GLASS WOOL WASTE AS CEMENT REPLACEMENT MATERIAL IN MORTAR

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CERTIFICATION OF APPROVAL

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Approved by,

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(Prof. Ir. Dr. Muhd Fadhil Nuruddin)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

December 2012

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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NUR ALWANI DIYANA NOR AZAHA

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ABSTRACT

Research on Glass Wool Waste material is an experimental project to investigate the capability of this new material to replace cement content in mortar mixture. The project is conducted by processing the new material first before being used in the mortar mixture which involve burning and grinding process. The new material need to be analysed to determine the best choice of material condition to optimize the mortar performance based on chemical analysis. After the new material is prepared, mortar specimens with 0%, 5%, 10%, 15% and 20% cement replacement are casted. Mechanical tests are conducted from time to time to determine which replacement percentage gives the best pozzolonic properties.

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CHAPTER 1

INTRODUCTION

1.1. Background of Study

The large scale production of Portland cement from the dredging and quarrying has a dramatic impact on the environment. Consequently extensive research is on-going into the use of Portland cement replacements, using many waste materials and industrial by-product, for example pulverised fuel ash (PFA) and ground granulated blast-furnace slag (GGBS).

In addition to the use of binder replacement materials from waste, there is somewhat regard given to the subject of using waste material to replace the cement content of concrete.

Glass wool waste is considered to be a material which could be used as binder and also as cement replacement. In Malaysia, the existing glass wool factory is Syarikat PGF Insulation Sdn. Bhd.,Seberang Perai, Penang. Glass wool waste which is most considered for recycling in terms of environmental protection is that from the glass wool production cut-offs. This study outlines the use of such recycled glass wool waste as a binder and cement replacement and details workability and strength development of concrete containing glass as partial replacement of traditional materials.

1.2. Problem Statement

Air pollution from cement production

Produce cement means produce pollution, in the form of carbon dioxide emissions. Cement plants account for 5% to 8% of global emissions of carbon dioxide, the main cause of global warming. Cement has no viable recycling potential; each new road, each new building, needs new cement.

There are two very different sources of carbon dioxide emissions during cement production. First is the combustion of fossil fuels to operate the rotary kiln is the largest source, approximately 3⁄4 tons of CO_2 per ton of cement. And second is the chemical process of calcining limestone into lime in the cement kiln.

Besides CO_2, both cement and concrete production generate considerable quantities of air-pollutant emissions. Dust is usually the most visible of these pollutants. It is estimated that total particulate (dust) emissions of 360 pounds per ton of cement produced.

Air pollutants commonly emitted from cement manufacturing plants include sulphur dioxide (SO_2) and nitrous oxides (NO_X). SO_2 emissions (and to a lesser extent SO_3, sulphuric acid, and hydrogen sulphide) result from sulphur content of both the raw materials and the fuel (especially coal).

As concrete is the most prevalent building material on earth that requires cement as its binding agent, an innovative research on new materials to replace cements it to be conducted to halt all the alarming pollution figures. The pollution due to production of cement is becoming worse as the cement demand currently exceeding 2.6 billion tons per year worldwide and growing at 5% annually.

Land pollution from glass wool waste

Glass wool is made from abundantly available natural resources. Raw materials include substance like sand, limestone and basalt and none of these are scarce resources. Besides that, manufactured glass wool also contains recycled glass or waste glass so that the usage of recycled material in glass wool manufacturing can be maximized.

As glass wool is manufactured in the form of rolls or in slabs, the production offcuts will be the solid waste produced. The most common practice to handle these production offcuts is waste disposal as landfill which soon will contribute to the land pollution.

Recycling the glass wool waste in the concrete material to solve the disposal problem of the waste may be the most feasible application. The method proposed in this study is suitable from both the technological and economic point of views. Therefore this study primarily focuses on evaluating the physical properties of concrete containing glass wool waste and its composition. Chemical analysis and physical test will be conducted on the concrete that contain certain percentage of glass wool waste.

1.3. Objective of the Project

The objectives of study can be categorised as follows;

i. To characterise of glass mineral wool waste in term of physical and chemical composition
ii. To obtain the right mixture proportions of concrete containing glass mineral wool waste
iii. To gather information data in respect of physical properties and mechanical properties of concrete containing glass mineral wool waste.

1.4. Scope of Study

Major problem of glass mineral wool waste is to dump it at a landfill. Indeed the waste product still has economic value if it to be treated in proper manner such as recycling to other useful products. One of the ideas to recycle the glass waste is to use it as a partial replacement for cement in concrete mixture. This modified product has a great potential for construction material which can be used together with normal silica sand and granite stone. Besides solving the landfilling problems, air pollution caused by the cement production process can also be reduced as the usage of cement is reduced.

Scope of research would be covered as followed;

i. Study on properties of glass mineral wool waste. It covers physical properties and chemical composition as well as components involving in glass wool product;
ii. Study on properties of concrete containing glass wool waste. With regard to this study, physical properties such as compressive strength, permeability and effect of acid on the concrete will be investigated by conducting all required test to the samples.
iii. Study on mixture proportions of concrete containing glass wool waste. In respect of this study area, amount of glass wool waste will be calculated and the right percentage of waste as replacement of aggregate will be identified.

Using glass sand as partial replacement in concrete have been studied by many researchers but study on application of glass wool wastes in concrete is still new in construction material.

CHAPTER 2

LITERATURE REVIEW

2.1. Ordinary Portland Cement (OPC)

Cement and concrete are key components of both commercial and residential construction in the world. The cement and concrete industries are huge. From one hand, cement industries manufacturers provide the required material for development and construction and from the other hand; therefore, the environmental effects of cement industry have been the focus of a significant amount of studies around the world. And as such, some standards and criteria have been presented to control the pollutants made by cement industry. (Wilson, 1993)

It is estimated that there are around 1500 integrated cement production plants in the world. World cement demand was 2,283 million Tons in 2005, with China accounting for 1,064 MT (47% of total).The expected demand for 2010 is estimated at 2836 MT. China will increase its demand by 250 million tons during the period, an increase higher than the total yearly European demand. (Lasserre, 2007)

Demand of Cement in Million Tons	**2005**	**2010**	**Growth rate**
North America	170	200	2.9%
Western Europe	208	236	2.2%
Asia/Pacific	1500	1900	5.2%
Other Regions	405	500	4.7%
WORLD Cement Demand	2283	2836	4.7%

With cement factories rapid development, environmental pollution problems have drawn increasing attention. There are quite a number of protests done by the villagers whenever a cement factory is built because of the pollution it gives. Of course, environmental pollution is the most direct, most likely to be felt consequences of the decline in the quality of the human living environment, quality of human life, health and production activities.

2.2. Problem with OPC Production

2.2.1. Paris, France (2007)

Cement poses a basic problem: the chemical reaction that creates it releases large amounts of carbon dioxide. 60% of emissions caused by making cement are from this chemical process alone. The remainder is produced from the fuels used in production, although those emissions may be mitigated with the use of greener technology.

One industry project called the Cement Sustainability Initiative suggests that concrete should be mixed using smaller portions of cement to reduce emissions. But there is less incentive for manufacturers to make fundamental changes in how buildings and roads are made.

The demand is growing so fast and continues to grow, and no one can cap that. Cement is a core business and it limits the changes to be made. Western cement manufacturers emphasize that the emissions problem cannot be solved until China and India and other booming economies realize that they must limit emissions as well. (ROSENTHAL, 2007)

2.2.2. Tibet, China (2010)

The cement factory is known to locals as the 'Amdo Cement Factory' and there are around 600 employees. The factory causes many problems for local Tibetans because it pollutes the area, producing chemical residues which have badly affected grasslands and forests, as well as livestock. There have been incidents before between local people and factory works, with a serious clash in 2008.

The cement factory gave no consideration to controlling pollution in step with production. Having expanded production, the amount of dust being discharged was an unknown number of times greater than before reconstruction. At times when the more serious pollutants are being emitted, even opening one's eyes can be very difficult.

Today, when the entire world is advocating harmonious development and the country is fully implementing a scientific viewpoint of development, the defendants at the same time as expanding production ignored state environment protection laws by seriously contravening effluent and dust and pollutant outputs, creating serious harm to the villagers' normal lives. (Police open fire at Tibetans protesting cement factory pollution, 2010)

2.2.3. California, United States (2008)

Cement, which is mostly commonly composed of calcium silicates, requires heating limestone and other ingredients to 2,640 degrees F (1,450 degrees C) by burning fossil fuels and is the third largest source of greenhouse gas pollution in the U.S., according to the U.S. Environmental Protection Agency. Making one ton of cement results in the emission of roughly one ton of CO2—and in some cases much more.

The U.S. used more than 122 million metric tons of Portland cement in 2006, according to the Portland Cement Association (PCA), an industry group, and China used at least 800 million metric tons.

Carbon capture and storage has been identified by experts ranging from the U.N.'s Intergovernmental Panel on Climate Change to the leaders of the world's eight richest nations (G8) as crucial to the fight against climate change. The idea is to capture the CO_2 and other greenhouse gases produced when burning fossil fuels, such as coal or natural gas, and then permanently store it, such as in deep-sea basalt formations. (Biello, 2008)

2.3. Glass Wool Insulation Product

Glass wool is the world’s leading choice of insulation products, and is the most versatile insulation products in use today. There are dozens of different insulation materials on the market but only glass wool insulation products have the all-round environmental benefits combines with proven thermal, acoustic and fire properties to provide the best insulation value.

