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THE DURABILITY PROPERTIES OF HIGH VOLUME OIL PALM BIOMASS WASTE MORTAR

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Doctor of Philosophy (Civil Engineering)

Faculty of Civil Engineering
Universiti Teknologi Malaysia

MAY 2016

DEDICATION

Praise be to Allah s.w.t, the Lord of the Worlds
All glory and honor to Him

I dedicate this work
to my beloved families especially my late mom, dad, mother and father in law.

I wish to express my sincere and profound gratitude to my beloved husband, Mostafa Samadi for his continuing assistance, the encouragement, guidance, critics and understanding throughout the period of my studies. The trust, patience, great insight, modesty and friendly personality demonstrated by him have always been my source of inspiration.

And also to all who supported me by Doa and work. Thank you for everything.
May Allah bless you. Amin

ACKNOWLEDGEMENT

Praise Be To Allah S.W.T, the Lord of the World

In preparing this thesis, I was in contact with many people, researchers, academicians, and practitioners. They have contributed towards my understanding and thoughts. In particular, I wish to express my sincere appreciation to my thesis supervisors, Prof. Ir. Dr. Mohd Warid Hussin, Assoc. Prof. Dr. Abdul Rahman Mohd Sam and Assoc. Prof. Mohamed A. Ismail for encouragement, guidance, critics and friendship. Without their continued support and interest, this thesis would not have been the same as presented here.

I am thankful to Materials and Structures laboratory, Faculty of Civil Engineering, Universiti Teknologi Malaysia (UTM) and Sustainable Building Materials and Construction lab, Hanyang University, Ansan, Korea for providing the facilities for my research. My thanks also go to Technicians and staff for their assistance in this research. The financial supports using Grant No. 03H36 from Research Management Centre (RMC), UTM, Grant No. 00C91 from Innovation and Commercialisation Centre (ICC), UTM and MyBrain 15, MyPhD from Ministry of Higher Education (MOHE) is sincerely appreciated.

My sincere appreciation also extends to all my colleagues and others who have provided assistance at various occasions. Their views and tips are useful indeed. I am grateful to all my family members.

To all of you, thank you for everything.

ABSTRACT

Research works on the use of pozzolanic waste materials have continued to gain attention worldwide in an attempt to reduce the environmental problem due to carbon dioxide emission from cement manufacturing process. Nowadays, biomass wastes from oil palm industry are abundantly available in many parts of the world. In relation to that an increase in the production of oil palm will create a major disposal problem of the wastes. It is estimated that for every tonne of oil processed, nearly five tonnes of agricultural waste known as biomass are generated including Palm Oil Fuel Ash (POFA), Oil Palm Kernel Shell (OPKS) and Oil Palm Fibre (OPF). This research therefore, focuses on investigating the effects of nano POFA as cement replacement, OPKS as fine aggregates replacement and OPF as tensile strength enhancement on the properties and performance of biomass mortar. The size of the POFA used had been successfully reduced from micromolecular to the nano-size range by ball milling and the LOI was reduced during treatment process. Various tests were carried out to determine the characteristics of materials including X-ray fluorescence, transmission electron microscopy, sieve analysis and balling effect. The effects of biomass waste on fresh and hardened properties of mortar such as hydration temperature, compressive strength, splitting tensile strength, flexural strength, modulus of elasticity, water absorption, total porosity, chloride penetration, acid resistance, sulphate resistance, dry-wet cycle test and ultrasonic pulse velocity were also investigated. Furthermore, various techniques including X-ray diffraction, scanning electron microscopy, thermo gravimetric analysis and fourier transform infrared spectroscopy analysis were used to study the microstructure of the biomass mortar. The results show that the use of 80% nano size POFA has reduced the hydration temperature by 30% and produced higher compressive strength at the age of 28 days by 32% compared to normal mortar. In addition, the compressive strength of the 80% nano POFA mortar at 365 days was 25% higher than its 28 days strength, while the mortar density and porosity was reduced by 50% and 51%, respectively. Thus, grinding the raw POFA to a nano size particle has significantly improved the reactivity of the ash. The inclusion of high volume nano POFA and OPKS reduces the density of mortar due to the low density of the materials itself. The experimental results also showed that the durability and microstructural characteristics of the biomass mortar were significantly improved and better than control mortar. The inclusion of 0.7 % OPF was found to increase the splitting tensile strength and flexural strength up to 69 % and 65 %, respectively, as compared to normal mortar. The overall results revealed that biomass waste with some treatment can be used to produce mortar that is sustainable, higher strength and good durability.

