UNIVERSITI PUTRA MALAYSIA

EFFECTS OF NANOMATERIALS ON HIGH PERFORMANCE CONCRETE MORTARS EXPOSED TO ELEVATED TEMPERATURES

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By

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

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DEDICATIONS

I dedicate this dissertation to all of the inspiring people who have enlightened not only my life but the life of others by unselfishly dedicating themselves to share love, knowledge and experience.

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

EFFECTS OF NANOMATERIALS ON HIGH PERFORMANCE CONCRETE MORTARS EXPOSED TO ELEVATED TEMPERATURES

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May 2014

Chair: Professor Dato Ir. Abang Abdullah Abang Ali

Faculty: Engineering

High performance concrete (HPC) is currently used in massive amounts in the construction industry due to its technical and economical advantages over normal concrete. The HPC is characterized by improved mechanical and durability properties resulting from the use of chemical and mineral admixtures as well as specialized production processes. However, sharp strength loss and reduction in elastic modulus at temperatures below 400 °C are major disadvantages of HPC compared to normal concrete; raising questions on its application in the construction industry at high temperatures. The demerit of HPC at elevated temperatures up to 400 °C is mostly attributed to a mechanism called hydrothermal process by which cracks and in some cases spalling occur due to vapor pressure. It is caused by the release of water from capillaries as well as degradation of hydration products entrapped in the impermeable microstructure of the HPC, increasing the internal vapor pressure. All this is brought about by the low water to binder ratio in the HPC with the presence of mineral admixtures such as silica fume. Nonetheless, some studies have mentioned the positive effect of mineral admixtures at elevated temperatures due to the formation of stronger clusters of calcium silicate hydrate and restriction in crystal growth of calcium hydroxide.

This study investigated the behavior of high performance concrete mortars with nanomaterials at elevated temperatures to simulate the behavior of binding matrix exposed to heat in order to increase the heat resistant behavior of the binding matrix with a focus on temperatures below 400 °C. Furthermore, since there is a debate on the effects of mineral admixtures on the HPC at elevated temperatures, the findings of this study helps to identify the critical influencing factors namely: chemical composition, moisture content, and permeability in behavior of the binding matrix at elevated temperatures. It is well established that nanomaterials can modify the above mentioned factors due to their size, shape, and solid state.

Four nanomaterials, namely: nano silica with amorphous state, nano titania with both amorphous and crystal states, nano alumina with pure crystal state, and halloysite nano clay with tubular shape were chosen for this study and fractions of 1, 2 and 3% by weight of cement were added to the mixes. XRD, DSC, SEM and gas permeability tests were conducted to investigate the chemical composition and microstructural changes of the HPC mortars after being exposed to elevated temperatures up to 1000 °C. The residual compressive strength, energy absorption, brittleness index and relative elastic modulus were studied to compare the mechanical properties of mortars with and without nanomaterials and to identify the most effective amount of each nanomaterials. Addition of a 1% nano silica, 2% nano titania, 1% nano alumina, and 3% halloysite nano clay , as most effective amounts, enhanced the heat resistant behavior of the mortars up to 400 °C in terms of residual mechanical properties and the microstructure. Up to 13% enhancement in the relative residual compressive strength, 28% enhancement in the relative elastic modulus, and 32% enhancement in the permeability of mortars were achieved when nanomaterials were added. The interlocking and filling effects of the nanomaterials played an important role in controlling the governing factor of vapor pressure in the binding matrix.

Keywords: High performance concrete mortars, Elevated temperatures, nanomaterials, Mechanical properties, Chemical composition.