Insulation products are one of the few building materials which make a positive contribution to improving the environment. They save many times more energy than is used to produce them. In the case of glass wool, the embodied energy is recovered more than 100 times over the lifetime of the products.

Glass wool insulation is a product made from molten glass which is then processed by a spinning process into fibres a bit like cotton wool. It is made from abundantly available natural resources. Raw materials include substances like sand, limestone and basalt. None of these are scarce resources. Glass wool is manufactured in large scale plants that are

professionally managed and incorporate modern process and energy control systems and pollution control equipment.

Glass wool insulation makes a substantial contribution to a better environment through the energy saved as a result of the use of this product. The energy conserved translates to reductions on the amount of fossil fuel burned to generate that energy. Not only does this mean a reduction in harmful greenhouse gas emissions, but scarce non-renewable resource consumption is also reduced. In addition, increased insulation usage will result in reduced energy demand and improved peak demand load management, reducing the need for investment in expensive power generation infrastructure. (Environmental Benefits of Insulation, 2005)

Basically, the demand for glass wool has increased from time to time. Glass wool has its special properties that make its demand increases from time to time which are: (AG, 2011)

1) Substantially reduce thermal losses which can saves energy costs and protects the environment.
2) Inflammable insulation material.
3) Contribute effectively to improving sound insulation.
4) Water repellent and resistant to moisture.
5) Dimensionally stable and consist of long fibres which make it very elastic.
6) Open for diffusion.
7) Easy to process and suit for all installation types.
8) Produced in elastic up to pressure-resistance slabs.
9) Made of natural quartz sand which is ageing-resistant and do not rot.

During the production, glass wool will be cut into specified size depending on the market demand. The production cut-off is considered as the waste and the common practice to handle this waste is by disposing them into a landfill. At first, landfilling is not a major problem as the amount of the cut-offs is not that much. Besides, it is a lightweight material and can be compressed to reduce the waste. In addition, glass wool is non-hazardous and there are no adverse environmental impacts as a result of using this product as landfill.

However, as time pass by, the demand of glass wool insulation products increases as well as the production factory. Glass wool landfill is now become one of the land pollution to the environment. (Environmental Benefits of Insulation, 2005)

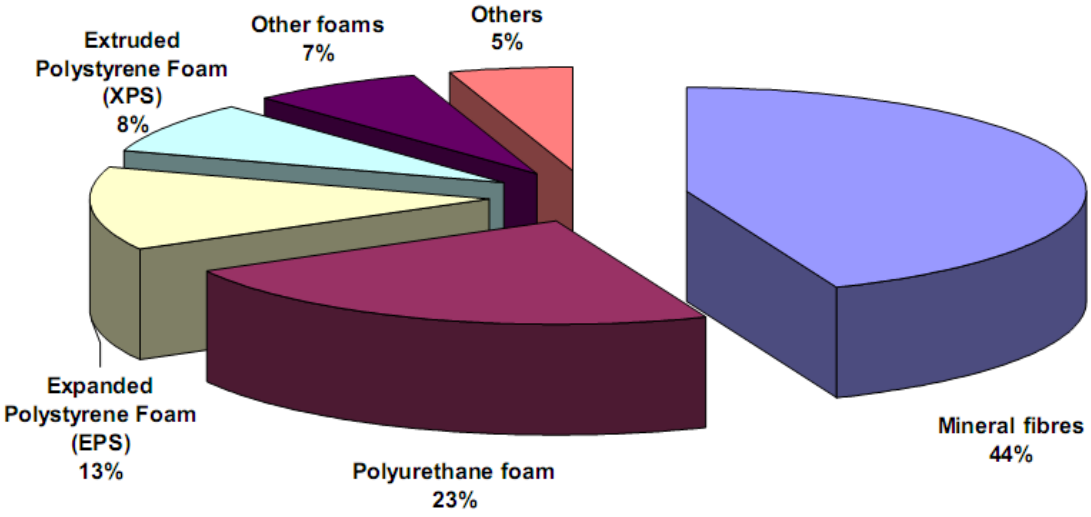


FIGURE (a): Insulation Market Value broken down by insulation type

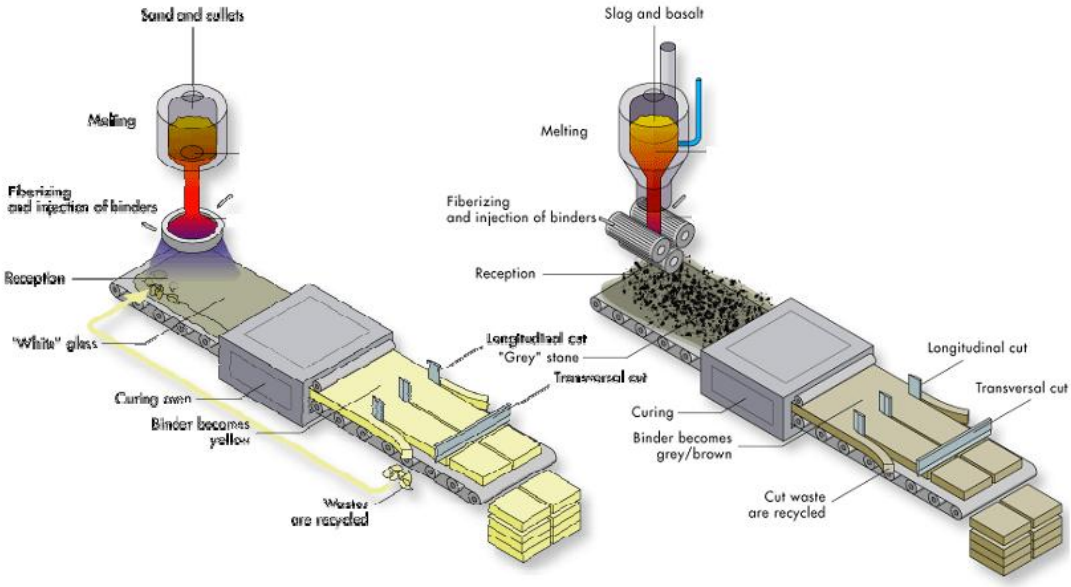


FIGURE (b): General plant configurations for glass wool production (left) and stone wool production (right)

2.4. Glass Wool Statistic from Syarikat PGF Insulation Sdn. Bhd.

FIGURE 1: Annual Glass Wool Production

FIGURE 2: Annual Glass Wool Waste Production

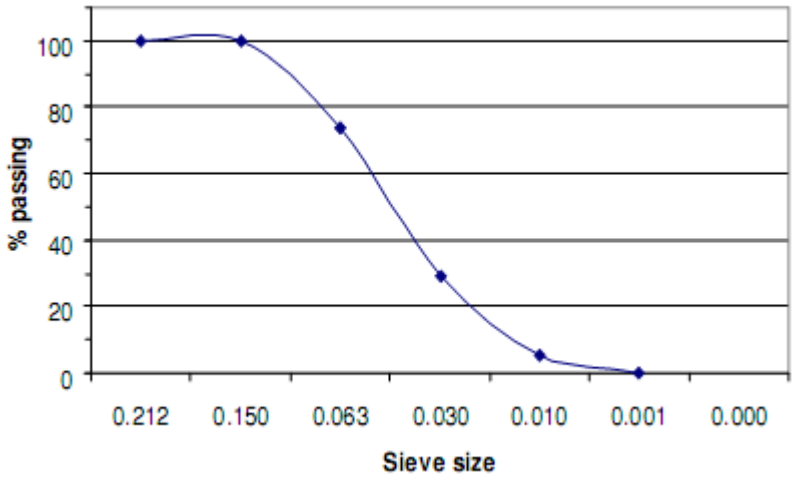
(W.F. Chin, PGF Insulation Sdn Bhd)

2.5. Development of Concrete Containing Glass Waste

2.5.1. G. D. Perkins

The glass being utilised in this research project is derived from post-consumer container glass, end of life (ELV) glass and architectural glass. The glass which is used in this project is, glass powder - a fine material which is collected in the air filtration which is collected in the air filtration system whilst producing the processed glass.

The glass powder is being used for partial binder replacement in proportions of 10%, 20% and 30% binder replacement. The glass powder has been analysed to establish chemical composition and size. The particle size of the glass powder was establish by using Malvern Instruments, Mastersizer 2000 unit.



Chemical composition and soluble oxide content of the material was determined. A range of concrete mixes was manufactured incorporating processed glass powder. Test mixes were undertaken to establish the effect of glass powder on the mix. Further to establishing a mix design an acceptable level of strength, additional mixes were produced using various quantities of the replacement material and comparisons made with the control mix.

The research concluded that, glass powder exhibits pozzolanic properties but is dependent upon fineness of the powder. (G. D. Perkins, 2008)

2.5.2. G. D Andina Sprince, Aleksandrs Korjakins, and Leonids Pakrastinsh

Glass waste requires recycling. Since there are different types of glass with different chemical compositions, there are also different possibilities of its use. Every year there is several hundred tons of waste glass produced in Latvia. Glass can be re-used as a fine raw material and it presents a possibility to save natural, non-renewable materials.

