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UTILIZING RADIOACTIVE WATER LEACH PURIFICATION (WLP) IN SELF COMPACTING CONCRETE (SCC)

by

HUZAIFAH MUHD FADHIL

FINAL YEAR PROJECT

Submitted to the Department of Civil Engineering as a Requirement for the Degree Bachelor of Engineering (Hons) (CIVIL ENGINEERING)

UNIVERSITI TEKNOLOGI PETRONAS

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ACKNOWLEDGMENTS

First and foremost, praised be to Allah, the Most Merciful and the Most Beneficent, who grants the author to complete his research within the given time. The author would like to take the opportunity to express his sincere thanks to his supervisor Dr Bashar S Mohammed, for his outstanding guidance and support throughout the duration of the study. It is a great honor to work and learn under his supervision.

The author would also like to thank his family namely his parents Muhd Fadhil Nuruddin and Latipah binti Yusof for their constant love, support, and encouragement. To the helpful colleagues, the author expresses his special thanks for continuous encouragement. They contributed significantly to the realization of the author's research studies.

Special thanks are extended to the following people and institutions for their help in conducting this research: Mr. Johan Ariff Mohamed, Mr. Hafiz Baharun and Mr. Ruzaimi Mohd Pouat Laboratory Technologist UTP Concrete Laboratory, for their technical advice and materials procurement. Lynas Malaysia Sdn Bhd for providing the WLP, Mr. Ahmad Postgraduate Student of Civil Engineering, Lab Technologist from Mechanical Engineering department, Mr. Adam of Universiti Teknologi PETRONAS, for their advice and discussion during the research.

Finally, the author would like to express his deepest gratitude to all parties who have supported him throughout the study and to Universiti Teknologi PETRONAS for providing him a chance and financial support.

ABSTRACT

Environmental issues aroused due to the concrete production have alarmed the world concerned into producing a novel concrete technology that is more environmental friendly. The performance of concrete is expected to be risen with the advancement of concrete technology in order to satisfy the increasing social demands. Currently, the production rate of cement in the world is approximated to be 1.2 billion tonnes per year in 2001 and this figure is expected to grow exponentially at about 3.5 billion tonnes per year in 2015. However, each tonne of Portland cement clinker production is associated with a similar amount of CO_2 emission that is released into the air. Water leach purification (WLP), the by-products of mining industries has attracted the attention of many parties due to the environmental issues its caused. Disposal of WLP has become a serious problem since it possesses radioactive material. Nevertheless, besides having disadvantages, WLP has its own beneficial properties which can be used in construction industry as an advancement of concrete technology based on its cementations properties. Thus, to study the effect of WLP, this project is conducted by focusing on WLP as an aggregate.WLP as aggregate is tested based on its physical properties, chemical properties and also mechanical properties / durability. The effect of WLP as CRM is conducted by performing several processes such as burning and grinding. For burning process, the dry WLP is burned into three different temperatures which are 300°C, 500°C and 700°C. X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF) analysis are conducted to analyze the chemical compositions of water leach purification (WLP). Later, WLP is used to replace 25%, 50%, 75% and 100% of cement content in concrete. After casting process, the sample is tested to determine its compressive strength As a result from this test, the strength development of WLP concrete is indeed comparable to normal OPC concrete with 62% and 86% strength at 7 & 14 days respectively.

Keywords: Water Leach Purification (WLP), X-ray Diffraction (XRD), X-ray Fluorescence (XRF), Scanning Electron Microscopy (SEM), Soil, Compressive Strength, Ordinary Portland Cement (OPC)

ABSTRAK

Isu-isu alam sekitar yang timbul disebabkan oleh pengeluaran konkrit telah mencemaskan dunia ke dalam menghasilkan teknologi baru konkrit yang lebih mesra alam sekitar. Prestasi konkrit dijangka akan meningkat dengan kemajuan teknologi konkrit untuk memenuhi permintaan yang semakin meningkat sosial. Pada masa ini, kadar pengeluaran simen di dunia ini hampir menjadi 1.2 bilion tan setahun pada tahun 2001 dan angka ini dijangka berkembang pesat pada kira-kira 3.5 bilion tan setahun pada tahun 2015. Walau bagaimanapun, setiap tan simen Portland pengeluaran klinker dikaitkan dengan jumlah yang sama pengeluaran CO2 yang dilepaskan ke udara. Air luluh pembersihan (WLP), undang- produk industri perlombongan telah menarik perhatian banyak pihak kerana isu-isu alam sekitar yang disebabkan . Penjualan WLP telah menjadi satu masalah yang serius kerana ia mempunyai bahan radioaktif. Namun, di samping mempunyai kelemahan, WLP mempunyai cirri tersendiri yang berfaedah yang boleh digunakan dalam industri pembinaan sebagai kemajuan teknologi konkrit berdasarkan sifat lekatannya. Oleh itu, untuk mengkaji kesan WLP, projek ini dijalankan dengan memberi tumpuan kepada WLP sebagai agregat.WLP sebagai agregat diuji berdasarkan sifat-sifat fizikal, sifatsifat kimia dan juga sifat-sifat mekanikal / ketahanan. Kesan WLP sebagai CRM dijalankan dengan melakukan beberapa proses seperti pembakaran dan pengisaran. Untuk proses pembakaran, WLP kering dibakar ke dalam tiga suhu yang berbeza iaitu 300 ° C, 500 ° C dan 700 ° C. X -Ray Pembelauan (XRD) dan X -Ray Pendarfluor (XRF) analisis dijalankan untuk menganalisis komposisi kimia air luluh pembersihan (WLP). Kemudian, WLP digunakan untuk menggantikan 25%, 50%, 75% dan 100% daripada kandungan simen di dalam konkrit. Selepas proses pemutus, sampel diuji untuk menentukan kekuatan mampatan. Hasil dari ujian ini, pembangunan kekuatan konkrit WLP memang setanding dengan konkrit OPC normal dengan 62% dan kekuatan 86% masing-masing pada 7 & 14 hari

