CHARACTEI ... ASH (POFA) AND FLY ASH ... ION PROCESS

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ABSTRACT

Malaysia as the world largest exporter of palm oil was succeeded continuously increasing the production of palm oil but has been facing problems in disposing palm oil fuel ash (POFA), a by-product of palm oil mill. Since the manufacturing process of Portland cement produced greenhouse gases, previous researchers were revealing the potential of this by-product as the partial cement replacement and reacted as pozzolans in concrete paste. Therefore, Geopolymer is proposed in this study since it nullifies the usage of ordinary Portland cement (OPC) in concrete while maintaining the consumption of by-product through its usage as the source material in geopolymer. The effectiveness of fly ash-POFA based geopolymer was developed as an alternative material to replace usage of OPC. POFA were used to replace fly ash in geopolymer by 0%, 12.5% and 25% by weight of fly ash. The compressive strength of fly ash-POFA based geopolymer concrete paste due to 1, 7 and 28 days of curing age days with different curing condition were determined. The porosity percentage of fly ash-POFA geopolymer concrete paste due to 1, 7 and 28 days also were tested. In addition, the energy dispersive x-ray (EDX) was conducted in order to determine the basic elemental characteristic of fly ash-POFA based geopolymer binder. It was found that the compressive strength of fly ash-POFA based geopolymer concrete paste was lower than fly ash based geopolymer concrete paste. It also revealed that the compressive strength of concrete paste was increasing due to curing day ages for ambient curing while for oven curing the compressive strength were decreasing. Based on the EDX analysis, the geopolymer formation in fly ash-POFA geopolymer based concrete paste are poly(silate-disiloxo) and sialate link.

Keywords: Geopolymer, POFA, Fly Ash, OPC, compressive strength.

ABSTRAK

Malaysia merupakan pengeksport minyak sawit terbesar dunia terus berjaya meningkatkan pengeluaran minyak sawit tetapi telah menghadapi masalah dalam pelupusan abu kelapa sawit (POFA), hasil daripada pemprosesan kelapa sawit. Memandangkan penghasilan simen Portland (OPC) menghasilkan gas rumah hijau, penyelidik sebelum ini telah mendedahkan potensi POFA sebagai pengganti separa simen dan bertindak sebagai pozolana dalam pes konkrit. Oleh itu, Geopolymer telah dicadangkan dalam kajian ini bagi menggantikan penggunaan OPC didalam konkrit disamping mengekalkan penggunaan hasil kelapa sawit sebagai bahan sumber didalam geopolymer. Keberkesanan abu terbang dan juga abu kelapa sawit diwujudkan sebagai bahan alternatif bagi menggantikan OPC. Sebanyak 0%, 12.5% dan 25% gantian POFA mengikut berat abu terbang digunakan dalam proses geopolymer ini. Kekuatan mampatan geopolymer pes konkrit berasaskan abu terbang-POFA akan diuji selepas proses pengawetan sehingga umur pes konrit 1, 7 dan 28 hari dengan keadaan pengawetan yang berbeza. Peratusan keliangan pes konkrit berasaskan abu terbang-POFAjuga diuji pada umur pes konkrit 1, 7 dan 28 hari. Serakan tenaga x-ray(EDX) telah dijalankan untuk menentukan ciri-ciri asas unsur geopolymer berasaskan abu terbang-POFA. Keputusan menunjukkan bahawa kekuatan pes konkrit berasaskan abu terbang lebih kuat daripada pes konkrit berasaskan abu terbang-POFA. Ia juga menunjukkan kekuatan mampatan abu terbang-POFA meningkat didalam kawasan pengawetan suhu bilik berbanding didalam pengawetan ketuhar. Berdasarkan analisis EDX, pembentukan geopolymer yang terhasil didalam abu terbang-POFA adalah poli(silat-disiloxo) dan juga lingkaran sialate.

Kata Kunci: Geopolymer, POFA, Abu terbang , OPC, kekuatan mampatan.