The use of glass powder in concrete production can make the construction industry more environmentally friendly. A study was conducted to examine the possibility of using glass powder as cement replacement in a new type of concrete The object of this study was lamp glass powder (LGP) obtained from fluorescent lamp waste.

Three different concrete mixtures were batched. Specimens of 20% and 40% cement replacement were compared with the specimens made of standard concrete. The samples were tested in two extreme conditions: in one case they were kept in 100% humidity ensured by preventing the desiccation of the concrete, and in the other case samples were air-dried by preventing them from becoming wet. Compression strength and modulus of elasticity of 7 and 28 days old cubic samples was determined.

Standard sample cubes 150x150x150 mm were produced in order to investigate the mechanical characteristics of the material. Concrete mixtures were cast into oiled steel moulds and compacted at the vibrating table. After two days the moulds were removed. Standard hardening conditions were provided.

After the hardening period, the samples were measured and tested in standard conditions. The samples were tested at the age of 7 and 28 days. Concrete samples containing glass powder as a micro filler displayed a reduction in compression strength when compared to the standard concrete samples. The concrete containing glass powder showed lower strength in the first 7 days, but on the 28th day the strength increased and was very similar to that of standard concrete. Fine glass powder caused a long-term hardening effect. Specimens with 40% cement replacement showed a 70.6% increase of compression strength, while the specimens with 20% cement replacement showed a 43.4% increase of compression strength. Standard specimens, however, showed only a 15.3% increase of compression strength. (G. D Andina Sprince, 2011)

2.5.3. G. S. Laldji, A. Tagnit-Hamou and G. Fares

In the last few years, mineral admixtures, either as addition to or replacement of Portland cement, are largely used throughout the world. Due to better performances of the fresh and hardened concrete properties, silica fume (SF), fly ash (FA) and ground granulated blast-furnace slag are normally incorporated in the production of concrete. Other alternative cementations materials can be used, as long as they meet the specifications requirements. While most of the known mineral admixtures are recycled by-products; thereby helping certain industries in solving their environmental problems, many other industries, including concrete and cement plants.

The characterization of Glass frit includes the main physico-chemical tests. Specific gravity and fineness were measured using a pycnometer and Blaine apparatus, respectively. The chemical analysis of Glass frit powder was conducted using chromatography and the atomic absorption test (AA), whereas the mineral phase composition was studied by X-ray diffraction (XRD).

Three mortar batches were prepared to conduct the test. Control mortar with a water to cement ratio of 0.485 and mortar having the same flow as the control and containing 20% of Glass frit as cement replacement were prepared, whereas the third mix was similar to the second one but incorporating ground granulated blast-furnace slag instead of Glass frit. Compressive strengths were measured on 50 x 50 x 50 mm cubes at different ages.

The strengths measured on mortar cubes and the resulting activity index are given as below:

Mortar	W/B	Compressive Strength						
		1 day	7 days	I_p 7 days	28 days	I_p 28 days	100 days	I_p 100 days
Control	0.485	16.19	34.83	--	46.79	--	52.13	--
20% glass frit	0.46	13.52	32.03	92%	46.32	99%	55.80	107%
20% slag	0.47	11.22	29.2	84%	42.97	92%	48.80	94%

* I_p is the strength activity index equal to the ration of the f'c of mortar incorporating in mineral admixture and f'c of the control mortar.

(G. S. Laldji, 2007)

2.6. Chemical Properties Analysis

2.6.1. Chemical Content by X-Ray Fluorescence (XRF) Test

X-ray fluorescence (XRF) is the emission of characteristic "secondary" (or fluorescent) X-rays from a material that has been excited by bombarding with high-energy X-rays or gamma rays. The phenomenon is widely used for elemental analysis and chemical analysis, particularly in the investigation of metals, glass, ceramics and building materials, and for research in geochemistry, forensic science and archaeology. (Erhan Guneyyisi, 2007)

Mortars contain rounded nodules of lime (CaO), which may indicate that the lime was slacked with minimum amount of water in order to convert lime into calcium hydroxide Ca(OH)2. The presence lime carbonate (CaCO3) in these mortars however, indicates that the decarbonation of calcareous rocks during the production of lime was incomplete, possibly due to the use of low temperature, or insufficient time of heating.

The result shows that the principle differences between green and clear glass exist in the Fe2O3 and Cr2O3 contents. These oxides, which are higher in the green glass than in the clear one, are responsible of the characteristic colour of the green glass. (A. Khmiri, February 2012)

Compared to silica fume, the waste glass has higher CaO, Na2O and Al2O3, but much lower SiO2 content. In agreement with ASTM C618-02 [20], which requires a sum of SiO2+Al2O3+Fe2O3 higher than 70% for good pozzolan, the sum for the investigated samples for the two glasses is 73%. The studied glass samples present satisfactory chemical composition. They can be classified as Class N natural pozzolan and therefore, they are likely to produce good pozzolan. (A. Khmiri, February 2012)

Table 1: Chemical composition of raw materials

%	Green glass	Clear glass	Silica fume	Cem I 42.5 N
SiO_2	71.44	71.48	93.17	20.59
Al_2O_3	1.70	1.60	0.32	6.62
Fe_2O_3	0.37	0.07	1.02	3.54
CaO	10.81	11.45	0.58	63.61
MgO	1.65	1.23	0.48	1.39
Na_2O	13.24	13.36	0.3	0.13
K_2O	0.36	0.48	1.17	0.61
SO_3	0.16	0.27	0.12	1.94
TiO_2	0.04	0.03	-	-
Cr_2O_3	0.19	0.01	-	-

2.6.2. Chemical Composition by X-Ray Diffraction (XRD) Test

The prepared sample material was mounted onto the XRD apparatus (Geigerflex Horizontal diffractometer with a graphite crystal monochrometer; Rigaku/MSC, Woodlands, TX). The x-ray beam angle 2 range was set between 3 degrees (3000) to 70 degrees (70000) and scanned at 2 degrees per minute. The Cu x-ray source was set at accelerating voltage of 45 KV and the current in the electron beam at 30 mA and on continuous scan mode. The peaks on the diffraction pattern were marked using the Rigaku software (version 2.8). Then the peaks were compared and matched with that of the standard material in the powder diffraction file (JCPDS International Center for Diffraction Data 1998, Pennsylvania) using a micro powder diffraction search and matching analysis program. (Jin-Seon Song, December 2006)

The figure shows the chemical composition and crystal structure of Portland cement. Portland cement did not contain bismuth oxide but it was composed of many different crystal phases. (Jin-Seon Song, December 2006)

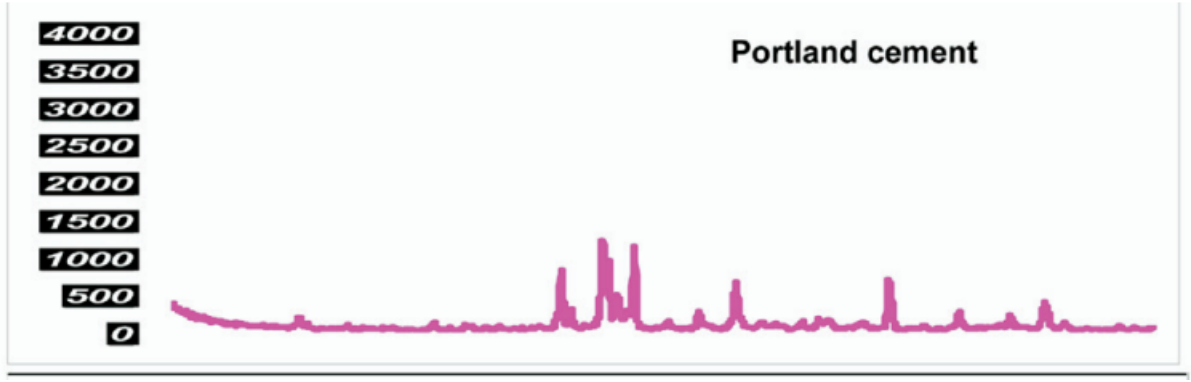


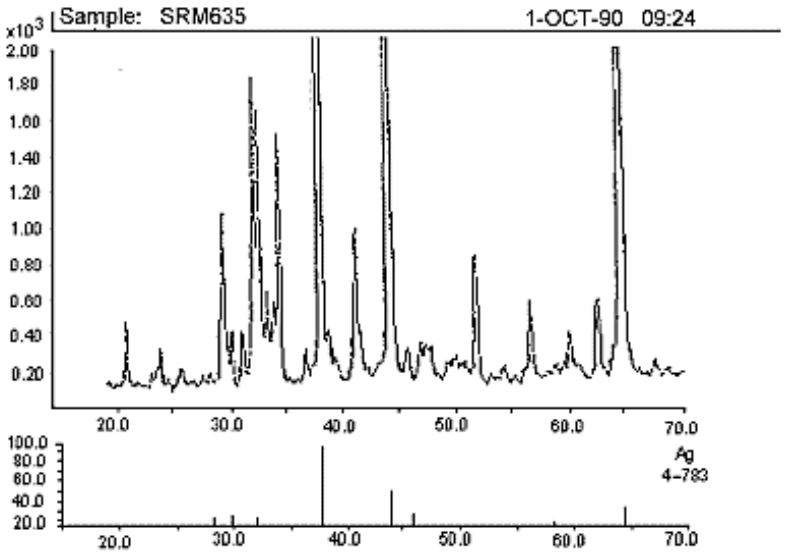
Fig: X-ray diffraction patterns of Portland cement, representing the crystal phases present in the material. (Jin-Seon Song, December 2006)