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

A rapidly growing economic in emerging developing country has caused an increase of environmental issues such as carbon footprint. These issues become more serious due to lack of environmental management system. Concrete is the largest production of all man-made materials with an annual global production of about one cubic meter for every person on earth. In other words, production of concrete was estimated to be 8.8 billion tons per year [1]. Concrete is basically a composite construction material made primarily with aggregates, cement and water. Ordinary Portland cement (OPC) is the most common type of cement used throughout the world. The cement basic ingredients are limestone, clay and other mineral, mixed in definite proportions to produce chemical reaction burnt at very high temperature.

However this mass production of Portland cement causes great concern on the environment because of the high carbon footprint. Besides CO_2 emissions, gases, noise and vibration when operating machinery and during blasting in quarries, consumption of large quantities of fuel during manufacture and damage to country side from quarrying also contribute to the environmental effects.

Due to a high growth of production in every sector, waste has been produce. Waste are materials that are not prime products (that is products produce for the market) for which the initial user has no further use in terms of his/her own purposes of production, transformation or consumption, and of which he/she wants to dispose. Wastes may be generated during the extraction of raw materials, the processing of raw materials into intermediate and final products, the consumption of final products, and other human activities. Residuals recycled or reused at the place of generation are excluded [2]. The economic costs of managing waste are high, and are often paid for by municipal governments [3].

There are several types of waste including mining, domestic and industrial. One type of waste that becomes very popular nowadays is WLP residue. Lynas operation in Gebeng, Pahang generally generates three types of residue, which are flue gas sulphurisation residue (FGD), neutralization underflow residue (NUF) and water leach purification residue (WLP). For FGD and NUF, Lynas company already know how to utilize it in making a product such as battery and many more but for WLP, based on its chemical compound, it has a potential in the production of concrete and still in research.

1.2 Problem Statement

The economic strength in every country and even degree of any civilization is reflected by the growth rate of the infrastructures and highlighted by the production rate of concrete. Concrete is the largest production of all man-made materials with an annual global production of about one cubic meter for every person on earth. In other words, production of concrete was estimated to be 8.8 billion tons per year [1]. Concrete is basically a composite construction material made primarily with aggregates, cement and water. Ordinary Portland cement (OPC) is the most common type of cement used throughout the world.

This mass production of concrete contributes a lot of environmental problems. The cement industry is one of two primary producers of carbon dioxide (CO2), a major greenhouse gas. Creating up to 5% of worldwide man-made emissions of this gas, of which 50% is from the chemical process and 40% from burning fuel [4]. The carbon dioxide CO2 produced for the manufacture of one tonne of structural concrete (using ~14% cement) is estimated at 410 kg/m3 (~180 kg/tonne @ density of 2.3 g/cm3) (reduced to 290 kg/m3 with 30% fly ash replacement of cement) [5]. The CO2 emission from the concrete production is directly proportional to the cement content used in the concrete mix; 900 kg of CO2 are emitted for the fabrication of

every ton of cement [6]. This shows that CO2 emission are very serious problem occur due to the production of concrete.

There also problems face in disposal of WLP residue. The economic costs of managing waste are high, and are often paid for by municipal governments [3]. Lynas operation in Gebeng, Pahang generally generate three types of residue, which are flue gas sulphurisation residue (FGD), neutralization underflow residue (NUF) and water leach purification residue (WLP). For the advancement of concrete production and clean environment, this project will be focusing on the potential utilizing of waste materials in the WLP residue that is produced by Lynas company. WLP are considered as a waste material and abundant. However it has a potential in becoming a part of concrete. If accomplished, this waste may change from waste to wealth.

In conclusion, below are the problems occurring:-

- Lack of compaction in cement leads to honeycombing.
- SCC is a cement that compact by itself
- The depletion of sand in concrete.
- WLP is a radioactive material that need costly disposal.

1.3 Objectives

The objectives of this study are as follows:

- 1. To establish the effect of WLP residue as aggregate on concrete strength development;
- 2. To identify the optimum amount of WLP residue as aggregate in cement.

1.4 Scope of Work

This project will be executed with a lot of experiment. It can be divided into two main compartments. First is study on WLP as an aggregate for concrete and second compartment is identify whether WLP can be cement replacement material (CRM) for concrete or not. Due to this two compartments, there are several test will be conduct to proof the objective can be accomplish.

1.4.1 WLP as Aggregate

WLP raw earth that has been collected will have a several test in finding its physical content. The raw earth will be test for their grading, density, moisture content and laser diffraction analysis. For grading test or know as sieve analysis test is to grade the WLP to which group they belongs either coarse, medium or fine. The soil density and moisture content is to know the density and moisture of a soil as a percentage of its oven-dried weight. It can be done at the geotechnical lab. Laser diffraction analysis is to determine the specific size particles that smaller than 63mm.