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
OPC	Ordinary Portland Cement
POFA	Palm Oil Fuel Ash
NaOH	Sodium Hydroxide Solution
Na_2SiO_3	Sodium Silicate Solution
EDX / EDS	Energy Dispersive X-ray Spectroscopy
FESEM	Field Emission Scanning Electron Microscopy

CHAPTER 1

INTRODUCTION

1.1 Background of study

Portland cement is widely used in concrete industry. But unfortunately, Portland cement releases greenhouse gases. Carbon Dioxide (CO_2) is one of the greenhouse gases that had been release by Portland cement during its manufacturing process. In order to reduce the uses of Portland cement in concrete, one of the new concrete technologies has been introduced, that is Geopolymer concrete. The name of geopolymer was formed by a French Professor Davidovitsin 1978 to represent a broad range of materials characterized by networks of inorganic molecules. The geopolymers depend on thermally activated natural materials. Example of natural materials is Meta kaolinite or industrial by-products like fly ash that provide a source of Silicon (Si) and Aluminium (Al). Geopolymer concrete has shown it has good properties such as high compressive strength, low creep, good acid resistance, and low shrinkage. The role of binder in geopolymer concrete is replaced by fly ash. Fly ash possesses pozzolanic properties as Ordinary Portland Cement (OPC) and rich with alumina and silicate.

Fly ash is the ash precipitated electrostatically or mechanically from the exhaust gases of coal-fired power station. It is also known as pulverized-fuel ash. Fly ash has spherical particles and very high fineness. The vast majority of particles have a diameter between 1μm and 100μm. Fly ash known as the finely divided residue that results from the combustion of ground or powdered coal and that is transported by flue gases from the combustion zone to particle removal system. By replacing cement with fly ash, especially low calcium fly ash (ASTM Class F) has been shown to have a significant effect on properties of concrete. It leads to lower early strength with a potential for later-age strength gain. A material with three dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds was resulted from the reaction of fly ash with an aqueous solution containing Sodium Hydroxide and Sodium Silicate in their mass ratio.

Palm oil fuel ash (POFA) is also a by-product from small power plant which uses palm fiber, shell and empty fruit bunches as a fuel and burnt at 800 – 1000 °C. The main chemical composition of POFA is silica. Since Malaysia is the second largest producer in palm oil industry, the wastage of palm oil can is having good prospect to be used as a cement replacement material. POFA has good pozzolanic properties; therefore, it is suitable in the production of high-strength concrete. A pozzolanic material has little or no cementing properties. However, in the presence of moisture, the fine particle of this pozzolan can react with calcium hydroxide at normal room temperature to provide the cementing property.

Abu (1990) has conducted research in Malaysian agriculture ash and acknowledge that POFA a pozzolanic material. POFA can be used as a partial cement replacement. Mortar mix with up to 35% POFA can show the same strength as the control mortar. Study by Awal & Hussin (1996) concrete gain maximum strength when 30% of the cement replaced by POFA. However, when POFA content getting added, the concrete strength decreases gradually.

Geopolymer technology is one of the new technologies attempted to reduce the use of Portland cement in concrete. Geopolymer was introduced by Davidovits in 1978 to represent a broad range of materials characterized by networks of inorganics molecules. The geopolymers depend on thermally activated natural materials. Example of natural materials is Meta kaolinite or industrial by-products like fly ash that provide a source of Silicon (Si) and Aluminium (Al). It shows good properties such as high compressive strength, low creep, good acid resistance and low shrinkage. The role of OPC binder in geopolymer concrete is replaced with fly ash which possesses pozzolanic properties and rich in alumina and silica, and combined together with potassium or sodium based alkaline solution.

Based on Awal & Hussin (1996), the higher the fineness of POFA, the higher strength of concrete can be gained. POFA also has been fund could produce a more durable concrete. POFA also has good characteristics for reaction with chemicals such as sulfate and acid and other chemicals when used as a replacement for cement in concrete mixes Awal (1998). Awal & Hussin (1997) also stated that incorporation of POFA as partial cement replacement in concrete result in significant increase in chemical resistance to acidic environment. POFA has been effective in suppressing expression due alkali-silica reaction despite the higher alkali content POFA. However, performance of POFA when incorporated as partial cement replacement in aerated concrete still remains undiscovered.