The XRD peak intensities should be deconvoluted and compare the diffraction patterns of standards with air samples having similar weights. X-ray diffraction peak intensities can be very sensitive to preferred orientations and particle-size distributions, and intensities may not always be comparable between samples and standards. (Rose, 1991)

Other diffraction peaks may be present in the sample that may present positive interferences:

Interferent	Peaks (2θ)
Ag	28.0, 32.2°
Gypsum ($CaSO_4 \cdot 2H_2O$)	29.2, 31.2, 33.5, 34.7, 50.5, 51.3°
Anhydrite ($CaSO_4$)	28.0, 31.5, 32.2, 52.5°

Typical PC X-ray Diffracton Patterns



CHAPTER 3

METHODOLOGY

3.1. Key Milestone for FYP 2

No	Details\Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Burning of Glass Wool															
2	Chemical Properties Analysis															
3	Grinding of Burned Glass Wool															
4	Mortar Cube Preparation															
5	Mechanical Properties Analysis															
6	Submission of Progress Report															
7	Pre-SEDEX presentation															
8	Submission of Draft Report															
9	Submission of Dissertation & Technical Paper															
10	Oral Presentation															
11	Submission of Hardbound Dissertation															

TABLE 1 : Key Milestone for FYP 2

3.2. Materials

Ordinary Portland cement (OPC) equivalent to ASTM type-1, silica sand and granite stone will be used in the experimental work. Glass mineral wool waste will be obtained from Syarikat PGF Insulation Company Sdn. Bhd., Perai, Penang.

Glass wool waste will be burned under four different burning temperatures which are 500˚C, 600˚C, 700˚C, 800˚C and 900 ˚C. There will be two method of burning process by using microwave and melting furnace. Observation and chemical analysis will be conducted to identify the optimum burning temperature and the best method of burning in order to get the best cement replacement material.

After the burning process, the burnt glass wool waste will be grinded into the rage of 75µm - 100µm fineness. As glass wool waste material is in powder form after the grinding process, it will be used as cement replacement material and mortar cube will be casted.

3.3. Mortar Mix Design

The experimental work will cover by preparation a total of 80 samples using different percentage of glass wool waste and control sample with fixed water-cementations material ratio 0.5.

The mixtures are designated as 5%, 10%, 15% and 20% of glass wool waste as cement replacement material. GW5, GW10, GW15 and GW20 represents 5%, 10%, 15% and 20% of cement replacement respectively. The control sample is a mixture without adding any glass wool waste and will be marked as GW0.

A drum cement mixer will be used in the batching process. All component materials will be fed into the mixer manually. Ordinary tap water is used all the mixes.

- Characteristic strength (specified): 20 N/mm^2 at 28 days
- Proportion defective: 5%
- Standard deviation (for 20 N/mm^2 strength): 8 N/mm^2
- Margin: (k=1.64) x 8 = 13.12 N/mm^2
- Target mean strength: 20 N/mm^2 + 13.12 N/mm^2 = 33.12 N/mm^2
- Cement type (specified): Ordinary Portland Cement (OPC)
- Aggregate type: Fine
- Free water/cement ratio: 0.5
- Free water content: 190 kg/m^3
- Cement content: 190/0.5 = 380 kg/m^3
- Aggregate/cement ratio: 2
- Aggregate content: 380 kg/m^3 x 2 = 760 kg/m^3

In conclusion, the mixture will be;

i. Cement: 380 kg/m^3
ii. Water: 190 kg/m^3
iii. Fine aggregate: 760 kg/m^3

Sample	Water (kg/m^3)	Cement (kg/m^3)	Glass wool (kg/m^3)	Sand (kg/m^3)	Remarks
GW0	190	380	0	760	0% of glass wool
GW5	190	361	19	760	5% of glass wool
GW10	190	342	38	760	10% of glass wool
GW15	190	323	57	760	15% of glass wool
GW20	190	304	76	760	20% of glass wool

TABLE 2: Mixture Proportions

Quantities per cube

Volume of 1 cube: 0.05 m x 0.05 m x 0.05 m = 1.25 x 10^{-4} m^3

i. Cement: 380 kg/m^3 x 1.25 x 10^{-4} m^3 = 0.0475 kg
ii. Water: 190 kg/m^3 x 1.25 x 10^{-4} m^3 = 0.02375 kg
iii. Fine aggregate: 760 kg/m^3 x 1.25 x 10^{-4} m^3 = 0.095 kg

As mortar will shrink at the end, so additional of 60% of each material will be added up.

i. Cement: 0.0475 kg + 60% = **0.076 kg**
ii. Water: 0.02375 kg + 60% = **0.038 kg**
iii. Fine aggregate: 0.095 kg + 60% = **0.152 kg**

3.4 Chemical Properties Analysis

3.4.1 X-ray fluorescence (XRF) Test

X-ray fluorescence (XRF) is the emission of fluorescent X-rays from a material that has been excited by attacking with high-energy X-rays or gamma rays. The test analysis is widely used for elemental analysis and chemical analysis, particularly in the study of metals, glass, ceramics and building materials, and for research in geochemistry, forensic science and archaeology.

FIGURE 3 : X-Ray Fluorescence Spectrometer

X-ray fluorescence (XRF) spectrometer is an instrument used for routine, relatively non-destructive chemical analyses of rocks, minerals, sediments and fluids. It works on wavelength-dispersive spectroscopic principles.

X-Ray fluorescence is used in a wide range of applications, including;

- research in igneous, sedimentary, and metamorphic petrology
- soil surveys
- mining (e.g., measuring the grade of ore)
- cement production
- ceramic and glass manufacturing
- metallurgy (e.g., quality control)
- environmental studies (e.g., analyses of particulate matter on air filters)
- petroleum industry (e.g., sulphur content of crude oils and petroleum products)
- field analysis in geological and environmental studies (using portable, hand-held XRF spectrometers)

It is typically used for bulk analyses of larger fractions of materials. This method or test is one of the most widely used methods for analysis of major and trace elements in rocks, minerals, and sediment since the relative ease and low cost of sample preparation, and the stability and ease of use of x-ray spectrometers make this

The analysis of major and trace elements in materials by x-ray fluorescence is made possible by the behaviour of atoms when they interact with radiation. When materials are excited with high-energy, short wavelength radiation (e.g., X-rays), they can become ionized. If the energy of the radiation is sufficient to dislodge a tightly-held inner electron, the atom becomes unstable and an outer electron replaces the missing inner electron. When this happens, energy is released due to the decreased binding energy of the inner electron orbital compared with an outer one.

The emitted radiation is of lower energy than the primary incident X-rays and is termed fluorescent radiation. Because the energy of the emitted photon is characteristic of a transition between specific electron orbitals in a particular element, the resulting fluorescent X-rays can be used to detect the abundances of elements that are present in the sample.

When conducting the test, calibration check is the first step to check the energy calibration to ensure that peak energies are accurately tied to specific elements. This procedure should be run every day before any analysis is performed. The energy adjustment involves measuring the Cu K" line (8041), and then determining the difference between the measured peak energy value and the ideal value. If any adjustments are required, the instrument software will perform it automatically.

Filter samples are then loaded into the sample cups after being removed from cold storage. Sample information should be recorded in the instrument logbook before the filters (in their sample cups) are loaded into the XRF sample tray in the same order as they are written into the instrument logbook. The instrument is then prepared for analysis by entering each filter aliquot number into the Method Tray List within the Win Trace software. The filter deposit analysis is then initiated.

This analysis protocol consists of each filter being analysed five separate times using five different excitation conditions. The specific excitation conditions have been optimized for specific groups of elements. The different excitation conditions are used to maximize the sensitivity of the measurement of the different groups of elements, which fluoresce over a wide range of excitation energies.

3.4.2 X-Ray Diffraction (XRD) Test

In definition, X-ray powder diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analysed material is finely ground, homogenized, and average bulk composition is determined.

X-ray powder diffraction (XRD) is one of the most powerful techniques for qualitative and quantitative analysis of crystalline compounds. The technique provides information that cannot be obtained any other way. The information obtained includes types and nature of crystalline phases present, structural make-up of phases, degree of crystallinity, amount of amorphous content, microstrain & size and orientation of crystallites.

FIGURE 4: X-Ray Diffraction Instrument (XRD)

The sample is irradiated with a beam of monochromatic x-rays over a variable incident angle range. Interaction with atoms in the sample results in diffracted x-rays when the Bragg equation is satisfied ($n\lambda=2d \sin \theta$). Resulting spectra are characteristic of chemical composition and phase. The technique uniquely provides phase identification (e.g. graphite or diamond), along with phase quantification, % crystallinity, crystallite size and unit cell size.

All diffraction methods are based on generation of X-rays in an X-ray tube. These X-rays are directed at the sample, and the diffracted rays are collected. A key component of all diffraction is the angle between the incident and diffracted rays. Powder and single crystal diffraction vary in instrumentation beyond this.

As for the test procedure, firstly, a few tenths of a gram of the material (as pure as possible) should be obtained. The sample must be in fine powder form, or else it should be grinded first. This is to minimize inducing extra strain (surface energy) that can offset peak positions, and to randomize orientation. Powder less than ~10 μm (or 200-mesh) in size is preferred.