1.4.2 WLP as Cement Replacement Material (CRM)

Cement replacement material in any type first need to go through a treatment process called burning and grading. For this project, the rare earth will be burn in three different temperatures which are 300°C, 500°C and 700°C. The burning process in different temperature is to determine the optimum amount of WLP needed in a concrete production. Then, this all three sample will be grading down to 100mm. These are the most suitable size for concrete mixture as almost all OPC are within this size and also due to time constraint.

1.4.3 WLP Characterization

WLP characterization needs to be done to see its chemical properties. Chemical test such as X-ray Diffraction (XRD), X-ray fluorescence (XRF), Scanning electron microscopy (SEM) will be made to see the WLP percentage of oxide composition, the amorphousness of WLP. After chemical test, for each temperature, there will be three other categories sample cast based on 25% of WLP, 50% of WLP, 75% of WLP and 100% of WLP mix with concrete mixture (OPC, sand, stone and w/c). Then the casting cube will be cured in water for 7, 14 and 28 days. After cured, the cube will be test for its compressive strength and Scan Electron Microscopy (SEM) to see the surface of the material. Fig. 1.1 shows the experimental detailed better understanding.



Figure 1.1: Experiment and test summary

CHAPTER 2

LITERATURE REVIEW

2.1 Chapter Overview

The mining industry has a large environmental footprint. There is no argument against this. It takes up vast tracts of land and uses an incredible amount of resources to operate a mine site especially when it comes to electricity and fuel. On top of that the waste product from mining needs to be stored somewhere or disposed off under strict environmental guidelines.

There is nothing good from mining but peoples do like smart phones and shiny gadgets. The components are made of metals that come predominantly from a mine site. Everyone yet to use a technologically advanced gadget comprising of recycled metal. As much as peoples don't like to admit it, we have become reliant on the mining industry to supply the materials to make our shiny toys.

Many mining companies do make efforts in reducing their impact on the environment through restoring the land and waterways to how their original state before mining. They also invest in ways to reduce the amount off chemicals used to extract minerals and metals.

Tailings, also called mine dumps, culm dumps, slimes, tails, refuse, leach residue or slickens [13], are the materials left over after the process of separating the valuable fraction from the uneconomic fraction of an ore. Tailings are distinct from overburden, which is the waste rock or materials overlying an ore or mineral body that are displaced during mining without being processed.

In some situations, tailings represent an external cost of mining [14]. This is particularly true of early mining operations which did not take adequate steps to make tailings areas environmentally safe after closure. Modern day mines, particularly in jurisdictions with well-developed mining regulations or operated by responsible mining companies, often incorporate the rehabilitation and proper closure of tailings areas in the mining costs and activities. For example, the province of Quebec, Canada, requires not only submission of closure plan before the start of mining activity, but also the deposit of a financial guarantee equal to 100% of the estimated rehabilitation costs [15]. Tailings dams are often the most significant environmental liability for a mining project [16].

Disposal of mine tailings is one of the most important environmental issues for any mine during the project's life. While significant pressure is placed on mining projects in developed countries to conform to stringent environmental standards, many projects in developing nations do not take significant steps to prevent or mitigate environmental damage [17]. The sustainability challenge in the management of tailings and waste rock is to dispose of material, such that it is inert or, if not, stable and contained, to minimise water and energy inputs and the surface footprint of wastes and to move toward finding alternate uses.

In order to prevent the uncontrolled release of tailings material into the environment, mines usually have a disposal facility which quite often takes the form of a dam or pond. This is a convenient method of storage since tailings are often in the form of slurry when they are discharged from the concentrator. These facilities often require the clearing of more land than the rest of the mine (including open-pit operations) combined, and failure of the wall can result in a massive release of tailings. As such they are of great environmental concern. Other way from using a disposal facility, this waste also can be used a cement replacement materials (recycle material) for a construction industry.

Besides using landfill and incineration, the waste also can be recycling to a useful material. Concrete recycling is an increasingly common method of disposing of concrete structures. Rare earth was once routinely shipped to landfills for disposal, but recycling is increasing due to improved environmental awareness, governmental laws and economic benefits. Mining waste also can be recycle into a cement replacement

materials and as an aggregate thus will be a solution due to the depletion of raw materials also with the cost of disposal landfill.

2.2 Cement Replacement Material (CRM)

The production of cement is a significant contributor to global warming. In addition to optimizing the energy efficiency of Portland cement production plants, the amount of cement used in concrete mixes can be reduced by using cement replacement materials. Environmental issues caused from Portland cement production have made researchers to create advance methods to obtain materials that are sufficiently reactive to replace cement portion in concrete. These materials are generally a waste by-product and contain highly reactive silica to react with calcium hydroxide resulted from hydration process between cement and water [5]. Rare earth that is resulted from burning process of raw rare earth is one of the cement replacement materials (CRM) produced from mining waste.

All cement substitutes have the dual benefit of replacing energy-intensive Portland cement and of using material that would otherwise be land filled. To varying degrees, cement replacement materials work in two ways which are they hydrate and cure like Portland cement and second they are pozzolanic material providing silica that reacts with hydrated lime, an unwanted by-product of concrete curing.

