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EFFECT OF PALM OIL FUEL ASH (POFA) AS CEMENT REPLACEMENT  
MATERIAL TOWARD CONCRETE MICROSTRUCTURE

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## ABSTRAK

Abu kelapa sawit (POFA) adalah bahan buangan yang dijana oleh kilang memproses kelapa sawit. Abu ini memiliki sifat pozolana yang bukan sahaja membolehkan ia berfungsi sebagai bahan pengganti simen tetapi turut memainkan peranan dalam menghasilkan konkrit yang kuat dan tahan lasak. Dalam kajian ini, keberkesanan menggunakan POFA sebagai bahan ganti simen dalam mikrostruktur konkrit telah di kaji., Penggunaan OPC telah diganti dengan 10%, 20 % dan 30% dengan POFA. Kiub konkrit 150 x 150 x 150 mm telah diuji kekuatan mampatannya pada 7, 28 dan 90 hari yang telah diawet dalam air. Walaupun kekuatan konkrit POFA tidak mencapai kekuatan OPC, kekuatan ujian mampatan konkrit POFA adalah hanya sedikit rendah daripada OPC pada awal usia akan tetapi memberikan kekuatan lebih tinggi berbanding konkrit OPC untuk usia yang lama . Hasil kajian menunjukkan konkrit 20% POFA memberikan nilai kekuatan paling tinggi dibandingkan dengan konkrit POFA lain. Konkrit POFA juga memberikan nilai paling rendah dibandingkan dengan konkrit OPC. Berdasarkan nilai ujian keliangan, 20% penggantian POFA memberikan nilai paling rendah. Analisis XRD membawa kepada fakta bahawa kewujudan CSH meningkat dengan umur pengawetan dan menunjukkan kemajuan tindak balas penghidratan simen dan tindak balas pozzalana. Daripada analisis mikrostruktur (SEM), kewujudan CSH yang memberikan ketumpatan struktur, dan peningkatan dalam kekuatan dapat diperhatikan. Platelet heksagon  $\text{Ca}(\text{OH})_2$  boleh dilihat dalam beberapa sampel.

## ABSTRACT

Palm Oil Fuel Ash (POFA) is a by-product produced in palm oil mills. These ash having pozzolanic properties that not only enables the replacement of cement but also plays an important role in making strong and durable concrete. For this research, the effectiveness using POFA as cement replacement material towards concrete microstructure was investigated. OPC replaced with 10%, 20 % and 30% of POFA respectively. For compressive strength, concrete cubes 150 x 150 x 150 mm was tested due to 7, 28, and 90 days cured in water. Although strength of POFA concrete did not exceed that of OPC, the compressive strength of POFA concrete is slightly lower than that of OPC at early age but at later ages it is found to be higher than that of OPC concrete. It was found that, the concrete with 20 % of POFA recorded highest compressive strength compare to those POFA mixes. POFA concrete exhibit lower value of porosity compared to OPC concrete. Based on porosity test results, the 20% replacement of the POFA, give the lower of porosity. XRD analysis leads to the fact that the existence of C-S-H increased with curing age indicating the progress of cement hydration reaction and pozzalanic reaction. From the microstructural analysis (SEM), shows existing of C-S-H gel have lead to a densification structure and increase in strength. Hexagonal platelets of  $\text{Ca}(\text{OH})_2$  could be observed in some samples.

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## **CHAPTER 1**

### **INTRODUCTION**

#### **1.1 Background of study**

Nowdays, thousands of tons of ash are produced annually by operation of 200 palm oil mills in Malaysia and are simply disposed of without any commercial return (Awal, 1997). Over half of the world's total palm oil is produced from the oil palm industry in Malaysia; the country has an aim to grow further with the global increase in vegetable oil demand. However, the nation's pollution problem is also increased for this sector which includes the annual production of 2.6 million tones of solid waste in the form of oil palm shells (Basri, 1999).