The use of recycled material as concrete ingredients is gaining popularity and development because of increasingly stringent environmental legislation. There is significant research on many different materials for cement usage substitutes and replacement such as palm oil fuel ash and many others fiber and pozzolanic material. Since Malaysia is second largest producer in palm oil industry, the wastage of palm oil can be used to replace in small amount of cement.

1.2 Problem Statement

Nowadays, the increasing wastage of palm oil from the palm oil industry has become a bigger problem because the wastages are not reused and recycled. Meanwhile the greenhouse gases produced from Portland cement manufacturing process has encouraged a further development on the cement replacement material technology. Geopolymer binder is proposed to provide an alternative solution to these issues. However basic material characteristics need to be understood, hence the optimum proportion of fly ash-POFA blend can be provided based on the fundamental analysis studied in this research.

1.3 Objective

The objectives of study are:

i- To determine the compressive strength performance of fly ash based geopolymer containing palm oil fuel ash (POFA).

ii- To determine the basic elemental characteristic of fly ash-palm oil fly ash (POFA) based geopolymer binder.

1.4 Scope of study

This study concentrates on the investigation of compressive strength, durability and porosity of geopolymer concrete palm oil fuel ash (POFA) based. The origin of fly ash is from Manjung Coal Fired Power Plant while Palm Oil Fuel Ash (POFA) is from Lepar Hilir 3, Gambang, Pahang.

The paste specimens compose of fly ash, sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) without POFA will be used as the control mix. Other specimen will be composing by adding 12.5% and 25% of POFA. 6M of NaOH also will be used in the entire specimen.

The Compressive Strength Test and also Porosity Test will be conducted at 1, 7, and 28 days specimen. The curing process for this research will be handled in room temperature and in oven. Other than that, Elemental Characteristic Test and also Crystallography Test will also be conducted for this research.

1.5 Research significance

Cement is an important material in concrete mix but cement also release greenhouse gases, so modification and development have been made to replace cement uses in concrete mix. Modification will be held by replacing cement with fly ash and also Palm oil fuel ash. This research is to investigate and propose another way as an alternative material in concrete and to reveal the strength of geopolymer palm oil fuel ash based.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

In this chapter, information from previous research related to geopolymer concrete will be discussed in term of engineering of properties of concrete itself. Topics discussed in this chapter are mainly about geopolymer concrete and material used in geopolymer concrete such as POFA and fly ash. Besides that, this chapter also discussed about test that can be used in obtain the geopolymer performance characterization.

The use of POFA in geopolymer concrete is actually to decrease the Portland cement usage in concrete as their manufacturing process produce greenhouse gases. As POFA has good pozzolanic properties, it is suitable in the production of higher concrete strength. The use of recycled material as concrete ingredients is gaining popularity and development because of increasingly stringent environmental legislation.

2.2 GEOPOLYMER

Concrete is the most prevalent building material. Portland cement is the important material in production of concrete which act as its binder to bind all the aggregate together. However, the utilization of cement causes pollution to the environment and reduction of raw material (limestone). The manufacturing of Portland cement requires the burning of large quantities of fuel and decomposition of limestone, resulting in significant emissions of carbon dioxide (Kong and Sanjayan, 2008). To avoid these alarming pollution figures, there is a research on geopolymer concrete with waste by-product from coal-fired powerplant.

Geopolymer concrete also showed good properties such as high compressive strength, low creep, good acid resistance and low shrinkage (Lodeiro et al., 2007). Geopolymers are environmental friendly materials which not emit greenhouse gases during polymerisation process. Besides, they need only moderate energy to produce. Geopolymers are made from sources material with silicon (Si) and Aluminium (Al) content, thus they can be made using fly ash, waste-product of coal fired power station, as the source materials (Hardjito, Wallah, Sumajouw, and Rangan, 2004a)

2.3 SOURCE MATERIAL IN GEOPOLYMER

2.3.1 Fly ash

Fly Ash is residue from the combustion of coal and it is captured in the chimney from the coal fired power plant. Fly ash particle are spherical and have very high finesse. It has diameter range from less than 1μm- 150μm. The use of fly ash prevents environmental pollution, and it contributes to a reduced need for natural resources (Siddique, 2008).

