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DEVELOPMENT OF GROUND DUNE SAND BLENDED CEMENT

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DEVELOPMENT OF GROUND DUNE SAND BLENDED CEMENT

By

OMER ABDALLA ALAWAD HASSAN

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

November 2014

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DEDICATION

To:
My Mother,
Your love is always with me no matter where I go.
My Father,
You enlighten me to do all right things and only the right,
reminding me that there can be no gain without pain.
My Brother,
Your unconditional support is treasured for always.
My wife,
For all that you have been, for all that you are and will always be,
I am grateful.

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 GROUND DUNE SAND BLENDED CEMENT

By

OMER ABDALLA ALAWAD HASSAN

November 2014

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Pozzolan materials (e.g. fly ash, slag, silica fume, rice husk ash) have been used successfully as a partial ordinary Portland cement (OPC) replacement material. However, there are some technical and economic drawbacks associated with the use of the existing pozzolan materials. Therefore, there is a growing interest to find an alternative material to be used as a source of siliceous materials for concrete production. This research aims to determine the potential of using ground dune sand (GDS) as partial cement replacement in binary (OPC-GDS) and ternary combinations of OPC-GDS-slag and OPC-GDS-lime. The proposed combinations of blended cement system are expected to save large amounts of OPC.

The primary objective of this study is to develop naturally available dune sand as an effective partial cementing material for use in the concrete industry. To achieve this objective, different treatment methods, namely, mechanical, chemical and thermal methods (autoclave curing) have been applied to determine the reactivity of GDS. For the ternary blended combinations, low (15%), moderate (30%) and high (45%) amounts of slag or lime were incorporated into a binder system containing 40% of GDS. Compressive strength, drying shrinkage and durability properties of the cast mixtures were investigated. Moreover, microstructure analyses were carried out using SEM, EDX, XRD, DTA and TGA analyses to characterize the hydrated products. A correlation between CaO/SiO_2 and compressive strength was then carried out.

The results revealed that autoclave curing is a promising curing system to utilize the GDS as partial cement replacement. The optimum level of replacement of OPC by GDS was found to be 30%, and up to 40% of GDS can be used without significant loss in the compressive strength. The inclusion of slag or lime as the ternary binder element to the mixture containing 40% of GDS yielded a compressive strength higher or comparable to the control mixture. The drying shrinkage and durability properties of blended autoclave cured mixes were significantly improved. The SEM, EDX, XRD, DTA and TGA analyses explained how GDS contributes to the strength and durability of blended mixtures. The outcome of this research will benefit the Middle East and other countries where supplies of natural dune sands are unlimited.

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

PEMBANGUNAN PASIR BUKIT TERKISAR CAMPURAN SIMEN

Oleh

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November 2014

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Bahan pozzolan (seperti abu terbang, jermang, wasap silika, abu sekam padi) telah digunakan dengan berkesan sebagai sebahagian bahan penggantian simen Portland (OPC). Walau bagaimanapun, terdapat beberapa kekangan teknikal dan ekonomi berkaitan dengan penggunaan bahan pozzolan yang sedia ada. Oleh itu, terdapat usaha untuk mencari pendekatan alternatif bahan yang akan digunakan sebagai sumber bahan bersilica dalam pembuatan konkrit. Penyelidikan ini bertujuan untuk menentukan potensi penggunaan pasir bukit terkisar (*ground dune sand* GDS) sebagai sebahagian pengganti simen dalam penduaan (OPC-GDS) dan pentiga kombinasi OPC-GDS- jermang dan OPC-GDS-kapur. Kombinasi sistem adunan simen yang dicadangkan dijangkakan dapat menjimat penggunaan OPC yang banyak.

Objektif utama dalam penyelidikan ini adalah untuk membangunkan pasir bukit sedia ada sebagai bahan gantian simen yang efektif untuk digunakan dalam industri konkrit. Untuk mencapai objektif ini, pelbagai kaedah rawatan antaranya; kaedah mekanikal, kimia dan terma (pengawetan autoklaf) digunapakai untuk menentukan kereaktifan GDS. Untuk kombinasi pentiga campuran, kuantiti rendah (15%), sederhana (30%) dan tinggi (45%) jermang atau kapur telah digabungkan dalam sistem pengikat yang mengandungi 40% GDS. Kekuatan mampatan, ciri pengecutan keringan, dan ciri ketahanlasakan campuran telah dikaji. Tambahan lagi, analisis mikrostruktur telah dijalankan menggunakan analisis SEM, EDX, XRD, DTA and TGA untuk mencirikan produk terhidrat. Korelasi antara CaO/SiO_2 dan kekuatan mampatan telah dijalankan.

