PROPERTIES OF CONCRETE WITH MODIFIED EGGSHELL POWDER AND GROUND PALM OIL FUEL ASH AS PARTIAL CEMENT REPLACEMENT

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ABSTRACT

The recent development in the construction industry has shown that some applications requires rapid achievement of early strength such as precast concrete. However, normal concrete achieves maturity at the age of 28 days. One of the methods to attain such early strength is through the incorporation of accelerators in concrete. Most of the accelerators applied in construction industry nowadays are chemically based and might not be environmental-friendly. Therefore, this study focus on the potential use of greener supplementary cementing materials from the avicultural wastes as accelerators. The aim of this study is to investigate the reactivity of Ground Palm Oil Fuel Ash (GPOFA) and eggshell powder (ESP) that contains silicon dioxide (SiO_2) and calcium oxide (CaO), respectively as the cementitious materials in order to achieve the early and later strength development of concrete. Various tests were carried out to determine the characteristics of binder including Differential Thermal and Thermo Gravimetry Analysis (DTA and TGA), X-Ray Fluorescence (XRF) spectroscopy, Scanning Electron Microscope (SEM), Particle Size Analyzer (PSA), X-Ray Diffraction (XRD), and Fourier Transform Infrared (FTIR) spectroscopy. The optimum amount of ESP and GPOFA was also determined based on fresh and hardened properties of concrete such as electrical conductivity, slump, hydration temperature, setting time, apparent density, strength activity index, ultrasonic pulse velocity, cube and cylinder compression strength, flexural strength, splitting tensile strength, chloride ion penetration, sulphate resistance, initial water absorption and carbonation test. The results show that the decarbonation ESP (DC-ESP) demonstrated the highest CaO percentage of 98% with the highest calcination temperature compared to uncarbonized ESP (UC-ESP) and carbonized ESP (C-ESP). The active CaO of ESP was obviously increased when the ESP was exposed to higher calcination temperature. The mix proportion with GPOFA and ESPs as a partial cement replacement was investigated and among all the concretes, G15DC5 (GPOFA 15% and DC-ESP 5%) concrete had achieved a significant increment of average compressive strength with 27% and 23% of improved strength compared to normal concrete and GPOFA concrete during the early and later ages. Furthermore, the DTA and TGA, XRD and SEM test on the paste sample based on optimum ESP and GPOFA concrete proven that the energetic effects of combination CaO and SiO_2 had dual function ability because its provide calcium hydroxide (($Ca(OH)_2$) and calcium silicates hydrate (CSH) gel during the early and later strength development of concrete. The abundant amount of ready stock active CaO in DC-ESP has produced more $Ca(OH)_2$ during cement hydration and produced more CSH gel during pozzolanic reaction. In addition, the concrete with optimum ESP and GPOFA also had demonstrated the least defects after the immersion in the sodium chloride solution, the highest compressive strength during sulphate resistance test, had the lowest value of absorption during initial surface absorption test and slightly higher depth in carbonation test. By combining pozzolan materials (GPOFA) with DC-ESP was found to have a superior synergy-reactivity for early and later age of concrete strength development. Hence, this study provides a positive impact for rapid construction technology. As a conclusion, modified ESP is a highly potential as partial cement replacement and accelerator for concrete.