ABSTRAK

Kajian tentang penggunaan bahan pozolana semakin mendapat perhatian di seluruh dunia dalam usaha mengurangkan masalah alam sekitar disebabkan pembebasan gas karbon dioksida dari proses pembuatan simen. Pada masa ini terdapat banyak bahan buangan dari industri kelapa sawit di beberapa tempat di dunia. Sehubungan dengan itu peningkatan pengeluaran minyak kelapa sawit akan menyebabkan masalah pembuangan sisa yang terhasil. Dianggarkan setiap satu tan minyak kelapa sawit akan menghasilkan lebih kurang lima tan bahan buangan pertanian yang dinamakan biomas termasuk abu kelapa sawit (POFA), tempurung kelapa sawit (OPKS) dan serat kelapa sawit (OPF). Oleh itu, kajian ini tertumpu kepada kesan POFA bersaiz nano sebagai pengganti simen, OPKS sebagai pengganti pasir dan OPF sebagai pemangkin kekuatan tegangan terhadap sifat-sifat dan prestasi biomas mortar. Saiz POFA yang digunakan telah berjaya dikurangkan daripada mikro molekul kepada saiz nano dengan menggunakan bola pengisar dan LOI telah dikurangkan semasa proses rawatan. Pelbagai ujian telah dijalankan untuk menentukan ciri-ciri bahan termasuk *X-ray Fluorescent*, *transmission electron microscopy*, analisis ayak dan kesan *balling*. Kesan penggunaan sisa biomas ke atas sifat segar dan keras mortar seperti suhu penghidratan, kekuatan mampatan, kekuatan tegangan, kekuatan lenturan, modulus keanjalan, penyerapan air, jumlah keliangan, penembusan ion klorida, rintangan asid, rintangan sulfat, ujian kitaran kering-basah dan halaju denyutan ultrasonik telah dikaji. Disamping itu, pelbagai teknik, termasuk *X-ray diffraction*, *scanning electron microscopy*, ujian *thermo gravimetric* dan ujian *fourier transform infrared spectroscopy* telah digunakan untuk mengkaji mikrostruktur mortar biomas. Keputusan kajian menunjukkan penggunaan 80% nano POFA telah mengurangkan suhu penghidratan sebanyak 30% dan menghasilkan kekuatan mampatan yang lebih tinggi pada umur 28 hari sebanyak 32% berbanding mortar biasa. Hasil kajian juga menunjukkan kekuatan mampatan untuk 80% nano POFA mortar pada umur 365 hari adalah 25% lebih tinggi daripada kekuatan 28 hari manakala ketumpatan dan keporosan berkurangan masing-masing sebanyak 50% dan 51%. Pengisaran abu mentah POFA kepada saiz zarah nano didapati telah meningkatkan kereaktifannya. Penggunaan isipadu nano POFA yang tinggi dan OPKS telah mengurangkan ketumpatan mortar disebabkan oleh ketumpatan rendah bahan itu sendiri. Keputusan kajian juga menunjukkan ciri-ciri ketahanan dan mikrostruktur mortar biomas telah meningkat dengan ketara. Penggunaan OPF sebanyak 0.7% didapati telah meningkatkan kekuatan tegangan dan kekuatan lenturan masing-masing sehingga 69% dan 65% berbanding dengan mortar biasa. Keputusan keseluruhan menunjukkan bahawa sisa biomas yang telah dirawat boleh digunakan untuk menghasilkan mortar yang mampan, berkekuatan tinggi dan mempunyai ketahanlasakan yang baik.