2.3 Aggregate

Construction aggregate, or simply "aggregate", is a broad category of coarse particulate material used in construction, including sand, gravel, crushed stone, slag, recycled concrete and geosynthetic aggregates. Aggregates are the most mined material in the world. Aggregates are a component of composite materials such as concrete and asphalt concrete; the aggregate serves as reinforcement to add strength to the overall composite material. Due to the relatively high hydraulic conductivity value as compared to most soils, aggregates are widely used in drainage applications such as foundation and French drains, septic drain fields, retaining wall drains, and road side edge drains [5]. Aggregates are also used as base material under foundations, roads, and railroads. In other words, aggregates are used as a stable foundation or road/rail base with predictable, uniform properties (e.g. to help prevent differential settling under the road or building), or as a low-cost extender that binds with more expensive cement or asphalt to form concrete [26].

2.4 Process Schematic for the waste WLP residue (Lynas Advanced Material Plant, LAMP)

Figure 2.1 shows how the WLP is been made from Mt. Weld lanthanide concenctration in Australia [7].



Figure 2.1: Process schematic for the waste WLP residue

2.5 Advantages and Disadvantages of By-Product and Re-Use Approach (Rare Earth)

There are numerous examples demonstrating that this approach can have beneficial and unbeneficial impacts during the life of a project. The following subsection explained both.

2.5.1 Advantages

- Minimization of construction costs and other site works related to stored materials such as lined cells, impervious capping, groundwater monitoring wells and interception and diversion channels [8];
- 2. Potential for cash flow generated by sale or disposal of by-products;
- Minimization of impacts and potential impacts arising from long term storage of wastes;
- 4. Reduction or elimination of ongoing monitoring costs and risks to regulatory agencies, which are often an expensive and long-term component of waste storage, particularly where emotive and complex technical issues such as the half-life of radioactive materials and the decay chains of other complex lanthanide materials are concerned.

2.5.2 Disadvantages of By-Product Approach

The main disadvantages of this approach are that for more complex materials it often requires additional time and investment at commencement to identify potential markets for by-product streams and to provide facilities to prepare the residues for commercial sale or further use. This requirement comes at a time when many projects are focused on short term project financing needs [8].

In addition, many benefits may have little, if any, current commercial value, which, however, may change in the future. These changes could arise due to

improvements in process technologies (e.g. residue re-processing for gold and tin), developments of markets for previously un-costed externalities (e.g. carbon trading) or demands brought about by changing societal values (e.g. paper, glass or plastic recycling).

2.6 Rare Earth

The lanthanide concentrate is treated with sulphuric acid at a moderately high temperature to decompose the more refractory lanthanide containing minerals. The resulting residue is then agitated with water and the resulting process liquor is treated via a series of process steps to remove various impurities. Following this, the liquor is mixed with a number of specialized reagents and then submitted to a solvent extraction process to recover a number of high quality product streams. Although complex, this is nevertheless a mature process technology and widely used around the world to recover lanthanide products. During the various process steps, additional materials are introduced to the process streams to enable recovery of the product or neutralization of off-gas streams for environmental or emissions requirements [8].

The principal waste product streams comprise:

- Water Leach Purification (WLP) residues resulting from the leaching and purification of the water soluble lanthanide components from the calcite, cracked concentrate;
- Neutralization Underflow Solids (NUF) consisting principally of the reaction product of acidic sulphuric acid derived liquor with calcium, magnesium and aluminum based minerals to produce calcium, magnesium and aluminum sulphates;
- Waste Gas Treatment residues referred to as Flue Gas Desulphurization (FGD) residues.

CHAPTER 3

METHODOLOGY

3.1 Project Identification

The project is carried out according to the process flow shown in Fig. 3.1



Figure 3.1: Process flow chart

3.2 Material Characterization

3.2.1 Chemical Properties

3.2.1.1 X-ray Diffraction (XRD)

Routine XRD-mineralogy profiles can provide qualitative and semi quantitative records of shifts in the source of sedimentary components to a lake sequence. XRD mainly displays information on autochthonous and authigenic minerals, but can give some indication of the abundance of amorphous silica phases. Set up with routine data collection, XRD is a rapid, accurate technique which can process 40 samples per day using an automated sample changer [17]. It is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimension (diffraction patterns) of WLP. Figure 3.2 shows XRD machine been used.



Figure 3.2: XRD machine

3.2.1.2 X-Ray Fluorescence (XRF)

X-Ray Fluorescence (XRF) is named as the process of emissions of characteristic x-rays. When a primary x-ray excitation source from an x-ray tube or a radioactive

source strikes a sample, the x-ray can either be absorbed by the atom or scattered through the material. The process in which an x-ray is absorbed by the atom by transferring all of its energy to an innermost electron is called the "photoelectric effect." During this process, if the primary x-ray had sufficient energy, electrons are ejected from the inner shells, creating vacancies. These vacancies present an unstable condition for the atom. As the atom returns to its stable condition, electrons from the outer shells are transferred to the inner shells and in the process give off a characteristic x-ray whose energy is the difference between the two binding energies of the corresponding shells. Because each element has a unique set of energy levels, each element produces x-rays at a unique set of energies, allowing one to non-destructively measure the elemental composition of a sample. Analysis using x-ray fluorescence is called "X-ray Fluorescence Spectroscopy" [18]. Figure 3.3 shows XRF machine.