The sample is then placed into a sample holder or onto the sample surface:

- smear uniformly onto a glass slide, assuring a flat upper surface
- pack into a sample container
- sprinkle on double sticky tape

Typically the substrate is amorphous to avoid interference. Care must be taken to create a flat upper surface and to achieve a random distribution of lattice orientations unless creating an oriented smear.

3.4.3 Nanoscale Imaging by Field Emission Scanning Electron Microscope (FESEM) Test

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties.

Areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using conventional SEM techniques. The SEM is also capable of performing analyses of selected point locations on the sample; this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions, crystalline structure, and crystal orientations.

FIGURE 5 : Field Emission Scanning Electron Microscope (FE-SEM)

While as for FESEM, it produces clearer, less electrostatically distorted images with spatial resolution down to 1 1/2 nm. That's 3 to 6 times better than conventional SEM. Smaller-area contamination spots can be examined at electron accelerating voltages compatible with Energy Dispersive X-ray Spectroscopy. It also reduces penetration of low kinetic energy electrons probes closer to the immediate material surface. High quality, low voltage images are obtained with negligible electrical charging of samples. (Accelerating voltages range from 0.5 to 30 kV.) And the need for placing conducting coatings on insulating materials is virtually eliminated.

3.5 Mechanical Properties Analysis

3.5.1 Compressive Strength

By definition, the compressive strength of a material is that value of uniaxial compressive stress reached when the material fails completely. The compressive strength is usually obtained experimentally by means of a compressive test. The apparatus used for this experiment is Compression Testing Machine (complies with the requirement of BS 1610).

FIGURE 6 : Compression Testing Machine

Compressive strength test is a mechanical test measuring the maximum amount of compressive load a material can bear before fracturing. The test piece, usually in the form of a cube, prism, or cylinder, is compressed between the platens of a compression-testing machine by a gradually applied load.

The test determines the capacity of a material or structure to withstand axially directed pushing forces. It provides data of force vs deformation for the conditions of the test method. When the limit of compressive strength is reached, brittle materials are crushed. Concrete can be made to have high compressive strength, e.g. many concrete structures have compressive strengths in excess of 50 MPa, whereas a material such as soft sandstone may have a compressive strength as low as 5 or 10 MPa.

The specimen that has been immersed in the curing tank for specified days are taken out. The surface of the specimen should be wiped out and grit off. And then it will be weighted before being tested. The platens of the testing machines must be ensured to be cleaned, or else it will affect the result of the test.

Center the specimen carefully on the lower platen and ensure that the lad will be applied to two opposite cast faces of the cube. Then the load is continuously applied and increade at nominal rate within the range 0.2 N/mm2 to 0.4 N/mm2until no greater load can be sustained. The machined will automatically prove the result to be recorded.

3.5.2 Chloride Penetration

Durability of reinforced concrete is largely controlled by the capability of the concrete cover to protect the steel reinforcement from corrosion. Chemical protection is provided by concrete's high alkalinity, and physical protection is afforded by the concrete acting as a barrier to the access of aggressive species. However, despite these inherent protective qualities, the corrosion of steel reinforcement has become the most common cause of failure in concrete structures.

It is the chloride ion which destroys the protective (passive) environment for the steel reinforcement and results in the corrosion of the steel reinforcement and eventual concrete distress. Moreover, carbonation in concrete normally involves a chemical reaction between carbon dioxide and the products of cement hydration. This reaction results in a significant reduction in the pH of the pore solution due to the removal of the hydroxyl ions, which may lead to steel depassivation and subsequent reinforcement corrosion. As a result, carbonation can be considered as a second cause of damaging the passivation layer over the reinforcement.

It is generally agreed that the ingress of chloride ions into concrete leads, in many structures, to long-term deterioration. In other words, chloride permeability of the concrete is one such intrinsic property of the concrete that needs to be assessed independently, especially in the design and construction of structures to be built in a salt-laden environment. The chloride-induced corrosion of reinforcing steel manifest through cracking, spalling, and delamination of the concrete cover, which eventually leads to the direct exposure of the reinforcing steel to aggressive environment.

The prismatic test specimens after being subjected to 28 days of initial curing procedure were soaked continuously in 4% NaCl solution for 28 days. During the 28-day soaking period, the test specimens were periodically withdrawn from the soaking tank at 7, 14, and finally 28 days to determine the depth of chloride penetration into concrete. For this purpose, the prisms were first split and the freshly split surfaces were soon sprayed with 0.1 N silver nitrate (AgNO3) solution.

The AgNO3 solution preferentially reacts with the free chloride present in the harden matrix to form a white precipitate of silver chloride (AgCl); whereas at greater depths, where free chlorides are absent, AgNO3 reacts with the hydroxides to form a brown precipitate of silver oxide (AgO). Thus, the depth of chloride penetration is clearly indicated as the boundary of colour change.

The white colouring (formation of silver chloride) occurs wherever the concentration of free chloride ion is greater than 0.15% by weight of cement. Measuring the depth of colour change was performed from the four sides of the split section.

CHAPTER 4

RESULT AND DISCUSSION

4.1. Chemical Properties Analysis

4.1.1. Chemical Content by X-ray fluorescence (XRF) Test

Oxides	Sample						
	OPC	GW original	GW burn @ 500^0C	GW burn @ 600^0C	GW burn @ 700^0C	GW burn @ 800^0C	GW burn @ 900^0C
SiO_2	21.343	67.143	65.695	55.53	55.35	62.11	66.36
Al_2O_3	3.745	3.581	3.375	4.37	3.99	3.28	2.78
Fe_2O_3	5.29	0.25	0.211	0.4	0.34	0.29	0.19
CaO	61.919	9.054	10.190	10.79	11.45	10.27	8.72
Na_2O	-	19.721	20.424	18.37	18.26	16.93	16.37
MgO	3.788	-	-	7.63	6.33	5.91	4.92
K_2O	0.165	-	-	0.61	0.58	0.53	0.39
ZrO_2	-	-	-	0.08	0.06	0.06	0.04
SO_3	2.635	-	-	-	0.31	0.42	0.22
TiO_2	0.263	0.094	0.104	-	0.19	0.20	-
BaO	-	0.089	-	-	-	-	-
PbO	-	0.045	-	-	-	-	-
ZnO	-	0.021	-	-	-	-	-
RuO_2	0.451	-	-	-	-	-	-