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LIST OF ABBREVIATIONS

ACI	-	American Concrete Institution
AI	-	Activity Index
ASTM	-	American Society for Testing and Materials
BS	-	British Standard
C-S-H	-	Calcium Silicate Hydrate
C_2S	-	Dicalcium Silicate
C_3A	-	Tricalcium Aluminate
C_3S	-	Tricalcium Silicate
Ca	-	Calcium
$CaCO_3$	-	Calcium Carbonate
CaO	-	Calcium Oxide
$Ca(OH)_2$	-	Calcium Hydroxide
CO_2	-	Carbon Dioxide
DTA	-	Differential Thermal Analysis
E	-	Ettringite
EDX	-	Energy Dispersive X-ray
FTIR	-	Fourier Transform Infrared Spectroscopy
G	-	Gypsum
GGBFS	-	Ground Granulated Blast Furnace Slag
GPOFA	-	Ground Palm Oil Fuel Ash
H_2SO_4	-	Sulphuric Acid
K_2O	-	Potassium
LOI	-	Loss of Ignition
LVDT	-	Linear Variable Differential Transducer
MK	-	Metakaolin

NaCl	-	Sodium Chloride
Na_2SO_4	-	Sodium Sulphate
NPOFA	-	Nano Palm Oil Fuel Ash
OPC	-	Ordinary Portland Cement
OPF	-	Oil Palm Fibre
OPKS	-	Oil Palm Kernel Shell
PFA	-	Pulverized Fuel Ash
POFA	-	Palm Oil Fuel Ash
Q	-	Quartz
RHA	-	Rice Husk Ash
SiO_2	-	Silica
SIRIM	-	Standards and Industrial Research Institute of Malaysia
SEM	-	Scanning Electron Microscopy
SF	-	Silica Fume
TEM	-	Transmission Electron Microscopy
TGA	-	Thermo Gravimetric Analysis
UTM	-	Universiti Teknologi Malaysia
UPV	-	Ultrasonic Pulse Velocity
XRD	-	X-ray Diffraction
XRF	-	X-ray Fluorescence Spectrometer

LIST OF SYMBOLS

A	-	Surface area
d	-	Diameter of specimen
E_c	-	Modulus of elasticity
ε_L	-	Longitudinal strain
ε_T	-	Transverse strain
$\varepsilon(t)$	-	Shrinkage strain at time
f_c	-	Compressive strength
f_{sp}	-	Tensile strength
f_r	-	Flexural strength
L	-	Length of specimen
L_t	-	Distance between transducers
m	-	Mass of composites
P	-	Ultimate compression load
T	-	Effective transit time
V	-	Volume of composites
v	-	Pulse velocity
W_a	-	Percentage of water absorption
W_d	-	Weight of dry specimen
W_w	-	Weight of wet specimen
W_{ssd}	-	Weight of saturated specimen in air
W_{sw}	-	Weight of specimen in water

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Nowadays, concrete has become predominant building material in the construction industry due to its excellent mechanical and physical properties when properly designed and manufactured. More than 10 billion tonnes of concrete is produced annually. As it has been estimated that by the year 2050, the rate of world's population will increase from 1.5 to 9 billions, thus, this will increase the demand for housing as well as the concrete materials which are estimated to be 18 billions tonnes by 2050 (Meyer, 2009). However, with the large quantities of concrete being produced, there are consequences that will affect the environment. It has been found that approximately one tonne of carbon dioxide (CO_2) is released with every one tonne of cement produced. Overall, around 5% of total CO_2 emission all over the world contributes by the cement manufacturing industry (Worrell *et al.*, 2001). Due to the implementation of the Kyoto protocol in February 2005, countries all over the world have to reduce their green house gases emissions. Therefore, the major challenges are to reduce the CO_2 emissions from the cement manufacturing industries.

Previously, researchers have focused on finding materials that have similar properties as cement but more sustainable and affordable. Since cement is the major building material, finding an affordable cost substitution material is a necessity to reduce the overall cost. The need towards sustainability and sustainable environment has made the use of pozzolanic material in construction popular. Within these few years, natural and industrial wastes have been used as construction materials namely, pulverized fuel ash (PFA), ceramic tile waste, ground granulated blast furnace slag (GGBFS), metakaolin (MK), silica fume (SF), palm oil fuel ash (POFA) and rice husk ash (RHA). Their usage as construction materials have achieved appreciable level since they offer advantages in terms of strength and durability (Meyer, 2009; Samadi *et al.*, 2015; Ismail *et al.*, 2013; Ramadhansyah *et al.*, 2012; Bamaga *et al.*, 2013). In addition, these pozzolanic materials also have been proven to improve the properties of concrete and reduce the hydration temperature and hence make the massive construction easier to construct.

