DEVELOPMENT OF GEOPOLYMER CONCRETE BY INCORPORATING WATER LEACH PURIFICATION RSIDUE (WLP) WITH MICROWAVE INCINERATED RICE HUSK ASH (MIRHA) AND FLY ASH-BASED

by

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Dissertation submitted in partial fulfillment of

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Universiti Teknologi PETRONAS

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CERTIFICATION OF APPROVAL

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ABSTRACT

The production of hazardous waste materials from industrial processes has cause major concern on the environment due to their intractable, harmful and costly disposal methods. Therefore, a new type of geopolymer composites are synthesized from three different industrial waste material namely water leach purification (WLP) residue, rice husk ash and fly ash as one of the alternative solution for the issues. The strength development of the geopolymer concrete produced at varying mix design proportion, different curing age and different concentration (in terms of Molar) of alkaline activator was studied in this research. It is to investigate the optimum synthesis condition based on the compressive strength of the specimen. Prior to the concreting work, few sets of characterization tests specifically X-Ray Fluorescence Analysis (XRF), Sieve Analysis test and Laser Diffraction Particle Sizing test on the WLP was carried out for selection of sample with optimum condition for geopolymer concrete production. The highest compressive strength achieved by the specimen activated with NAOH 8 M is 17.94 Mpa after 28^{th} days of curing in which the first 48 h was 80°C oven curing.

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CHAPTER 1: INTRODUCTION

1.1 Background of Study

In the last decade, geopolymer binders have emerged as one of the alternatives to cement binders for application in concrete industry. Geopolymers can be produced by polymerization of alumino-silicate oxides with alkali polysilicates yielding Si-O-Al bonds [1]. The method of producing geopolymer is known as geopolymerisation whereby many small molecules were combined into a covalently bonded network. While geopolymer concrete was produced as a replacement for the existing conventional concrete, it was actually manufactured by reusing and recycling of industrial solid wastes and by-products. Not only it is acknowledged as a type of sustainable concrete, it is also cost-effective.

In this modern world, numbers of devices and appliances were utilizing the rare earth elements in their production. Mobile phones, plasma and Liquid Crystal Display (LCD) televisions, disk drives and hybrid vehicles are some of the equipments that incorporating rare earth elements for instance [2]. Based on 2009 USGS report and report from the Ministry of Industry and Information Technology of China, there are about six countries that can provide the world with rare earth products namely China, Russia, India, USA, Brazil and Malaysia. Even though there are plenty of rare earth reserves in Australia, however, no processing plant could be built for environmental problems. Hence, Lynas had moved its separation and smelting factories to Malaysia [3].

The refinery processes of rare earth by Lynas generated few different kind of waste products namely water leach purification residue (WLP), flue gas sulphurisation residue (FGD) and neutralization underflow residue (NUF) which consist of thorium, uranium and their decay product at concentrations of about 1,6000 ppm (Th) and 30 ppm (U). Those wastes will be disposed of in at least three separate waste ponds which are referred to as residue storage facilities [4].

On the other hand, the agriculture industry related to rice production has increase the amount of rice husk produced throughout the year. Total world production of rice husk has reached 130 million tons annually with 446 thousand tons of them are produced in Malaysia [5].

1.2 Problem of Statement

Geopolymers are materials that were used in various applications such as fire-resistant and heat-resistant coatings and adhesives, medicinal applications, high-temperature ceramics, new binders for fire-resistant fiber composites, toxic and radioactive waste encapsulation and new cements for concrete. In construction industry, geopolymers were first introduced in the 1957 by Viktor Glukhovsky, Kiev, Ukraine, in the former Soviet Union which had developed concrete materials originally known under the names "soil silicate concretes" and "soil cements", derived from the low basic calcium or calcium-free aluminosilicate (clays) and alkali metal solution used in the cement production [6]. Although, geopolymerisation is not a new concept, however, the application of this technology in incorporating waste materials from industrial processes is relatively recent. Fly-ash based geopolymer concrete that was considered as one of the earliest waste incorporated geopolymer concrete were still lack in popularity compared than the conventional concrete produced by utilizing ordinary Portland cement (OPC). This is probably due to the lack of awareness on the significant benefits offered by geopolymer concrete among the users as well as the short number of geopolymer concrete manufacturers.

Quite a number of industrial and mining waste materials such as rice husk ash, fly ash and lead smelting slag create environmental problems and health problems. The current available method for disposing these waste materials not only could led to another type of environmental issues but costly at the same time. Rice husk for instance is an agricultural by-product of rice mills which were abundant in countries that consume rice as their staple food including Malaysia. The worldwide annual rice husk output is about 80 million tonnes and over 97% of the husk is generated in the developing countries. According to the statistics compiled by the Malaysia Ministry of Agriculture, Malaysia produces about 408,000 metric tonnes of rice husk each year [7]. Essentially, about 20% of the paddy weight is husk. It is one of the most intractable agriculture wastes known to man, because it's tough, woody and abrasive nature along with high silica content makes its proper disposal very difficult [8]. Rice husk ash on the other hand is a by-product due to the burning of rice husk for electricity generation. Both rice husk and rice husk ash create environmental hazard through pollution and land dereliction problems where the most common method of rice husk ash disposal is dumping on waste land.

Conversely, Water Leach Purification residue (WLP) is a major waste materials produced by the water leaching and purification process of the lanthanide concentrate in the rare earth industry. This process utilizes Magnesium Oxide and water which produces WLP as the most toxic waste materials in the rare earth refinery process. According to a report done by the National Toxic Network (NTN), WLP generated from Malaysia's Lynas Waste Advanced Materials Plant (LAMP) is expected to be about 478,800 m^3 after 10 years of operation which make it the most abundant wastes out of the total solid waste generated by the whole processes [9]. At present, due to the unfeasible total disposal of the waste; the storage of the WLP itself has become a major concern along with the potential threats it might pose to the environment and public health. Therefore, new trustworthy and environmentally friendly disposal methods are crucial.

In order to solve the tricky disposal issues as well as the health and environmental issues posed by these waste materials, geopolymerisation of rise husk ash and WLP has became one of the possible alternative environmental and commercial approach to treat the problems. Thus, by utilising these waste materials into concrete mixture will not only consume a significant amount of this by-product and leaving no room for waste disposal or storage problems. At the same time it could minimize environmental issues rooted from the unreliable disposal method such as burning and dumping. Indirectly, environmental pollution posed by OPC which has reportedly causes the increase of carbon footprint and the depletion of raw materials due to its manufacturing activity could be lessen. It is believed that this resolution could produce a new type of sustainable construction material which not only economical but eventually contribute to the preservation of environment quality in the future.

1.3 Objectives

There are two main objectives for this research. The first objective is to investigate the potential utilization of the water leach purification residue (WLP) together with Microwave Incinerated Rice Husk Ash (MIRHA) and fly ash as raw materials for geopolymer production. Next, is to study the strength development of the geopolymer concrete with varying mix design and different molarity of alkaline activator.