A large area is required for the disposal of these POFA waste materials. The landfill of POFA is the problem for the palm oil industry when it is not reused for any work. The production of POFA is rising every year, it is disposed for landfills, now become an important environmental disposal issue. Government needs to focus for assigning more hectares of land for disposal of these huge amounts of waste; and financial losses are also increased for transporting as well as maintenance purposes of these wastes. However, reduction of dumped waste and environment sustainability can be ensured by proper consumption or recycling of these materials.

Increasing world population and life demand are continuously raising the price of raw materials and reducing the natural resources; for these reasons researches have been concentrated to use waste materials as a potential alternative in the construction industry. Waste materials, when properly processed, have shown to be effective as construction materials and readily meet the design specifications.

During recent decades, many researchers have been conducted for the use of agro waste ashes - POFA, rice husk ash (Awal, 1997; Basri, 1999; Tangchirapat, 2009; Chindaprasirt, 2007; Chindaprasirt, 2008; Tay, 1995; Jaturapitakkul, 2007; Sata, 2010; Rukzon, 2009; Tangchirapat, 2007), sawdust ash and bagasse ash -as constituents in concrete. All of these agro waste ashes contained a high amount of silicon dioxide in amorphous form and as a result they could be used as pozzolanic materials. Pozzolans are such fine materials, with containing silica and/or alumina, that they do not exhibit any cementing properties of their own; in the presence of calcium oxide (CaO) or calcium hydroxide (Ca(OH<sub>2</sub>)), silica and alumina in the pozzolans react and form cementitious materials (ASTM, 2001).

POFA is an agro waste ash that contains a large amount of silicon dioxide and has high potential to be used as a cement replacement. For producing high-strength concrete, POFA can be used as a pozzolanic material; it improves the durability, reduces cost due to less use of cement. It will also be beneficial for the environment with respect to reducing the waste disposal volume of landfills (Tangchirapat, 2009). POFA contains the silica oxide that can react with calcium hydroxide (Ca(OH)<sub>2</sub>) generated from the hydration process; and the pozzolanic reactions produce more calcium silicate hydrate (C-S-H) gel compound as well as reducing the amount of calcium hydroxide (Eldagal, 2008).

Thus, for the concrete production, POFA contributes to make stronger, denser and more durable concrete. Abundant agricultural and industrial wastes are discharged from the developing countries; these wastes, including POFA, can be reutilized as potential cement replacement material in the concrete construction.

Therefore, effective consumption of POFA as a replacement for cement will also encourage researchers to investigate sustainable way of saving material, especially cement. The use of POFA in concrete as cement replacement material is logical, worthy and attributable for the present situation demand in concrete industry. Finally, this reutilization of POFA will have the double advantages reduction in the cost of construction material and minimization of waste disposal problem.

## 1.2 Problem Statement

Study about the effectiveness of Palm Oil Fuel Ash (POFA) is exactly to continue since the engineers and expertise always faced limited compressive strength when cement hydration only produce limited of C-S-H. Many researchers have studied the use of agro waste ashes as constituents in concrete, namely rice-husk ash (Mehta, 1977), sawdust ash (Udoeyo and Dashibil, 2002) and bagasse ash (Singh et al., 2000). Their results have revealed that these agro waste ashes contained a high amount of silica in amorphous form and could be used as a pozzolanic material. This limitation can be addressed by suitable "pozzolanic" admixtures inducing a secondary cementitious reaction.

Primary reaction:  $\text{Cement} + \text{Water} \rightarrow \text{CSH} + \text{Ca(OH)}_2$

Secondary reaction:  $\text{Ca(OH)}_2 + \text{SiO}_2 + \text{Water} \rightarrow \text{CSH}$  ("Pozzolanic Reaction")

Other than that, effective consumption of POFA as pozzolanic material in concrete, would decrease the cost of concrete production, could reduce negative environmental effect, and also would solve the landfill problem for the disposal of these wastes.

### 1.3 Objective of Study

The objectives of this study are:

- i. To study the compressive strength of concrete containing palm oil fuel ash (POFA) as a cement replacement material.
- ii. To study the effect of Palm Oil Fuel Ash (POFA) in porosity.
- iii. To investigate the microstructure of Palm Oil Fuel Ash (POFA) concrete by using Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD).