TABLE 3 : Chemical Content by XRF Test

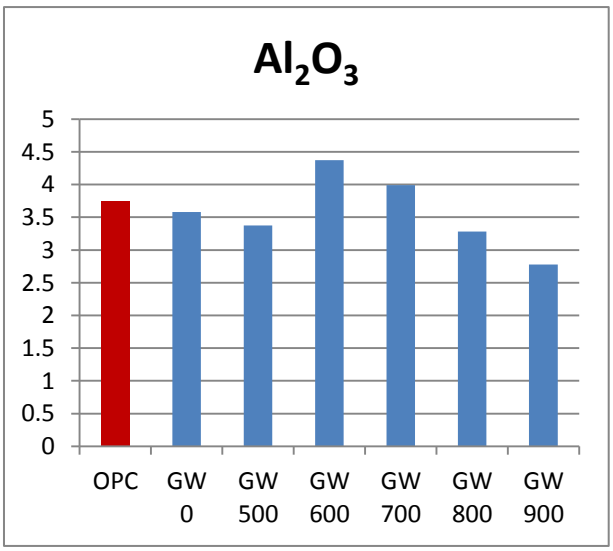


FIGURE 7 : Comparison of Al_2O_3 content

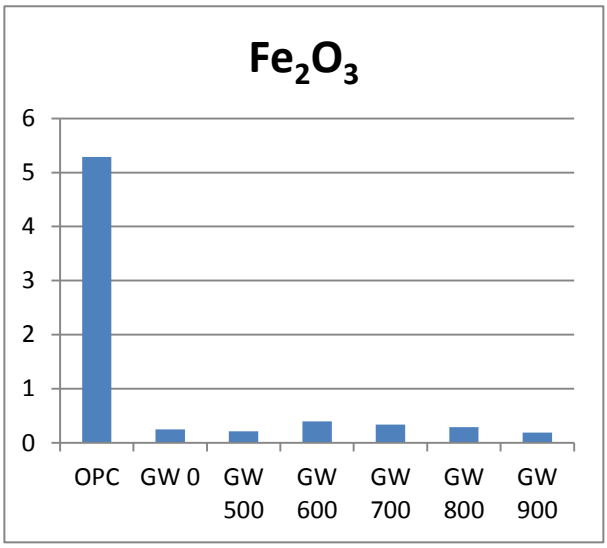


FIGURE 8 : Comparison of Fe_2O_3 content

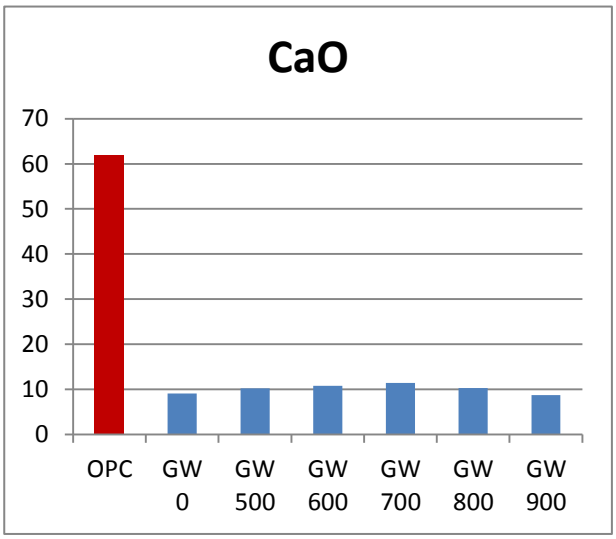


FIGURE 9 : Comparison of CaO content

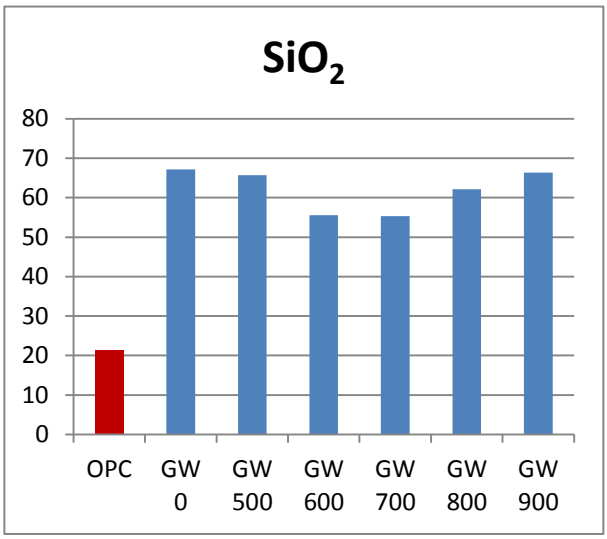


FIGURE 10 : Comparison of SiO_2 content

Based on ASTM C618-01, it requires a sum of SiO2+Al2O3+Fe2O3 higher than 70% for good Pozzolan. According to the result recorded, the sums of all these four (4) compounds are as below;

1) Glass Wool burned at 0 0C ; 80%
2) Glass Wool burned at 500 0C; 79%
3) Glass Wool burned at 600 0C; 71%
4) Glass Wool burned at 700 0C; 71%
5) Glass Wool burned at 800 0C; 76%
6) Glass Wool burned at 900 0C; 78%

Since the XRF results for glass wool burned at these various temperature give the value of more than 70% of Al2O3, Fe2O3, CaO and SiO2 contents, it means they have pass this test to be considered of having pozzolonic properties.

The chemical composition, however, should not be used as the only criterion for prediction of the pozzolanic activity. The amorphous state is also required. The test to determine either the material is in amorphous state or crystalline state will be discussed after this.

4.1.2. Chemical Composition by X-Ray Diffraction (XRD) Test

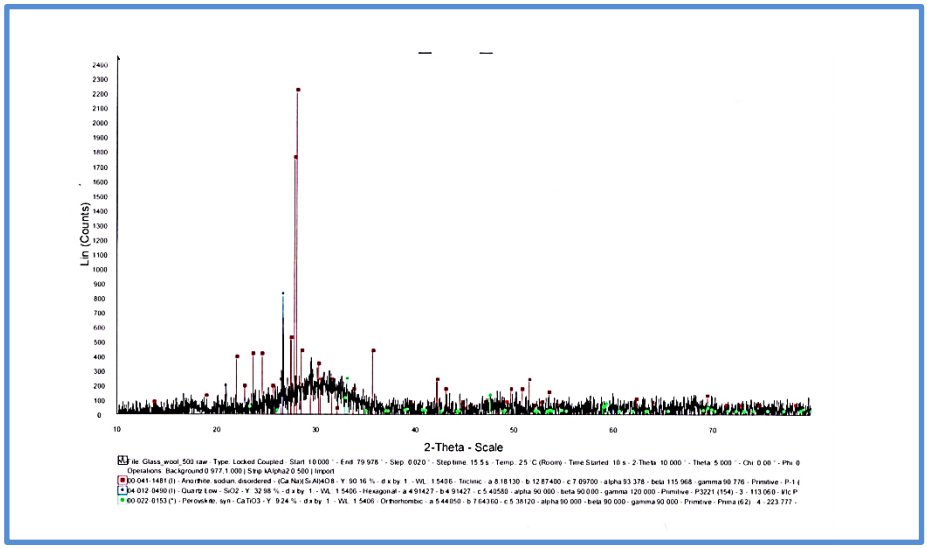


FIGURE 11 : XRD test result for glass wool burned at 600 ^{0}C

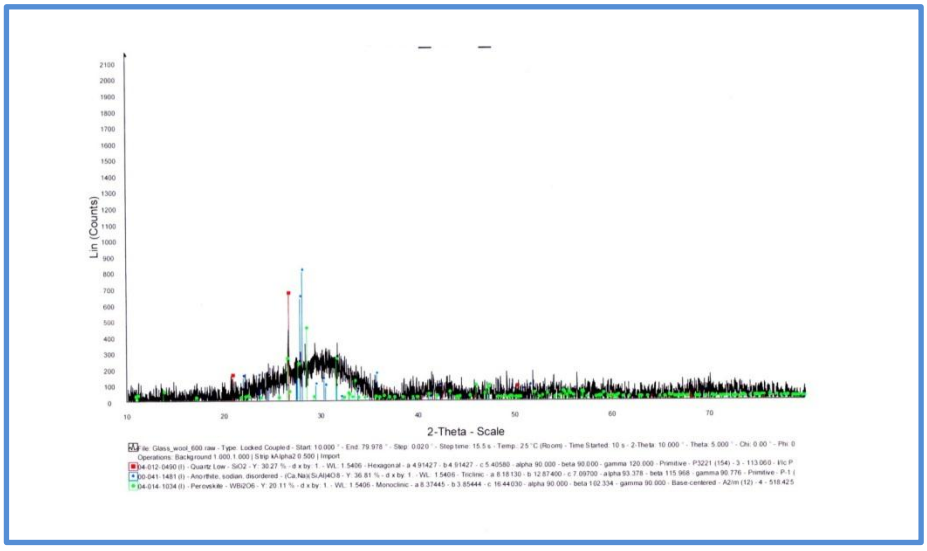


FIGURE 12 : XRD test result for glass wool burned at 700 ^{0}C

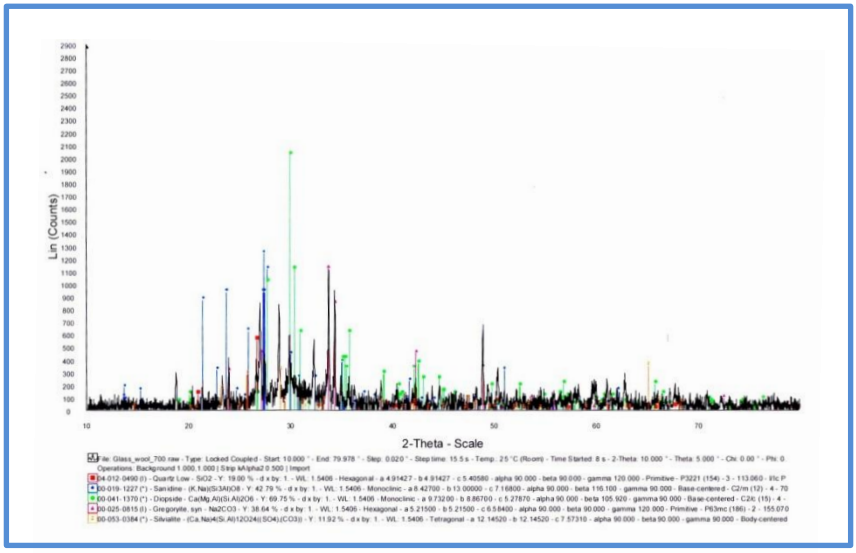


FIGURE 13 : XRD test result for glass wool burned at 800 ^{0}C

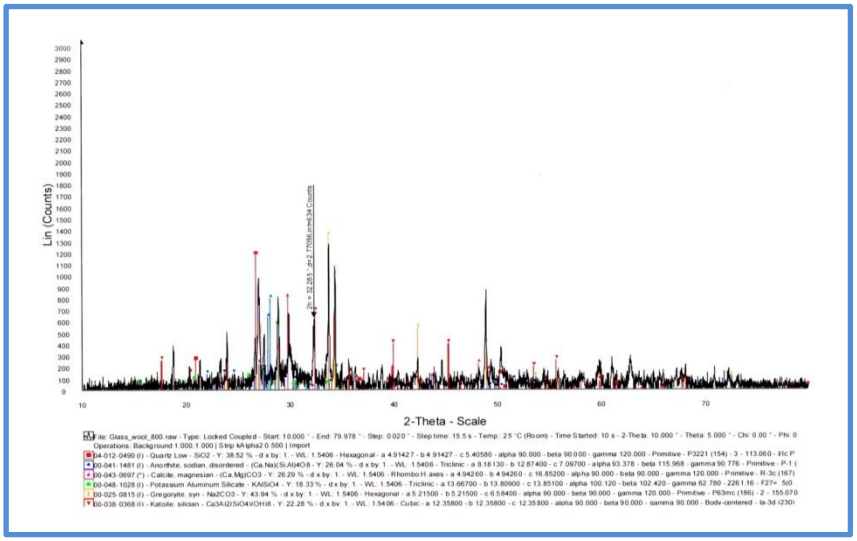


FIGURE 14 : XRD test result for glass wool burned at 900 ^{0}C

The results showed that glass wool burned at 600 ^{0}C and 700 ^{0}C is at amorphous state while the same material burned at 800 0C and 900 ^{0}C is in crystalline state. As what we can see from the graph, the phase started to change at 800 0C burning temperature. Graph for material burned at 600 ^{0}C and 700 ^{0}C have less peak compared to 800 0C and 900 ^{0}C. The first and second graphs also show amorphous pattern graph line.