On the whole, this research intends to convert WLP into a cement replacement material as a substitute for ordinary Portland cement (OPC) by incorporating it with MIRHA and fly ash and turn it into a beneficial construction material through geopolymerization whereby the final product are geopolymer concrete.

1.4 Scope of Study

Before developing the geopolymer concrete, study on the characterisation of the water leach purification residue (WLP) was carried out in order to select the best characteristic of the WLP to be incorporated into the cementitious composite. A few set of tests are necessary in understanding the properties of both raw WLP and burnt WLP. Tests such as the X-Ray Fluorescence Analysis (XRF), Sieve Analysis test and Laser Diffraction Particle Sizing test was carried out on the materials.

The X-Ray Fluorescence Analysis (XRF) is an analytical technique used to determine the elemental composition of inorganic based materials. X-rays irradiate a sample and the elements present emit a fluorescent X-ray radiation that is characteristic for those elements. Analysing crystals are used to disperse the different energies. A detector is used to measure the intensity of the X-rays and wavelength.

The sieve analysis is a practice or procedure used to assess the particle size distribution of a granular material. The size distribution is often of critical importance to the way the material performs in use. Laser Diffraction Particle Sizing test is a rapid and accurate particle size distributions test for a sample. The laser beam passes through a dispersion of particles in air or in a liquid. The angle of diffraction increases as particle size decreases. This technique is relatively fast and can be performed on very small samples. A particular advantage is that the technique can generate a continuous measurement for analyzing process streams.

Conversely, to study the development of geopolymer concrete incorporating the WLP together with fly ash and microwave incinerated rice husk ash (MIRHA) by carrying out compressive strength test; a mechanical test measuring the maximum amount of compressive load a material can bear before fracturing. The test piece in the form of a cube is compressed between the platens of a compression-testing machine by a gradually applied load.

1.5 The Relevancy of the Project

Since the world has been facing a number of environmental issues related to the construction activities like the greenhouse gases emission by the manufacturing of Portland cement and land dereliction problem due to the disposal of industrial waste, the world is in need of a green and sustainable engineering solution for the affair. Study on incorporating the most abundant waste materials produced by the industrial and agricultural process into concrete is crucial in resolving the disposal problem. Given that UTP has carried out similar research on this topic with rice husk ash as the focal point and had proven succeed. With the preceding experience, research on WLP as a raw material in geopolymer concrete has become straightforward. Additionally, UTP has all the necessary equipments for the experimental lab.

1.6 Feasibility of the Project

With all the required equipments for experimental lab such as the machine for characterization tests and the strength test machine available in UTP, it is believed that this project is feasible in terms of resources. In cases where the equipments are unavailable due to some constraints, the option is to outsource the facility from other universities or independent laboratories. In terms of time, the research should be completed within 28 weeks where the first 14 weeks will be focusing on the characterization tests while the last 14 weeks will be focusing on the development of geopolymer concretes.

CHAPTER 2: LITERATURE REVIEW

This chapter discussed about environmental issues due to the manufacturing of Portland cement and the use of water leach purification residue (WLP) in geopolymer concrete through some published literature.

2.1 Ordinary Portland Cement (OPC) in Concrete and Environment

Production of cement that was used in concrete manufacturing has contributed to numerous environmental problems. Production of cement that generates CO_2 with similar amount manufactured, depletion of raw material, and enormous amounts of toxic gases have driven world's concern to a utilization of greener cement technology [9]. Meanwhile concrete industry is the main material and increasing demand of concrete in the world already surpasses 8.8 billion tons production per year [10]. Due to the production of OPC, it is estimated that by the year 2020, the CO_2 emissions will rise by about 50% from the current levels [11], [12]. Basically, production of 1.5 billion ton of cement generates 1.5 billion ton of CO_2 which are responsible for 5% CO_2 production in the world [13]. The cement sustainability initiative progress report shows that combustion acquires 40% whilst calcinations acquire 60% of the total CO_2 emissions from cement manufacturing process. The emissions from combustion are related to fuel use and the emissions due to calcinations are formed when the raw materials (limestone and clay) are heated to over 1500°C and CO_2 is liberated from the decomposed limestone [14]. This has contributes to global warming and thus climate changes all over the world particularly in the developed and developing country.

Environmental issues due to the manufacturing of OPC have lead to the finding of substitute materials by the researchers. A new type of binder is needed to replace the role of OPC so as to reduce the associated environmental problems with its production. Geopolymer has recently emerged as a novel engineering binder material with environmentally sustainable properties [15]. The production of geopolymer requires much lower calcining temperature (600-800°C) and emits 80-90% less CO_2 than Portland cement [16]. Thus reduces the emission of CO_2 due to manufacturing of OPC.

2.2 Water Leach Purification Residue (WLP)

The question of what to do with Water Leach Purification residue (WLP) in Malaysia arose with the operation of the rare earth refinery industry by the Lynas Advanced Materials Plant (LAMP) in Gebeng, Pahang towards the end of the year 2012. According to the research done by the National Toxic Network (NTN), Lynas assumed that the waste materials will be utilised in other industries such as construction industry due to its storage and environmental issues [17]. The waste water treatment plant will generate up to 2000tpa of biosludge which will be disposed of to the WLP tailing pond [18]. The biosludge is likely to contain residual uranium, thorium and other hazardous materials as a result of the concentration of contaminants in the water filtration process [19].

However, due to the limited near to non research has been done on the characterisation of WLP, the closest materials to it would be phosphogypsum and red mud. Though from recent research, only red mud had been incorporated as geopolymer concrete whereas research done on phosphogypsum as source material for geopolymer concrete is hardly available. Therefore, to be able to understand the characteristic of WLP, this section will review briefly the characteristic of phosphogypsum as a reference while the next section will be presenting red mud role in geopolymer concrete.

Phosphogypsum is an industrial byproduct of phosphoric acid manufacture using a single (dihydrate) or two-step (hemihydrate –dihydrate) process. Over 6.0 million tons of phosphogypsum is produced per annum in India and poses various environmental and storage problems. Phosphogypsum contains impurities of free phosphoric acid, phosphates, fluorides and organic matter that adhere to the surface of gypsum crystals and also substituted in the crystal lattice of gypsum [20-22]. Phosphogypsum also contains radioactive elements such as U 238, U234, Ra226, Pb210 and Po210, which are derived from the phosphate rocks [23-24]. The contents of radioactive elements vary in a wide range, depending on the composition of rock phosphate [25]. It is essential to remove these impurities to use phosphogypsum for controlling the cement hydration. Some beneficiation processes comprising washing with water, thermal as well as chemical treatments have been applied to gypsum sludge [26]. It has been found that water-soluble impurities can be removed by washing with water, whereas HPO_4^{2-} and FPO_3^{2-} substituted in the crystal lattice of gypsum with SO_4^{2-} ions form solid solutions and are difficult to remove by washing. In heating phosphogypsum towards hemihydrate stage, HPO_4^{2-} ions get freed, which form inactive compounds on neutralization with $Ca(OH)_2$ [27-28]. The impurities of phosphates and fluorides can be reduced by treating phosphogypsum with a mixture of sulphuric acid and silica [29], ammonium hydroxide [30] or aqueous ammonium sulphate solutions [31]. Recently, a process has been developed at the Central Building Research Institute for the beneficiation of phosphogypsum wherein impurities, particularly undecomposed phosphate rock, organic matter, alkalis, quartz, etc., retained over a 300-mm sieve, are discarded as they are rich in impurities while the bulk of gypsum passing through the sieve is further washed, centrifuged and dried for making value-added building materials [32].