ABSTRAK

Perkembangan terkini dalam industri pembinaan telah menunjukkan bahawa beberapa aplikasi memerlukan pencapaian kekuatan awal konkrit yang cepat seperti konkrit pratuang. Walau bagaimanapun, konkrit biasa mencapai kematangan pada usia 28 hari. Salah satu kaedah untuk mencapai kekuatan awal konkrit adalah dengan penggunaan pencepat dalam konkrit. Kebanyakan bahan pencepat yang digunakan dalam industri pembinaan pada masa kini adalah berasaskan bahan kimia dan tidak mesra alam. Oleh itu, kajian ini memberi tumpuan kepada potensi penggunaan bahan pengganti simen yang lebih mesra alam dari sisa penternakan sebagai bahan pencepat. Tujuan kajian ini adalah untuk mengkaji keaktifan Bahan Bakar Kelapa Sawit Dikisar (GPOFA) dan serbuk kulit telur (ESP) yang masing-masing mengandungi silikon dioksida (SiO_2) dan kalsium oksida (CaO) sebagai bahan bersimen untuk mencapai perkembangan kekuatan awal dan akhir konkrit. Kerja pencirian pada ESP telah dilakukan di bawah *Differential Thermal* dan *Thermo Gravimetry Analysis* (DTA/TGA), *X-Ray Fluorescence* (XRF) spektroskopi, *Scanning Electron Microscope* (SEM), *Particle Size Analyzer* (PSA), *X-Ray Diffraction* (XRD) dan *Fourier Transform Infrared* (FTIR) spektroskopi. Jumlah ESP dan GPOFA yang optimum juga telah ditentukan berdasarkan sifat segar dan keras konkrit seperti kekonduksian elektrik, ujian penurunan, suhu penghidratan, masa pengerasan, ketumpatan ketara, index aktiviti kekuatan, halaju denyut ultrabunyi, kekuatan mampatan kiub dan silinder, kekuatan lenturan, kekuatan tegangan, penembusan ion klorida, rintangan sulfat, penyerapan air dan ujian karbonasi. ESP nyahkarbon (DC-ESP) menunjukkan peratusan CaO yang paling tinggi iaitu sebanyak 98% dengan suhu pengkalsinan yang tinggi berbanding ESP tanpa berkarbon (UC-ESP) dan ESP berkarbon (C-ESP). CaO yang aktif jelas meningkat apabila terdedah kepada suhu pengkalsinan yang tinggi. Nisbah kandungan campuran GPOFA dan ESP sebagai separa pengganti simen telah dikaji dan di antara semua konkrit, konkrit G15DC5 (GPOFA 15% dan DC-ESP 5%) telah mencapai purata kekuatan mampatan dengan peningkatan kekuatan sebanyak 27% dan 23% berbanding konkrit biasa dan konkrit GPOFA pada usia awal dan akhir perkembangan konkrit. Di samping itu, ujian DTA dan TGA, XRD dan SEM pada adunan sampel berdasarkan konkrit ESP dan GPOFA yang optimum membuktikan bahawa kesan bertenaga antara kombinasi CaO dan SiO_2 mempunyai keupayaan fungsi berganda kerana memberikan kalsium hidroksida ($(Ca(OH)_2$) dan gel kalsium silikat hidrat (CSH) semasa usia awal dan akhir perkembangan konkrit. Jumlah CaO yang aktif di dalam DC-ESP telah menghasilkan lebih $Ca(OH)_2$ semasa penghidratan simen dan gel CSH semasa tindak balas pozzolanik. Tambahan pula, konkrit dengan kandungan ESP dan GPOFA yang optimum juga telah menunjukkan kecacatan yang paling sedikit selepas direndamkan ke dalam larutan natrium klorida, kekuatan mampatan yang paling tinggi semasa ujian rintangan sulfat, nilai penyerapan yang paling rendah semasa ujian penyerapan air dan nilai yang sedikit tinggi kedalaman dalam ujian karbonasi. Penggabungan bahan pozzolan (GPOFA) dengan DC-ESP menunjukkan ia mempunyai kereaktifan yang unggul untuk usia awal dan akhir perkembangan kekuatan konkrit. Oleh itu, kajian ini memberikan impak yang positif kepada teknologi pembinaan yang pesat. Kesimpulannya, ESP yang diubahsuai sangat berpontensi sebagai bahan separa pengganti simen dan pencepat untuk konkrit.

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LIST OF ABBREVIATIONS

ASTM	-	American Society for Testing and Materials
BS	-	British Standard
BET	-	Brunauer/Emmett/Teller nitrogen absorption test
$CaCO_3$	-	Calcium carbonate
CaO	-	Calcium oxide
$Ca(OH)_2$	-	Calcium hydroxide /Portlandite
CH	-	Calcium hydroxide /Portlandite
CO_2	-	Carbon dioxide
C-S-H	-	Calcium silicate hydrate
C_2S	-	Belite
C_3S	-	Alite
C_3A	-	Tricalcium aluminate
C_4AF	-	Tetracalcium aluminoferrite
C-ESP	-	Carbonized eggshell powder
CSA	-	Calcium sulpho-aluminate
CFA	-	Classified fly ash
DC-ESP	-	Decarbonized eggshell powder
DTA	-	Differential thermal analysis
ESA	-	Eggshell ash
ESP	-	Eggshell powder
EDX	-	Energy dispersive X-ray
FTIR	-	Fourier transform infrared
GPOFA	-	Ground palm oil fuel ash
GGBS	-	Ground granulated blast-furnace slag
ISAT	-	Initial surface absorption test
LOI	-	Loss on ignitions
NC	-	Normal concrete
NP	-	Normal paste
NaCl	-	Sodium chloride
Na_2SO_4	-	Sodium sulphate