Palm oil fuel ash is generally known as one of the pozzolanic material from natural waste. By 2014, Malaysia has recorded a staggering 5.39 million hectares of oil palm plantations, an increase of 11.0% from the previous 4.85 million hectares in 2010 (MPOB, 2015). Malaysia is the second largest producer of crude palm oil in the world, consequently producing a large amount of oil palm waste. It is estimated that "the total potential palm biomass from 4.69 million hectares of oil palm planted area in Malaysia in 2009 is 77.24 million tonnes per year comprising of 13.0 million tonnes of oil palm trunks, 47.7 million tonnes of oil palm fronds, 6.7 million tonnes of empty fruit bunches, 4.0 million tonnes of palm kernal shell and 7.1 million tonnes of mesocarp fibre (all dry weight)" (Ng *et al.*, 2011; Zwart, 2013).

These wastes are usually used as fuel in oil palm mill to generate electricity and produced approximately 5% another solid waste namely, POFA (Tay and Show, 1995). In Malaysia alone, about 3 million tonnes of ashes are generated annually. Even though the wastes have been used for another purpose, there is still left and dumped in open field creating an environmental hazard. Therefore, the utilization of oil palm biomass waste presents an opportunity to make the industry more environmentally safe and sustainable.

1.2 Research Background

The global need for the preservation of natural resources, reduction of CO_2 emission, durability and sustainability of concrete structure have all heightened the search for alternative cementitious materials. POFA is the source of silicate that produced after the combustion of oil palm fibre, shell and mesocarp as fuel to generate electricity (Khalil *et al.*, 2013). POFA as one of the cementitious materials has been widely used in Malaysia since 1990. However, the results showed very low pozzolanic properties and used until 10% replacement (Tay, 1990). It was then continued by Awal and Hussin, (1997) who reported that the development and application of palm oil fuel ash in concrete enabled the replacement of OPC up to 40% without any adverse effect on the strength of concrete. It was a significant development.

Later other researchers also reported that ground POFA can be used successfully as a supplementary cementitious material in concrete due to its good pozzolanic properties (Chandara *et al.*, 2010; Kroehong *et al.*, 2011b; Tangchirapat *et al.*, 2012). The results showed that with the replacement of 5 to 40% POFA with 45 µm size, the compressive strength decreased at the early age but increased after the age of 28 days. However, mortar containing POFA showed better performance in terms of the durability and fire resistance (Jaturapitakkul *et al.*, 2007; Abdullah and Hussin, 2010; Tangchirapat *et al.*, 2012).

Recently, high volume ground POFA (70%) have been studied by Awal and Shehu, (2014) where the results showed similar properties of strength gained at later ages, but lower than OPC mortar. The treated POFA was then studied by Johari *et al.*, (2012) to improve the material properties. A 60% ultrafine size (<1 µm) POFA replacement shows better strength than normal concrete at later age. Furthermore, the hydration rates for micro size POFA have been studied and the results showed that POFA concrete with or without high volume reduced the hydration temperature (Awal and Hussin, 2011; Mehmannavaz *et al*., 2014). This result proved that POFA can be improved and used in high volume provided it is treated well. Even though many studies have been done by other researchers about the partial replacement of cement by POFA, but there is still high amount of ash abundant in the landfills which lead to environmental problems. Therefore, further research on the other possible application of the waste is highly required.

Annually, about 4 million tonnes of Oil Palm Kernel Shell (OPKS) are produced in Malaysia (Mannan and Ganapathy, 2002). During the past 26 years, many researchers focused on the utilization of OPKS as lightweight aggregates to minimize the harmful effect of this waste on the environment (Basri *et al*., 1999; Okpala, 1990; Mannan and Ganapathy, 2001a; Shafigh *et al*., 2011a). The results revealed that OPKS can be used for natural aggregates replacement to produce structural concrete with density of 20 to 25% lower than normal concrete. The mechanical and structural properties of OPKS concrete have been compared with normal weight concrete by many researchers proving the effectiveness of OPKS as coarse aggregate replacement. Generally, the compressive strength of OPKS concrete in the range of 13 to 22 MPa was reported by many researchers (Teo *et al.*, 2007; Alengaram *et al*., 2011b; Alengaram *et al.*, 2013b).

