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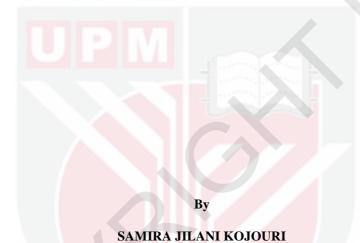
DEVELOPMENT OF HIGH VOLUME POFA SELF- COMPACTING CONCRETE USING COLLOIDAL NANO SILICA AS A VISCOSITY MODIFYING AGENT

SAMIRA JILANI KOJOURI

FK 2016 55



DEVELOPMENT OF HIGH VOLUME POFA SELF- COMPACTING CONCRETE USING COLLOIDAL NANO SILICA AS A VISCOSITY MODIFYING AGENT



Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirement for the Degree of Doctor of Philosophy

May 2016

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

DEVELOPMENT OF HIGH VOLUME POFA SELF- COMPACTING CONCRETE USING COLLOIDAL NANO SILICA AS A VISCOSITY MODIFYING AGENT

By

SAMIRA JILANI KOJOURI

May 2016

Chairman : Farah Nora Aznieta Binti Abd Aziz, PhD Faculty : Engineering

This study delineates the effects of nano silica on self-compacting concrete (SCC) with high volume replacement of palm oil fuel ash (POFA). POFA is an agro waste found in hefty amounts in Malaysia. It is a by-product of burning residues of palm oil industry which reportedly has pozzolanic behavior. Pozzolanic mineral admixtures are used in concrete to enhance the mechanical properties and the durability. However, the addition of mineral admixtures especially in high volumes reduces the workability and increases the demand for superplasticizer. The negative effect of the explained phenomenon is of great importance in SCC in which high workability as well as cohesiveness is required. Furthermore, inclusion of mineral admixture as a replacement for cement brings about the delay in hydration for which unbound water increases in the matrix at early ages. This increases the risk of segregation and bleeding and as a result the hardened properties of concrete including the mechanical properties and the durability are disadvantageously overshadowed.

The overarching purpose of this study was to overcome the above mentioned demerits of inclusion of high volume POFA in the SCC matrix. Accordingly, cement was replaced with 20%, 30% and 50% of POFA to investigate the behavior and reactions at early and late age properties of SCC. Slump flow, J-ring, L-box, and sieve stability test were conducted to assess the fresh properties of SCC with POFA. Also, the compressive strength at ages of 1, 3, 7, 28, and 90 days were recorded. Moreover, the durability tests including chloride permeation, gas permeability, sulfate attack and drying shrinkage were conducted at late ages up to six months. The SEM, XRD, FTIR, TGA, DSC and calorimetry tests were also performed to study the underlying mechanism which the microstructure and chemical composition of samples were changed. The same tests were carried out for samples with 1% colloidal nano silica. For samples with the highest level of replacement (50% POFA), dosages of 0.5% and 1.5% colloidal nano silica were also used.

The results showed that the replacement of cement with more than 20% dosage of POFA notably increased the bleeding and segregation of the mixture. The chemical tests revealed that the delayed hydration increased the amount of free water in the matrix. The free water diffused out of the matrix and bleeding and segregation occurred

consequently. However, when nano silica was added, the bleeding and segregation were controlled by accelerating the hydration through its pozzolanic and dissolving effects. Furthermore, results from the hardened properties showed that the addition of nano silica compensated the strength depletion caused by high volume of cement replacement, albeit, the target strength of 30 MPa was achieved at 90 days. Similarly, the addition of nano silica reduced the drying shrinkage, gas permeability, sulfate attack and chloride ingression in high volume POFA-SCC. In general, the results were conclusive that the addition of nano silica enhances the properties of self-compacting concrete with high volume of mineral admixture with low cementing properties.

Keywords: Self-compacting concrete, POFA, Colloidal nano silica, Stability, Compressive strength, Durability.



Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PEMBANGUNAN KONKRIT PEMAMPATAN DIRI POFA BERISIPADU TINGGI MENGGUNAKAN NANO SILIKA BERKOLOID SEBAGAI EJEN MENGUBAHSUAI KELIKATAN

Oleh

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Mei 2016

Pengerusi : Farah Nora Aznieta Binti Abd Aziz, PhD Fakulti : Kejuruteraan

Kajian ini menggariskan kesan-kesan nano silica terhadap konkrit memadat-diri (SCC) dengan kandungar isipadu yang tinggi menggantikan abu sisa bahan api kelapa sawit (POFA). POFA merupakan sisa pertanian yang didapati dengan jumlah yang banyak di Malaysia. Ia adalah hasil sampingan pembakaran sisa industri minyak sawit yang dilaporkan mempunyai tingkah laku pozzolanik. Bahan tambah mineral pozzolanik konkrit untuk meningkatkan digunakan dalam ciri-ciri mekanikal dan ketahanlasakannya. Walau bagaimanapun, penambahan bahan tambah mineral terutama dalam jumlah yang tinggi mengurangkan kebolehkerjaan dan meningkatkan permintaan untuk superplasticizer. Kesan negatif fenomena yang dijelaskan adalah amat penting dalam SCC di mana kebolehkerjaan yang tinggi serta kejeleketan diperlukan. Tambahan pula, kemasukan bahan tambah mineral sebagai pengganti simen membawa lengah dalam penghidratan yang mana air tak terikat bertambah dalam matriks pada awal umur. Ini meningkatkan risiko pengasingan dan penjujuhan dan kesannya sifat konkrit terkeras termasuk sifat-sifat mekanikal dan ketahanlasakan dibayangi secara negatif.

Tujuan paling utama kajian ini adalah untuk mengatasi kelemahan yang dinyatakan di atas hasil kemasukan POFA berisipadu tinggi dalam matriks SCC. Oleh itu, simen telah digantikan dengan 20%, 30%, dan 50% POFA untuk menyiasat perilaka dan tindakbalas yang terhasil pada umur awal dan lewat dalam sifat-sifat SCC. Ujian-ujian aliran turunan, J-ring, kotak-L, dan ujian kestabilan ayak telah dijalankan untuk menilai sifat-sifat baru POFA SCC berisipadu tinggi. Kekuatan mampatan sampel pada umur 1, 3, 7, 28, dan 90 hari telah direkodkan. Selain itu, ujian ketahanlasakan termasuk penyerapan klorida, kebolehtelapan gas, serangan sulfat dan pengecutan keringan telah dijalankan pada umur lewat sehingga enam bulan. Ujian-ujian seperti SEM, XRD, FTIR, TGA, DSC, dan ujian kalorimeter juga telah dilakukan untuk mengkaji mekanisme yang mendasari di mana mikrostruktur dan kimia komposisi sampel telah berubah. Prosedur yang sama telah dijalankan untuk sampel dengan 1% nano silica berkoloid. Bagi sampel dengan tahap tertinggi penggantian (50% POFA), dos nano silika berkoloidal yang lain sebanyak 0.5% dan 1.5% juga digunakan.

Hasil kajian menunjukkan bahawa penggantian simen dengan POFA terutamanya pada dos yang lebih daripada 20% meningkatkan penjujuhan dan pengasingan. Ujian kimia mendedahkan bahawa penghidratan yang tertangguh meningkatkan jumlah air bebas di dalam matriks. Air bebas diresap daripada matriks dan penjujuhan dan menyebabkan pengasingan berlaku seterusnya. Walau bagaimanapun, apabila nano silika ditambah, penjujuhan dan pengasingan dapat dikawal dengan mempercepatkan penghidratan melalui kesan pozzolanik dan perlarutan. Tambahan pula, hasil daripada sifat keras konkrit menunjukkan bahawa penambahan nano silika menyebabkan kekurangan kekuatan yang disebabkan oleh isipadu penggantian simen yang tinggi walaupun kekuatan sasaran 30 MPa dicapai pada 90 hari. Begitu juga penambahan nano silika mengurangkan pengecutan terkering, kebolehtelapan gas, serangan sulfat dan kemasukan klorida dalam POFA-SCC berisipadu tinggi. Secara umumnya, keputusan menunjukkan bahawa penambahan nano silika meningkatkan sifat-sifat SCC dengan bahan tambah mineral berisipadu tinggi dengan sifat-sifat penyimenan yang rendah.