### 1.4 Scope of Study

This study focused on the investigation of effectiveness concrete containing POFA substituted as ordinary Portland cement (OPC) replacement. The study was used different percentage of POFA subjected to same curing types, which is water curing. The percentages of POFA were calculated based on total weight of the OPC which is 0%, 10%, 20% and 30% as compare with control mix. The specimens were labeled as POFA-0, POFA-10, POFA-20 and POFA-30 respectively. After designation of the mix proportion, the concrete were cast into the standard steel mould with dimensions of 150 mm x 150 mm x 150 mm. The POFA-concretes where cured in water until the test date of compressive strength test. The specimens were cured at 7, 28 and 90 days.

Concrete with grade 30 were designed and poured into standard concrete mould. For porosity tests, all specimens were tested on 28 days and 1 year ages. Other than that, analysis POFA concrete monitored by X-Ray Diffraction was applied to identify the peak patterns of C-S-H after 60 days age. The hydration

products were determined through the length intensity of C-S-H collected by X-Ray scans recorded as intensity in unit counts. Lastly, microstructure analysis was monitored by Scanning Electron Microscope to determine the chemical compound and microstructure of POFA concrete. From the microstructure analysis it proved that have a changes on the microstructure between control mixture and POFA concrete within 10 days and 1 year because of increases of C-S-H.

### **1.5 Significant of the Study**

From cement hydration, C-S-H and  $\text{Ca(OH)}_2$  is main product of Portland cement concrete. The engineering properties of concrete such as setting and hardening, strength and dimensional stability depend primarily on C-S-H gel. By adding POFA it has potential as a cement replacement material toward concrete microstructure. By adding POFA the compression strength of concrete were increased and porosity of concrete were decreased because of pozzolanic reaction that formed of additional C-S-H gel that would fill the existing voids in concrete thus creating denser concrete. Furthermore, reduction in amount of  $\text{Ca(OH)}_2$  that vulnerable to aggressive environment improves the durability of concrete.

## **CHAPTER 2**

### **LITERATURE RIVIEW**

#### **2.1 Introduction**

Concrete is a suitable and popular construction material all over the world and cement is the most important constituent for its production. Due to continuous increasing of the cost of cement, the use of supplementary cementing materials such as industrial by-product (slag, fly ash, silica fume) and biogenic wastes (palm oil fuel ash, rice husk ash, ash from timber) have become significant in concrete industry. One of these important biogenic waste is the POFA generated as by-product from palm oil mills. The production of POFA increases every year, it is disposed for landfills without any return value and now becomes a burden. It contains a non-crystalline silicon dioxide with high specific surface area and high pozzolanic reactivity. Many researches have been conducted for the use of pozzolans, especially waste pozzolans such as POFA, rice husk ash, and silica fume as a replacement of OPC.

Test results regarding compressive strength and durability of concrete from these researches confirmed the use of POFA as a pozzolanic material for cement replacement in mortar and concrete. In this paper, a review on the strength of

concrete as influenced by the use of POFA as partial replacement of cement in concrete has been presented. Based on the information available in literature on the utilization of POFA in concrete, the characteristic and behavior of POFA as a pozzolana material, the compressive strength, porosity of concrete are affected by the percent replacement of POFA in concrete is mainly discussed here. Other than that, the existing of main product from cement hydration monitored by XRD and microstructure analysis of POFA concrete also discussed.

## **2.2 Pozzolanic Materials**

Many researchers have studied the use of agrowaste ashes as constituents in concrete, namely rice-husk ash (Mehta, 1977), sawdust ash (Udoeyo and Dashibil, 2002) and bagasse ash (Singh et al., 2000). Their results have revealed that these agro waste ashes contained a high amount of silica in amorphous form and could be used as a pozzolanic material. ASTM C 618 (2001) defines pozzolanic material as a material that contains siliceous or siliceous and aluminous material by composition.

In general, a pozzolanic material has little or no cementing property; however, when it has a fine particle size, in the presence of moisture it can react with calcium hydroxide at ordinary temperatures to provide the cementing property. POFA is one of the agrowaste ashes whose chemical composition contains a large amount of silica and that has high potential to be used as a cement replacement. A pozzolana (Neville, 2005) is a siliceous or siliceous and aluminous material which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties.



