THE EFFECT OF PALM OIL FUEL ASH (POFA) AS A PARTIAL CEMENT REPLACEMENT TOWARDS THE FORMATION OF DELAYED ETTRINGITE FORMATION (DEF) IN CONCRETE

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ABSTRACT

Malaysia is one of the largest producers of palm oil and the amount of waste generated by the palm oil plant increasing from day to day. Due to the increasing of waste, a lot of research has been done to recycle palm oil fuel ash (POFA) utilization in Malaysia as construction material to replace ordinary Portland cement (OPC). The utilization of POFA which is generated by the residual of Malaysian palm oil industry can produced a new construction material. It is one of the solutions to reduce the quantity of POFA disposed at landfills. This study was focus on the delayed ettringite formation (DEF), compressive strength and microstructure of concrete containing POFA. Four types of mixes are prepared. The percentage of POFA used was 0%, 10%, 20% and 30% as a partial cement replacement. The concrete cured for 7, 28 and 90 days were tested for the compressive strength. The specimens of 0% and 20% of POFA at 28 days and 90 days were testing for microstructure by using Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD) test. All specimens were tested for DEF after heated about 90 days. From the results obtained, the compressive strength of the concrete for 10%, 20% and 30% produced slightly lower than the control mix (0%) at each of age curing period. Specimen with 30% of POFA produced the lowest compressive strength compared to the others. The maximum compressive strength was determined for control mix (0% of POFA) about 29.349 MPa. This shows that the 20% POFA replacement for cement produces the optimum compressive strength results. The results generated from XRD test shows that at 29.411(deg) is the peak of C-S-H for 0% of POFA and 29.546 (deg) is the peak of C-S-H for 20% of POFA at 90 days. It shows that reaction occur in concrete. The image generated from (SEM) shows the microstructure of the POFA in concrete. The samples do not generate a lot of momentous, so the images are not as clear as metals at high magnifications. 0% of POFA shows larger extend and size ettringite needles (2μm) while on the 10% to 30% of POFA substitution concrete, the needles' size are 1μm. As a conclusion, POFA can be used as a replacement of cement in concrete. The use of finer pozzolan as a partial cement replacement can be eliminated the formation of ettringite and better effect to the concrete. POFA can be used as an alternative material in concrete because it has pozzolanic characteristic prove the reaction in the hydration process.

ABSTRAK

Malaysia merupakan salah satu pengeluar terbesar minyak sawit dan jumlah sisa yang dihasilkan oleh kilang minyak sawit yang semakin meningkat dari hari ke hari. Oleh kerana peningkatan sisa abu kelapa sawit banyak kajian telah dilakukan untuk mengitar semula penggunaan abu bahan api kelapa sawit (POFA) di Malaysia sebagai bahan pembinaan bagi menggantikan simen Portland biasa (OPC). Penggunaan POFA yang dihasilkan oleh sisa industri minyak sawit Malaysia boleh menghasilkan bahan pembinaan baru. Ia adalah salah satu penyelesaian untuk mengurangkan kuantiti POFA dilupuskan di tapak pelupusan. Kajian ini memberi tumpuan kepada pembentukan ettringite kedua (DEF), kekuatan mampatan dan mikrostruktur POFA. Empat jenis campuran disediakan. Peratusan POFA yang digunakan adalah 0%, 10%, 20% dan 30% penggantian simen. Konkrit sembuh selama 7, 28 dan 90 hari telah diuji untuk kekuatan mampatan. Spesimen 0% dan 20% daripada POFA pada 28 hari dan 90 hari telah menguji untuk mikrostruktur dengan menggunakan Imbasan Mikroskopi Elektron (SEM) dan Sinar-X Pembelauan (XRD) ujian. Kemudian, semua spesimen telah diuji untuk DEF selepas dipanaskan kira-kira 90 hari. Daripada keputusan yang diperolehi, kekuatan mampatan konkrit untuk 10%, 20% dan 30% dihasilkan sedikit lebih rendah daripada campuran kawalan (0%) pada setiap tempoh umur pengawetan. Spesimen dengan 30% POFA menghasilkan kekuatan mampatan yang paling rendah berbanding dengan yang lain. Kekuatan mampatan maksimum telah ditentukan bagi campuran kawalan (0% daripada POFA) kira-kira 29,349 MPa. Ini menunjukkan bahawa 20% penggantian POFA bagi simen menghasilkan keputusan kekuatan mampatan yang optimum. Keputusan yang dihasilkan daripada ujian XRD menunjukkan bahawa pada 29,411 (darjah) adalah puncak CSH untuk 0% daripada POFA dan 29,546 (darjah) adalah puncak CSH untuk 20% daripada POFA pada 90 hari. Ia menunjukkan bahawa tindak balas berlaku dalam konkrit. Imej yang dihasilkan dari (SEM) menunjukkan mikrostruktur yang POFA di dalam konkrit. Sampel tidak menghasilkan banyak imej, sehingga imej yang tidak begitu jelas logam pada pembesaran yang tinggi. 0% daripada POFA menunjukkan lebih besar dan memanjangkan saiz jarum 'ettringite (2μm) manakala pada 10% kepada 30% daripada penggantian POFA konkrit, jarum' saiz adalah 1μm. Kesimpulannya, POFA boleh digunakan sebagai pengganti simen dalam konkrit. Penggunaan pozolan lebih halus sebagai pengganti simen separa boleh menghapuskan pembentukan 'ettringite dan memberi kesan yang lebih baik kepada konkrit. POFA boleh digunakan sebagai bahan alternatif kerana ia mempunyai ciri-ciri bahan pozzolanic dengan membuktikan tindak balas dalam proses penghidratan.