Keputusan menunjukkan bahawa pengawetan autoklaf adalah sistem pengawetan yang menjanjikan penggunaan GDS sebagai bahan pengganti simen separa. Tahap optimum penggantian OPC oleh GDS adalah 30%, di mana, sehingga 40% GDS boleh digunakan tanpa kehilangan yang signifikan pada kekuatan mampatan.

Memasukan jermang atau kapur sebagai elemen pengikat ketiga dalam campuran mengandungi 40% GDS menghasilkan kekuatan mampatan lebih tinggi atau setanding dengan campuran kawalan. Pengecutan keringan dan ciri ketahanan adunan campuran terawet secara autoklaf telah meningkat dengan signifikan. Analisis SEM, EDX, XRD, DTA and TGA menerangkan bagaimana GDS menyumbang pada kekuatan dan ketahanan campuran. Hasil daripada penyelidikan ini akan menguntungkan Negara Timur Tengah dan negara lain di mana bekalan pasir bukit adalah hampir tidak terhad.

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Omer Alawad,
November 2014

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LIST OF ABBREVIATIONS

SEM	Scanning Electron Microscopy
EDX	Energy DispersiveX-Ray
TGA	Themor-gravimetric Analysis
XRD	X-Ray Diffraction
OPC	Ordinary Portland cement
C3S	$3CaO. SiO_2$ Tricalcium silicate
C2S	$2CaO. SiO_2$ Dicalcium silicate
C3A	$3CaO. Al_2O_3$ Tricalcium aluminate
C4AF	$4CaO. Al_2O_3. Fe_2O_3$ Ferrite
CH	$Ca(OH)_2$ Calcium hydroxide
C-S-H	$CaO. Al_2O_3. SiO_2. H_2O$ Calcium silicate aluminate hydrate
AFt	$3CaO. Al_2O_3. 3CaSO_4. 32H_2O$ Ettringite
AFm	$3CaO. Al_2O_3. CaSO_4. 12H_2O$ Tricalcium monosulfo aluminate
DTA	Differential Thermal Analysis
PSD	Particle Size Distribution
ASTM	American Society for Testing and Materials
FA	Fly Ash
FT-IR	Fourier Transform Infrared Spectroscopy
SCMs	Supplementary cementing materials
SF	Silica Fume
W/C	Water-to-Cement ratio
XRF	X-ray Fluorescence
MK	Metakaolin
MPa	Megapascals
C	CaO
CO_2	Carbon dioxide
S	SiO_2
A	Al_2O_3
F	Fe_2O_3
H	H_2O
M	MgO
N	Na_2O
K	K_2O
KW	Kilo watt
$\bar{S}$	$SO^{=}_3$
GJ	Giga Joule
Q	Quartz

CHAPTER 1

INTRODUCTION

1.1 General

Concrete is the most extensively used construction material in the world [1]. The consumption rate of concrete is estimated to be one ton for every living human being [2]. Perhaps, the worldwide consumption of concrete is only second to that of water. This could be due to the low cost per cubic meter, availability of raw materials, ease of casting, excellent resistance to water, and ability to be formed in various shapes and sizes [3]. The main ingredients of concrete are ordinary Portland cement (OPC or PC), water and fine and coarse aggregates. OPC is the most important ingredient because it reacts with water to make glue, which bonds the coarse and fine aggregates together.

OPC comprises four main components, which are tri-calcium silicate (C_3S), di-calcium silicate (C_2S), tri-calcium aluminate (C_3A) and tetra-calcium aluminoferrite (C_4FA). When OPC comes into contact with water, several chemical reactions (hydration) occur, resulting in different hydrated products. The hydration of C_3S and C_2S, which is considered to be about 75% of the total weight of OPC, produces calcium silicate hydrate, also known as CSH gel and calcium hydroxide ($Ca(OH)_2$ or CH). Where, the hydration of C_3A and C_4FA forms ettringite (AFt) and monsolfmonate (AFm) phases [4, 5].