POFA	-	Palm oil fuel ash
PSA	-	Particle size analyzer
Q	-	Quartz
RFA	-	Run-of-station fly ash
SAI	-	Strength activity index
SCM	-	Supplementary cementing materials
SiO_2	-	Silica oxide
SEM	-	Scanning electron microscope
SSD	-	Saturated surface dry
TGA	-	Thermogravimetry analysis
UC-ESP	-	Uncarbonized eggshell powder
UPOFA	-	Unground palm oil fuel ash
UPV	-	Ultrasonic pulse velocity
XRD	-	X-Ray Diffraction
XRF	-	X-Ray fluorescence

LIST OF SYMBOLS

A_C	-	Cross sectional area
D	-	Cross sectional dimension
E_c	-	Compression young's modulus of concrete
F	-	Maximum load
f_c	-	Flexural strength
f_t	-	Tensile strength
L	-	Length of specimen
ρ_c	-	Apparent density
V_c	-	Volume of cured concrete
W_c	-	Mass of cured concrete

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Generally, concrete is one of the most important building materials widely used in the construction industry. The major component in a normal concrete mixture consists of cement with fine and coarse aggregates together with water. Cement is an important ingredient in concrete which acts as a binding agent. The concrete strength development is divided into two phases, i.e. the early strength development and later strength development. Early strength define as the compressive strength of the concrete at the first 24 hours after achieve concrete quality (Yasin et al., 2017). Meanwhile, later strength development is ability of pozzolanic reaction that consumes the calcium hydroxide ($Ca(OH)_2$), available in cement and develop calcium silicate hydrate (C-S-H) gel which are responsible for gaining strength in concrete (Khalid et al., 2018). Portland cement is one of the commonly used cement with alite and belite are among the major constituents in modern Portland cement. During the early strength development, alite is very reactive in the presence of water. It is the main strength-giving component of cement. However, belite is less reactive at early ages but contributes appreciably to concrete strength at later strength development. These behaviour at early and later strength development of concrete could be altered when any suitable cementitious materials are added into the concrete (Liew et al., 2017). In chemistry, reactivity is a measure of how readily a substance undergoes a chemical reaction. The reactivity between alite and belite reaction which is important constituent about 70 to 80 % of the cement were control most of the strength development of concrete properties (Abdul Awal and Abubakar, 2011). The reactivity of pozzolanic reaction only occurred when the reactive silica material in the concrete is used. Reactive silica is the principal parameter which determines the tendency of potential pozzolanic reaction that can be found in the silica material (Antiohos and Tsimas, 2007).

In this rapid construction development era, there is a huge demand to obtain the early strength development of concrete such as in precast concrete. Calcium carbonate ($CaCO_3$) is a chemical substance found in rocks most notably as limestone (Cree and Rutter, 2015) and the addition of $CaCO_3$ could contribute to higher early strength in concrete (Antoni et al., 2015). Generally, limestone and clay are the major constituents in the production of Portland cement. However, carbon dioxide (CO_2) is released during cement production, whereby approximately 1 tonne of CO_2 (greenhouse gasses) are generated in the chemical conversion process to produce 1 tonne of cement. This will lead to high energy consumption during the cement production process (Aprianti et al., 2015). The strength development of concrete with different types of materials is shown in Figure 1.1.