Figure 3.3: XRF machine

3.2.1.3 Scanning Electron Microscopy (SEM)

The Scanning Electron Microscope (SEM) is a microscope that uses electron rather than light to form an image. In optical microscope, lenses are used to bend the light waves and the lenses are adjusted for focus. In the SEM, electromagnets are used to bend an electron beam which is used to produce the images on a screen. Beam of electron is produced in an electron gun by heating of a metallic filament. This electron beam will follow a vertical path through the column of the microscope and pass electromagnetic lenses, which focus and direct the beam towards the sample. When the electron beam hits the sample, other electrons, called as backscattered electron and secondary electron, are ejected from the sample. The detectors will collect these secondary and backscattered electrons and convert them to a signal that is sent to a display screen [21]. The SEM is designed for direct studying of the surfaces of solid object. SEM machine is shown in Figure 3.4.



Figure 3.4: SEM machine

3.2.2 Physical Properties

Four test are conducted to obtain the physical properties of WLP which consists of moisture content, density and lazer analysis. The test is performed at geotech lab consult by the lab technician. The experimental detailed is provided in Table 3.1.

Table 3.1: Physical test details

3.3 Mechanical Properties

The self compacting concrete will be cast in $50 \ge 50 \ge 50$ mm cube size. The concrete will be mix in different proportion of WLP based on 3.2 table.

MIX	WID	CA	FA	W/C	Cement	W	SP
.NO	VV LF	(kg/m^3)	(kg/m^3)	w/C	(kg/m^3)	(kg/m^3)	(kg/m^3)
1	0%	500	700	0.32	500	160	0.055
2	25%	500	525	0.38	500	190	0.055
3	50%	500	350	0.43	500	215	0.055
4	75%	500	175	0.48	500	240	0.055
5	100%	500	0	0.54	500	270	0.055

Table 3.2: Mix proportion

From above mix proportion table, concrete will be cast. 6 cubes and 3 cylinders will be used for each mix. Thus,

$$6(0.1 \times 0.1 \times 0.1) = 0.006$$
$$3(\frac{\pi (0.1)^2}{4}) \times 0.2 = 0.0047$$
$$0.006 + 0.0047 = 0.011$$

The total value will be multiply with cement (OPC), fine aggregate (FA), coarse aggregate (CA), water cement ratio (w/c) and super plasticizer (s/p). After multiply, the total amounts that will be used are in table 3.3:

Table 3.3: Amount of each mixing

Material	Amount (kg)
Cement (OPC)	5.5
FA	7.7
CA	5.5
W/C	1.76
S/P	0.055

For water and fine aggregate (FA) the total will be different for each mixture. The amount will be based on the Table 3.2. Figure 3.4 - 3.6 shows several images related to mixing procedure and compressive strength test. Procedure of mixing are as followed:

• First, fine aggregate and coarse aggregate will be mix together for 2 minutes in the concrete mixer. Add cement (OPC) and WLP into mixing and mix it for 3 minutes. Then add half of water and let it mix it for another 3 minutes. After that, mix another half of water + S/P and mix for 2 minutes. After one day (24 hours) concrete will cured in water for 7, 14 and 28 days. After curing, cube and cylinder will be test on its compressive strength, permeability and porosity.



Figure 3.4 : Concrete mixture machine



Figure 3.5 : Cast concrete in the 50x50x50mm cube



Figure 3.6 : Comprehensive strength test

Table 3.4 explained in detailed the procedure taken in each mechanical test.

Table 3.4: Mechanical test details

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

The experimental results and analysis of the study are presented in this chapter. In this chapter there are three sections which are the physical properties, chemical properties and also mechanical properties / durability. The chemical properties (X-ray fluorescence, X-ray diffraction and Scanning electron microscopy), physical properties (sieve analysis moisture content, density and Laser diffraction analysis), durability (compressive strength, permeability and porosity) is evaluated.

4.2 Chemical Properties

For chemical properties, the WLP will be test on its amorphousness, oxide composition and its inner condition. The tests are XRD, XRF and SEM.

4.2.1 X-Ray Diffraction Analysis

X-Ray Diffraction (XRD) was used to analyze the crystalline properties of a material. Graph patterns of XRD analysis can show whether the material is in amorphous, partially crystalline, or crystalline state. Fig. 4.1 shows the chemical composition of the raw WLP was evaluated by XRD.



Figure 4.1: Raw WLP

Result indicates that the main spectrum from 0 keV-8keV contains the characteristics peaks of the main constituent element of Iron (Fe), Carbon (C), Phosphorus (P) and Sulphur (S).

The XRD for WLP burning at 300°C is shown in Fig. 4.2. The characteristics peak of the aim constituent element found in WLP as Iron (Fe), Phosphorus (P), Sulphur (S) and Silicon (Si).



Figure 4.2: WLP at 300°C

The XRD for WLP burning at 500°C is shown in Figure 4.3. Based on the results, after 500°C burning, WLP contained more element compared to 300°C burning. The

characteristics peak of the main constituent element found in WLP as Cerium (Coe), Iron (Fe), Phosphorus (P), Silicon (Si) and Sulphur (S).



Figure 4.3: WLP at 500°C

The XRD analysis on the WLP at 700°C is presented in Figure 4.4. The component elements found after 700°C burning are Titanium (Ti), Ferum (Fe), Phosphorus (P) and Calcium (Ca).