2.3 Geopolymers

The geopolymer technology has recently attracted increasing attention as a viable solution to reusing and recycling industrial solid wastes and by-products, which provides a sustainable and cost-effective development for many problems where hazardous residues have to be treated and stored under critical environmental conditions [33]. Therefore, countless studies and researches on geopolymer have been going on for few decades with various waste materials, hazardous or not, utilized to replace Ordinary Portland Cement (OPC) in the conventional cement concrete.

Geopolymers belong to the group of strong and durable cementitious materials that harden at temperatures below 100°C [34-36]. Geopolymer can be produced by polymerization of alumino-silicate oxides with alkali polysilicates yielding Si-O-Al bonds [37]. The two main ingredients of geopolymer binder are alkali liquids and source materials. The alkali liquids are usually sodium or potassium based solutions. The source materials should be rich in silicon (Si) and aluminium (Al) from geological origin or by-product such as clays, metakaolin, fly ash, bottom ash, slag, rice husk ash. The use of geopolymer involves a lesser amount of green house gas and is, therefore, a more environmentally-friendly binding material compared to the conventional Portland cement [38].

Throughout the year, a number of waste materials have been successfully utilized in replacing OPC such as Pulverized Fly Ash (PFA), Ground Granulated Blast Furnace

Slag (GGBS) and Microwave Incinerated Rice Husk Ash (MIRHA). In respect to hazardous waste materials, few researches have been conducted on geopolymerization of these materials in construction industry. Among them are the geopolymerization of lead smelting slag (LSS), bauxite residue and red mud (RM).

2.4 Use of WLP in Concrete

Red mud which posses the similar qualities to the LAMP tailing but with a lower concentration of radioactive uranium and thorium and their decay products [39] was reviewed as it is the closest example which incorporating radioactive waste materials in construction materials. J. He, 2012 has developed geopolymer composites from two types of waste materials namely red mud (RM) and rice husk ash (RHA). For this research. J. He prepared 4 specimens to study different parameters on the geopolymer. He studies the characteristics of the geopolymer with different curing durations. The geopolymer was cured in a laboratory ambient environment for 14 days after casting. The specimens where then demoulded, followed by prolonged curing in exposed condition and at elevated temperatures [40]. It is to study the effect of curing time on geopolymer. From this experiment, J. He et al. [41] concluded that the complete curing time for RM-RHA was about 35 days and with curing progressing, there is an obvious transition from a more ductile to a brittle failure. He stated that if the stabilization of peak strength is adopted as a criterion to judge whether curing is complete, then some geopolymerization or side reactions may still occur after complete curing, as indicated by the continuous increase in stiffness or decrease in failure strain while under a constant peak strength. [42]. Therefore, J. He et al. [43] concluded that much longer reaction or curing time is required for this type of RM-RHA based geopolymers to develop its maximum strength and stiffness and that a great amount of impurities in the final geopolymeric products from the two raw materials may cause negative impact on the geopolymerization rate. The effect of RHA particle size was also studied by J. He et al. [44] where he found out that a finer RHA that was ground to #100 mesh produces geopolymer with improved compressive strength, Young’s modulus and failure strain since finer particle sizes improve the reactivity of the RHA so that a higher degree of geopolymerization of the materials can be achieved [45], which makes the resulting geopolymer specimens stronger and more ductile and the higher specific surface area of a finer RHA particles also results in stronger and more ductile geopolymer [46]. The other two parameters used to investigate the synthesis of RM-RHA geopolymer were the RHA/RM ratio which essentially controls the Si/Al ratio of the end product. The alkalinity or Na/Si ratio was also altered by changing the concentration of NaOH solutions [47]. This geopolymer which posses the compressive strengths of up to 20.5 Mpa, which is comparable to most Portland cements, suggesting that the RM-RHA geopolymers can be a potential cementitious construction material [48].

For WLP to be utilised in the construction industry as one of the cement replacement materials, a study and experimental research on the geopolymer development are eventually will become the focus of this paper. One of the most distinctive ways adopted by researchers in geopolymer concrete study is by observing the compressive strength development of the geopolymer versus the curing time of the geopolymer (A. Kusbiantoro et al., 2012) [49]. The geopolymer comprises of varying mixing ratios of the raw materials will be studied by observing their respective compressive strength development over the days. In an experimental research apart from the RM-RHA geopolymer, study on Immobilisation of Lead Smelting Slag within Spent Aluminate – Fly Ash Based Geopolymers shows that hazardous waste material such as lead smelting slags (LSS) generated from the production of lead have a potential to be turned into useful building materials under controlled conditions in specific situations [50]. The experimental result of this LSS geopolymers was plotted as compressive strength versus curing time graph. The plot shows that the higher the value of LSS incorporated into the geopolymer the higher the compressive strength of the geopolymer at 28 days of curing (M.B. Ogundiran et al., 2013). Research on Synthesis and Heavy Metal Immobilization Behaviours of Slag Based Geopolymer was a work done for the synthesis of slag based geopolymer with four different slag content (10%, 30%, 50%, and 70%) and three types of curing regimes (standard curing, steam curing and autoclave curing) to obtain the optimum synthesis condition based on the compressive and flexural strength. In this research, the slag based geopolymer achieved 75.2 MPa compressive strength and 10.1 Mpa flexural strength [51]. Consequently, these methods should be considered in the study of WLP geopolymer development.

It is proven that hazardous materials can be utilised into construction product by incorporating its optimum amount into the concrete to produce acceptable concrete with high compressive strength. This optimum amount needs to be studied by carrying out development study of the material which comprises of a range of experiments. WLP waste may contain some useful and valuable elements but these are frequently bound up with toxic or radioactive elements that make other uses hazardous to workers and consumers of the final product or the environment [52].

CHAPTER 3: METHODOLOGY

3.1 Introduction

This chapter presents the details of development process of making geopolymer concrete incorporating water leach purification residue (WLP), microwave incinerated rice husk ask (MIRHA) and fly ash (FA) as the source materials.

Since WLP has never been used in geopolymer concrete production, a trial-and-error process was adopted. The focus of the study was to discover the potential of WLP as a cementitious material and introducing it as a new material to the concrete construction industry at once.

The manufacturing and testing method for this research was done using the same method used in the production of other type of geopolymer concrete. Compressive strength of the produced specimen was selected as the research parameter for this development process.

However, due to the absence of literature review or information on the characteristics of the water leach purification residue (WLP) itself, the present study on the geopolymer concrete strength was carried out after a few sets of characterisation tests on the material was completed. Therefore, Figure 1 below shows the sequence of the research including the characterisation test of the WLP.