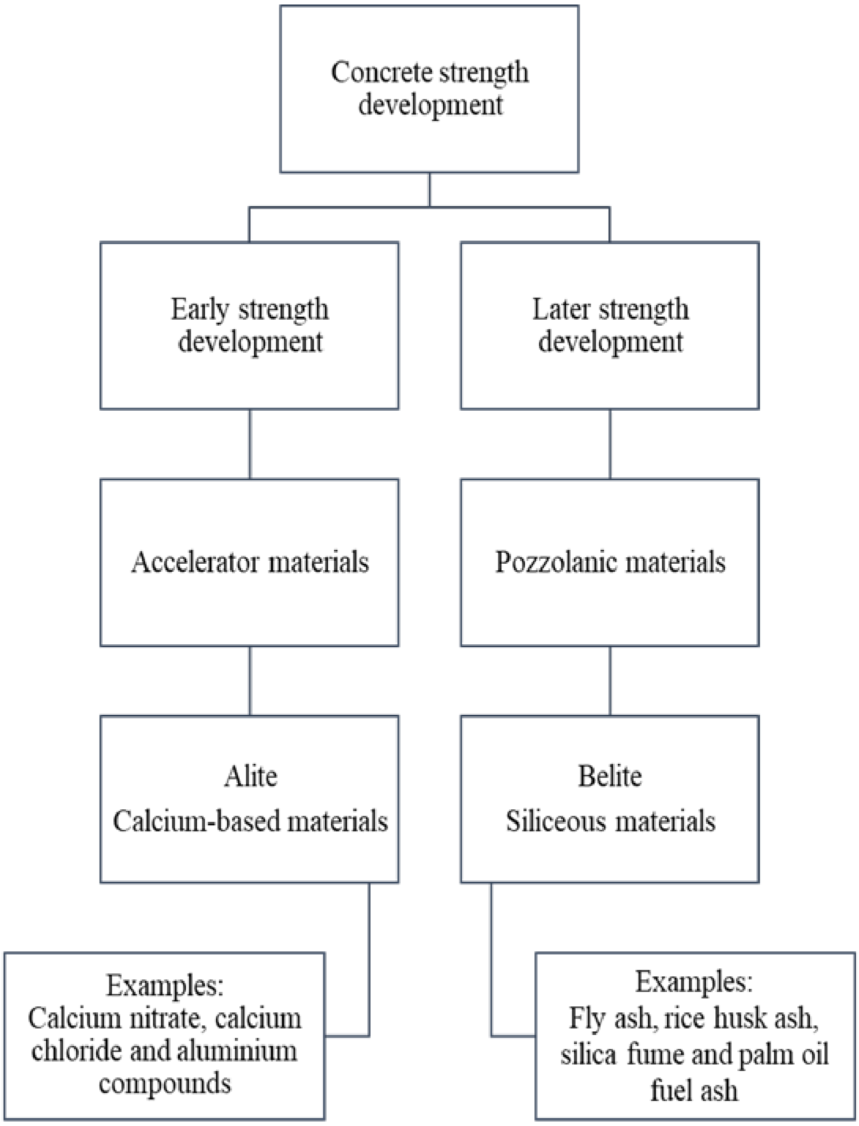

Figure 1.1 Phases of concrete strength development (Winter, 2012)

1.2 Background of Study

In the recent development of the construction industry, some applications require the rapid achievement of early strength such as precast concrete. One of the methods to attain such early strength is through the incorporation of accelerators in concrete. As such, most of the accelerators applied in concrete development nowadays are chemically based and might not be environmental-friendly. Therefore, the research focus of the concrete industry has shifted to the potential use of greener supplementary cementing materials from the agricultural or avicultural wastes as accelerators. These alternatives are essentially important as it has resolved the waste disposal issues in the agricultural industry while simultaneously creating greener cementitious materials which could be beneficial to the concrete industry.

The research on the implementation of supplementary cementing materials (SCM) in the cement matrix has grown rapidly. For instance, the addition of ground granulated blast-furnace slag (GGBS) and fly ash as partial cement replacement in concrete, which are high in calcium oxide (CaO) and silicon dioxide (SiO_2), has resulted in the enhancement of compressive strength during early and later stage (Abhilash et al., 2016). The utilization of SiO_2 - CaO has been a new method to improve the concrete strength for the development of dual-function concrete (early and later strength concrete). In view of the development of composite materials have enhanced strength and durability as compared to conventional materials, and the environmental sustainability benefits have become very important in construction fields, green composite materials have sparked the interest of experts from various fields to develop sustainable composite materials to become superior construction materials (Laca et al., 2017).