### 2.3 Pozzolanic Behaviour

A common feature of nearly all Cement Replacement Materials is that they exhibit pozzolanic behavior to a greater or lesser extent, and so we will define this before discussing the individual materials (Illston and Domone, 2001). A pozzolanic material is one which contains active silica ( $\text{SiO}_2$ ) and is not cementitious by itself, but will, in a finely divided form, and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form cementitious compounds.

The key to the pozzolanic behavior is the structure of the silica; this must be in a glassy or amorphous form with a disordered structure, which is formed in rapid cooling from a molten state. A uniform crystalline structure which is formed in slower cooling, such as is found in silica sand, is not chemically active. Naturally occurring pozzolanic materials were used in early concretes, as mentioned in the Introduction to this part of the book, but when a pozzolanic material is used in conjunction with a Portland cement, the calcium hydroxide that takes part in the pozzolanic reaction is that produced from the cement hydration.

“Cement Reaction” Primary reaction:  $\text{Cement} + \text{Water} \rightarrow \text{CSH} + \text{Ca(OH)}_2$

“Pozzolanic Reaction” Secondary reaction:  $\text{Ca(OH)}_2 + \text{SiO}_2 + \text{Water} \rightarrow \text{CSH}$

The products of the pozzolanic reaction cannot be distinguished from those of the primary cement hydration, and therefore make their own contribution to the strength and other properties of the hardened cement paste and concrete.

## 2.4 Palm Oil Fuel Ash (POFA)

Palm oil industries can play a major role in the economic development of different tropical countries. These industries produce a large amount of solid waste by-products - fibers, nut shells, and empty fruit bunches during the processing oil-palm fruit for oil extraction. There will be approximately 20 tonnes of nut shells, 7 tonnes of fibers, and 25 tonnes of empty bunches discharged from the mill for every 100 tonnes of fresh fruit bunches processed (Tay, 1995). Palm oil fuel ash is commonly known as POFA, which is about 5% of solid waste product, have the potentiality to be used as pozzolanic materials in concrete industry (Sata, 2010). It is the ashes produced from fiber and shell of palm oil burning for the generation of energy in palm oil mill. It is a promising pozzolanic and available material in many parts of the world.

The by product from palm oil industry such as palm oil residue, palm fiber and shells, when burnt at temperatures of about 800-10000C to produce steam for electricity generation in biomass thermal power plants are known as POFA (Tangchirapat,2009). In Thailand, more than 100,000 tons of POFA are produced annually (Chindaprasirt, 2007), and this amount increases every year because palm oil is one of the major raw materials used in the production of bio diesel. It has recently been accepted as a pozzolanic material in concrete due to its large amounts of silica. The material is similar to other pozzolanic materials, such as silica fume and fly ash. However, due to the pozzolanic properties of POFA, it is not only sed as replacement of cement but also used in making strong and durable concrete (Awal, 1997). In practice, POFA produced in Malaysia palm oil mill is dumped as waste without any profitable return (Sumadi and Hussin, 1995). Either in 20<sup>th</sup> or 21<sup>st</sup> century, POFA is still used and considered as a nuisance to the environment and disposed without being put for any other use as compared to other type of palm oil by-product (Abdullah et al., 2006). Since Malaysia is continuously to increase production of palm oil, therefore more ashes will be produced and failure to find any solution in making use of this by-product will create severe environmental problems.

## 2.5 Advantages of POFA in Concrete

POFA exhibits a good pozzolanic reactivity as investigated in different literatures (Awal, 1997; Basri, 1999; Tangchirapat, 2009; Chindaprasirt, 2007; Chindaprasirt, 2008; Tay, 1995; Jaturapitakkul, 2007; Sata, 2010; Rukzon, 2009; Tangchirapat, 2007), besides that, it contributes to produce denser and durable concrete with a particular level (about 20-30%) of replacement. Based on the discussions presented in this paper, the following advantages of POFA have been found to use in cement or concrete. Utilization of POFA in concrete could reduce the cost of concrete production due to less cement use disposal problem can be minimized (Rukzon, 2009; Tangchirapat, 2007).