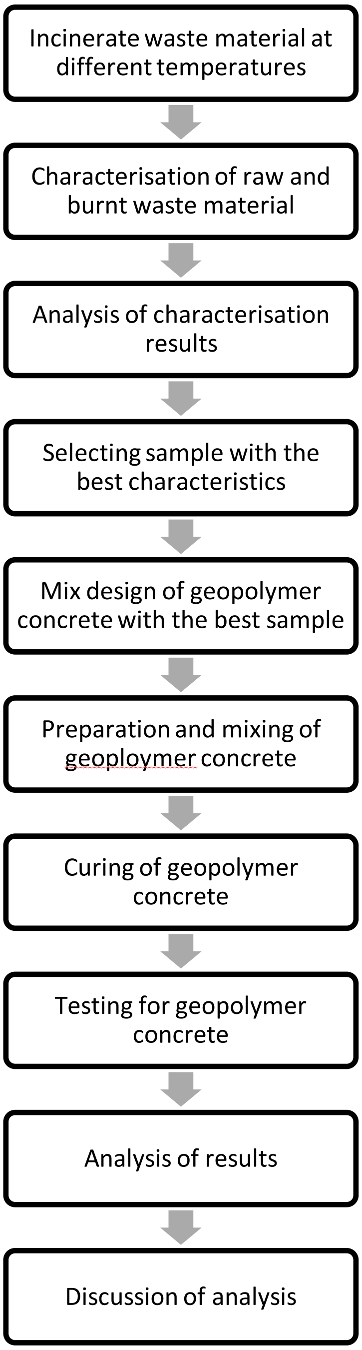


Figure 1: Sequence of research

3.2 Materials

Materials like coarse aggregates and fine aggregates were prepared as according to the appropriate standard set in the guidelines. Equally, the main constituents of geopolymer are source materials and alkaline liquid, therefore, alkaline liquid consists of the combination of sodium hydroxide or potassium hydroxide with sodium silicate or potassium silicate will be needed [53]. As for the source materials which comprises of fly ash (FA), microwave incinerated rice husk ash (MIRHA) and water leach purification residue (WLP), all was obtained from the respective industries related to the generation of the wastes.

3.2.1 Water Leach Purification Residue (WLP)

In this experimental research, the raw water leach purification residue (WLP) obtained from Lynas Advanced Materials Plant (LAMP), Gebeng, Pahang, Malaysia was used as one of the source material together with microwave incinerated rice husk ash (MIRHA) and fly ash (FA). The raw WLP was stored in the concrete technology laboratory of Department of Civil Engineering, PETRONAS University of Technology, Perak, Malaysia before it is sun dried and divided into two portions. The first portion of the dried WLP was crushed manually and sieved to 600 µm while the other portion was ground by the grinding machine.

Both the sieved and ground WLP was further divided into 3 small portions respectively. Each of these small portions was then burnt at 300°C, 500°C and 700°C correspondingly. The Earth Material Characterisation Laboratory, University of Science, Malaysia carried out the XRF analysis.

From the Table 10 and Table 11 below, for all the tested portions, the molar Si-toAl ration was about 3 and the calcium oxide content was very low. On the other hand, the iron oxide (Fe_2O_3) contents from all of the portions are quite high especially WLP which was burnt at the highest temperature. This gave the 700°C WLP darker colour than the other samples.

The particle size distributions of the WLP were given in Figure 2 and Figure 3. Figure 2 shows the particle size distribution in percentage by volume passing size or cumulative. The final cumulative percentage is 93.68%, short of approximately 6.32% 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 test was conducted by using manual sieving with the BS sieve aperture size of 2.00 mm to 63 µm. 80% of the particles were bigger than 0.1 mm with approximately 4.68% passing through 63 µm sieve. Another particle size distribution tests were performed using the Malvern Instruments Mastersizer MS2000 on the sample that pass through the finest manual sieve. It is shown in Figure 3 whereby the particle size distribution is in percentage by volume in interval. 80% of the particles were smaller than 2.884 µm and the Specific Surface Area was 1.37 m^2/g.

	WLP RAW (G)	WLP 300'C (G)	WLP 500'C (G)	WLP 700'C (G)
SiO_2	8.64	9.76	11.42	9.30
TiO_2	0.89	0.91	0.95	1.02
Al_2O_3	2.69	2.81	3.01	2.79
Fe_2O_3 (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_2O	0.36	0.40	0.48	0.33
K_20	0.48	0.44	0.48	0.42
P_2O_5	20.91	21.31	22.51	24.09
Jumlah	65.36	68.35	72.90	74.30

Table 1: XRF result for ground WLP – raw and burnt at 300°C, 500°C and 700

	WLP RAW (S)	WLP 300'C (S)	WLP 500'C (S)	WLP 700'C (S)
SiO_2	5.82	5.46	6.51	6.08
TiO_2	0.95	0.97	1.07	1.07
Al_2O_3	2.08	2.08	2.36	2.37
Fe_2O_3 (t)	32.61	33.41	36.50	37.07
MnO	0.11	0.11	0.12	0.12
MgO	0.12	0.13	0.13	0.14
CaO	1.73	1.75	1.85	1.82
Na_2O	0.35	0.19	0.23	0.20
K20	0.16	0.15	0.22	0.17
P_2O_5	20.85	21.49	23.14	23.45
Jumlah	64.78	65.74	72.13	72.49