In Malaysia, large amount of waste generated from palm oil mills and other Asian-Pacific is commonly used as landfills due to lack of economically attractive use opportunities. Furthermore, Malaysia aspires to maintain its leadership in palm oil output, increasing palm oil plantation from 400 hectares in 1920 to around 3.6 million in 2002, with a goal expansion of 5.2 million by 2020. (Basiron and Simeh, 2005). As a result, it is expected that more waste will be discharged to the environment by industry in the near future. To address the problem of waste generating from various palm oil mills in Malaysia, it has become vital to discover technically possible and economically befitting cost-effective solutions to waste disposal from the sector by means of changing wastes into low or ideal high value products. Reuse and recycling of waste will not only save the huge disposal costs, but also save natural resources, and in some situations, bring technical benefits. As a result effort in numerous forms of exploitation. Ash has shown to be a valuable material, particularly in construction. Some of the benefits of using palm oil fuel ash (POFA) in concrete mix include; improved workability, reduced permeability (Sumadi and Hussin, 1995), early strength may be depressed with an increased ultimate strength (Tangchirapat et al., 2009). Furthermore, the ash played a positive role in the reduction of bleeding in concrete (Tay and Show, 1995), better surface finish and influenced heat of hydration (Abdul Awal and Abubakar, 2011).

Laboratory tests have revealed that this ash not only allowed for the replacement of standard Portland cement, but also played an important role in managing concrete durability problems (Tangchirapat et al., 2009). Research initiated on palm oil fuel ash at the Faculty of Civil Engineering, Universiti Teknologi Malaysia has identified POFA as a pozzolanic material. Studies elsewhere have also confirmed (Tangchirapat et al., 2009; Sata et al., 2004; Tay and Show, 1995) the research findings of the institution. Therefore, an effort to explore the potentials of application POFA in concrete as it can be potential as partial cementitious replacement materials in the concrete.

In this study, bio-materials such as palm oil fuel ash (POFA) were considered as the cementitious replacement materials in the concrete. Palm oil fuel ash (POFA) can be categorized into two types; unground palm oil fuel ash (UPOFA) and ground palm oil fuel ash (GPOFA). POFA has a natural open cellulose/porous structure which has resulted in the decrement of concrete strength. After a thoroughly- ground process, the cellulose structure can be broken. The GPOFA materials should undergo some modification via the grinding process to get sufficient fineness in order to obtain comparable fineness to the cementitious materials. Therefore, only GPOFA was considered as cement replacement in concrete for this study. Besides, GPOFA is enriched with silica (SiO_2) content which could speed up the concrete compressive strength at a later age (Skariah et al., 2017).

In addition for this study, the chicken eggshells waste was selected as the composite materials with GPOFA due to its excellent properties and the rich content of calcium oxide (CaO), which could act as catalysts and help in accelerating the early strength of concrete development (Laca et al., 2017). There are some transformation processes that need to be done to convert eggshell waste to eggshell powder (ESP) under a thorough calcination process, in which three types of ESP were produced under various calcination temperature range in order to obtain uncarbonized eggshell powder (UC-ESP), carbonized eggshell powder (C-ESP) and decarbonized eggshell powder (DC-ESP). Several studies have investigated the effectiveness of UC-ESP and C-ESP on concrete (Hassan and Aigbodion, 2015). However, no previous study has used this investigation on the effect of decarbonized ESP in the concrete. Therefore, this is potential area for developing a new material by investigating the DC-ESP in concrete as well. Hence, the combination of GPOFA and ESP is seen as a highly preferable green cementitious material in enhancing the dual strength of concrete, in which the combination of SiO_2-CaO derived from GPOFA-ESP could act as booster or accelerator for both early and later compressive strength in blended cement concrete.

Therefore, this research was conducted for the enhancement of concrete properties with the incorporation of agricultural waste (palm oil fuel ash) and avicultural waste (chicken eggshell) as alternative materials in order to make cement greener, environmentally safe and sustainable. The focus of the study was on the transformation of ESP with different calcination temperature range from 0 ºC to 250 ºC (UC-ESP), 251 ºC to 800 ºC (C-ESP), 800 ºC and onwards (DC-ESP). The GPOFA-ESP is an essential alternative to conventional cementitious materials which only contribute to either early compressive strength or later compressive strength. The green cementitious materials produced by GPOFA and ESP have definitely become a popular trend as cement replacement materials due to their dual-function advantage which could lead to a significant contribution in the building and construction industry and at the same time observing the environmental sustainability values by converting agricultural wastes into value-added products.