The use of OPKS in concrete resulted in low workability due to the existence of numerous pores in the OPKS surface which are responsible for high water absorption in the range of 14 to 33% (Alengaram *et al,* 2013b). The effectiveness of crushed OPKS with smaller size of 2.36 to 10 mm as coarse aggregates in concrete also have been reported by Alengaram *et al.,* (2010b) where crushed OPKS with smaller size showed better performance compared with normal size. The crushed OPKS showed lower strength loss of approximately 6 to 11% compared with normal size OPKS (Shafigh *et al.*, 2012). However, there is still lack of information on the use of OPKS as fine aggregates replacement. It is believed that by using waste OPKS as fine aggregates replacement can help to preserve the natural resources.

It was reported that the oil palm industry must dispose about 1.1 tonnes of OPF per every tonne of oil produced (Karina *et al*., 2008). Evidently, the use of natural fibre as filler in cement matrix offers several advantages over conventional inorganic fillers in terms of their lower cost, lower density and environmentally beneficial (Shinoj *et al.*, 2011). OPF is hard and tough and its porous surface morphology is useful for better mechanical interlocking with cement matrix bonding. The use of fibres is to control plastic shrinkage and drying shrinkage cracking. Another function of OPF is to lower the permeability of concrete and thus reduce bleeding. Without the fibre, the concrete manifests certain characteristics where it is relatively strong in compression but weak in tension and brittle in nature. The weakness in tension can be overcome by the use of conventional steel reinforcement and to some extent by the inclusion of a sufficient volume of certain types of fibre.

1.3 Problem Statement

The rapid developments in the construction industry in Malaysia have led to huge demand for Portland cement and natural aggregates for construction. However, the use of these materials not only poses environmental risks but depletes the natural resources as well. Therefore, this study was undertaken to find a way to reduce the use of Portland cement and natural aggregates by replacing them with high volume oil palm biomass waste namely POFA, OPKS and OPF. Furthermore, the brittle characteristic of concrete or mortar requires another material to be added to increase the performance life of concrete. Increasing the use of waste materials reduces the use of cement and natural aggregates automatically. Malaysia produces large amount of oil palm biomass waste which is locally available in large quantities. Hence the use of this waste would solve the problem of disposal effectively.

Oil palm biomass waste is a recommended material to be used in order to reduce the CO_2 emission and to recycle the waste materials for saving the environment. Despite the application of POFA as a pozzolanic material in mortar up to 30% as cement replacement, the problem of ash disposal still present as large amount of the ash remains unutilized. Therefore, by replacing cement with high volume nano POFA will improve the strength and durability characteristic of mortar. On the other hand, the addition of OPKS will reduce the density of high filler loading mortar while the addition of OPF solves the issues of brittle characteristic of mortar. In order to completely evaluate the potential of oil palm biomass wastes for new applications, a comprehensive and detailed study of fundamental properties is truly necessary.

1.4 Aim and Objectives

The main aim of this research is to use biomass waste including POFA as cement replacement, OPKS as fine aggregates replacement and OPF as strength enhancement. Hence the measurable objectives of this study were as follows:

i) To characterize the properties of biomass waste from oil palm industries including POFA, OPKS and OPF.

ii) To determine the appropriate mix proportions and its effect on fresh and hardened state properties of biomass mortar.

iii) To examine the effects of biomass waste on strength and mechanical properties of biomass mortar.

iv) To evaluate the durability performance of biomass mortar especially in aggressive environments.

v) To study the microstructural characterization of biomass mortar in relation to strength and durability performance.

1.5 Research Questions

The research work conducted sought to address the following questions:

i) Can nano size POFA with high volume improve the performance of mortar in terms of the strength and durability?

ii) Does OPKS help to reduce the density of mortar without much effect on the strength performance?

iii) Can OPF enhance the splitting tensile strength and flexural strength of the biomass mortar?

iv) What is the effect of microstructure, chemical composition and physical characteristic of NPOFA, OPKS and OPF on the fresh and hardened state properties of mortar?