Kata Kunci: Konkrit memadat-diri, POFA, Nano silica berkoloid, Kestabilan, Kekuatan mampatan, ketahanlasakan

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I certify that a Thesis Examination Committee has met on 6 May 2016 to conduct the final examination of Samira Jilani Kojouri on her thesis entitled "Development of High Volume POFA Self-Compacting Concrete using Colloidal Nano Silica as a Viscosity Modifying Agent" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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LIST OF ABBREVIATIONS AND ACRONYMS

NVC	Normal vibrated concrete
POFA	Palm oil fuel ash
OPC	Ordinary Portland cement
SP	Superplasticizer
PNS	Polynaphtalane sulforlate
PCE	polycarbxylate ether
PA	Polyacrylate
VMA	Viscosity modifying admixtures
NS	Nano silica
CNS	Colloidal nano silica
Ctr	Control
C ₃ S	Tricalcium silicate
C_2S	Dicalcium silicate
C-S-H	Calcium silicate hydrate
СН	Calcium hydroxide
ITZ	Interfacial transition zone
AFm	Alumina, Ferric oxide, monosulfate
W/b	Water to binder ratio
TGA	Thermo gravimetric analysis
FTIR	Fourier transforms infrared spectroscopy
SEM	Scanning electron microscope
XRD	X-Ray Diffraction Analysis
DSC	Differential scanning calorimeter
RCPT	Rapid chloride penetration test

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CHAPTER 1

INTRODUCTION

1.1 Background

Concrete is the most used building material in the world and reportedly more than 10 billion tons are produced annually (Mehta, 2002). Being easily engineered for different purposes and its rather cheap and available constituents are major success keys in its mass production around the world. Many forms of concrete, so far, have been proposed and cast adaptable to its use and the required strength, namely, self-compacting concrete, ductile concrete, and foam concrete. Of all types of concrete being introduced to construction industry, self-compacting concrete (SCC) or also known as self-consolidating concrete, has a great importance due to its high workability and viscosity which eases up the pouring and cast in members with congested reinforcement, foundations, and underwater concreting. Furthermore, removal of the vibration process cuts off the labour and hence the costs are reduced extensively.

All the above mentioned advantages of SCC comes with a requirement of high volume of cement (up to 600 kg/m³) to achieve an appropriate matrix cohesiveness to hold the aggregates, which is known as stability. Production of cement is believed to trigger more than 7% of emitted carbon dioxide to the atmosphere (Huntzinger and Eatmon, 2009). Furthermore, high volume of cement induces thermal crack and also jeopardizes dimensional stability of concrete due to mechanisms such as shrinkage and creep (Rozière et al., 2007). Experts have proposed usage of viscosity modifying agents to reduce the cement content and to control the stability. On the other hand, mineral admixtures are suggested into the matrix to lessen the amount of cement through cement replacement. Admixing mineral admixtures not only reduces the cement content but enhances the qualities of concrete such as mechanical properties and the durability. However, only limited amounts of replacement are suggested in order to maintain the appropriate stability and avoid the strength depletion of samples (Hassan et al., 2012, Sheinn et al., 2003).