1.3 Problem Statement

Generally, a normal cement concrete achieves maturity age at 28 days. However, the normal cement has demonstrated low durability performance. A pozzolanic material rich with silicon dioxide (SiO_2) is considered as a partial cement replacement in the concrete mix in order to achieve higher strength and durability. However, the maturity age of concrete with SiO_2 could only extend up to 56 days. Despite being durable, the occurrence of double phenomenon (maturity and durability) in concrete may cause a prolonged or delay of construction. This delaying problem can be solved by using an accelerator or catalyst, but the safety of the materials used need much consideration since they are chemically based materials.

According to the concrete strength development as shown in Figure 1.1, there are two major stages of strength development namely, at the early stage and later stage. These strengths development is governed by different types of the accelerator and pozzolanic materials used in the mix. The accelerators for concrete early strength development could be enhanced by alites which contains calcium-based materials. Common accelerators used in the cement industry nowadays are commercial materials such as calcium nitrate, calcium chloride and aluminium-based compounds. On the other hand, the pozzolanic materials which are generally used in the industry are belites with siliceous materials such as ground granulated blast-furnace slag, silica fume, fly ash and natural pozzolans like calcined shale, and calcined clay or metakaolin.

Despite the availability together with costs of commercial or natural pozzolans and accelerator materials, the use of alternative waste materials from agricultural or avicultural industries is gaining attention nowadays in order to achieve environmental sustainability. In the study by Munir et al. (2015), palm oil fuel ash (POFA) from the palm oil industry is chosen to be the alternative constituents as partial cement replacement due to its pozzolanic properties. These agro-waste ashes from the palm oil industry contain a significant amount of silica that has perfectly the pozzolanic properties which are essentially needed for concrete strength development. POFA has a high potential to be utilized as partial cement replacement in order to produce concrete with great strength and durability. Besides, POFA is generally used to produce various types of concrete such as high strength concrete, lightweight concrete and foamed concrete (Munir et al., 2015). However, POFA could only contribute to the later strength of concrete. The strength development of concrete is influenced by the fineness of POFA. Awal and Abubakar, (2011) reported that for same replacement of POFA in concrete, finer POFA with 45 µm size would lead to greater strength development than the coarser POFA (Awal and Abubakar, 2011).

Apart from that, the poultry waste from avicultural industry is also seen a popular alternative as the cementitious materials replacement in concrete. One of the common waste materials used is calcium-rich eggshell. Eggshell waste is highly preferable to be the natural lime replacement in concrete due to the presence of a large amount of calcium carbonate, which has a high similarity with the calcium content in the commercial accelerator materials. The utilization of eggshell waste is highly recommended in cement replacement due to several advantages such as the binding properties and its ability to enhance the stability in subgrade soil (Tan *et. al*., 2018). Additionally, eggshell waste also acts as a great accelerator in expediting the early strength development of concrete. In view of the need to conserve natural limestone and to minimize cement usage, it is vital to promote the utilization of waste materials as cement replacement components. Since ESP was derived from calcium carbonate compound, thus the heating process was required in order to transform calcium carbonate into active calcium oxide. The high calcium oxide content has great potential to be cementitious accelerators in concrete since the pozzolanic reaction only occur involved calcium oxide reaction.

Consequently, it is highly recommended to produce cementitious replacement materials from the agro-waste as mentioned earlier which could contribute to both the early and later strength of concrete. One of the alternatives to solve this problem is by selecting green materials that could help in boosting the early age strength of concrete (acting as a catalyst), such as bio-lime inspired eggshell that is rich in CaO and the enhancement in later concrete strength with materials that are rich in SiO_2 such as GPOFA. This study presents the idea by utilizing a combination of bio SiO_2-CaO, which is a new trend in providing the dual function of early and later concrete strength development. Two materials of SiO_2-CaO will be introduced which are Palm Oil Fuel

Ash (POFA) and Eggshell Powder (ESP). Both materials are from agriculture and aviculture waste, respectively, that are abundantly available in Malaysia and also South-East Asia countries. The combination of bio SiO_2-CaO is highly preferable as ESP has the potential to boost up the development of early-age strength of concrete without changing the overall concrete properties. Hence, this study provides an impact for rapid and green construction technology towards materials sustainability.

