengeluaran simen. Gipsum merah (RG), juga lumpur separa pepejal coklat kemerah-merahan adalah sisa yang dihasilkan daripada proses sulfat bijih ilmenit yang kaya dengan titanium dan besi untuk memperoleh titanium oksida. Kandungan besi yang tinggi pada gipsum menghalang penguraian gipsum merah (RG). Penambahan besi boleh meningkatkan kuantiti fasa cecair dan memberi kesan kepada penguraian gipsum merah. Walau bagaimanapun, lebihan fasa cecair yang disebabkan oleh kandungan besi yang tinggi akan membalut gipsum dan dengan itu menghalang penguraiannya sebagai pengikat dalam penghasilan simen. Pencirian RG seperti sifat fizikal dan kimia diuji menggunakan piawaian makmal piawai. Pengujian termasuk pengukuran analisis saiz zarah, Pembelauan dan X-Ray (XRF) dan beberapa sifat penting termasuk kandungan lembapan dan graviti tentu. Jadi, saya percaya bahawa pemprosesan fizikal boleh menaik taraf kandungan RG dan boleh menghasilkan maklumat tentang kemungkinan penggunaan RG pada industri simen bersama-sama dengan ujian standard untuk maklumat yang lebih lengkap.

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CHAPTER 1

INTRODUCTION

1.1 Introduction.

In Malaysia, construction industry plays a vital role in the economic growth through the development of commercial buildings and the infrastructure of the country. In 2015, the Construction Industry Development Board (CIDB) of Malaysia stated that the ‘construction industry’ stood as the third-biggest sector in term of productivity, only next to manufacturing an agriculture sector.

Red Gypsum (RG) is another potential waste material that can replace cement in cement production. In Malaysia, more than 1,000,000 tonnes of Red Gypsum waste are generated every year. Most of this waste is dumped in secured landfills without being utilized. With the current trend of production, eventually there will be a new application from Red Gypsum waste generator to build an additional landfill to cater the growing demands of its RG waste handling. Utilizing RG in cement manufacturing can reduce the piling of RG at landfills significantly. Furthermore, RG has been suggested as a raw material that can be added into natural gypsum.

The main purpose of using gypsum in cement is to slow down the hydration of the cement once it has been combined with water. When water is introduced to cement, it begins to react with the Tricalcium aluminate ,C_3A and hardens, which is the process involved in hydration. This method takes extremely little time, therefore there is no time for transportation, mixing, or positioning. When gypsum is mixed with water in cement, a reaction with Tricalcium aluminate,C_3A particles occurs, resulting in ettringite. The main purpose of using gypsum in cement is to slow down the hydration of the cement once it has been combined with water. When water is introduced to cement, it begins to react with the Tricalcium aluminate, C3A and hardens, which is the process involved in hydration. This method takes extremely little time, therefore there is no time for transportation, mixing, or positioning. When gypsum is mixed with water in cement, a reaction with Tricalcium aluminate,C_3A particles occurs, resulting in ettringite.

This ettringite starts out as very fine-grained crystals that create a covering on the Tricalcium aluminate, C_3A particles' surfaces. These crystals are too tiny to fill the spaces between the cement particles. As a result, the cement mix remains plastic and workable. The amount of time allotted for mixing, transporting, and installing concrete has a significant impact on its strength, composition, and workability. Gypsum is used as a cement retarding agent because it slows down the hydration process.

1.2 Problem Statement.

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 oxide. High iron content on gypsum inhibits the decomposition of the red gypsum (RG). The addition of iron can increase the quantity of the liquid Phase and effect the decomposition of red gypsum. However, the excess liquid Phase caused by the high iron content will wrap the gypsum and thus inhibits its decomposition as a binder in cement production.

1.3 Objective.

The objectives of the research are:

1. To decrease the iron content in the decomposition of the red gypsum for the cement production.

2. To analyses the effect of the several parameters of hydro cyclone in which that can affect the iron content in red gypsum which is pH condition, vortex finder, percent solids and spigot sizes.