One of the mineral admixtures is palm oil fuel ash (POFA) which is the residue from burning shells and fibres of palm fruit and is produced in hefty amounts in Malaysia. Based on statistics of Malaysian Palm Oil Board (2015), more than 5.6 million hectares of land are occupied with palm plants in Malaysia that produce approximately 15 million tons of POFA annually. Many studies suggest that POFA has pozzolanic behavior and hence can lead to formation of secondary C-S-H and strengthen the matrix. Also, different works verified that POFA can enhance the durability of concrete in terms of chloride and sulfate resistance (Jaturapitakkul et al., 2007, Johari et al., 2012). However, a comprehensive study on application of high volumes of POFA in self-compacting concrete is indispensable.

The other recently exploited mineral admixture is nano silica which is available in two forms of powder and colloidal gel. In general, nano silica influences the cement matrix by its pozzolanic behavior originated from its amorphous nature of nano sized SiO_2

(Björnström et al., 2004). Its nano size particle boosts up its effectiveness compared to silica fume, albeit at a shorter duration also the addition of nano-TiO₂, nano-Al₂O₃ and nano silica to the self-compacting mortar showed that among all samples, nano silica increased the strength to a higher level compared to other nano materials (Mohseni et al., 2015). Furthermore, high surface energy of particles can enhance the dissolution of cement compounds to render higher amounts of hydration products at very early stages of hydration. The hydration products can later envelop the nano particles which act as nuclei for a better distribution of C-S-H all over the matrix (Said et al., 2012). The mentioned phenomena besides nano filling effect of nano silica can further densify the matrix. Studies show that nano silica can enhance both mechanical properties and the durability of cement based materials.

1.2 Problem Statement

One of the major problems with self-compacting concrete is the usage of high content of cement in the matrix (Okamura and Ouchi, 1998). Cement should be used in high amounts to create enough cohesiveness in the matrix to hold the aggregates together as well as to facilitate its flow ability which consequently causes higher rate of shrinkage and induces cracks in the matrix especially in the perimeter of the aggregates (Persson, 2001). To solve the problem, experts have proposed usage of viscosity modifying agents (Domone, 2007).

Cement replacement especially in high volumes is suggested since cement production is merely responsible for 7% of increasing carbon footprint into the ecosystem (Huntzinger and Eatmon, 2009). However, addition of mineral admixtures as cement replacement increases the water demand and reduces the workability. So, superplasticizers are added to overcome the reduced workability. The addition of high volume of mineral admixtures and superplasticizers negatively affects the hydration evolution and in some cases chemically conflicts the viscosity modifying agent. The delayed hydration may cause the bleeding and segregation of the fresh concrete which depletes the strength of hardened matrix later and lowers the durability qualities. The delayed hydration is not only as a result of superplasticizer but also can be caused by high volume replacement of cement with mineral admixtures especially with the one's with lower cementitous qualities such as POFA.

According to the statistics of Malaysian palm oil board (Malaysian Palm Oil Board, 2015), it is predicted that production of palm oil is increasing on an annual basis. POFA a by-product of palm oil plantation in Malaysia is dumped in landfills as a waste without any profitable return and causes environmental problems. Nevertheless, Awal and Hussin (2011) reported that the use of ground POFA as a supplementary cementing material due to its high content of SiO₂ is accepted. Due to low reactivity of POFA comparing other mineral admixtures such as silica fume and its porous morphology, POFA can facilitate and control stability of fresh SCC by delaying the hydration at lower contents of replacement.

Farzadnia et al. (2015) reported that porous surface of POFA imbibes water and consequently, increases the water demand at high content of replacement. However, the solution to dispose POFA as a highly produced agro waste is a core motive for researcher to come up with scientific solutions to use POFA as a cement replacement in concrete especially in high volume. However, Tangchirapat et al. (2009a) mentioned

that only up to 30% of POFA can be used as a cement replacement.