Besides, precast concrete materials have been extensively utilized in civil infrastructure as it helps to reduce construction time and environmental benefits compared to cast-in-place concrete. However, this advantage also brings to setback if the quality of individual components does not achieve good early concrete strength. There was about 5–16% of the construction cost spent on the maintenance or repair in order to resolve the defects associated with the poor quality of concrete materials at the last stage of construction (Kim et al., 2014). Hence, it is important to ensure that the quality of precast concrete materials is well performed at an early stage. Therefore, this study was undertaken to determine the best way to achieve excellent concrete strength development in both early and later ages by altering the cement properties through the use of waste materials.

1.4 Aim and Objectives

The aim of this study is to investigate the reactivity of GPOFA and ESP that contains silica (SiO_2) and calcium oxide (CaO), respectively in the cementitious materials towards the early and later strength development. Four (4) main objectives were constructed to achieve the aim of this study:

1. To characterize the properties of three different forms of eggshell powder (ESP), i.e. uncarbonized ESP (UC-ESP), carbonized ESP (C-ESP) and decarbonized ESP (DC-ESP).
2. To determine the optimum proportioning ratio of GPOFA with different forms of ESP (GPOFA-ESPs) in reactive silica concrete.

3. To investigate the reactivity of GPOFA-ESPs concrete in terms of mechanical and microstructural properties.

4. To evaluate the durability performance of GPOFA-ESPs concrete especially in aggressive environments.

1.5 Scope of the Study

The scope of this study was established to achieve the objectives by using bio-lime as cement replacement in POFA concrete. All the experimental procedures were specified according to several standards, i.e. the British Standard (BS), Eurocode Standard (BS-EN), American Society for Testing and Materials (ASTM), and other recommended test procedures proposed by previous researchers. In this study, GPOFA and three categories of ESP (i.e. UC-ESP, C-ESP, DC-ESP) were used as partial cement replacement and their microstructural properties and performance were evaluated.

The raw materials such as UC-ESP, C-ESP and DC-ESP were prepared and these materials was evaluated based on several microstructural analyses. The tests were includes; differential thermal and thermogravimetry analysis (DTA/TGA), X-Ray Fluorescence (XRF) spectroscopy, Scanning Electron Microscope (SEM), particle size analyzer (PSA), X-Ray Diffraction (XRD) spectroscopy and Fourier Transform Infrared (FTIR) spectroscopy test. The main contribution of these tests is to identify the significant microstructural properties and the differences between each material and its beneficial effect as cementitious replacement material.

Subsequently, the optimum proportioning of the concrete mixture with the presence of GPOFA, UC-ESP, C-ESP and DC-ESP as cement replacement was investigated. The replacement level of both combination materials was from 0 to 20%. This phase comprised of the properties evaluation of fresh and hardened state concrete. In the first part before the investigation on fresh and hardened concretes, all the materials were investigated under the electrical conductivity test. This test gave comprehensive results on the cementitious reactivity behaviour. Then, the evaluation of fresh and hardened concrete tests conducted includes the workability, ultrasonic pulse velocity (UPV), apparent density and compression strength test. The specimens were cured for 1, 3, 7, 28, 56 and 90 days. The early strength development of concrete was determined by concrete cured at the age below 7 days, while for the later strength development of concrete was determined using concrete cured at the age after 28, 56 and 90 days. After the optimum mix proportions of the specimens were determined, strength activity index (SAI), setting time and hydration temperature test were conducted.

The reactivity analysis of GPOFA-ESP concrete was conducted based on the desired mix proportion specimens. In this phase, the reactivity of GPOFA concrete containing different forms of ESP, i.e. UC-ESP, C-ESP and DC-ESP were tested. The tests conducted were mechanical and microstructural analyses. For the mechanical test, compressive strength, flexural strength and splitting tensile strength tests were conducted. The specimens were tested at the age of 1, 3, 7, 28, 56 and 90 days. Normal concrete was tested as a control experiment in comparison with the mixed-proportion concretes. The microstructural analyses of the cement paste were tested using XRD, TGA / DTA and SEM, EDX.