1.4 Thesis Outline.

The thesis has been divided into five chapter, which is for the chapter 1 is introduction, chapter 2 is literature review, chapter 3 is methodology, chapter 4 is results and discussion and lastly for the final chapter, which is chapter 5 is conclusion and recommendation.

In the first chapter, it is about the introduction of the composition of the red gypsum, problem statement that involved in this research, followed by objective of this research, scope of research and the thesis outline.

Next, chapter 2 is about literature review. In this chapter explain about thermotical mechanism of the hydro cyclone, described the related past work on the research of the characterization of Red Gypsum (RG) and parameters that effects the efficiency in separation on hydro cyclone.

For the following chapter, which is, chapter 3 is about the methodology that is use in this research. It consists of the steps and stages of work that already done to achieve the objective of the research. Flowchart also has been for a better understanding of the processes that has been one.

For the chapter 4, it is about the results and discussion of the overall study was done. In the discussion, it was explained about the reason why the results show like this and like that. At last, but not least, chapter 5 is all about the conclusion and the recommendation of this study and the recommendation for the future work.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction.

Gypsum ($CaSO_4.2H_2O$) crusts are found in many of the world's deserts where monthly potential evaporation exceeds mean monthly precipitation throughout the year (Watson, 1985). In southern Tunisia and the central Namib Desert, gypsum crusts are found around Ephemeral lakes. The crusts have gypsum contents of 50%–80% by weight and contain relatively high concentrations of sodium, potassium, magnesium, and iron. Subsurface crusts are not restricted to low lying landscape positions and are characterized by gypsum contents up to 90% by weight (Watson, 1985).

However, another type of gypsum is Red Gypsum (RG). 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 Red Gypsum with a high concentration of high concentration of sulphur trioxide, calcium oxide and iron hydroxide. Red Gypsum also has high concentrations of iron hydroxides (8%), as detailed in previous investigations (Fauziah,1966), which explain its characteristic reddish colour. Normal gypsum would be white in colour but as for Red Gypsum, the main contribution factor to its reddish-brown appearance it its high composition of Fe_2O_3 consisting of 28.99%.

2.2 Gypsum mineralization.

Gypsum, common sulphate mineral of great commercial importance, composed of hydrated calcium sulphate (CaSO $\cdot 2H_2O$). Gypsum ($CaSO_4.2H_2O$) is very similar to Anhydrite ($CaSO_4$). The chemical difference is that gypsum contains two waters and anhydrite is without water. Gypsum is the most common sulphate mineral.

2.2.1 Physical and chemical properties of Red Gypsum (RG)

Red Gypsum (RG) has a high specific gravity which has may be due to the presence of iron which usually iron-rich soil which have specific gravity in range of 2.75 to 3.0 but could be higher. Normally, calcium carbonate which have specific gravity of 3 to 7 depending on its state of being cast, ore, slag or compound like iron carbonate may alter the Red Gypsum specific gravity to a higher value if compared to ordinary gypsum.

For particle size determination where it may require that Red Gypsum to be pestle carefully without really breakdown 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, Red Gypsum 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. Table 2.1 show that the properties of Red Gypsum (RG).

Table 2.1 The properties of Red Gypsum (RG). (August,A.E, 2004)

pH	7.4
Free Moisture (%)	10-50
Particle density	2.71
Dry density	1.21
Liquid limit (%)	105
Plastic limit (%)	Non-plastic
Strain to Failure (%)	10

2.2.2 Physical and chemical properties of gypsum.

Gypsum rock (calcium sulphate dihydrate—$CaSO_4 \cdot 2H_2O$) was formed in geological times through the evaporation of seawater. It is often laid down in beds, ranging in thickness from a few centimetres to several tens of meters.