Based on ASTM C 618 (1993), natural pozzolan and fly ashes has been categorized into three categories which is Class N for raw and calcined natural pozzolans such as volcanic ashes. Class F is for fly ash which normally produced from burning anthracite or bituminous coal. Class F fly ash exhibits pozzolanic property but rarely. Class C, normally only fly ash was produced from lignite or sub-bituminous coal included in this category. This class has both pozzolanic and cementitious properties.

2.3.2 The effect of fly ash

The fly ash particle is very similar in size and shape to entrained air bubbles and has many very similar effects.it helps in reduction of bleeding, improved cohesion and plasticity, improve pumpability and reduced slump loss with time. Fly ash is not compressible and it has the benefit that is resent as a clearly defined quantity. Because of the PFA particle is so fine, so they are very valueable as pore-blockers, substantially reducing permeability in the hardened concrete (R. Siddique, 2008).

Fly ash needs to be activated, usually using alkaline solutions. The reactions between fly ash with an aqueous solution containing Sodium Hydroxide and Sodium Silicate in their mass ratio, results in a material with three dimensional polymeric chain and ring structure containing Si-O-Al-O bonds (Aleem and Arumairaj, 2012).

2.3.3 Palm Oil Fuel Ash (POFA)

Palm oil fuel ash is a by-product in palm oil mill. After palm oil is extracted from the palm oil fruit, both palm oil husk and palm oil shell are burned as fuel in the boiler of palm oil mill. Generally, after combustion about 5% palm oil fuel ash by weight of solid wastes is produced (Abdullah and Hussin, 2004).

The ash produced sometimes varies in tone of colour from whitish grey to darker shade based on carbon content in it. In other words, the physical characteristic of POFA is very much influenced by the operating system in palm oil factory. In practice, POFA produced in Malaysia palm oil mill is dumped as waste without any profitable return (Sumadi and Hussin, 1995).

2.3.4 Pozzolana properties of POFA

Both physical properties and chemical analysis indicated that POFA is a pozzolanic material (Awal and Hussin, 1993). This pozzolanic material is grouped in between Class C and Class F as specified in ASTMC618-92a (Awal and Hussin, 1997). POFA is moderately rich in silica content meanwhile lime content is very low as compared to ordinary Portland cement (OPC) (Awal and Hussin, 1997).

The pozzolanic of any material is closely related to the ability of silica to react with calcium hydroxide to produce calcium silicate hydrate. For an assessment of pozzolanic activity with cement, the method of pozzolanic activity index which determines the total activity of pozzolana (Neville, 2005)

2.3.5 Strength and Durability of POFA

The pioneer in POFA research has embarked on studying agricultural ash in Malaysia and finally acknowledge that POFA is pozzolanic material and able to be replace as partial cement replacement up to 35% in mortar mix that could exhibit similar strength as control mortar (Abu, 1990).

POFA concrete gain maximum strength when 30% of the cement was replaced by POFA. It is reported that the maximum strength gain occurred at the replacement level of 30% but further increase in the ash content would reduce the strength of concrete gradually. However, the result of POFA performance once added in concrete still yet to be studied. Besides that, added that increasing in fineness of POFA would lead to greater concrete strength development than the coarser one (Awal and Hussin, 1996).

2.4 GEOPOLYMER PERFORMANCE CHARACTERIZATION

2.4.1 Compressive strength

The compressive strength was increase when the finest of fly ash increase. Hence the nature and the concentration of the activators were dominant factors in the reaction of alkali activation. The highest compressive strength was obtained using a solution of sodium silicate as an activator (n = 1.5; 10% Na_2O). Sodium silicate is the most suitable as alkaline activator because it contains dissolved and partially polymerized silicon which reacts easily, incorporates into the reaction products and significantly contributes to improving the mortar characteristics (Al Bakri et al., 2011)

Hardijito & Rangan (2004) observed that higher concentration of sodium hydroxide (molar) resulted higher compressive strength and higher the ratio of sodium silicate-to-sodium hydroxide liquid ratio by mass, showed higher compressive strength of geopolymer concrete. They also found that the increased in curing temperature in the range of 30 to 90 °C increased the compressive strength of geopolymer concrete and longer curing time also increased the compressive strength. They handled the geopolymer concrete up to 120 minutes without any sign of setting and without any degradation in the compressive strength, resulted very little drying shrinkage and low creep.