Therefore, material burned at 800 ^{0}C and 900 ^{0}C are eliminated from the choices because we are looking for material with amorphous state. In definition, amorphous means to have no defined shape, or an easily altered shape, like liquid or a rubber. Crystalline, on the other hand, insinuates that there is a regular pattern to the molecular aggregates.

The amorphous is analogous to a plateful of spaghetti; loose and randomly coiled. While the crystalline state is more like the uncooked spaghetti in the box; the chains are all tightly bundled and ordered in the same direction. Hence, material in amorphous state will bind with the aggregates and cement better, compared to the one with crystalline state.

Since we are looking for a good binder to replace cement content, the choices left are glass wool burned at 600 ^{0}C and 700 ^{0}C. However, after burning the glass wool under 600 ^{0}C of temperature, the material is still in fibre form. While, under 700 ^{0}C burning temperature, the glass wool which is originally in fibre form changes into solid stone-like form.

As we need to do the grinding process after this burning process, material with solid stone-like form will be a better choice. Hence, it left us with the only one choice which is glass wool waste burned at 700 ^{0}C.

4.1.3. Nanoscale Imaging by Field Emission Scanning Electron Microscope (FESEM) Test

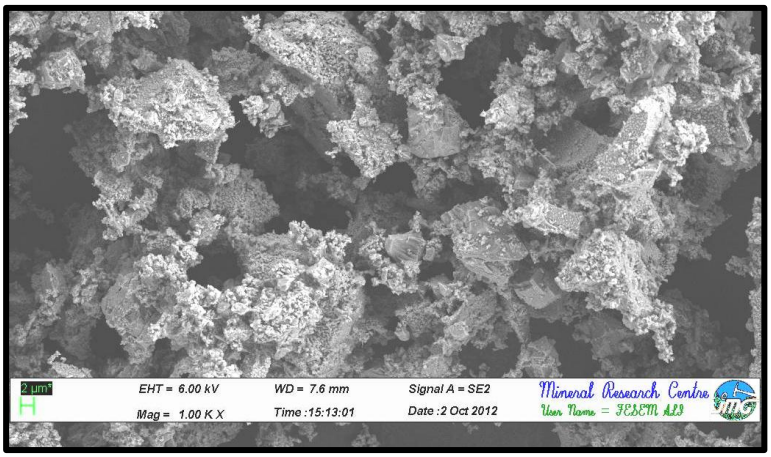


FIGURE 15 : Microstructure of OPC

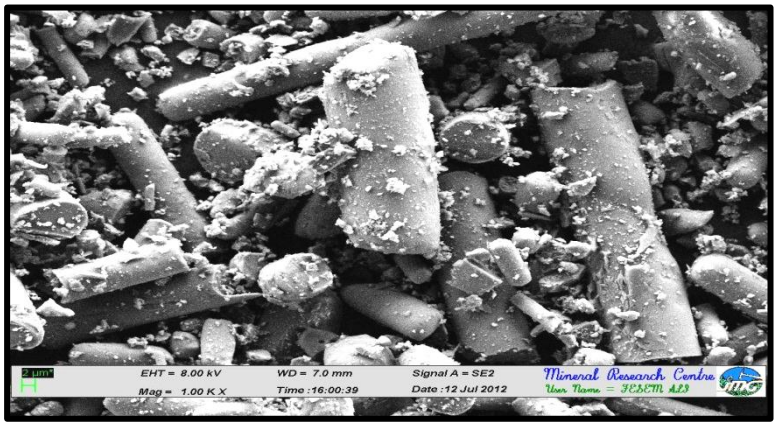


FIGURE 16 : Microstructure of Glass Wool burned under 600 ^{0}C

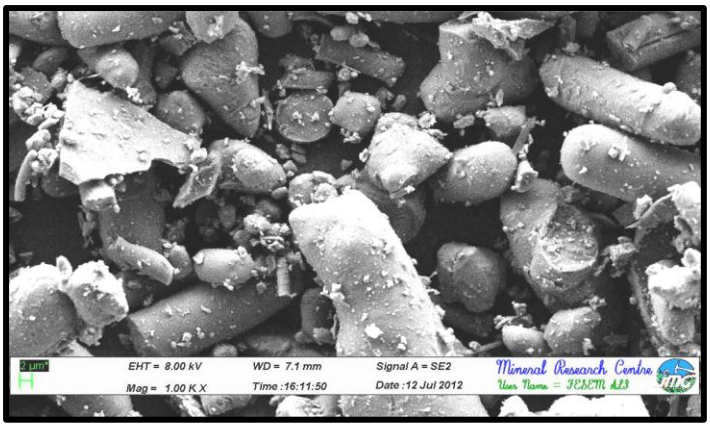


FIGURE 17 : Microstructure of Glass Wool burned under 700 ^{0}C

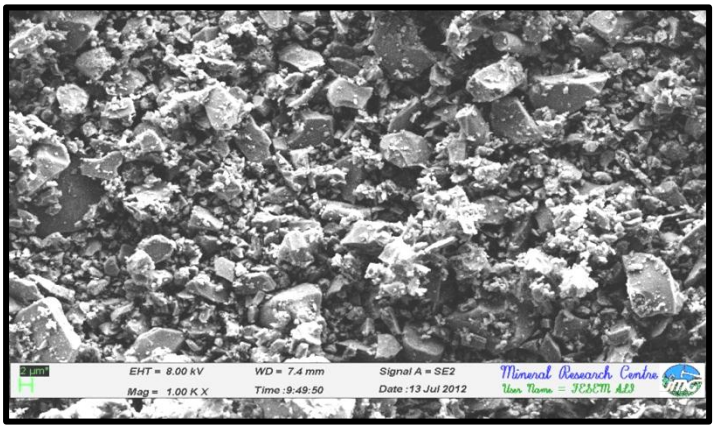


FIGURE 18 : Microstructure of Glass Wool burned under 800 ^{0}C

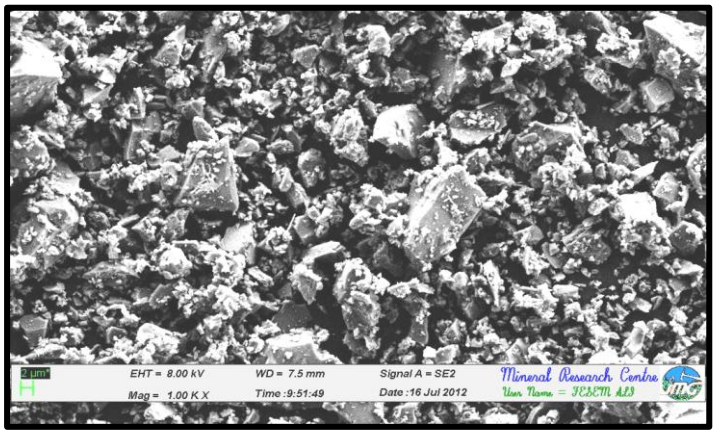


FIGURE 19 : Microstructure of Glass Wool burned under 900 ^{0}C

As shown in the figure above, the micro structure of glass wool burned under 700 ^{0}C still have its fibre form even though visually, the material has changed from fibre into solid stone-like form. While for temperature 800 ^{0}C and 900 ^{0}C, the microstructure image shows that all the fibres are already been destroyed and this explain the crystalline state earlier.

4.2. Mechanical Properties Analysis

4.2.1. Compressive Strength

Days	Stress (MPa)				
	GW0	GW5	GW10	GW15	GW20
3	27.26	24.07	21.47	26.15	21.68
	24.89	26.6	22.99	27.89	20.15
	26.6	25.08	23.76	25.53	21.52
average	26.25	25.25	22.74	26.52	21.12
7	28.79	29.08	25.13	32.97	25.93
	32.12	29.26	28.79	37.85	26.01
	34.86	35.34	29.18	29.9	28.58
average	31.92	31.23	27.7	33.57	26.84
28	54.35	36.01	44.52	43.13	40.14
	49.01	38.32	41.81	31.59	41.43
	46.64	40.34	42.17	41.81	34.73
average	50	38.22	42.83	38.84	38.86