Because gypsum rock is slightly soluble in water, it is not usually found above ground in wet or damp areas, but it may be found underground, where is it not affected by the water table. In dry regions it may also be found on the surface, sometimes in the form of gypsum sand. The presence of gypsum on or just below the surface of the ground is often indicated by changes in vegetation. Some plants thrive on gypsum-rich (alkaline) soil, whereas others are not at all tolerant of those conditions.

Gypsum rock is usually white or colourless, although it may sometimes have gray, yellow, pink, or brown hues. Gypsum is much softer than minerals of similar color, such as calcite or quartz, and is the only one that can be scratched with a fingernail. If a piece of gypsum is held over a flame, it will turn cloudy and opaque and give off water. Table 2.2 show that the physical and chemical properties of gypsum.

Table 2.2 Physical and chemical properties of gypsum.

Chemical classification	**Sulphate**
Color	Clear, colorless, white, gray, yellow, red, brown
Streak	White
Luster	Vitreous, silky, sugary
Mohr's Hardness	2
Specific gravity	2.3
Crystal system	Monoclinic
Cleavage	Perfect
pH	7.4
Free moisture (%)	10-17

Some gypsum sand deposits contain only about 60 percent gypsum, and these are not very suitable for producing a plaster. Those containing more than 80 percent would be most suitable.

2.2.3 Use of gypsum.

gypsum is used as a fluxing agent, fertilizer, filler in paper and textiles, and retarder in Portland cement. About three-fourths of the total production is calcined for use as plaster of Paris and as building materials in plaster, Keene's cement, board products, and tiles and blocks. Gypsum plaster is a white cementing material made by partial or complete dehydration of the mineral gypsum, commonly with special retarders or hardeners added. Applied in a plastic state (with water), it sets and hardens by chemical recombination of the gypsum with water.

2.3 Component of ordinary Portland cement.

The principal raw materials used in the manufacture of Ordinary Portland Cement are argillaceous or silicates of alumina in the form of clays and shales and calcareous or calcium carbonate, in the form of limestone, chalk and marl which is a mixture of clay and calcium carbonate.

The ingredients are mixed in the proportion of about two parts of calcareous materials to one part of argillaceous materials and then crushed and ground in ball mills in a dry state or mixed in wet state. The dry powder or the wet slurry is then burnt in a rotary kiln at a temperature between 1400-degree C to 1500-degree C. the clinker obtained from the kiln is first cooled and then passed on to ball mills where gypsum is added, and it is ground to the requisite fineness according to the class of product. The below constituents forming the raw materials undergo chemical reactions during burning and fusion and combine to form the following compounds called Bogue compound. Table 2.2 shows the main components for Ordinary Portland Cement.

Table 2.1 The main composition for Ordinary Portland cement.

Lime (CaO)	60 to 67%
Silica (SiO_2)	17 to 25%
Alumina (Al_2O_3)	3 to 8%
Iron oxide (Fe_2O_3)	0.5 to 6%
Magnesia (MgO)	0.1 to 4%
Sulphur trioxide (SO_3)	1 to 3%
Soda and/or Potash (Na_2O+K_2O)	0.5 to 1.3%

The proportions of the below four compounds vary in the various Portland cements. Tricalcium silicate and dicalcium silicates contribute most to the eventual strength. Initial setting of Portland cement is due to tricalcium aluminate. Tricalcium silicate hydrates quickly and contributes more to the early strength.

The contribution of dicalcium silicate takes place after 7 days and may continue for up to 1 year. Tricalcium aluminate hydrates quickly, generates much heat and makes only a small contribution to the strength within the first 24 hours. Tetra calcium alumina-ferrite is comparatively inactive. All the four compounds generate heat when mixed with water, the aluminate generating the maximum heat and the dicalcium silicate generating the minimum. Table 2.3 shows the four-compound various Portland Cement.

Table 2.2 Four compound vary in various Portland cement.