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

KESAN BAHAN NANO KE ATAS MORTAR KONKRIT BERPRESTASI TINGGI TERDEDAH PADA SUHU MENINGKAT

Oleh

NIMA FARZADNIA

Mei 2014

Pengerusi: Professor Dato Ir. Abang Abdullah Abang Ali

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Konkrit prestasi tinggi (HPC) digunakan dalam jumlah yang besar dalam industri pembinaan pada masa kini disebabkan kelebihannya dari segi teknikal dan juga ekonomi berbanding konkrit normal. Peningkatan ciri mekanikal dan ketahanlasakan HPC terhasil daripada penggunaan bahan tambah kimia dan mineral serta proses penghasilan yang khusus. Walaubagaimanapun, kehilangan deripada kekuatan yang mendadak dan pengurangan modulus kenyal pada suhu kurang 400 °C adalah kelemahan utama HPC berbanding konkrit normal; rintangan haba ini menambahkan persoalan terhadap penggunaannya di dalam industri pembinaan. Kelemahan HPC pada suhu meningkat sehingga 400 °C kebanyakannya diperolehi daripada mekanisma yang dipanggil proses hidrotermal dimana retakan dan di dalam beberapa kes serpihan berlaku disebabkan tekanan wap. Ini disebabkan oleh air yang dibebaskan daripada rerambut serta penurunan produk penghidratan yang terperangkap di dalam mikrostruktur tidak boleh telap HPC dan meningkatkan tekanan wap dalaman. Semua ini disebabkan oleh nisbah air kepada pengikat yang rendah di dalam HPC dengan kehadiran bahan tambah mineral seperti wasap silika. Namun begitu, beberapa kajian ada menyatakan tentang kesan positif bahan tambah mineral pada suhu meningkat disebabkan oleh pembentukan kelompok kalsium silikat hidrat yang lebih kuat dan pembatasan dalam tumbesaran hablur kalsium hidroksida.

Kajian ini tertumpu kepada kelakuan mortar konkrit prestasi tinggi dengan bahan nano pada suhu meningkat untuk mensimulasi kelakuan pengikatan matriks yang didedahkan kepada haba bertujuan untuk meningkatkan kelakuan rintangan haba pengikatan matriks dengan fokus pada suhu kurang daripada 400 °C. Tambahan pula, kerana wujudnya perdebatan terhadap kesan bahan tambah mineral terhadap HPC pada suhu meningkat, penemuan kajian ini dapat membantu dalam mengenalpasti faktor kritikal yang mempengaruhi iaitu: komposisi kimia, kandungan lembapan, dan kebolehtelapan dalam kelakuan pengikatan matriks pada suhu meningkat. Adalah diketahui bahawa bahan nano dapat mengubahsuai faktor yang disebutkan di atas disebabkan saiz, bentuk dan keadaan pepejal bahan tersebut.

Empat bahan nano yang biasa digunakan iaitu: silika nano dengan keadaan amorfus, titania nano dengan keadaan amorfus dan juga hablur, alumina nano dengan keadaan hablur tulen, dan tanah liat nano haloisit berbentuk tiub dipilih untuk kajian ini dan pecahan 1, 2 dan 3% dari berat simen telah ditambah ke dalam campuran. Ujian XRD, DSC, SEM dan kebolehtelapan telah dijalankan untuk mengkaji komposisi kimia dan perubahan mikrostruktur HPC mortar selepas didedahkan kepada suhu meningkat sehingga 1000 °C. Baki kekuatan mampat, serapan tenaga, indeks kerapuhan dan nisbi modulus kenyal dikaji untuk dibandingkan dengan ciri mekanikal mortar dengan dan tanpa bahan nano untuk mengenalpasti jumlah paling berkesan untuk setiap jenis bahan nano. Penambahan 1% silika nano, 2% titania nano, 1% alumina nano dan 3% tanah liat nano haloisit adalah jumlah paling berkesan untuk meningkatkan kelakuan rintangan haba mortar hingga 400 °C dalam sebutan ciri mekanikal dan mikrostruktur. Sehingga 13% peningkatan dalam nisbi baki kekuatan mampat, 28% peningkatan dalam nisbi modulus kenyal, dan 32% peningkatan dalam kebolehtelapan mortar berjaya dicapai apabila bahan nano ditambah. Kesan saling mengunci dan isian oleh bahan nano memainkan peranan paling penting untuk mengawal faktor pengawal tekanan wap dalam matriks pengikat.

Kata kunci: Mortar konkrit prestasi tinggi, Suhu meningkat, Bahan nano, Ciri mekanikal, Komposisi kimia.

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I certify that a Thesis Examination Committee has met on (**30 May 2014**) to conduct the final examination of (**NIMA FARZADNIA**) on his (or her) thesis entitled "**EFEFCTS OF NANO MATERIALS IN HIGH PERFORMANCE CONCRETE MORTARS EXPOSED TO ELEVATED TEMPERATURES**" 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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CHAPTER 1

INTRODUCTION

1.1 Background

Concrete is a widely used construction material around the world and its properties have undergone changes through technological advancements. Numerous types of concrete have been developed to enhance its properties. Currently, high-performance concrete (HPC) is used in massive amounts due to its technical and economical advantages over normal concrete.The HPC is characterized by improved mechanical and durability properties resulting from the use of chemical and mineral admixtures as well as specialized production processes.