Figure 4.4: WLP at 700°C

4.2.2 Scan Electron Microscopy Analysis

The scanning electron microscopy (SEM) permits the observation and characterization of heterogeneous organic and inorganic materials on a nanometer (nm) to (mm) scale. In addition, SEM is more versatile instrument available for the examination and analysis of the microstructures characteristics of solid objects. Figure 4.5 illustrate the microstructure of WLP from raw, burn 300°C, 500°C until burn 700°C up to 30000 times magnification.



b) Burn 500°C

b) Burn 300°C



d) Burn 700°C

Figure 4.5: Parameters obtained from SEM analysis

4.2.3 X-ray Fluorescence Analysis

X-Ray Fluorescence (XRF) analysis was performed to determine the content of various chemical oxides in WLP. Since the pozzolanic reactivity of WLP is determined by the silica content and amorphousness of WLP, description about its content will determine the quality of WLP to be used in this research.

Both XRD and XRF tests was carried out to study the chemical composition of the WLP. The sample was dried under the sun before one portion was sieved to 600mm and the other portion was ground into finer pieces. They were then burnt at 300°C, 500°C and 700°C respectively. Though, from the result in table 4.1 it shows

that ground WLP burnt at 500°C has the highest content of SiO₂. However the ratio between SiO₂ and Al₂O₃ for all the samples was more than 2.0. To act as a good base material replacing cement in a concrete, the ratio of SiO₂ has to be between 1.5-2.0.

	WLP RAW (G)	WLP 300'C (G)	WLP 500'C (G)	WLP 700'C (G)
SiO2	8.64	9.76	11.42	9.30
TiO2	0.89	0.91	0.95	1.02
Al ₂ O ₃	2.69	2.81	3.01	2.79
Fe2O3 (t)	28.96	30.23	31.48	33.59
MnO	0.11	0.11	0.11	0.12
MgO	0.11	0.11	0.11	0.14
CaO	2.21	2.27	2.34	2.50
Na ₂ O	0.36	0.40	0.48	0.33
K20	0.48	0.44	0.48	0.42
P2O5	20.91	21.31	22.51	24.09
Jumlah	65.36	68.35	72.90	74.30

Table 4.1: XRF analysis

4.3 Physical Characterization

For physical properties the WLP will have four tests which are grading test also known as sieve analysis test to determine the size distribution of soil and moisture content test to see the moisture content in the soil. The other two tests are density test to see its density in soil and laser diffraction analysis test to determine the specific size of soil particles that passing through 63mm.

4.3.1 Bulk Density

This bulk density is used to convert quantities by mass to quantities by volume. The procedures are as followed [22]; First, convert inner diameter into radius in m³ and height into m³. Then convert weight of steel casing and weight of steel casing + WLP into kg. Weight of steel casing + WLP – weight of steel casing. Weight volume to get the bulk density in kg/m³. Figure 4.6 and 4.7 shows on filling the steel casing with raw WLP. Table 4.2 is the data for bulk density.



Figure 4.6: Fill the steel casing with WLP



Figure 4.7: Level the steel casing with plate

	Casing's Label	Casing's Inner Diameter		
Empty Casing	BD 1 =	57.78	mm	
	BD 2 =	57.78	mm	
	BD 3 =	56.87	mm	
	Average =	57.48	mm	
	Casing's Label	Casing's He	eight	
	BD 1 =	97.27	mm	
Empty Casing	BD 2 =	94.45	mm	
	BD 3 =	98.14	mm	
	Average =	96.62	mm	
	Casing's Label	Casing's Weight		
	BD 1 =	174.18	g	
Empty Casing	BD 2 =	170.32	g	
	BD 3 =	175.58	g	
	Average =	173.36	g	
Casing with WLP	Casing's Label	Casing + WLP Weigh		
	BD 1 =	542.64	g	
	BD 2 =	528.95	g	
	BD 3 =	546.70	g	
	Average =	539.43	g	

Table 4.2: Bulk Density Data

The bulk density test on WLP residue was carried out in accordance with the requirements of the ASTM. The bulk density of WLP residue was found to be 1460 kg/m3, which is comparable to the density of loose sand (circa 1440 - 1500 kg/m3).

4.3.2 Moisture Content

Clean and dry the moisture content tin and weight it to the nearest 0.01g (mw_1). Take a sample of at least 30g of soil, crumble and place loosely in container, and replace the lid. Then weight the container to the nearest 0.01g (mw_2). Remove the lid, and place the container with its lid and contents in oven and dry at 105°C to 110°C for a period of 24 hours. Do not replace the lid as the sample is in the oven. After drying, remove the container and contents from the oven and place the whole sample in the

desiccators to cool. Replace the lid and then weight the container and content from the nearest $0.01g (mw_3)$ [23]. Calculate the moisture content of soil specimen.

The moisture content on soil specimen, w, as a percentage of the dry soil mass to the nearest 0.1%. From the Equation 1,

$$W = (mw_2 - mw_3) / (mw_3 - mw_1) \times 100\%$$
(1)

where mw_1 is the mass span, mw_2 is the mass of pan with wet soil and mw_3 is the mass of pan with dry soil. Figure 4.8 and 4.9 shows the raw WLP after drying. Table 4.3 are the data for moisture content.