2.4.2 Workability

Water also play important role in geopolymer concrete as much as normal concrete. The used of water in geopolymer is to improve the workability, but it will increase the porosity in concrete due to evaporation of water during curing process at elevated temperature (Sathia et al., 2008)

The higher content of POFA exhibits a lower slump and lower degree of compaction. POFA concrete needs more water than OPC concrete for lubrication to maintain the same workability. This is due to high porosity of

POFA particles, which absorb some water and thus reduce the free water content needed for workability (Safiuddin et al., 2011)

2.4.3 Setting Time

Several studies showed the use of POFA delays the setting time of the concrete. Therefore, the initial and final setting time increase with the increase of POFA content. The long setting times of POFA concrete are due to the pozzolanic reaction. The setting time varies with the degree of ash fineness and replacement level of cement. The higher replacement level of cement with POFA reduces the amount of tricalcium silicate and thus increases the setting time of concrete (Safiuddin et al., 2011)

2.4.4 X-ray Fluorescence (XRF) Analysis

Table 2.1: Chemical composition of materials

Chemical composition (%)	Cement Type I	Cement Type V	GPA(Ground POFA)
Silicon dioxide (SiO_2)	20.9	22.1	65.3
Aluminium oxide (Al_2O_3)	4.7	3.5	2.5
Iron oxide (Fe_2O_3)	3.4	5.5	1.9
Calcium oxide (CaO)	65.4	62.4	6.4
Magnesium oxide (MgO)	1.2	0.9	3.0
Sodium Oxide (Na_2O)	0.2	0.0	0.3
Potassium oxide (K_2O)	0.3	0.1	5.7
Sulphur trioxide (SO_3)	2.7	1.0	0.4
Loss on ignition (LOI)	0.9	1.6	10.0
$SiO_2 + Al_2O_3 + Fe_2O_3$			69.7
Bouge chemical composition (%)			
Tricalcium silicate (C_3S)	62.8	51.2	
Dicalcium silicate (C_2S)	12.5	24.8	
Tricalcium aluminate (C_3A)	6.8	0.0	
Tetracalcium aluminoferrite (C_4AF)	10.3	16.9	

The chemical composition of ground POFA with high fineness (GPA) is listed in Table 1. The main component of GPA is SiO_2 which is 65.3%. The total amount of SiO_2, Al_2O_3, and Fe_2O_3 was 69.7%. The amounts of LOI and SO_3 were within the respective limits of 10.0% and 4.0% as specified by ASTM C618. Although GPA is not natural pozzolan, it can be classified as Class N (Natural) pozzolan based on the chemical composition according to ASTM C618 (Tangcharipat, Jaturapitakkul & Chindaprasirt, 2009

Table 2.2: Physical properties and chemical composition of OPC and POFA

Tests	OPC	POFA
Physical properties		
Fineness- Sp. Surface area (m2/kg)	315	520
Soundness - LeChatelier method (mm)	1	1
Specific gravity	3.28	2.22
Chemical composition (%)		
Silicon dioxide (SiO2)	20.20	43.60
Aluminium oxide (Al2O3)	5.70	11.40
Ferric oxide (Fe2O3)	3.00	4.70
Calcium oxide (CaO)	62.50	8.40
Magnesium oxide (MgO)	2.60	4.80
Sulphur trioxide (SO3)	1.80	2.80
Sodium Oxide (Na2O)	0.16	0.39
Potassium oxide (K2O)	0.87	3.50
Loss on ignition (LOI)	2.70	18.00
28-days strength activity index with OPC		112