1.6 Scope of the Research

The research utilizes high volume oil palm biomass waste where the nano POFA as the base material for cement replacement, OPKS as fine aggregates replacement to reduce the density and OPF addition to enhance the flexural and tensile strength in biomass mortar as shown in Figure 1.1. The waste materials were obtained from only one source of Oil Palm Factory, which is situated in Kahang, Johor. As far as possible, the technology and the equipment currently used to manufacture OPC mortar were used to make the biomass mortar. The POFA used had undergone treatment and used in nano size. Replacement levels of NPOFA were 20, 40, 60, 80 and 100% from the weight of cement content. The fine aggregates were then replaced in the percentage of 25, 50, 75 and 100% with OPKS in the size of 300 µm to 2.36 mm. Finally, the OPF added in the range of 0.3 to 0.9%. A substantial number of intensive investigations and analysis were executed as mention in the following paragraphs. These investigations are a representative of the research scope and are limited only to mortar application.

The first phase of the experimental study was based on the preparation and testing of the physical properties, chemical composition and microstructural analysis of materials including POFA, OPKS and OPF. This include the visual analysis, pozzolanic activity index, 45 µm wet sieve, morphology, composite density and particle size using Transmission Electron Microscopy (TEM). It also deals with the determination of chemical composition by using X-Ray Fluorescence (XRF), the determination of the degree of amorphous by using X-Ray Diffraction (XRD) and the degree of weight loss under specific temperature using Thermo Gravimetric Analysis (TGA).

The second phase deals with the mix design and proportioning of the ash replacement level, fine aggregates replacement level and percentage of fibre added. The water binder ratio was fixed at 0.4 and the binder to fine aggregates used was 1:3

ratios. The appropriate mix designs that were close in agreement to the target strength of 30 MPa at 28 days were selected as compared with normal mortar.

The third phase comprised of evaluating fresh and hardened state properties of mortar. The mortar properties included the workability of fresh mortar, setting time, hydration temperature, compressive strength, flexural strength, splitting tensile strength, porosity, water absorption, modulus of elasticity, drying shrinkage and durability in aggressive environment such as chloride penetration, acid attack, sulphate resistance and dry-wet cycles.

The fourth phase deals with the microstructural analysis of mortar paste in relation to strength and durability by using SEM, EDX, XRD, TGA, DTA and FTIR.