Figure 4.8: Raw WLP



Figure 4.9: WLP after drying process

	Pan's Label	Weight	Weight	
Empty Pan	MW 1 =	18.49	g	
(Mw_1)	MW 2 =	20.65	g	
	MW 3 =	20.21	g	
	Average =	19.78	g	
	Pan's Label	Weight		
W,LP (before	MW 1 =	20.06	g	
oven drying) (Mw ₂)	MW 2 =	20.05	g	
	MW 3 =	20.05	g	
	Average =	20.05	g	
Pan + WI P	Pan's Label	Weight		
(after oven drying) (Mw ₃)	MW 1 =	29.87	g	
	MW 2 =	31.85	g	
	MW 3 =	31.52	g	
	Average =	31.08	g	

Table 4.3: Moisture content data

Pan + WLP after drying – empty pan

= 31.08g - 19.78g = 11.3g

Moisture content = (20.05-11.3)/11.3

$$= 0.774$$

In percentage , $0.774 \ge 100\% = 77.4\%$

Based on the result produced from the experimental test, average weight for empty pan is 19.78g. For the sample which is WLP, we take only $20g (\pm 0.06g)$ for the standardize value. After dry for 24 hours, the average value (moisture content) for the WLP is 31.08g. Using the calculation given, moisture content for WLP residue is **77.4%**.

4.3.3 Sieve Analysis

The oven dried sample is weighed to 0.1% of its total mass. Eight numbers of test sieves are stacked on the mechanical shaker with the largest size test sieve appropriated to the maximum size of material present at the bottom of the stack. This is followed by the smaller size test sieves and a receiver at the bottom of the stack. The sample is placed on the top sieve and the sieve is covered with a lid. The test sieves are agitated on the mechanical sieve shaker for 15 minutes. The amount retained on each of the test sieves are weighed to 0.1% of its total mass [24]. Figure 4.10-4.12 shows some of the sieve analysis process. Figure 4.13 and table 4.4 shows the sieve analysis result.



Figure 4.10: Sieve dry WLP



Figure 4.11: After Sieve



Figure 4.12: Pan weighing

Sieve (mm)	% passing
3.3500	100.00
2.0000	77.83
1.1800	66.30
0.6000	54.66
0.4250	52.88
0.3000	49.73
0.2120	37.88
0.1500	20.23
0.0630	11.84
0.0359	6.32
0.0129	0.00
0.0014	

Table 4.4: Sieve analysis result



Figure 4.13: Percentage passing vs. particle size

From graph above, we found that the percentage which is tabulated in Table 4.5.

Soil types	Soil percentage (%)		
Gravels	4.64		
Sand	85.56		
Silt	8.3		
Clay	1.5		

Table 4.5: Percentage of soil

The final cumulative percentage is at 93.68%, short of approximately 6.5% from the total supposed collection of 100%. During the lab testing the soil is transferred from one container to the other, causing some lost of soil during the transfer, and hence the little discrepancy of percentage with the ideal situation. Yet the percentage of difference is small enough that the data is representative and conclusive. The soil sample used was crushed and ovened before proceeding with sieve analysis particle distribution test. The data shows a rather uniform distribution of the soil, with approximately 5.5% of the soil sample passing through the finest sieve of 63 μ m. WLP contains very fine particle grain size that is unable to be determined by particle distribution test using sieve analysis alone. Therefore laser diffraction test shall be conducted to further analyze the fine grain particles of the soil sample.

4.3.4 Laser Diffraction Analysis

Carry out manual sieve analysis test on the sample with the smallest sieve opening of 63 μ m. Sample which passes 63 μ m sieve will be tested with laser diffraction analyzer to get the particle size distribution by putting in a small amount of the sample into the laser diffraction particle size analyzer. The result of the test was generated by a computer attached to the analyzer [25].

MAINERN	MASTEF	RSIZER 2000		
	Result Analysis R	eport		
Sample Name: Mining Waste	SOP Name: Mining waste	Measured: Tuesday, April 16, 2013 11:33:30 AM		
Sample Source & type: Huzaifah	Measured by: Administrator	Analysed:		
Sample bulk lot ref: 123-ABC	Result Source: Measurement	1000007, April 10, 2013 11:00.02 AW		
Particle Name: Mining waste	Accessory Name: Scirocco 2000	Analysis model: Sensitivity:		
Particle RI: 1.570 Dispersant Name:	Absorption: 0 Dispersant RI: 1.000	Size range: Obscuration: 0.020 to 2000.000 um 9.01 % Weighted Residual: Result Emulation: 0.357 % Off		
Concentration: 0.0011 %Vol	Span : 4.532	Uniformity: Result units: 2.75 Volume		
Specific Surface Area: 1.37 m²/g	Surface Weighted Mean D[3,2]: 4.369 um	Vol. Weighted Mean D[4,3]: 24.861 um		
d(0.1): 1.791 um	d(0.5): 7.665 un	n .3* d(0.9): 36.526 um		

Figure 4.14: Laser diffraction analysis report



Figure 4.15: Laser diffraction analysis graph and result

From Table 4.5 and figure 4.14-4.15, it can be seen that the fine particles of the WLP residue are well graded with specific surface area obtained as $1.37 \text{ m}^2/\text{g}$. Therefore, the fine particles of WLP residue will serve as a good filler material for WLP geopolymer concrete. This also shows that the absorption of water by WLP is quiet high.