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LIST OF ABBREVIATIONS

ASTM	-	American Society for Testing and Materials
DEF	-	Delayed ettringite formation
ESA	-	External sulfate attack
FA	-	Fly ash
FELDA	-	Federal Land Development Authority
FELCRA	-	Federal Land Consolidation and Rehabilitation Authority
FKASA	-	Faculty of Civil Engineering and Earth Resources
FKKSA	-	Faculty of Chemical Engineering and Natural Resources
JKR	-	Malaysian Public Works Department
MARDI	-	Malaysian Agricultural Research and Development
	-	Institute
MRR2	-	Middle Ring Road 2
OPC	-	Ordinary Portland cement
PFA	-	Pulverized fuel ash
POFA	-	Palm oil fuel ash
PORIM	-	Palm Oil Research Institute of Malaysia
PORLA	-	Palm Oil Registration and Licensing Authority
RISDA	-	Rubber Industry Smallholders Development Authority
SEM	-	Scanning Electron Microscopy
XRD	-	X-ray Diffraction
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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Cement concrete is generally used as a construction material in the worldwide. Concrete is a mixing of cement and aggregates likes fine and course aggregates. All these materials are building up by specific mixing rate as established. Cement concrete widely used because of its strength, durability, economy and others. The strength of concrete is depends on the curing age, shape surface of aggregate, the use of admixture and others.

Delayed ettringite formation (DEF) can be defined as a cracking and expansion of concrete or formation of ettringite in the cement materials with a cement hydration process after the concrete has hardened. Usually, the concrete related with the delayed formation of mineral ettringite. The mineral ettringite is a normal product at early stage of cement hydration. DEF is occurs from the highly temperature (above 70ºC to 80ºC). Any damage in a concrete that caused by DEF is a common phenomenon. A few external sources needed for the reaction to occur likes water or moisture from surrounding. Taylor et al., (2001) define the formation of ettringite in a cementitious material by a process that begins after hardening in substantially complete and in which none of the sulfate comes from outside the cement paste. DEF also can related to the sulphate attack (Collepardi, 2003). This formation occurs because of the expansion. However, not necessarily any sulphate attack is caused by ettringite formation (Collepardi, 2003). For example, at early

stage of ettringite formation (EEF), it can occurs immediately (within hours) (Collepardi, 2003). Delayed ettringite formation (DEF) usually occurs at late ages. It is related to heterogeneous expansion in a very rigid hardened concrete that can cause cracking and spalling. There is two different mechanisms of DEF. Firstly, it based on the thermal decomposition of ettringite in high temperature cured concrete elements and the subsequent re-formation of ettringite at ambient temperature in a saturated atmosphere. Based on the second mechanism, it is depends on a chain of three essential events such as micro cracking, late sulfate release and exposure to water. Delayed ettringite formation will occur even at room temperature.

An admixture is defined as an ingredient that's added in concrete other than Portland cement, water, and aggregates. Those admixtures are added directly during mixing or before mixing concrete. There are two types of admixtures such as chemical admixtures and mineral admixtures. A chemical admixture likes plasticizers, super plasticizers, accelerators and set retarders. While, a mineral admixture such as POFA, fly ash, silica fume, rice husk and others. Basically, the use of admixture is to reduce the cost of concrete during construction at site. It is also can increase the quality of concrete during mixing and curing.