Compound	Abbreviated designation
Tricalcium silicate ($3CaO.SiO_2$)	C_3S
Dicalcium silicate ($2CaO.SiO_2$)	C_2S
Tricalcium aluminate ($3CaO.Al_2O_3$)	C_3A
Tetra calcium aluminate ($4CaO.Al_2O_3.Fe_2O_3$)	C_4AF

Due to this, tricalcium aluminate is responsible for most of the undesirable properties of concrete. Cement having less Tricalcium aluminate,C_3A will have higher ultimate strength, less generation of heat and less cracking. Table below gives the composition and percentage of found compounds for normal and rapid hardening and low heat Portland cement. Table 2.4 shows the Composition and compound content of Portland Cement.

Table 2.3 Composition and compound content of Portland Cement

(a) Composition: Percent			
Portland Cement	Normal	Rapid hardening	Low heat
Lime	63.1	64.5	60
Silica	20.6	20.7	22.5
Alumina	6.3	5.2	5.2
Iron Oxide	3.6	2.9	4.6
(b) Compound: Percent			
C_3S	40	50	25
C_2S	30	21	35
C_3A	11	9	6
	12	9	14

2.3.1 Gypsum for Portland cement production.

In the manufacture process of cement, clinkers are formed. These cement clinkers are cooled down and added with small amount of gypsum. The mixture then sent to final grinding process. For ordinary Portland cement it remains between 3 to 4% and in case of Quick setting cement it can be reduced up to 2.5%.

2.3.2 Role of gypsum in cement.

The main purpose of adding gypsum in the cement is to slow down the hydration process of cement once it is mixed with water. The process involved in hydration of cement is that, when the water is added into cement, it starts reacting with the

Tricalcium aluminate, C_3A and hardens. The time taken in this process is very less, which doesn't allow time for transporting, mixing, and placing.

When gypsum is added into the cement and water is added to it, reaction with Tricalcium aluminate, C_3A particles tales place to form ettringite. This ettringite is initially formed as very fine-grained crystals, which form a coating on the surface of the Tricalcium aluminate,C_3A particles. These crystals are too small to bridge the gaps between the particles of cement. The cement mix therefore remains plastic and workable. The time allowed for mixing, transporting and placing plays a important role in strength, composition and workability of concrete. As gypsum retards the process of hydration, it is termed as retarding agent of cement.

2.3.3 Effect of Gypsum in cement

The effect of gypsum in cement is to prevent flash setting time of cement during manufacturing and it retards the setting time of cement during cement production. However, gypsum also allows a longer working time for mixing, transporting, and placing in construction purposes. When water is mixed to cement Aluminates and sulfates get react and evolve some heat but gypsum acts as coolant and brings down the heat of hydration. Therefore, Water required in gypsum-based cement for hydration process is less. Lastly, Gypsum cements possess considerably greater strength and hardness when compared to non-gypsum cement.

2.4 Processing of Portland cement.

2.4.1 Introduction

When it comes to hard rocks like limestone, slate, and some types of shales, raw materials used to make cement are extracted by quarrying, sometimes with the help of blasting. Underground techniques are used to mine some deposits. Excavators may immediately excavate through limestone and clay, which are softer rocks. Trucks, rail freight carriages, conveyor belts, or ropeways are used to move the excavated materials to the crusher plant. By pipeline, they can also be carried in a wet form or as slurry. Some form of beneficiation can be utilised in areas where there aren't any limestones with a high enough lime concentration. Froth flotation will improve the limestone by removing excess silica or alumina, but it is an expensive procedure that is only performed when there is no other option.

2.4.2 Processing of Portland cement.

There are four stages in the manufacture of Portland cement:

(1) crushing and grinding the raw materials,

(2) blending the materials in the correct proportions,

(3) burning the prepared mix in a kiln,

(4) grinding the burned product, known as “clinker,” together with some 5 percent of gypsum (to control the time of set of the cement).