4.4 Mechanical Properties/Durability

For mechanical properties / durability will be having a test which are compressive strength test, permeability and porosity.

4.4.1 Compressive Strength Test

Compressive strength test was conducted to analyze the impact of WLP addition into the concrete mix proportion. The strength development of the concrete samples was measured at 7, 14, and 28 days of age. The data analysis was made for each w/c concrete samples and compared to control concrete (without WLP) and concrete samples containing various percentages of WLP. Table 4.6 shows the mix proportion of concrete.

MIX.N	W/I D	CA	FA	W/C	Cement	W	$SD(1ra/m^3)$
0	WLP	(kg/m^3)	(kg/m ³)	W/C	(kg/m^3)	(kg/m ³)	sf (kg/III)
1	0%	500	700	0.32	500	160	0.055
2	25%	500	525	0.38	500	190	0.055
3	50%	500	350	0.43	500	215	0.055
4	75%	500	175	0.48	500	240	0.055
5	100%	500	0	0.54	500	270	0.055

Table 4.6: Mix proportion

Table 4.7: Compressive strength result

WLP Days	0%, (MPa)	25%, (MPa)	50%, (MPa)	75%, (MPa)	100%, (MPa)
7	65.56	31.83	23.12	11.23	5.99
14	75.45	43.94	32.11	20.45	13.65
28	85	51.11	43.02	33.02	22.48

The strength development of WLP concrete is similar to OPC concrete with 62% and 86% strength at 7 & 14 days respectively. Table 4.7 shows that compressive strength decrease proportionally based on its WLP percentage used.



Figure 4.16: Compressive strength vs. WLP percentage

4.4.2 Permeability Test

The relationship of the permeability of the concrete using different percentage of WLP plays an important role. Based on the result obtained, the permeability of the concrete decreased as the percentage of WLP been used increased. This statement can be proved with the result obtained where the permeability of the concrete is higher when 100% of WLP used = 0.9 cm/s while for 25% of WLP used = 0.5 cm/s. Figure 4.17 shows the results obtained for permeability.



Figure 4.17: Permeability vs. WLP percentage

4.4.3 Porosity Test

The relationship of the porosity of the concrete using different percentage of WLP plays an important role. Based on the result obtained, the porosity of the concrete decreased as the percentage of WLP been used increased. This statement can be proved with the result obtained where the permeability of the concrete is higher when 100% of WLP used = 20 % while for 25% of WLP used = 15 %. Figure 4.18 shows the results obtained for permeability.



Figure 4.18: Porosity vs. WLP percentage

4.4.4 Relationship between Compressive Strength, Permeability and Porosity

The strength development of WLP concrete is similar to OPC concrete with 62% and 86% strength at 7 & 14 days respectively. Compressive strength decrease proportionally based on its WLP percentage used. The higher the percentage of WLP was used, the lower the compressive strength will get.

The relationship of the permeability of the concrete and the porosity using different percentage of WLP play an important role. Based on the result obtained, the permeability of the concrete and the porosity decreased as the percentage of WLP been used increased. Figure 4.19 - 4.21 shows the relation between compressive strength, permeability and porosity based on its percentage of WLP been used.



Figure 4.19: Compressive strength, porosity vs. WLP percentage



Figure 4.20: Compressive strength, permeability vs. WLP percentage



Figure 4.21: Porosity, permeability vs. WLP percentage

CHAPTER 5

CONCLUSION

5.1 Conclusion

This project is carried out to determine the possibility of utilizing WLP residue and also identify the optimum amount of WLP residue as cement replacement material in concrete. Besides that the project is to establish the effect of WLP residue as aggregate in concrete. The following conclusions can be drawn from the study.

- Physical properties show that the bulk density test on WLP residue was carried out in accordance with the requirements of the ASTM. The bulk density of WLP residue was found to be 1460 kg/m3, which is comparable to the density of loose sand (circa 1440 – 1500 kg/m3). For moisture content, the test on WLP residue (as received) was carried out in accordance with the requirement of the BS 1337: Part 2. The average moisture content was found to be 77.4%. For sieve analysis, the data shows a rather uniform distribution of the soil, with approximately 5.5% of the soil sample passing through the finest sieve of 63 μm. WLP contains very fine particle grain size that is unable to be determined by particle distribution test using sieve analysis alone. Therefore laser diffraction test shall be conducted to further analyze the fine grain particles of the soil sample. From laser diffraction analysis, it can be seen that the fine particles of WLP residue are well graded with specific surface are obtained as 1.37 m²/g.
- The optimum value for compressive strength is 51.11 MPa at 25% and this is good in structural application where structural concrete > 17 MPa.

5.2 Recommendations for Future Research

Future expansion of this research can be made to explore the potential of WLP application in construction industries. There are some adjustments that can be taken to analyze the concrete properties more comprehensively. For mixture proportion, this research observed the effect of WLP for 25%, 50%, 75% and 100% mix in the concrete mixture. However, this value can be change and the w/c may differ to see its optimum compressive and tensile strength for future research. The temperature for burning also can be change up to 900°C to see whether the silica in WLP will crystallize or not. Other test and experiment also can be carry out to see the WLP characteristic and other potential lies behind the WLP for a betterment construction industries. Utilize WLP in geopolymer concrete as the surface are in fine particles and will serve as a good filler material.

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