The contributions of CSH gel, CH, AFt, and AFm to concrete properties vary. For instance, the AFt and CSH gel are responsible for initial setting and early strength development, respectively. These phases are mainly formed due to the hydration of C_3A and C_3S, hence their hydration starts after several minutes (i.e. 15 minutes) of mixing. The hydration of C_2S starts after several days of mixing and continues up to hundreds of days. Therefore, C_2S is responsible for the late strength development. On the other hand, CH generated from the hydration of C_3S and C_2S does not induce strengthening properties. CH may weaken the transition zone between the cement paste and aggregate and become a source of concrete deterioration (i.e. carbonation and expansive gypsum formation) [3]. The AFm phase is usually generated from AFt when the amount of C_3A is more than the supplied sulfate ions, or as in the case of elevated curing temperature. AFm has a minor contribution to strength, but could be converted to AFt causing harmful expansion of the hardened concrete properties [6, 7].

The manufacturing of OPC consumes a considerable amount of energy and resources [8]. The production of one ton of OPC requires about four GJ of energy and about two tons of raw materials (limestone, shale, etc.). In addition, the manufacturing of one ton of OPC emits approximately one ton of carbon dioxide (CO_2) into the atmosphere. From the projection made by the cement companies, the consumption rate of cement has risen from two million tons per year in 1880 to about two billion tons in 2006 [9]. Moreover, this proportion is expected to remain steady in the next decade. In particular, the manufacturing of OPC accounts for about seven per cent of the total world CO_2 emissions [10]. However, environmental concern has placed considerable pressure on cement plants to reduce the CO_2 emissions and use alternative eco- friendly materials with lower environmental impact. In fact, the Rio de Junior (1992), Kyoto (1997) and Copenhagen (2009) protocols were essentially established to reduce the total greenhouse gas emissions [11, 12].

1.2 Use of Supplementary Cementitious Materials in Concrete

One option to reduce the CO_2 emissions is to replace large amounts of OPC with supplementary cementitious materials (SCMs) - natural, industrial by product, or agricultural waste materials - which have been used successfully as partial cement replacement materials in concrete production [13-17]. Natural pozzolan materials, such as volcanic tuff, diatomaceous earth and volcanic glass have been used since the days of the ancient Romans. Whereas, the industrial by-product and agricultural waste materials, including ground granulated blast furnace slag (slag or GGBS), fly ash (FA), silica fume (SF), metakaolin (MK), rice husk ash (RHA), and sugar cane, were only introduced to the concrete industry during the last two centuries.

The incorporation of SCMs in concrete production provides technical, economic and ecological benefits [18]. Introducing SCMs in fine form enhances the concrete density due to the filling effect of pores between cement particles and provides a physicochemical effect (nucleating effect), which promotes the hydration of OPC [19]. Most SCMs contain a high amount of siliceous or siliceous and aluminous materials in a non-crystalline (i.e. amorphous or glassy) state. These materials are favoured components in concrete production because they have the ability to react with CH in the presence of moisture to form additional CSH phases [20]. Converting CH to CSH through pozzolanic reaction not only improves the strength but also enhances the physical and durability properties of concrete mixtures. The benefits of using SCMs include: improved workability, increased ultimate strength, low heat of hydration, reduced permeability, and enhanced resistance to chemical attack [16, 21].

1.3 Problem Statement

The incorporation of SCMs as cement replacement material can provide numerous benefits to the concrete industry. In addition, by enhancing the engineering properties of concrete, a significant reduction in the total consumption of OPC can be achieved. This reduction will have a significant positive impact on the environment by way of the reduced total CO_2 emissions. However, there are some technical and economic barriers associated with the use of the existing SCMs. The technical shortfalls include slow rate of strength development; prolonged period of curing; increased water demand; increased chemical admixture dosage; and difficulties in the placing of concrete [22, 23].