New technologies may bring new problems in some applications and should be studied carefully before being applied practically in industry. One important precaution to take into consideration is when high temperatures are involved. Normal concrete meets certain minimum safety standards since it limits the extension of damage in fire due to its heat resistance. Recently, drawbacks in the HPC have attracted the attention of researchers after some real vehicle accident fire exposures such as at the Great Belt tunnel in Denmark and the Chunnel tunnel. A sharp strength loss and in some cases explosive spalling were some of the major disadvantages of the HPC at elevated temperatures raising questions on its application in the construction industry, especially in tunnels, power plants and military buildings. Furthermore, concrete is used in some heat exposed environments such as runway areas, air field pavements, molten metal splash areas, hot material storage areas, fire resistant plastering works and some other possible applications which necessitate a further study on processes by which heat resistance is enhanced.

Some of the most important factors affecting the resistance of concrete at elevated temperatures are aggregate type, cement type, and water to binder ratio. A number of studies have investigated the effect of the above-mentioned factors on the behavior of both normal concrete and HPC.For the HPC and its behavior at elevated temperatures, researchers attach great importance to the effect of mineral admixtures since they are the main distinguishing constituents of HPC from conventional concrete.

Mineral admixtures are available in the market as industrial by-products such as silica fume and fly ash or agro-wastes such as rice husk and palm oil fuel ash. They modify the binding matrix of concrete and the interfacial transition zone (ITZ) by their effect on the formation of hydration products through their pozzolanic behavior and nucleation effect due to their high surface area. Today, a number of efforts have been made to study the addition of mineral additives into the binding matrix in order to reduce consumption of cement as well as to enhance the properties of concrete.

1.2 Statement of the Problem

Results from the latest studies on mineral admixtures in the HPC at elevated temperatures have encouraged discussions on their effect on the behavior of HPC binding matrix at elevated temperatures. A number of researchers have addressed the adverse effect of incorporation of mineral admixtures, which resulted in high impermeability of the binding matrix, causing sharp strength loss due to evaporation of bound and unbound water in the binding matrix. Others reported an enhanced heat resistance due to the formation of stronger clusters of C-S-H and restricted size of calcium hydroxide crystals as a result of pozzolanic behavior of the mineral admixtures.

Recently, with the introduction of nanomaterials to the concrete industry, new opportunities have arisen to study the effects of additives on microstructural changes and chemical composition of the binding matrix. This is due to remarkable effect of their nano sized particles on the binding matrix. Incorporation of nanomaterials in the heat exposed binding matrix may clarify the mechanisms by which concrete is affected by high temperatures. It is also well established that the incorporation of nanomaterials can refine the binding matrix by chemical effects such as pozzolanic reactivity, and that the physical filling and nucleation effects enhance the mechanical properties and durability of concrete.

So far , some aspects of incorporation of nanomaterials in cementitous composites have been studied but there are still areas that need to be covered before mass production of such concretes. Since nanomaterials considerably influence the formation of hydrated products, moisture content and permeability of the matrix, an investigation on the behavior of the binding matrix with nanomaterials after exposure to elevated temperature is indispensable. Moreover, the interlocking effect of nanomaterials in failure planes caused by heat exposure as well as modification in hydration products may enhance the heat resistance of concrete. This study intended to investigate the physical filling and nucleation effects, as well as the chemical effect of pozzolanic reactivity and shape of nanomaterials on the heat resistant behavior of HPC mortars and to identify the influencing factors on the behavior of HPC binding matrix at elevated temperatures.

1.3 Objectives

The main aim of this research was to study the effect of adding nanomaterials in high performance concrete mortars when exposed to elevated temperatures.

This was realized through the following three objectives;

- To investigate the effects of nanomaterials on the microstructure and chemical composition of the binding matrix at ambient room temperature and after exposure to elevated temperatures.

- To investigate the effects of nanomaterials on the mechanical properties of the binding matrix at ambient room temperature and after exposure to elevated temperatures.

- To identify the influencing factors on the behavior of the binding matrix in high performance concrete at elevated temperatures.