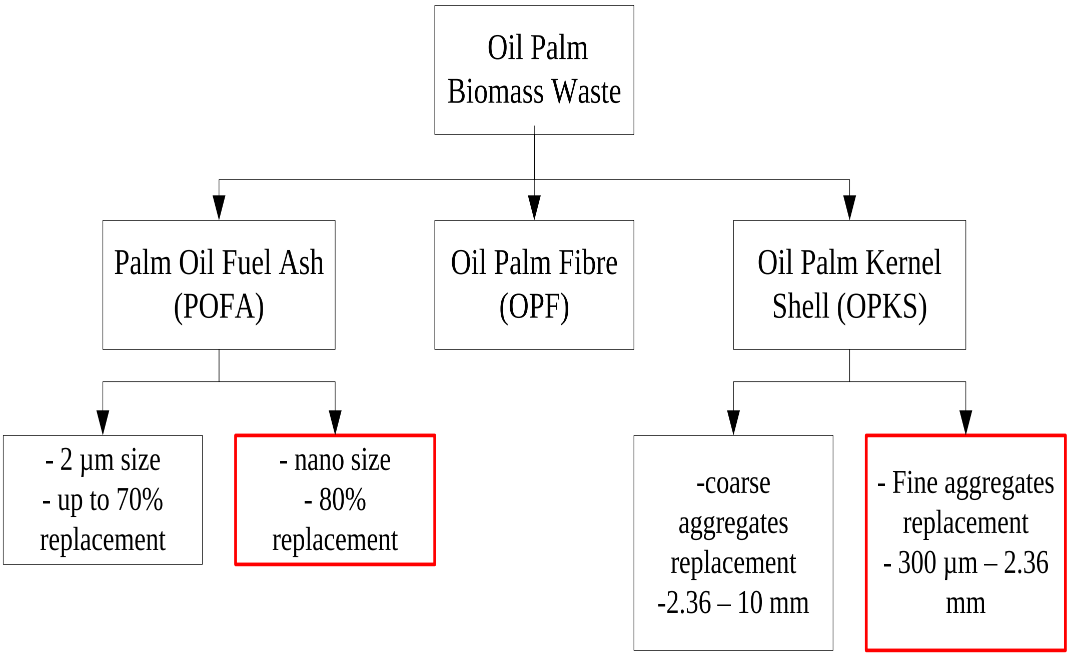

Figure 1.1 Proposed scope of research

1.7 Significance of Study

Since biomass mortar consumed high volume oil palm biomass waste along with the use of high volume NPOFA, OPKS and OPF, a substantial amount of waste generated from oil palm mills thus was diverted from landfill. Replacing high amount of cement is regarded as a move toward low-carbon footprint. Furthermore, replacing an appropriate amount of waste meant the mechanical, deformation and durability properties of biomass mortar could be greatly improved. The significance of using high volume oil palm biomass waste is shown in Figure 1.2.

The use of treated NPOFA, OPKS and OPF in high volume would increase more crystalline formation even at early age since the materials give better performance between reactive silica and cement hydration products. Thus, the porosity and cracks in the mortar will be reduced and the durability performance of mortar will be improved. In addition, with the enhancement of durability performance, the service life of mortar can be extended and cost of maintenance will be reduced. Moreover, immense environmental concerns such as air and water pollution, saving energy, saving natural resources etc. could be possible. Finally, the use of oil palm biomass waste for the production of biomass mortar will open up new research opportunities in the area of concrete technology.

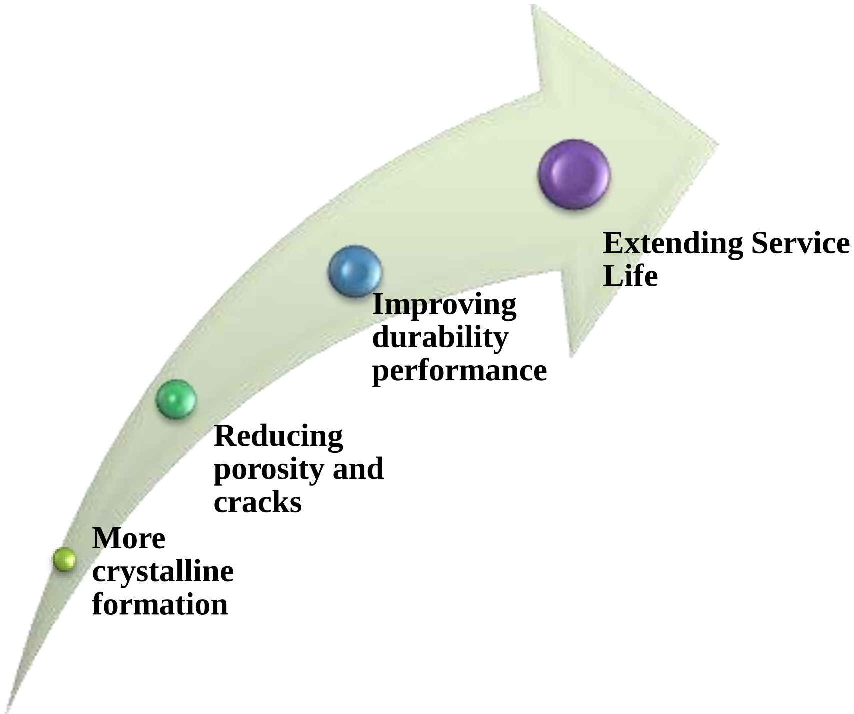

Figure 1.2 Significance of using oil palm biomass waste

1.8 Thesis Organization

The thesis was prepared according to the UTM thesis manual, 2015. Thus, the thesis was designed to consist of seven chapters.

Chapter 1: Provides an introduction of the research, overview about the background of the study to address the problem statement, highlight the aim and objectives of the study and the research methodology. The scope of the study is also clearly mentioned in this chapter as well as the significance of research was also highlighted in this chapter.

Chapter 2: Discusses the relevant and critical review of literature related to the area of research and previous studies that have been conducted by other researchers.

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