Table 11: XRF result for sieved WLP – raw and burnt at 300°C, 500°C and 700°C

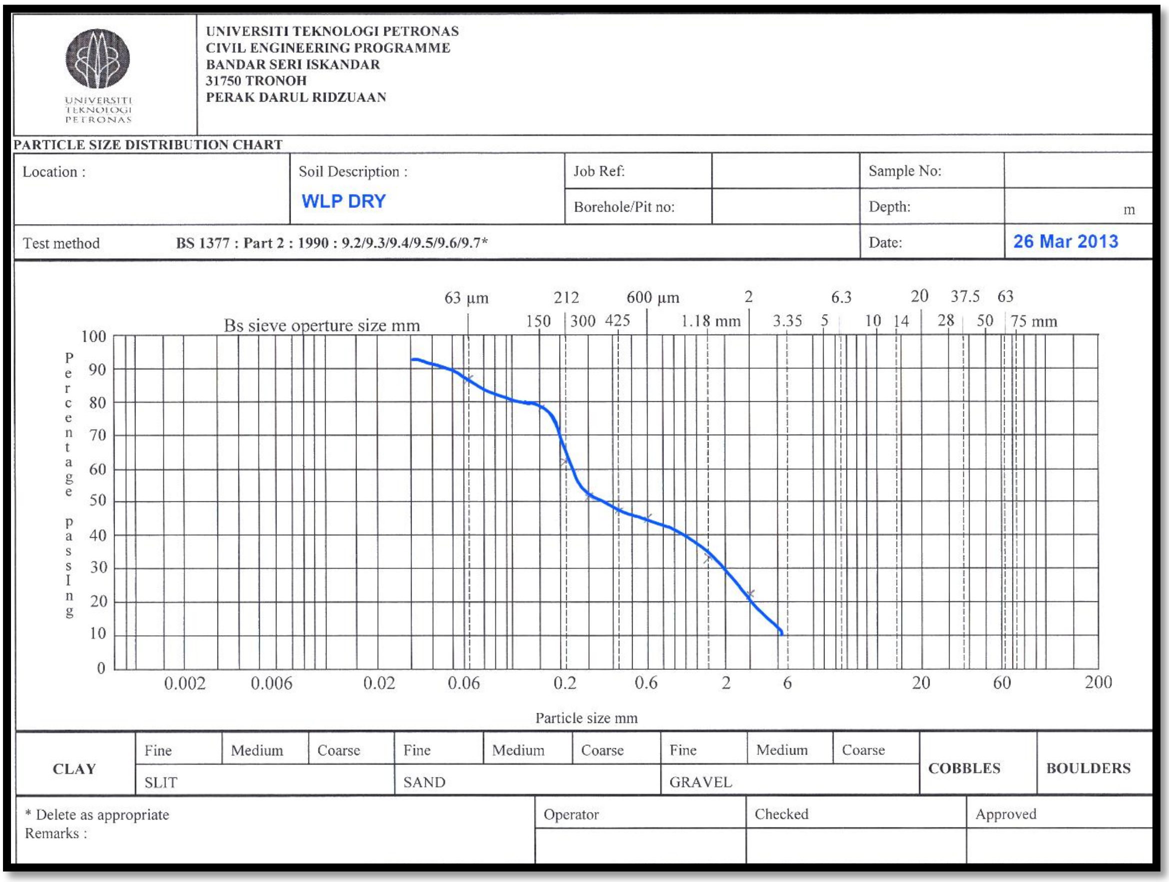
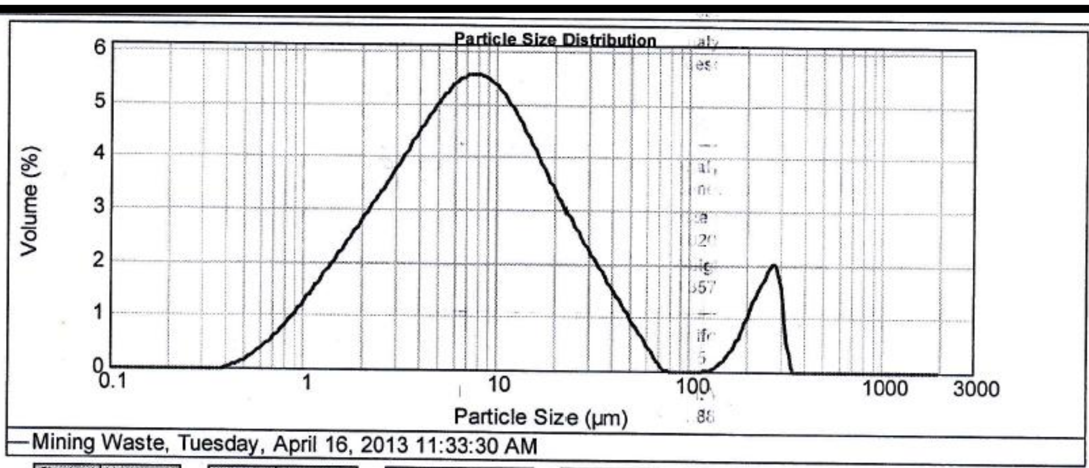


Figure 2: Particle size distribution



Size (µm)	Volume in %
0.010	0.00
0.011	0.00
0.013	0.00
0.015	0.00
0.017	0.00
0.020	0.00
0.023	0.00
0.026	0.00
0.030	0.00
0.035	0.00
0.040	0.00
0.046	0.00
0.052	0.00
0.060	0.00
0.069	0.00
0.079	0.00
0.091	0.00
0.105	

Size (µm)	Volume in %
0.105	0.00
0.120	0.00
0.138	0.00
0.158	0.00
0.182	0.00
0.209	0.00
0.240	0.00
0.275	0.00
0.316	0.00
0.363	0.00
0.417	0.08
0.479	0.16
0.550	0.30
0.631	0.46
0.724	0.67
0.832	0.90
0.955	1.15
1.096	

Size (µm)	Volume in %
1.096	1.41
1.259	1.69
1.445	1.97
1.660	2.25
1.905	2.53
2.188	2.82
2.512	3.12
2.884	3.43
3.311	3.75
3.802	4.07
4.365	4.38
5.012	4.65
5.754	4.86
6.607	4.99
7.586	5.02
8.710	4.93
10.000	4.74
11.482	

Size (µm)	Volume in %
11.482	4.46
13.183	4.10
15.136	3.71
17.378	3.30
19.953	2.91
22.909	2.54
26.303	2.19
30.200	1.86
34.674	1.55
39.811	1.23
45.709	0.91
52.481	0.60
60.256	0.30
69.183	0.03
79.433	0.00
91.201	0.00
104.713	0.00
120.226	

Size (µm)	Volume in %
120.226	0.02
138.038	0.14
158.489	0.38
181.970	0.79
208.930	1.28
239.883	1.64
275.423	1.66
316.228	0.10
363.078	0.00
416.869	0.00
478.630	0.00
549.541	0.00
630.957	0.00
724.436	0.00
831.764	0.00
954.993	0.00
1096.478	0.00
1258.925	

Size (µm)	Volume in %
1258.925	0.00
1445.440	0.00
1659.587	0.00
1905.461	0.00
2187.762	0.00
2511.886	0.00
2884.032	0.00
3311.311	0.00
3801.894	0.00
4365.158	0.00
5011.872	0.00
5754.399	0.00
6606.934	0.00
7585.776	0.00
8709.636	0.00
10000.000	

Figure 3: Particle size distribution using laser diffraction

3.2.2 Alkaline Liquid

Alkaline solution for the manufacturing of geopolymer concrete typically comprises of sodium hydroxide and sodium silicate. The sodium hydroxide used in this research was in the form of pellet. The mass of NaOH solids in a solution varied depending on the concentration of the solution expressed in terms of molar, M [curtin-flyash].

In this experiment, two types of concentration were used which is 8 M and 12 M. to prepare the NaOH solution, the NaOH pellet with the calculated mass in grams was dissolved in distilled water. For example, to produce NaOH solution with concentration of 8 M, it is 8 x 40 g (molecular weight of NaOH) = 320 g per litre of the solution. Likewise, for the NaOH solution with concentration of 12 M, 480 g of the pallet was needed.

Conversely, sodium silicate solution was obtained from the concrete technology laboratory. The chemical composition of the sodium silicate solution was Na_2O = 14.7%, SiO_2 = 29.4%, and H_2O = 55.9% by mass. The other characteristics of the sodium silicate solution were specific gravity = 1.53 g/cc and viscosity at 20°C = 400 cp. [curtin-flyash]

3.3 Laboratory Work

Initially, several trial mixtures of geopolymer concrete were created and the casting was done in 100x100x100 mm cubes formwork.