POFA is grayish in colour becomes darker with increasing proportions of unburned carbon. However, it is much finer than OPC and its specific gravity is 2.22. The particles have a wide range of size but they are relatively spherical. The chemical analysis in Table 2 shows that POFA satisfies the requirements to be classified in between Class C and Class F according to the standard specified in ASTM C618-01(2001) (Awal and Hussin, 2011)

2.4.5 Energy Dispersive X-Ray (EDX) Analysis

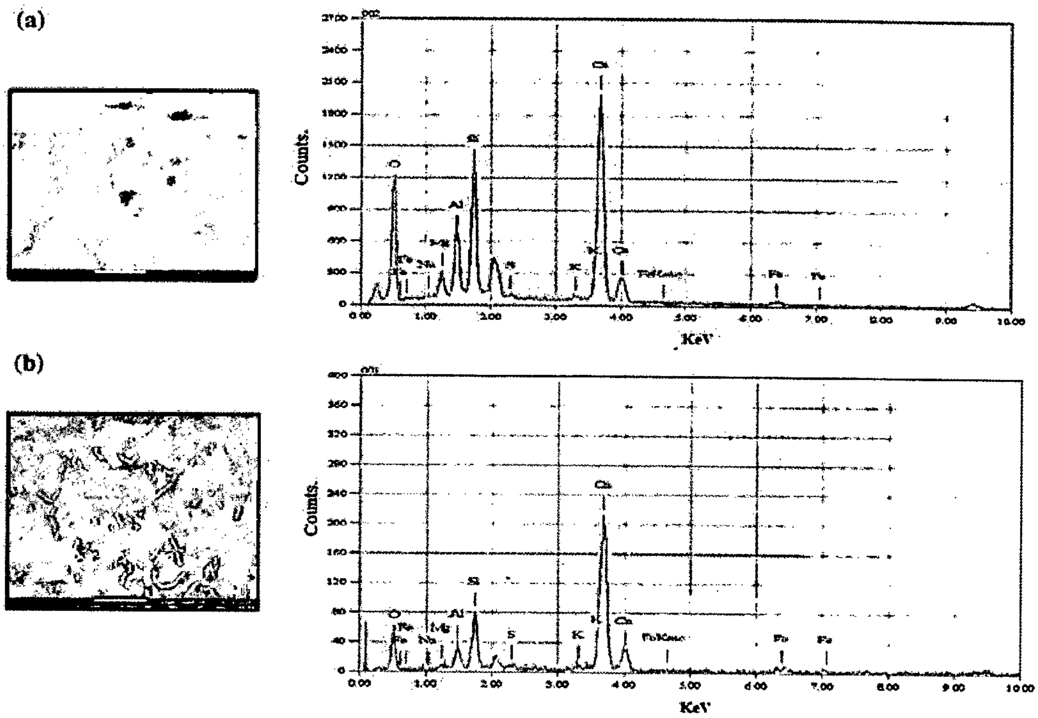

Figure 2.1: FESEM and EDS analysis of mortars immersed in sodium sulphate
(a) OPC, (b) POFA

A typical field emission scanning electron microscopy (FESEM) and EDS analysis of OPC and POFA mortars immersed in sodium sulphate solutions are shown in Figure 1. The FESEM results in general show porous structure with different degree for all mortar types. Based on EDS analysis, POFA mortar cured in sodium sulphate solution exhibits higer amount of sulphur compared with OPC (Bamaga et al., 2011)

CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

This chapter were explained about the research that had been conducted and explain the study that has been carried out. In this chapter also included the explanation about the materials used, the research planning and also the testing has been conducted to determine the characteristic of POFA in concrete. This part also gives the clear point of view about the research and clearly shows how the objective of this research can be achieved.

For this study, the materials used are Fly Ash, Sodium Hydroxide (NaOH), Sodium Silicate (Na_2SiO3) and also POFA. After casting and curing are done, all specimens had been tested. Tests including compressive strength test, porosity test, and setting time test. Specimen also had been analyzed by X-ray Fluorescence analysis and Energy Dispersive X-ray analysis.