Lastly, the durability performance of GPOFA-ESP was evaluated based on chloride ion penetration test and sulphate resistance test. The samples after the destructive test were used for the microstructural analysis. The initial surface absorption and carbonation of concrete tests were also conducted.

1.6 Significance of the Study

The utilization of GPOFA-ESP as partial cement replacement in concrete is an essential and sustainable alternative to enhance the cement properties which could contribute to the building and construction industries. The ESP which acts as a bio-lime, possesses superb properties that might contribute significantly to the study. The significance of this research are as follows:

1. High volume utilization of waste materials such as POFA and ESP as an attractive alternative for partial cement replacement in concrete.

2. Enrichment on concrete properties especially in the early strength development for any blended cement concrete.

3. Improvement on the quality of normal concrete properties with bio-lime as cement replacement and its applicability evaluation in the building and construction industry by microstructural testing.

4. Promote environmental awareness by reviving the local agricultural waste with less carbon dioxide emission and reduce the negative impact on human health during cement production.

5. Encouragement on the innovative usage of waste material in agricultural industry for the development of value-added products in construction industry.

1.7 Thesis Outlines

This research is presented in eight chapters to achieve the research aim and four objectives. This thesis has been structured to present the research in such arrangement:

Chapter 1: Provides an introduction of the research, overview of the background of the study to address the problem statement. In addition, the aim and objectives are also highlighted and the research question. The scope of the study is also clearly mentioned as well as the significance of the 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.

Chapter 3: Provides the materials used, processes and methodology that is employed to conduct the research.

Chapter 4: Focused on the characterization of the raw materials (Portland cement, GPOFA and different forms of ESP) including the test of differential thermal and thermogravimetry analysis (DTA/TGA), X-Ray Fluorescence (XRF) spectroscopy, particle size analyzer (PSA), Scanning Electron Microscope (SEM), Fourier Transform Infrared (FTIR) spectroscopy and X-Ray Diffraction (XRD) spectroscopy test.

Chapter 5: Reveals the results of fresh and hardened state properties of the concrete mixture. Parameters studied in this chapter include conductivity behaviour of binder materials, workability in terms of the slump of fresh concrete, apparent density, ultrasonic pulse velocity and cube compressive strength. It also presents the results obtained from the strength activity index, setting time and hydration behaviour of concretes.

Chapter 6: Consists of the results and discussion arising from the cementitious reactivity of concrete mixtures containing OPC, GPOFA and different forms of optimized ESPs. Tests falling in this category include; compressive, flexural and splitting tensile strength. Microstructure involving XRD, TGA/DTA and SEM results are presented and discussed in this chapter.

Chapter 7: Discusses the durability performance of concrete considered in this chapter. The tests include chloride penetration, sulphate attack, initial surface absorption and carbonation of concrete test.

Chapter 8: Discusses the conclusion and recommendations based on the research findings.

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LIST OF PUBLICATIONS

Conference papers

1. **Rasid, N. N. A.**, Sam, A. R. M., Mohamed, A., Shukor Lim, N. H. A., Majid, Z. A. and Khalid, N. H. A. (2020) 'The Effect of Eggshell Powder as an Accelerator for Blended Cement Concrete', Journal of Computational and Theoretical Nanoscience.

2. Abd Khalid, N. H., **Abdul Rasid, N. N**., Mohd Sam, A. R., Abdul Shukor Lim, N. H., Zardasti, L., Ismail, M., Mohamed, A. and Majid, Z. A. (2019) 'The hydration effect on palm oil fuel ash concrete containing eggshell powder', in IOP Conference Series: Earth and Environmental Science.

3. Khalid, N. H. A., **Rasid, N. N. A**., Mohd.Sam, A. R., Lim, N. H. A. S., Ismail, M., Zardasti, L., Mohamed, A., Majid, Z. A. and Ariffin, N. F. (2018) 'Characterization of palm oil fuel ash and eggshell powder as partial cement replacement in concrete', IOP Conference Series: Materials Science and Engineering, 431, p. 032002.