Based on this information, this study is carried out with a hypothesis that nano silica can facilitate use high volume of POFA as a cement replacement in SCC. Hypnotically nano silica reduce the water absorbed in POFA by filling it pore structure. The excessive water can be released over time causing segregation and bleeding at high content of POFA. Furthermore, the amount of available water for hydration depletes which delays the hydration rate remarkably. Nano silica can accelerate hydration by its dissolution effect on C_3S . On the other hand, POFA was selected due to its low cementing and pozzolanic properties which accentuates the negative effect of high volume use. Besides, its hybrid effect with nano silica in SCC is not known yet.

In this study, nano modification of POFA is used to overcome the disadvantage of POFA and to change it to an advantage. Nano sized silica with high surface energy accelerates the hydration at the very first moments of contact with cement particles (Said et al., 2012), which is beneficial to the matrix in order to use it as a viscosity modifying agent. Other additives that have the same potential used are silica fume, limestone and metakaolin, however the reactivity of these materials are reported to initiate at later age of concrete i.e. up to three days from mixing (Wild et al., 1996). This is further reported by Jiang et al. (2015), whom investigated the pozzolanic reactivity of metakaolin and silica fume and concluded that most part of CH has not react with metakaolin and silica fume at first 3 days of curing due to their lower pozzolanic reactivity and lower surface area.

Based on above mentioned researches, nano particles are chosen because it increases the packing density of particles as a wider range of sizes are exploited in the matrix. Also, the pore structure of POFA can be modified in favour of segregation and bleeding when high volume of POFA is used. Besides, the gradual release of free water can render higher rate of C-S-H in long run especially with presence of POFA, for which the pozzolanic reactivity initiates at ages as late as 14 to 28 days of curing (Altwair et al., 2011). This can enhance the durability of high volume POFA- SCC as well as compensate the strength loss at later ages.

The aim of this study is to exploit nano silica as a modifying agent to overcome the delayed hydration due to either high volume replacement of cement with POFA or the increased demand for superplasticizers in order to control the stability. Furthermore, nano silica hypothetically, can compensate the depleted compressive strength caused by high volume of replacement and can enhance the durability issues of SCC.

1.3 Research Questions

Although the advantages of incorporation of low content of POFA in concrete is well investigated, Chindaprasirt et al. (2007a) agree that using POFA in high replacement levels negatively affect fresh properties, especially in self-compacting concrete in which the stability is highly influenced by cement content. The partial replacement of cement with POFA can beneficially reduce thermal cracking due to excessive heat augmentation, however, reduction in the peak temperature and also the hydration delay at peak temperature may disadvantageously change the fresh properties of concrete and may cause bleeding and segregation, so addition of appropriate viscosity modifying

agents can help to enhance the fresh properties of SCC and consequently improve the hardened properties of SCC. Nevertheless additions of chemical viscosity modifying agents are not beneficial as they may conflict with superplasticizers. So, introducing a viscosity modifying agent with enhancing effects on properties of self-compacting concrete such as stability, early age strength, and durability in long run is indispensable.

This study focused on effects of nano silica as a viscosity modifying agent on chemical and physical characteristics of self-compacting concrete with high volume POFA. This study tents to answer the following questions;

- 1. What are the effects of high volume replacement of POFA on early age and late age properties of SCC?
- 2. How can physical and chemical properties of nano silica affect the fresh properties of high volume POFA self-compacting concrete?
- 3. To what extent can nano silica compensate the possible shortcoming caused by high volume cement replacement in terms of mechanical properties and durability?
- 4. What is the governing mechanism when the binary mix of nano silica and high volume POFA is used in the SCC matrix?