A. Mix Design of Geopolymer Concrete

For this study, few types of different waste materials were replacing the function of ordinary Portland cement as the binder for the concrete. The manufacturing of WLP geopolymer concrete was applying the trial and error concrete technology methods with a number of varying mixtures tested. The sample mixtures were tested with compressive strength test. Tables below shows the varying mixture proportion detail tested.

Mixture Proportion Detail

Percentage of WLP & MIRHA (%)	Fly Ash	MIRHA	WLP	Coarse Aggregate	Fine Aggregate	Sodium Hydroxide	Sodium Silicate	Water
5	315.0	17.5	17.5	1200.0	645.0	41.0	103.0	140.3
10	280.0	35.0	35.0	1200.0	645.0	41.0	103.0	140.3
15	245.0	52.5	52.5	1200.0	645.0	41.0	103.0	140.3

Table 3: Fly ash, MIRHA and WLP (with same inclusion percentage) as source materials

Mixture Proportion Detail

WLP	Coarse Aggregate	Fine Aggregate	Sodium Hydroxide	Sodium Silicate	Water
350.0	1200.0	645.0	41.0	103.0	234.0

Table 4: WLP alone as source material

Mixture Proportion Detail

WLP	MIRHA	Coarse Aggregate	Fine Aggregate	Sodium Hydroxide	Sodium Silicate	Water
17.5	332.5	1200.0	645.0	41.0	103.0	234.0

Table 5: WLP and MIRHA as source materials

B. Preparation of Geopolymer Concrete

Before mixing process, all the essential materials will be prepared in advance due to certain reasons. Alkaline solutions for instance were prepared 1 hour before mixing process starts to prevent precipitation of NaOH in the solution [54]. Mixing process starts with dry mixing for about two minutes before proceeding with the wet mixing in order to ensure the mixture homogeneity so as to prevent problems like concrete bleeding or segregation afterwards.

C. Curing of Geopolymer Concrete

After casting the specimens, they were kept in rest for some times before being demoulded. The curing process of a concrete is extremely crucial since this is the time where concrete will achieve their best strength. Thus, the method adopted for curing will determine the best method to achieve the optimum concrete compressive strength. Few available curing methods are ambient curing, oven curing, external exposure curing and hot gunny curing where the specimens will be kept under different controlled temperature and environment according to the varying methods. However, for this research oven curing was adopted with temperature kept at 80°C for 48 h before demoulded. The specimens then were kept at ambient cured (at room temperature) until the testing age.

3.4 Project Activities

Various activities have been carried out throughout this research and it has been divided into two major parts. The first part is the WLP characterisation while the second part was the geopolymer concrete synthesis. Table 4 and table 5 below summarize details of the activities done.

ACTIVITY	INCENARATION OF WLP	CHARACTERISATION TEST
	▪ 300◦C ▪ 500◦C ▪ 700◦C	▪ X-Ray Diffraction Analysis (XRD) ▪ Laser Diffraction Particle Sizing test ▪ Sieve Analysis Test

Table 4: Activities for characterisation part

ACTIVITY	MIX DESIGN	GEOPOLYMER CONCRETE	CURING	TEST ON CONCRETE CUBE
	▪ Coarse aggregate ▪ Fine aggregate ▪ Fly ash ▪ MIRHA ▪ WLP ▪ Sodium Hydroxide ▪ Sodium Silicate ▪ Water	▪ Dimension: 100 mm X 100 mm X 100 mm ▪ Testing age: 7 and 14 days ▪ Geopolymer: 1) Fly ash + MIRHA + WLP (both MIRHA & WLP replace 5%, 10% & 15% of fly ash)	▪ Oven curing at 80°C	▪ Compression Strength Test

Table 5: Activities for geopolymer concrete synthesis

3.5 Key Milestone and Gantt Chart

To keep track on the progress of the project as well as the key milestone, Gantt chart was prepared to provide a visual timeline for starting and finishing specific task. Table below shows the timelines for the project.