Today, combination of cement POFA, fly ash (FA), rice husk, silica fume or pozzolanic in concrete become a new practice in construction, especially the using of POFA. The properties of concrete in both fresh and hardened states can be improved when using POFA. It can show the differences in the rate on strength development and formation of ettringite in concrete. When properly rounded and places, POFA concrete widely shows an improvement in workability, durability, ultimate strength, drying shrinkage and others.

Since Malaysia is one of the largest producers in palm oil industry, the wastage of the palm oil can be used in concrete as a partial cement replacement. Nowadays, the use of recycled materials as concrete materials in the development increase because of environmental laws increasingly stringent. In addition, there is a

large study on different materials for the use of cement substitute and replacement such as POFA, fly ash (FA), and others pozzolanic material.

The palm oil industry is one of the most important agricultural industries in Malaysia. In addition to the production of crude palm oil, a big amount of solid waste output from the palm oil industry. As has been proven (Zarina, et al,. 2013) every year, 4 million tons of solid waste from oil palm residues produced The waste are from coconut fibers, shells, and empty fruit bunches. The use of POFA is minimal and not control, while the quantity increases every year and most of POFA are disposed of as waste in landfills causing environmental problems. Many researchers have studied the use of agricultural waste ash as a component of concrete, for example rice husk ash (Ventura, et al., 2015) wood waste ash (Cheah and Ramli, 2011) and bagasse ash (Chusilp et al., 2009). The results showed that agricultural waste ash contained high amounts of amorphous silica in the form and can be used as a pozzolanic material. According to ASTM C 618 (2001) defines pozzolanic materials as containing siliceous or siliceous and aluminous by composition.

POFA is one of the residual ash agricultural chemical composition contains a large amount of silica and that has a high potential to be used as a substitute for cement (Rahmat et al., 2012).

Most of the concrete produced today is a multi-component product that contains one or more additives as an additional to the four basic components of cement, water, fine aggregate and coarse aggregate. For each component, one usually has a few options that can affect the cost of the final product and in service behavior. Among the constituent components, however, cement or cementitious materials as a whole plays an important role in producing the concrete is strong and durable. For various purposes pozzolan which has been considered as a replacement for part of the cement in concrete.

1.2 PROBLEM STATEMENT

Due to the current building in worldwide, many types of problems will be found in concrete building structure. When a few testing is done, usually it shows more than one type of failure towards the building structure. The major problem is crack on the concrete such as the failure of Kuala Lumpur Middle Ring Road 2 (MRR2). In a few study (Ismail, 2013) and (Azlan and Rahman, 2011), they found that the cracking occur because of the formation of ettringite in concrete. MRR2 is build up by Malaysian Public Works Department (JKR) to connect the area around the boundary of Federal Territory of Kuala Lumpur and Selangor. Kepong residents reported that 7000 cracks were founded on 31 from 33 pillars of the viaduct. The viaduct was closed for the third time at 28th pillar on 3 August 2008.Ismail (2013),

However, the crack on the concrete is caused by movement, shrinkage, thermal changes, temperature, DEF and others. DEF is a common problem as many countries have reported about this type of contamination in concrete element. When DEF occurs, it can cause the contraction and expansion that took place on the concrete of a building. For every type of the problems have their own method to solve it. In this study, focus on the DEF which is the problems can reduce by adding some pozzolan (POFA) in concrete and to study the effectiveness of pozzolan towards the formation of DEF. The cracking and expansion of the concrete caused by the delayed ettringite formation can reduces the strength of concrete structures. Moreover the processes that caused by formation of ettringite are not too understood and researchers have proposed many different theories on the causes of DEF (Ismail, 2013). The other focus of this research is to observe and check the structure of crack that caused by ettringite formation.

1.3 RESEARCH OBJECTIVES

The primary objectives of this research is to determine if the growth of DEF in existing concrete can be reduced or even prevented with commercial products (POFA) ,while the sub-objectives of this research are:

1. To study the process of DEF in the concrete.

2. To study the effectiveness of POFA as a partial replacement on compressive strength.

3. To determine the microstructure of POFA in concrete.

1.4 SCOPE OF STUDY

This study concentrated on quality of concrete when adding a special admixture (POFA). The purposes of this study are focus on identifying and observe the initial cracking caused by ettringite formation. Types of raw materials, selection methods and selection product characterization methods have been identified before the study was conducted.