1.4 Objectives

The main aim of this research is to overcome the negative effects of high volume replacement of POFA on fresh and hardened properties of self-compacting concrete by addition of nano silica. The enhancement in the durability of the proposed concrete such as drying shrinkage, chloride ingression, and sulfate attack and gas permeability was also investigated. This was realized through the following four objectives;

- 1. To determine the stability, fresh properties, and early and late age compressive properties of high volume POFA self-compacting concrete with Colloidal Nano Silica (CNS).
- 2. To ascertain the durability behaviour of high volume POFA-SCC with Colloidal Nano Silica (CNS).
- 3. To determine the microstructure and chemical composition of high volume of POFA-SCC with Colloidal Nano Silica (CNS)

1.5 Significance of the Study

This study intended to use nano silica as a viscosity modifying agent in high volume POFA self-compacting concrete. The replacement of cement with high volume of POFA reduces the carbon dioxide footprint to a great extent. Furthermore, it solves the environmental problem caused by hefty amount of agro waste in Malaysia. Nano silica can control the bleeding and segregation caused by high volume replacement of POFA due to its high surface energy and chemical reactivity. The advantage of nano silica over other viscosity modifying agents is that there is no conflicting effect with the

superplasticizers. Furthermore, unlike other viscosity modifying agents, nano silica can compensate the strength loss caused by replacement of cementing materials with low cementitous properties. In addition, this study unveils the mechanisms by which nano silica interacts with POFA which is considered as a low cementitous material due to a low content of Ca and Al.

1.6 Scope and Limitations of the Study

This study focused on cement replacement with up to 50% POFA. The 50% is considered as a high volume for mineral admixtures with low cementitous properties due to its low contents of Ca and Al ions. The ground POFA was used to increase the reactivity of its particles. On the other hand nano silica in colloidal state dosage was limited to 1% for mixtures 20 and 30% POFA; and up to 1.5% for 50% replacement of POFA. The volume of water and the type of aggregate were constant throughout the study.

In the first phase of the study the fresh properties of SCC are determined from slump flow, J-ring, L-box, and sieve stability test. However, to optimize the workability, the dosage of superplasticizer was varying with constant W/b ratio. The maximum amount of superplasticizer used is 8%. In the second stage of the study, the compressive strength of SCC was measured and reported at ages of 1, 3, 7, 28 and 90 days. The target strength of control samples was set to be 30MPa. To investigate the underlying mechanism by which POFA and nano silica modify the SCC matrix, the following tests were conducted: XRF, XRD, TGA, SEM, FTIR, and DSC tested and qualitatively analysed. On the other hand, , the durability of high volume POFA self-compacting concrete with and without nano silica was evaluated through permeability tests, shrinkage, chloride ingression, and attack at ages for a period of six month. The growing use of SCC in infrastructures especially underwater structures and foundations, which exposes SCC to environments, necessitated the performance assessment of samples against attack over other durability issues such as carbonation hence less focus on the later are made in this research. The energy consumption, CO_2 emission, cost analysis and carbonation were not investigated in this study.

1.7 Overview of the Thesis

The thesis encompasses four chapters to report and discuss the research work done in this study as follows;

Chapter two initially introduces the history, characteristics and properties of selfcompacting concrete. Modification of SCC matrix using mineral admixture at different replacement levels including low and high volume is also discussed. Following the given reviews on SCC, the POFA and its chemical and physical properties are presented and its effects on concrete are reviewed. In the final section of the chapter, properties of nano silica and mechanisms by which it enhances the cement based materials are discussed. Chapter two is finally ended with concluding remarks to help readers better comprehend the purpose of the dissertation. Chapter three describes the methodology and the approach used to achieve the objectives of the study. It consists of introduction of materials, sample preparation, testing procedure as well as the purpose behind the selection of the tests. Flowcharts are provided to depict the methodology step by step.

Chapter four comprises of three sections; the fresh properties, the hardened properties and the durability of the high volume POFA SCC with/without nano silica. The orders of sections are arranged according to the objectives of the study for more clarification. The mechanism by which POFA and nano silica influenced the matrix has been discussed along the chapter.

Finally, Chapter 5 provides a summary and conclusion of the study and suggests some recommendation for further research in this area of knowledge.



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