1.4 Research Questions

This study focused on the chemical and physical characteristics of nanomaterials in order to answer the following questions;

1. How can different characteristics of nanomaterials namely interlocking, nucleation, and pozzolanic activity affect the heat resistant behavior of the binding matrix?

2. Which is the most dominant factor influencing the behavior of mortars after being exposed to elevated temperatures: permeability, formation of hydration products or moisture content?

1.5 Significance of the Study

This study intended to investigate modification of the binding matrix by incorporation of nanomaterials at elevated temperatures. The selection of nanomaterials followed two criteria; their potential to enhance heat resistance of the binding matrix, and secondly to investigate the most dominant factor influencing the behavior of binding matrix after being exposed to elevated temperatures when additives were present in the mix. Knowledge derived from the findings of this study could elaborate the behavior of the binding matrix at elevated temperatures which may increase the knowhow of the industry to avoid structural disasters when concrete structures are subjected to high temperatures and ensure the built infrastructures are able to endure accidental exposures to fires. Also, it may introduce a process by which the binding matrix of concrete can be modified in order to be used in environments where temperature may be elevated to 400 °C to 600 °C. The introduction of a heat resistant Portland cement concrete may also change the heat related building codes in terms of concrete cover thickness and etc. Furthermore, the results of this study may solve the arising problems related to the incorporation of silica fume, as the mostly used mineral admixtures in the HPC, at elevated temperatures.

1.6 Scope of Research

The overarching purpose of this study is to introduce mechanisms by which nanomaterials modify the heat resistant behavior of the binding matrix in high performance Portland cement concrete at elevated temperatures. To do so, this study focused on the microstructural changes and chemical composition of the binding matrix of high performance mortars with the presence of four different types of nanomaterial at different temperatures of 28 °C, 100 °C, 200 °C, 300 °C, 400 °C, 600 °C, 800 °C and 1000 °C. Aggregates with size bigger than 4.75 mm were excluded from this study in order to minimize the effect of aggregates on behavior of the binding matrix due to their volume concentration and the amount of interfacial area per unit volume. Nano silica, nano titania, nano alumina, and halloysite nano clay were chosen as additives due to their characteristics either in shape or chemical compositions and were added in fractions of 1, 2 and 3%. The target temperature at which the heat resistance needs to be enhanced is 600 °C where using refractory castables are not economical. The effects of the modification of the matrix at 28 days on behavior of the mortars at elevated temperatures were considered and early hydration was not studied.

In this study, compressive strength, XRD, DSC, SEM, gas permeability, and mass loss were major tests conducted to trace properties of mortars both at room temperature and at elevated temperatures. All results were obtained in residual state after exposure to elevated temperatures with 1 hour steady state and the effect of cyclic heating was not investigated in this study. Also, spalling was not targeted in this study since it mostly occurs in large surfaces of the structural members and the heat exposed damages in mortars can be traced by cracks and sharp strength loss.

1.7 Overview of the Thesis

The following four chapters shall describe the research work done in this thesis as follows;

Chapter 2 initially discusses the microstructural differences between normal and high performance concrete. Then the effects of elevated temperatures on the chemical composition, microstructure and residual mechanical properties of concrete are mentioned, and how high performance concrete varies in behavior after being exposed to elevated temperatures is further discussed. This chapter also reviews the influencing factors on the behavior of high performance concrete when exposed to high temperatures. Moreover, some efforts to enhance the heat resistance of cementitous composites are reviewed. In the remaining sections of this chapter the mechanical properties, durability and microstructural changes of paste, mortar and concrete containing targeted nanomaterials are reviewed.

In chapter 3, the methodology including materials, sample preparation and temperature exposure are described and the different tests which were conducted are mentioned. These tests were implemented to study the mechanical properties of mortars which included compression, brittleness index, energy absorption, and relative elastic modulus as well as chemical composition and microstructural changes namely XRD, SEM, DSC , gas permeability, and mass loss of samples after being exposed to elevated temperatures. Figures and images are used to illustrate the tests clearly.

Chapter 4 discusses test results for halloysite nano clay, nano silica, nano titania, and nano alumina, respectively. This chapter tends to compare and introduce the most advantageously effective amount of nanomaterials to be used in mortars with the behavior at elevated temperatures. In this chapter the author aimed to discuss the most important effect of nanomaterials on the behavior of binding matrix after being exposed to elevated temperatures.

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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