The concrete cube dimension of 0.15 x 0.15 x 0.15 m used for tested the microstructure of palm oil fuel ash (POFA) and formation of DEF. Then, the concrete cube dimensions 0.10 x 0.10 x 0.10 m were tested for compression strength.

In this study, four batches of concrete specimens were tested. POFA was used as a partial cement replacement. The first batch as a control, mean no adding fly ash in concrete. The second batch is the concrete was mixed with 10% of POFA by the total weight of ordinary Portland cement. The third batch is the concrete was mixed with 20% of POFA while the last batch was mixed with 30% of POFA.

Concrete were taken out from the mould after 24 hours. After that, all the specimens were cured on 7, 28 and 90 days. The cubes of concrete from each set were prepared for testing the compressive strength will be measured at 7, 28 and 90 days. The testing conduct accordance to BS1881: Part 119: 1983. The testing that used to detect the microstructure of POFA in the concrete is by using X-ray Diffration (XRD) and Scanning Electron Microscopy (SEM) test. While, Scanning Electron Microscopy (SEM) also is used to detect the formation of ettringite after the concrete was heated about 90 days.

At the end of this research, the result was expected that by using POFA as a partial cement replacement will give a better result towards the DEF and compressive strength of concrete. By using POFA, the formations of ettringite reduce. So, this study was to investigate whether the POFA have a potential as a replacement of cement.

1.5 RESEARCH SIGNIFICANCE

The studies were focused on the effectiveness of the methods implemented for repair work carried out. This study can provide understanding in making the assessment, design and selection of appropriate repair methods. Furthermore, it provides an understanding of how the repair process is acting and assesses the extent of the repair work that it is effectively implemented. Knowledge of how to control the cracking of the concrete is still inadequate at present, (Alfredsson, 2008). This study is also important to provide guidance and solutions for common methods carried out in the repair of concrete structures cracking. The study will also provide guidance in dealing with similar problems in the future.

In the past, coal combustion produced by fly ash was simply entrained in flue gases and dispersed into the atmosphere. The created environmental and health concerns that prompted laws which have reduced FA emissions to less than 1% of

FA produced. Worldwide, coal power stations were produced more than 65% of FA where it is disposed in landfills and ash ponds.

U.S coal fired power plants (2005) reported that 71.1 million tons of fly ash was produced, and 29.1 million tons were reused in various applications. Nowadays, because of increased costs of landfill and the current interest is sustainable development, a lot of research was done to recycle FA utilization in Malaysia.

Due to environmental factors, many studies had conducted on FA recycling to be used as a replacement for Portland cement. But, this study focuses on the compressive strength, hydration process and also the formation of ettringite in concrete.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

Concrete is one of the earliest construction materials that used in construction. Concrete is a combination of cement (commonly Portland cement), fine aggregate, course aggregate and water. All the materials are mixed together due to its proportion as established.

Concrete is one of the most important materials for the most widely used in construction project. Therefore, the techniques used to produce good concrete must be understood and considered carefully. The raw of materials used such as cement, aggregates and water can influence the result of concrete quality. Other than that, the result of concrete quality depends on mixing rate including transportation and ways of compacting. When the raw materials used are not qualified, the resulting concrete will have low standard. Apart from that, the concrete result not strong and also does not meet the specifications. Therefore, all materials should be tested first before used. Concrete technology has determined that all materials must get the approval of the standards set before it can be used in construction work.

Concrete hardens after mixing and pouring process. Water reacts with the cement in a chemical process known as hydration. The water is absorbed by the cement, which hardens, bind with other components simultaneously and eventually forming materials such as stone. Concrete is used more than man-made material on

earth. It is used to make walkways, foundation, road, building structures, parking structures, brick walls and others.

Concrete technology has been modified the properties of concrete by adding pozzolanic materials. A lot of research has been done to find the best admixture of concrete that can improve the quality of concrete likewise fresh and hardened concrete. The pozzolanic materials will help to reduce the cost of project. Otherwise it also can reduce the usage of cement in concrete.

In this chapter include all information about concrete material. The pozzolanic materials needed for this research is POFA.