With high fineness, POFA can be used as a cement replacement to produce high-strength concrete it also reduces the water permeability of concrete; shows smaller degree of expansion and loss in compressive strength with compare to concrete made with OPC type I cement (Tangchirapat, 2009). POFA can be used as pozzolans to replace part of OPC in making mortar with relatively high strength and good resistance to chloride penetration (Chindaprasirt, 2008). Concrete using POFA of high fineness (retained 1% on a No. 325 sieve) as a replacement of 20-30% achieved 100% strength compare to ordinary concrete (Jaturapitakkul, 2007).

Concrete made with palm oil shell can be used as structural light weight concrete (Basri, 1999; Mannan, 2001). Awal and Hussin (Awal, 1997) suggested that POFA has a good potential in suppressing expansion due to alkali-silica reaction. POFA can be used as a cement replacement to produce good resistance against sulfate attack (Chindaprasirt, 2008; Jaturapitakkul, 2007; Tangchirapat, 2007). For concrete with POFA, the temperature rise of fresh concrete decreased as POFA content increased (Sata, 2010).

## **2.6 Pozzolanicity of Palm Oil Fuel Ash (POFA)**

The pozzolanic activity indices of palm oil fuel ash was obtained by (Abu, 1990) and (Sumadi and Hussin 1993) are 78.6 and 87.6 % respectively. However, for all classes of ash; the pozzolanic activity is 75 % (ASTM C 618-94 a).

The pozzolanicity 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 totals activity of pozzolana (Neville, 2005).

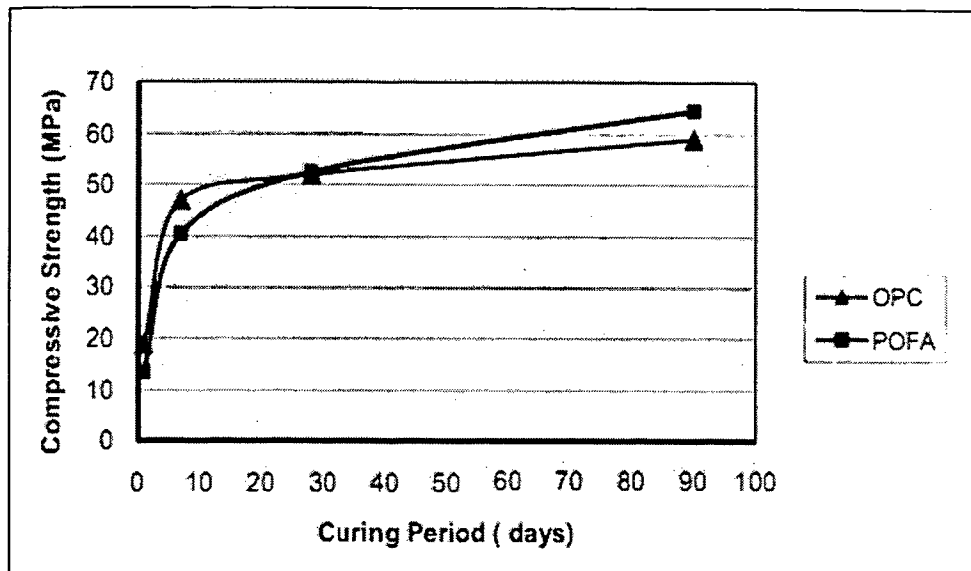
## **2.7 Chemical Composition of POFA**

Both physical properties and chemical analysis indicated that POFA is a pozzoianic material (Sumadi & Hussin, 1993). This pozzolanic material can be grouped to Class C and Class F as specified in (ASTMC618-92a, 1994). POFA is moderately rich in silica content meanwhile lime content is very low as compared to OPC (Awal & Hussin 1997). However, the chemical composition of POFA can be varied due to operating system in palm oil mill.