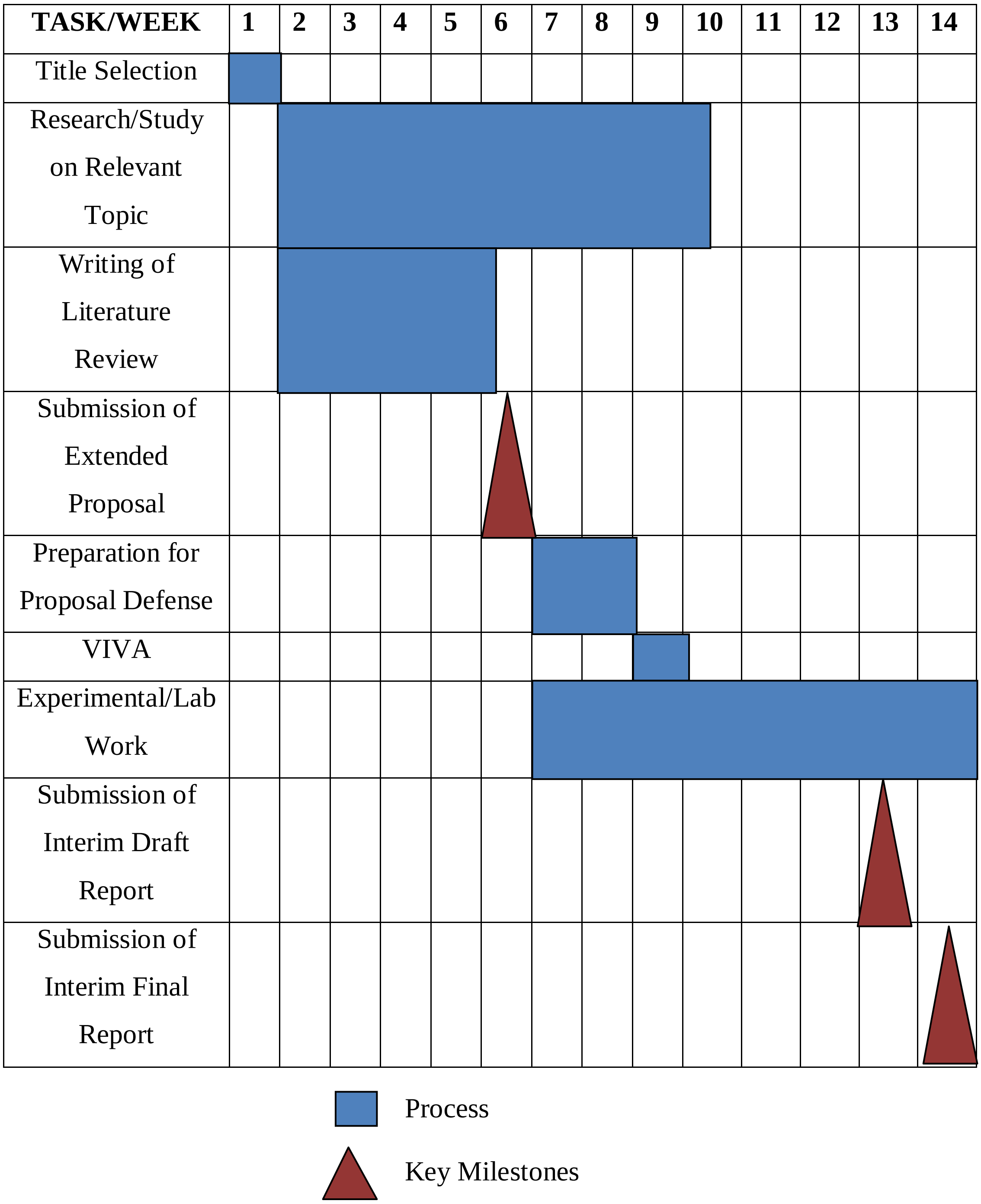


Table 8: Timelines for FYP I

TASK/WEEK	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Project Work Continues															
Submission of Progress Study															
Project Work Continues															
Pre-SEDEX															
Submission of Draft Report															
Submission of Dissertation (soft bound)															
Submission of Technical Paper															
VIVA															
Submission of Project Dissertation (hard bound)															

Process

Key Milestones

Table 9: Timelines for FYP II

3.6 Tools

Tools and equipments are crucial in ensuring the research activities can be carried out smoothly and efficiently. Table below shows the major equipments used in carrying out the characterisation test for the WLP as well as the machine used to test the strength of the produced geopolymer concrete.

Experimental Test	**Equipment**
X-Ray Diffraction Analysis (XRD)	 X-Ray Diffraction (XRD) machine
Laser Diffraction Particle Sizing The objective of particle size distribution test by using laser diffraction method is to measure the size and distribution of the soil sample smaller than 63 µm.	 Laser Diffraction Particle Sizing machine

Sieve Analysis To determine the size distribution of soil using the dry sieving method	 Sieve mesh  Sieve shaker
Compression Strength Test	 Compression Testing machine

Table 10: Tools and equipments used for the research

CHAPTER 4: RESULT AND DISCUSSION

4.1 Introduction

In this chapter, the experimental results are presented and discussed. The compressive strength test data and results were plotted in Figures and Tables. The compressive strength test was performed on 100x100x100 mm specimens.

In Section 4.2 of the chapter, the effects of few parameters on the compressive strength of WLP+MIRHA+FA-based geopolymer concrete are discussed. The parameters considered are as follows:

1. Concentration of sodium hydroxide (NaOH) solution, in Molar
2. Age of concrete
3. Percentage of WLP, MIRHA and FA as source material

4.2 Effect of Parameters

4.2.1 Concentration Of Sodium Hydroxide (Naoh) Solution, in Molar

Concrete casting was done by using fly ash as the base material while MIRHA and WLP replacing it by 5%, 10% and 15% respectively. For mix design using NaOH – 8 M, it is observed that geopolymer concrete produced with WLP+MIRHA+FA (5%,5%,90%) mix and WLP+MIRHA+FA (10%,10%,80%) mix provide some reading when tested while for WLP+MIRHA+FA (15%,15%,70%) mix result was not available. On the other hand, all of the mix design with NaOH – 12 M produces results. The curing time was 48 hours at 80°C. The measured 7th day compressive strengths of the tested cubes are given in Table 11.

Mix Design	NaOH Molarity	Compressive strength at 7th day (Mpa)
		Cured for 48 hours at 80°C
WLP+MIRHA+FA (5%,5%,90%)	8 M	9.52
WLP+MIRHA+FA (10%,10%,80%)		3.45
WLP+MIRHA+FA (15%,15%,70%)		n/a
WLP+MIRHA+FA (5%,5%,90%)	12 M	12.23
WLP+MIRHA+FA (10%,10%,80%)		6.57
WLP+MIRHA+FA (15%,15%,70%)		3.17

Table 11: Effect of Alkaline Solutions

In Table 11, the difference between all the mixtures is the concentration of NaOH solution in terms of Molar (second column). Mixture design with higher concentration of NaOH solution produced higher compressive strength than the one with lower concentration of NaOH.

4.2.2 Age of Concrete

The effect of concrete age on compressive strength of the geopolymer concrete was shown in the Figure 4 and Figure 5. When WLP and MIRHA replacing FA by 5% and 10% respectively – NaOH 8M, the compressive strength increases as the curing age increase. However, when the curing age reaches 28th days, the compressive strength of specimen with WLP+MIRHA+FA (10%,10%,80%) starts to decline while specimen WLP+MIRHA+FA (10%,10%,80%) kept increasing. On the contrary, the compressive strength of specimens with NaOH – 12 M were higher by almost twice of the specimens with NaOh – 8 M during the early curing age except that the compressive strength of 12 M drops faster than the 8 M specimen which is at 14th days.