2.2 MATERIAL TO PRODUCE CONCRETE

2.2.1 Cement

Portland cement is the most commonly used cement for construction. It is the basic ingredient of concrete, mortar and plaster construction. Cement is synthesized through chemical combination that control by calcium, aluminum, iron, silicon and other ingredients.

In cement manufacturing, the common material used likes limestone, chalk, shells, slate, clay, silica sand, blast furnace slag and iron. When heated at high temperature, all the ingredients will form a rock-like substance that is terrain into the fine powder. We commonly think the fine powder as cement.

Joseph Aspdin as a British engineer first made Portland cement in early 1824. It was named for the limestone cliffs on the island of Portland Isle of Portland in England based on its color. The color of cement is likes stone quarried in situ. It consists of a mixture of calcium oxide, aluminum oxide and silicon oxide. Portland cement and similar materials produced by heating limestone (calcium sources) with

mud and clinker, sulfate source (usually gypsum). After mixed with water, the resulting powder will form a solid hydrate with a certain period.

Cement plant laboratories will check every step in the manufacture of Portland cement through frequent chemical and physical tests. In this lab, the finished product will be analyze and test to make sure it follow all specification given by industry.

At high temperature likes oven bricks, usually it is require the use of concrete reflection. This is occur because of Portland cement based concrete can be destroyed or damaged at high temperature, while the concrete refractory are able to withstand such conditions.

Figure 2.1: Ordinary Portland cement (OPC).

2.2.2 Aggregates

The aggregates commonly used for concrete are gravel and natural deposits of sand. Aggregates are divided into two types, which are fine aggregates and course aggregates. The fine and coarse are delivered separately. Both aggregates need to be sieved, so the preparation of mixing of coarse aggregates more expensive

compared to fine aggregates. Crushed aggregate are costly to produce and need more cement paste because of its shape.

There are a few reasons why need to mix fine and course aggregate. One of the reasons is to produce a strong concrete by combining the in correct proportion. A concrete with less voids or spaces in it can be made and it will reduce the quantity of cement that is quite expensive.

2.2.2.1 Course Aggregate

Coarse aggregate is gravel which has been, crushed, sieved and washed. This aggregate need to be sieved, it is to make sure the particles vary from 5 up to 50mm in size. The course aggregate should has rounded shape. The other considerations must be accounted, although the definition seems likes to limit the size of course aggregates.

When proportioned and mixed perfectly with cement, both two groups produce void less that is strong and durable. Due to strength and durability, the aggregates should be better than the hardened cement to support the designed loads and the effects of weathering.

One rule commonly assigned to coarse aggregates is its maximum size. The larger pieces of coarse aggregate can interlink within the concrete form. The maximum size of aggregates must be not larger that the sizes in table 2.1 below.

Table 2.1: Minimum recommended size of coarse aggregate.

Structure	Minimum dimension (mm)
Reinforced cement concrete lintels and slabs (50-120 mm), posts and battens (area 40 cm^2).	15
Unreinforced cement concrete work between 50-150 mm, conglomerate floors, reinforced cement concrete work exceeding 120 mm but not 450 mm.	20
Unreinforced mass cement concrete work on small jobs over 150 mm, for reinforced work over 450mm.	40
Unreinforced mass concrete work on ordinary work.	65

Figure 2.2: Course aggregate

2.2.2.2 Fine Aggregate

Fine aggregate is an extremely needed material in the construction. Fine aggregate is natural sand which is must be clean, impurities and free from waste stone. This aggregate must be sieved to remove particles larger than 5mm. It is very important to know what type of sand that good for construction. Sand can be classified into three different forms which is used specific type of construction. Sand is classified as:

- Fine Sand (0.075 to 0.425 mm)
- Medium Sand (0.425 to 2 mm)
- Coarse Sand (2.0 to4.75 mm).

Figure 2.3: Fine aggregate

2.2.3 Water

Water can be used to produce concrete. The major factor to determine the strength of concrete is the water cement ratio. The best of water cement ratio will produce high strength concrete, while the higher water cement ratio will produce low strength of concrete. Cement paste is a material that formed by a mixed of water and cementations materials which is without aggregate parts or support. Elasticity or viscosity is determined by the water content, amount of cement paste in the total mixture and physical characteristics of aggregates (maximum size, shape and grade).

In cement hydration, the water used to form a binding which is inert aggregate held in termination until the matrix hard. The water remaining represented as a lubricant between fine and coarse aggregates where it make concrete can be used.

Figure 2.4: Tap water