## **2.8 Strength Development**

POFA concrete is weaker at early age, but at later ages the compressive strength is found to be higher than that of OPC concrete (Awal and Hussin, 1997) as

shown in figure 2.13. In general, specimens consisting POFA exhibit lesser strength at early age but compressive continue to increase as curing age become higher. This is because pozzolana starts reacting somewhat belatedly with the calcium hydroxide produced by clinker hydration and therefore it behaves like an inert diluting agent towards the Portland cement with which it has been mixed (Massaza, 1993).



**Figure 2.1:** Strength development of OPC and POFA concretes

(Awal and Hussin, 1997)

## 2.9 Particle Size Distribution

It is a well known fact that the increase in the fineness of pozzolana material would lead to significant increase in strength. The extremely fine particles in concrete act as lubricant in the concrete mix and permits a reduction in water content, thereby, increasing strength. Additionally, The ash spheres with their multi sized spherical morphology promote a high packing density of plastic concrete. The

influence of ash fineness toward strength development of concrete has been investigated by many researchers. The fineness of pozzolanic ash also tends to affect both the fresh and hardened state properties of concrete (Awal, 1998). Generally, the ash used as pozzolanic material needs to produce in a finer size so that can function effectively in increasing the strength of concrete.

### **2.10 Curing Length**

Curing is the process of controlling the rate and extent of moisture loss from concrete during cement hydration. It may be either after it has been placed in position (or during the manufacture of concrete products), thereby providing time for the hydration of the cement to occur. Since the hydration of cement does take time – days, and even weeks rather than hours curing must be undertaken for a reasonable period of time if the concrete is to achieve its potential strength and durability.

The hydration of Portland cement is the chemical reaction between grains of Portland cement and water to form the hydration product, cement gel and cement gel can be laid down only in water-filled space. Hydration can proceed until all the cement reaches its maximum degree of hydration or until all the space available for the hydration product is filled by cement gel, whichever limit is reached first. However, curing of pozzolanic concretes or mortars, however needs more care than Portland Cements. Also the strength development of concrete containing pozzalans is more adversely affected by very short curing periods under water than the plain one (Neville, 2005).

### 2.11 Porosity in Concrete

Concrete porosity; voids in concrete can be filled with air or with water. Air voids are an obvious and easily-visible example of pores in concrete. Broadly speaking, the more porous the concrete, the weaker it will be. Probably the most important source of porosity in concrete is the ratio of water to cement in the mix, known as the 'water to cement' ratio.

The decreasing of porosity was attributed to the post-reactions of the natural pozzolan that can cause the formation of reaction products which close porosities and cracks and decrease the rate of water penetration. The water penetration into the Portland pozzolan cement concrete specimens was lower than that of the control specimens at the age of 28 days, (Najimiet al. 2008). Sumadi and Hussin (1995) investigated the water permeability of concrete with ground POFA. They found that the permeability of POFA concrete decreased with increased age due to the formation of additional gel from the pozzolanic reaction of ash.

### 2.12 Microstructural Analysis

The structural development of cement is a result of complex hydration reactions that stiffens, densify and impart structural integrity on the product (Conner, 1990). The main hydration phases under normal conditions are C-S-H gel of variable stoichiometry and calcium hydroxide which together form about 90 % (w/w) of the solid hydration products in an OPC paste. The other solid hydration products are hexacalcium aluminoferrite trisulfate or "ettringite" type phases and tetracalcium aluminate monosulfate or "monosulfate".

Scanning electron microscopy (SEM) was used to observe the morphology of the POFA and concrete samples. Samples that were tested for strength were used. Samples were manually fractured mounted on to aluminum stubs using conductive carbon coated cement double sided tape. Samples were coated with gold-palladium for microstructural scanning. All samples were kept in desiccator prior to analysis.

In the presence of POFA, pozzolanic reaction occurs, involving the reaction between calcium hydroxide with  $\text{SiO}_2$  or  $\text{Al}_2\text{O}_3\text{-SiO}_2$  framework to form calcium silicate hydrate, calcium aluminate hydrate and calcium aluminate ferrite hydrate. The main reaction product of pozzolanic reaction is C-S-H gel that enhanced the strength of cement (James and Rao, 1986). Hydration processes that result in densification of the cement matrix can improve the ability of the product to combat aggressive condition.