Moreover, some SCMs may need a further treatment process, such as grinding and calcined under controlled conditions before being used in concrete production [14, 24]. Furthermore, due to market demand and transportation costs, SCMs can end up being more expensive than the OPC itself when they are imported from other countries [25, 26]. Therefore, there is an urgent need to find alternative materials as good sources for siliceous materials to be used as partial cement replacement material.

In many parts of the world, there is an abundance of natural dune sand. The particle size distribution of dune sand has shown that it does not meet the standard limit of fine aggregate gradations of either BS 882 and ASTM C 33 [27]. This is because the maximum size of the dune sand grains is less than 900 µm. Therefore, the applications of dune sand have been limited to partial fine aggregate replacement, road construction, backfilling material, and sand concrete [27-30].

The characterization of the dune sand shows that it contains a high amount of SiO_2 (91%) in crystalline form. Unlike amorphous silica, crystalline silica does not react or hardly reacts with CH under normal conditions. However, the reactivity of this silica could be improved by a further grinding process or by thermal treatment under special conditions [5, 7, 31] To the best knowledge of the researcher, the potential for using ground dune sand (GDS) as a partial cement replacement material in binary and ternary combinations with slag and hydrated lime has not yet been investigated.

1.4 Objectives of the Study

The main objective of this study is to examine the potential of using GDS as partial cement replacement in binary and ternary blended system of OPC-GDS- slag and OPC-GDS- lime. The ultimate goal of this study is to establish broad scientific knowledge and the engineering behaviour of concrete containing GDS in binary and ternary combinations of blended cement. The general objectives of this study are shown below:

1. To identify an effective method that is suitable to activate the GDS to form a latent reaction in cement-blended mixtures.
2. To determine the optimum level of GDS in developing blended cement mixtures and the optimum combinations for the ternary blended system.
3. To examine the effectiveness of developing blended cement mixtures on the physical and durability properties.
4. To ascertain the microstructural features and underlying mechanism of hydration of mixtures containing GDS blended cement system.

1.5 Scope of the Study

In this study, the examination is limited to the development of blended cement mixtures incorporating GDS, slag and lime in binary and ternary blended mixtures. The scope of this study is designed as follows:

1. To examine the reactivity of GDS, a control and blended mixture containing 30% GDS as cement replacement material were fabricated.
2. To examine the suitable treatment methods (mechanical, chemical and thermal methods), six different mixtures incorporating treated GDS were cast.
3. To examine the optimum replacement level of GDS in binary blended cement, five mixtures with different replacement levels (0%, 10%, 20%, 30% and 40%) were fabricated.
4. To examine the suitable combination of slag or lime with blended system containing OPC and GDS as base cementing materials, 12 preliminary mixtures were cast. Then, eight ternary blended cement mixtures containing low (15%), moderate (30%) and high (45%) amounts of slag or lime were introduced into the blended system containing 40% GDS and different amounts of OPC as base cementing materials.
5. To examine the fresh properties, compressive strength and physical and durability properties of binary and ternary blended mixtures the normal consistency, setting time, workability, compressive strength, chloride ions permeability, drying shrinkage and resistance to sulfate attack tests were conducted.
6. To study the microstructure of control and blended cement mixtures, SEM, EDX, XRD, DTA and TGA analyses were conducted to ascertain and understand the underlying mechanisms of the micro-scale changes that occur in the hydrated blended cement pastes.

1.6 Significance of the Study

The successful use of GDS to reduce OPC consumption has a potential impact on the sustainability and economy of concrete production in the Middle East and other countries, where resources of natural dune sands are unlimited. Moreover, the use of GDS and the proposed combinations of blended cement mixtures would contribute to saving a large amount of OPC and reduce the negative environmental effects of OPC manufacturing.

1.7 Overview of the Thesis

The thesis consists of five chapters. Chapter 1 presents the introduction to the thesis in terms of general background, objectives of the study and the scope of the study. Chapter 2 provides a literature review related to OPC, SCMs, activation methods, and microstructure study on the hydrated cementing mixtures. A critical discussion focusing on the research objectives is presented at the end of this chapter. Chapter 3 discusses the material properties and experimental programme used to carry out this study. Chapter 4 presents the results and discussion of the data obtained from the developed experimental programme. Chapter 5 concludes the problems discussed throughout this thesis and highlights the contributions of this research and recommendations for future studies.

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