The raw materials are ground wet and fed to the kiln as a slurry, ground dry and fed as a dry powder, or ground dry and then moistened to form nodules that are fed to the kiln. These three manufacturing methods are known as the wet, dry, and semidry processes. Crushing and grinding the raw material

All materials, except for the soft ones, are first crushed, sometimes in two steps, and then ground, typically in cylindrical ball mills or tube mills that rotate and contain a charge of grinding steel balls. Depending on the procedure being used, this grinding can be done wet or dry, although for dry grinding, the raw materials may first need to be dried in cylindrical, rotating dryers. In wash mills, water and vigorous swirling break down soft materials to create a fine slurry that is then fed through screens to filter out bigger particles.

Selective quarrying and control of the raw material delivered to the crushing and grinding mill allow for the initial approximation of the chemical composition needed for a specific cement, which is then used in the blending process. Drawing material from two or more batches that comprise raw mixes with somewhat varied compositions allows for a finer level of control. These mixtures are kept in silos for the dry process; slurry tanks are utilised for the wet phase. The silos' dry contents are thoroughly mixed by intense circulation and agitation brought on by compressed air. The slurry tanks are agitated mechanically, with compressed air, or with both during the wet process. The water content of the slurry, which is typically between 35 and 45 percent, is occasionally filtered down to between 20 and 30 percent, and the filter cake is then fed into the kiln. As a result, less fuel is needed to burn the material.

2.4.2(a) Burning Process.

Bottle kilns were the first kilns used to burn cement in batches, followed by chamber kilns and subsequently continuous shaft kilns. In certain countries, the shaft kiln is still in use in a modernised version, although the rotary kiln is the most common type of burning apparatus. These kilns are made of a steel, cylindrical shell coated with refractory materials, and may be up to 200 metres (660 feet) long and six metres in diameter in wet process facilities but shorter for the dry process. They move slowly along an axis that is slightly angled toward the horizontal. The raw material feed is fed from the top and steadily makes its way to the bottom, or firing end, of the kiln. Pulverized coal, oil, or natural gas pumped through a pipe can all be used as the fuel for a fire. Depending on the raw materials being burnt, the temperature at the firing end ranges from approximately 1,350 to 1,550 °C (2,460 to 2,820 °F).

In order to improve heat transmission to the entering raw materials and decrease the heat wasted in the waste gases, some type of heat exchanger is frequently added at the rear end of the kiln. The burnt product comes out of the kiln as tiny clinker nodules. These enter coolers, where the product is cooled, and the heat is transferred to the entering air. Clinker may either be used right away to make cement or kept in stockpiles for future use. Before moving on to the smaller rotating kiln in the semidry process, the raw materials in the form of nodules containing 10 to 15 percent water are fed onto a travelling chain grate. Raw nodules on the grate are preheated by hot gases being drawn through them from the kiln.

Cement kiln dust emissions can be a major annoyance. Between the kiln outlet and the chimney stack, it is customary and even required to install cyclone arrestors, bag-filter systems, or electrostatic dust precipitators in populated regions. The formation of clinker, a by-product of the chemical reaction that powers the present process, is integrally connected to the generation of more than 50% of the emissions from cement manufacture. The requirement for clinker itself might be decreased by the use of alternative materials, which would help lessen the effects of the cement-making process on the environment.

2.4.2(b) Grinding process

In horizontal mills similar to those used for grinding raw materials, the clinker and necessary quantity of gypsum are crushed to a fine powder. The material may be processed continuously in the mill (open circuit grinding) or it may be removed from the finished product and ground again in the mill (closed-circuit grinding). The feed material may occasionally have a tiny quantity of a grinding assist added. An air-entraining agent is similarly added to air-entraining cements. Cement that has reached its completion is pneumatically pushed to storage silos, where it is pulled for packaging in paper or shipping in bulk containers.