TABLE 4 : Stress (MPa) for every mixture

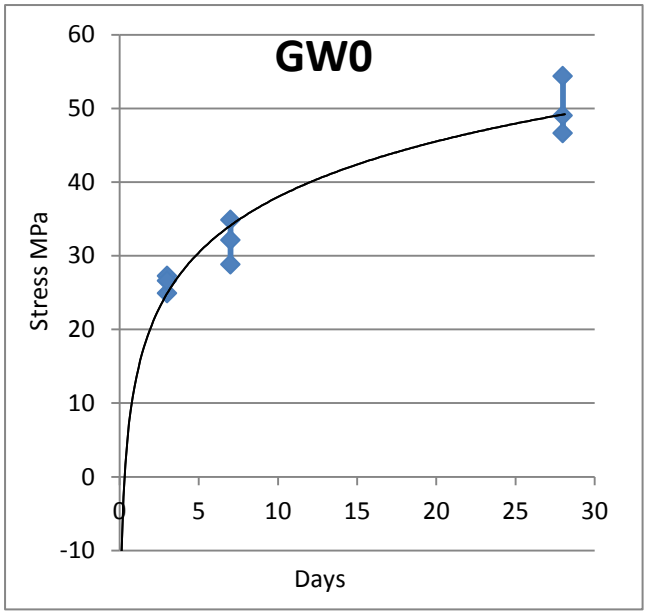


FIGURE 20 : Stress (MPa) for 0% cement replacement

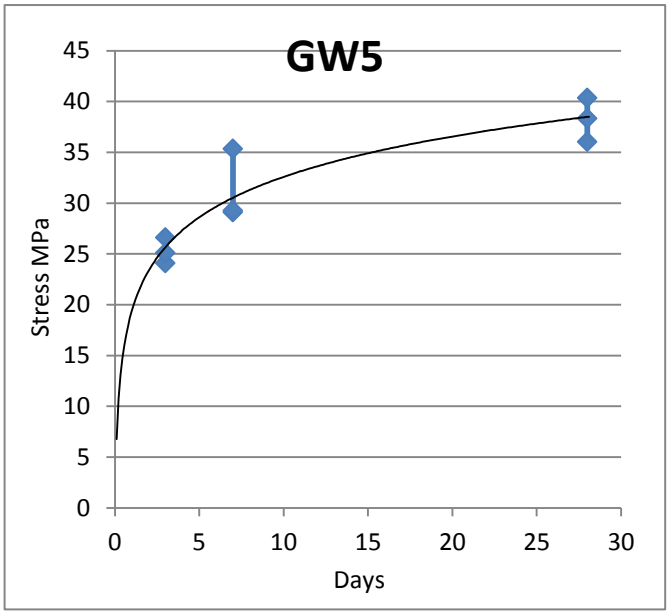


FIGURE 21 : Stress (MPa) for 5% cement replacement

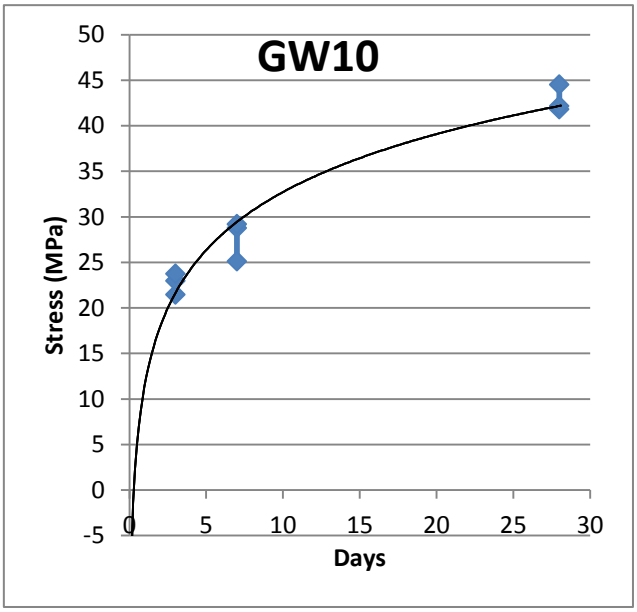


FIGURE 22 : Stress (MPa) for 10% cement replacement

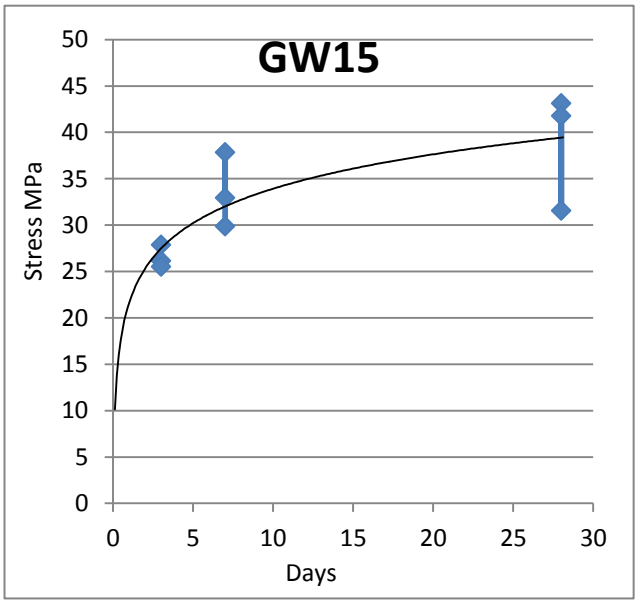


FIGURE 23 : Stress (MPa) for 15% cement replacement

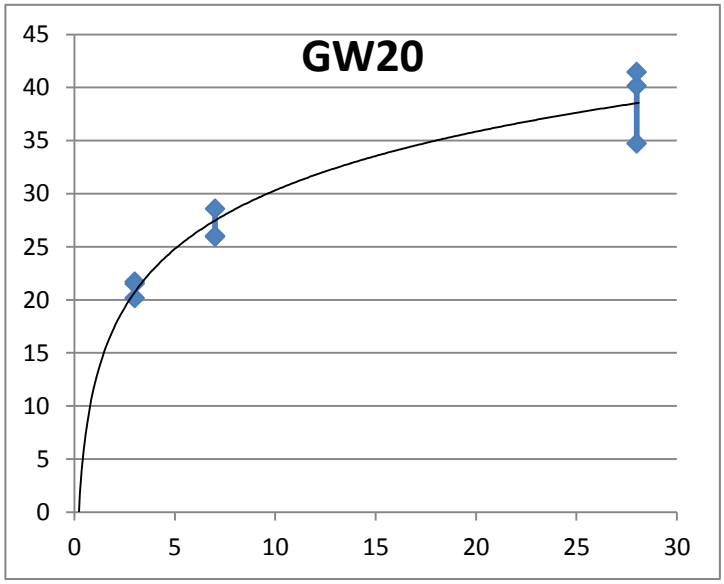


FIGURE 24 : Stress (MPa) for 20% cement replacement

Based on the result recorded, the mixture with 15% Glass Wool content shows higher strength on day 3 and day 7 compared to the controlled mixture. However, its strength on day 28 is much lower than the mixture with 10% cement replacement.

The result is expected to exhibit pozzolonic properties at the later age. However, it still depends on the fineness of the powder. The result that is not really satisfactory might be due to the fineness of the powder and curing ages. So, further research should be observed to see how the graph goes on day 56 and day 90.

Some error or mistakes may occur during the lab work since the pattern of the strength test result is not accordingly. If the time constraint is not being considered, the lab work whould be repeated

4.2.2. Chloride Penetration

Days	Chloride Penetration (mm)				
	GW0	GW5	GW10	CW15	CW20
7	2.00	4.00	4.30	4.50	4.90
14	5.50	5.25	4.90	4.75	4.55
28	6.30	6.10	5.80	5.65	5.30

TABLE 5 : Chloride Penetration

The result shows that chloride ions penetrate more as the replacement percentage is higher on day 3. However, the ion penetration becomes slower as the days go until day 28. It indicates that the degree in the rate of increment of the chloride penetration reduce as the percentage of cement replacement increase. The lesser the chloride ion penetration, the better the specimen as it means corrosion can be avoided if the material is being used in reinforce concrete.

Since the penetration rate of increment is also depended on type of cement used in the mixture, the result does make sense.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Since the world now is focusing on greener technology in construction, this new material has the potential to be one of cement replacement material in mortar and concrete. Besides can reduce the amount cement usage in which contribute to the air pollution by emitting carbon dioxide, it also help to reduce the pollution cause by the waste from the glass wool production itself. With further research and study, this new material can gives more contribution towards the performance of the mortar or concrete. So, this research are done in order to increase the understanding and also give extra view for future research that will be done based on cement replacement material development.

As the conclusion, these experiments are able to achieve the objectives that have been set at the beginning of the project:

i. To characterise of glass mineral wool waste in term of physical and chemical composition
ii. To obtain the right mixture proportions of concrete containing glass mineral wool waste
iii. To gather information data in respect of physical properties and mechanical properties of concrete containing glass mineral wool waste.

Firstly is to characterise of glass mineral wool waste in term of physical and chemical composition. Glass wool is first burned under several different temperatures and then being grinded into powder form.

Second it to i. obtain the right mixture proportions of concrete containing glass mineral wool waste by preparing a few mixture batches and conducted the mechanical test on each batches.

And the last one is to gather and interpret information data in respect of physical properties and mechanical properties of concrete containing glass mineral wool waste. It is proven that this material have the potential to be develop as cement replacement material.

5.2 Recommendation

Based on this research, a few recommendations can be suggested for future guideline. First is to use the material on concrete in term of mortar and see whether it give the same result. Besides that, instead of replacing the cement content, it may give different views if this material is used as additives in the mixture. More tests should also being conducted for example, acid test, carbonation test and so on, in order to study and investigate more on the properties it gives to the concrete. And the one of the most important recommendation is to test the specimens at later ages, such as 56 days, 90 days and so on. This is because, during this project, time constraint is a matter. While, based on the literature review, this material will show pozzolonic properties at the later ages.

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