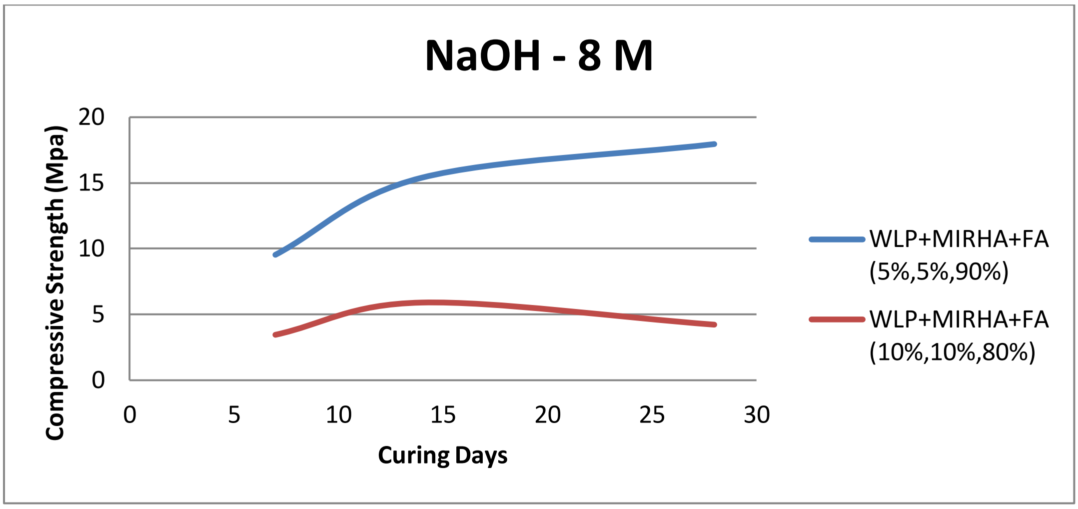


Figure 4: Compressive strength at different ages with 8 M NaOH

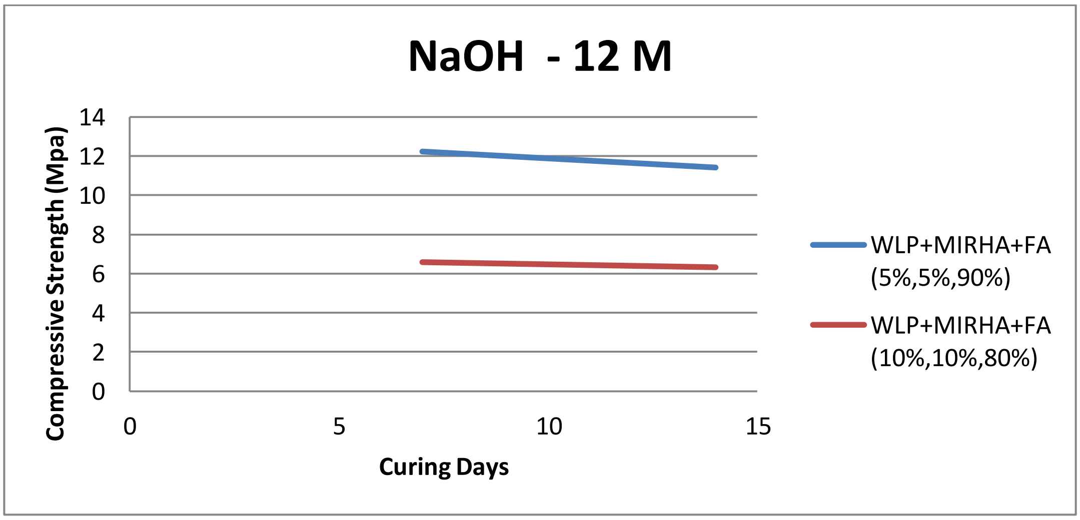


Figure 5: Compressive strength at different ages with 12 M NaOH

4.2.3 Percentage of WLP, MIRHA and FA as source material

Table below shows the compressive strength of the geopolymer concrete produced from varying mix design proportion. In the beginning, the casting was done using 95% of MIRHA as a base material, however, MIRHA failed to bind all the aggregates together and crumbles with slight pressure. Casting was also done by using 100% WLP as source materials and by looking at the result, WLP alone has no potential to become a cementitious material. It is noted that the mix design of WLP (100%) and WLP+MIRHA (5%,95%) fall short as a geopolymer concrete.

Therefore, it is best to conclude that WLP+MIRHA+FA (5%,5%,90%) with NaOH 8 M is the best mix design proportion due to its ability to produce geopolymer concrete with the highest strength of 17.94 Mpa. It is approaching minimum concrete strength of 20 Mpa used in construction industry. With a longer curing time, the potential of this geopolymer concrete to achieve higher compressive strength is also high.

Mix Design	**NaOH Molarity**	**Curing Days**			
		3	7	14	28
WLP+MIRHA+FA (5%,5%,90%)			9.52	15.38	17.94
WLP+MIRHA+FA (10%,10%,80%)	8 M		3.45	5.92	4.23
WLP+MIRHA+FA (15%,15%,70%)			n/a	n/a	n/a
WLP+MIRHA+FA (5%,5%,90%)			12.23	11.42	
WLP+MIRHA+FA (10%,10%,80%)	12 M		6.57	6.31	
WLP+MIRHA+FA (15%,15%,70%)			3.17	n/a	
WLP (100%)	12 M	n/a			
WLP+MIRHA (5%,95%)	8 M	n/a			

Table 12: Compressive strength results

CHAPTER 5: CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Based on the experimental work and the analysis of the results reported in this study, the following conclusions were drawn:

1. WLP can be included as source material in the production of geopolymer concrete provided that it is incorporated together with other source materials which in this case are MIRHA and fly ash. However, the inclusion of WLP should be limited to certain amount while fly ash should be the major component.
2. The higher the concentration (in terms of molar) of sodium hydroxide solution, the higher the compressive strength produced by the geopolymer concrete.
3. The best mixture design after adopting the trial-and-error method was to include MIRHA and WLP at 5% respectively and the rest 90% should be FA. This is the mixture that can produced geopolymer concrete with highest strength.

5.2 Recommendation

There are a lot of improvement can be done on this research, some of the recommendation that can be considered are as follows:

1. Researcher can consider various other parameters on the compressive strength of water leach purification residue, microwave incinerated rice husk ash and fly ash-based geopolymer concrete such as:
   i. The addition of super plasticizer
   ii. Dry curing versus steam curing
   iii. Curing temperature
   iv. Ratio of sodium silicate solution-to-sodium hydroxide solution, by mass
   v. Mixing time [55]
2. Other than incorporating WLP with fly ash, fresh research can be done by incorporating this waste material with some other cement replacement materials such as Metakaolin or Ground Granulated Blast Furnace Slag (GGBS)

3. Further research on WLP can be performed in other area to diverse the utilization of the waste material. Geopolymer concrete should not be the only focal point for the exploitation of this waste material. A study can be done to observe the suitability of WLP as a subgrade material for the construction of road and highway.

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APPENDICES

APPENDIX 1-1

BURNING OF RICE HUSK INTO MIRHA

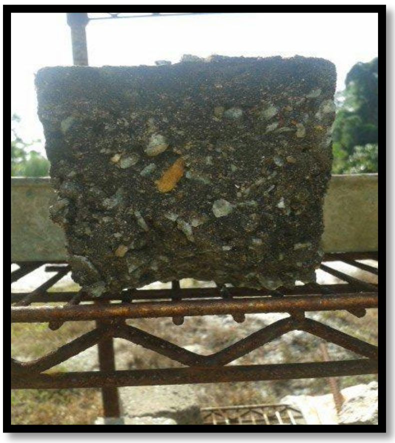
<u>Furnace used to burn rice husk:</u>

<u>MIRHA after burning for 8 hours:</u>

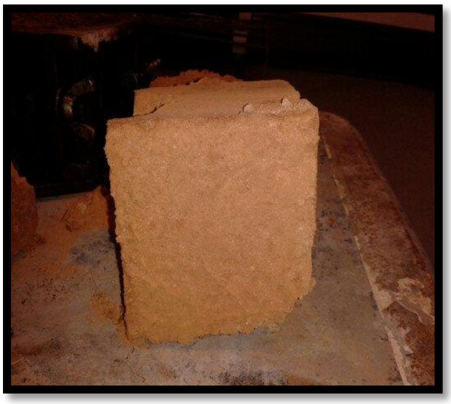
APPENDIX 1-2

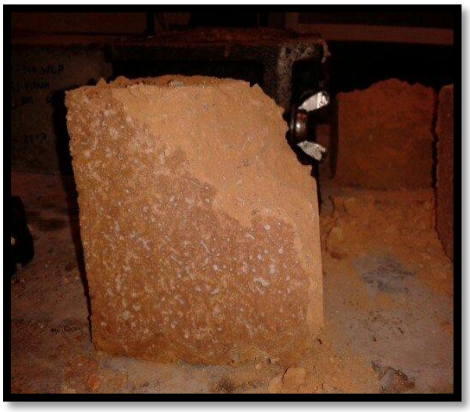
GEOPOLYMER CONCRETE

WLP+MIRHA:



WLP alone:





WLP+MIRHA+FA: