SHORT-TERM EFFECT OF SULPHATE ATTACK ON CONCRETE

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DEDICATION

This project report is dedicated to my husband, Chung Chuan Hin, who has been a constant source of strength and motivation during the challenges of postgraduate life. I dedicated this project to my son, Chung Sze Kai and my daughter, Chung Sze Qi, as a guide to working hard for the things that we strive to accomplish. This project is also dedicated to my parents, Chan Tuck Choy and Foong See Moi, who have always supported me. I am deeply thankful that I have all of you in my life. We live and learn.

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ABSTRACT

The concrete structures subject to aggressive environment under sulphate attack is of key importance to the service life of the structures. Previous studies have considered the full immersion method to evaluate the concrete resistance to sulphate attack. However, the integration of dry-wet cycles under short-term exposure of external sulphate attack as representing environmental factors in the service conditions was not fully explored in literature. The purpose of this study is to evaluate the properties of concrete under external sulphate attack and drying-wetting cycles for short-term exposure. The dry-wet cycle in this study was defined as immersion of concrete specimens in 5% and 10% sodium sulphate solutions for 6 days and placing at oven at 60^{o}C for 21 hours, followed by 3 hours at room temperature for drying purpose as representing environment factors in the service conditions. The drying process was conducted at the end of 7, 14, 21, 28, 35, 42, 49, 56, 63, 70, 77 and 84 days. Twelve drying-wetting cycles were conducted with regards to the actual weather conditions in Malaysia and each drying-wetting cycle was lasted for 7 days. Changes on properties over time in term of visual observation, change in mass, ultrasonic pulse velocity (UPV) and residual compressive strength of the concrete under different exposure conditions were performed. Concentration of sulphate ions (SO_4^{2-}) across the depth of specimens was measured by Ion Chromatography instrument. Microstructure study of the concrete degradation were observed under Field-Emission Scanning Electron Microscopy (FE-SEM) with Energy Dispersive X-ray Spectroscopy (EDX) analysis and X-ray Diffraction (XRD). From all the tests carried out, it was found the sulphate attack in 10% concentration after 84 days of drying-wetting cycles have a noticeable impact on the degradation of concrete. The results reported that exposing concrete to higher concentration of 10% sodium sulphate after 84 days of drying-wetting cycle, leading to higher mass loss at 0.75%, 9.55% reduction in UPV, 5.30% reduction in compressive strength and sulphate ions penetrated into concrete by reaching depth at 20 to 25 mm within 56 days of drying-wetting cycle. Gypsum, ettringite and calcite are the identified phases after 84 days drying-wetting cycles. Under repetitive drying-wetting cycles, such physical process can significantly accelerate the sulphate attack when high concentration of sodium sulphate travel through the concrete and get deposited at the surface, causing it to scale physically. Therefore, concrete would experience more severe deterioration when drying and wetting process existed in the environments with a higher sulphate content.

ABSTRAK

Struktur konkrit tertakluk kepada persekitaran yang agresif di bawah serangan sulfat sangat penting untuk jangka hayat struktur tersebut. Kajian terdahulu telah mempertimbang kaedah rendaman penuh untuk menilai ketahan konkrit terhadap serangan sulfat. Walau bagaimanapun, kitaran rendaman-pengeringan di bawah jangka pendek bagi serangan sulfat luaran sebagai faktor persekitaran tidak diterokai sepenuhnya dalam kajian lepas. Tujuan kajian ini adalah untuk menilai sifat konkrit di bawah serangan sulfat luaran dan kitaran rendaman-pengeringan untuk pendedahan jangka pendek. Kitaran rendaman-pengeringan dalam kajian ini didefinasikan sebagai rendaman specimen konkrit dalam larutan natrium sulfat 5% dan 10% selama 6 hari dan meletakkan pada ketuhar pada suhu 60^{o}C selama 21 jam, diikuti oleh suhu bilik 3 jam untuk tujuan pengeringan, mewakili faktor persekitaran. Proses pengeringan dilakukan pada akhir 7, 14, 21, 28, 35, 42, 49, 56, 63, 70, 77 dan 84 hari. Dua belas kitaran rendaman-pengeringan dilakukan mewakili keadaan cuaca sebenar di Malaysia dan setiap kitaran rendaman-pengeringan berlangsung selama 7 hari. Perubahan sifat dari semasa ke semasa dari segi pemerhatian visual, perubahan jisim, ujian pecutan denyutan ultrasonik (UPV) dan ujian mampatan konkrit dalam keadaan pendedahan yang berbeza dilakukan. Kepekatan ion sulfat (SO_4^{2-}) di kedalaman spesimen diukur dengan instrument Kromatografi Ion. Kajian struktur mikro degradasi konkrit diperhatikan di bawah pengimbasan pelepasan mikroskop (FE-SEM) dengan analisis Spektroskopi sinar-X Penyebaran Tenaga (EDX) dan Difraksi sinar-X (XRD). Dari semua ujian yang dilakukan, didapati serangan sulfat dalam kepekatan 10% setelah 84 hari kitaran rendaman-pengeringan mempunyai kesan yang nyata terhadap degradasi konkrit. Hasil yang dilaporkan menunjukkan bahawa pendedahan konkrit kepada kepekatan natrium sulfat yang lebih tinggi, 10% selepas 84 hari kitaran rendaman-pengeringan, menyebabkan penurunan berat yang lebih tinggi iaitu 0.75%, pengurangan nilai UPV sebanyak 9.55% dan pengurangan kekuatan mampatan sebanyak 5.30% serta ion sulfat menembusi konkrit dengan mencapai kedalaman pada 20 hingga 25 mm dalam 56 hari kitaran rendaman-pengeringan. Gipsum, ettringit dan kalsit adalah fasa yang dikenal pasti setelah 84 hari kitaran rendaman-pengeringan. Di bawah kitaran rendaman-pengeringan berulang, proses fizikal seperti itu dapat mempercepat serangan sulfat dengan ketara apabila kepekatan natrium sulfat yang tinggi bergerak melalui konkrit dan didepositkan ke permukaan, menyebabkannya mengelupas secara fizikal. Oleh itu, konkrit akan mengalami kemerosotan yang lebih teruk apabila proses rendaman-pengeringan wujud di persekitaran dengan kandungan sulfat yang lebih tinggi.

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LIST OF ABBREVIATIONS

Al	-	Aluminium
ASTM	-	American Society for Testing and Materials
BS	-	British Standards
Ca	-	Calcium
$Ca(OH)_2$	-	Calcium Hydroxide
C-S-H	-	Calcium Silicate Hydrate
C3A	-	Tricalcium Aluminate Hydrates
DOE	-	Department of the Environment
EDX	-	Energy Dispersive X-ray Spectroscopy
FE-SEM	-	Field-Emission Scanning Electron Microscopy
OPC	-	Ordinary Portland Cement
O_2	-	Oxygen
PPA	-	Pusat Perkhidmatan Analysis
S	-	Sulfur
SEM	-	Scanning Electron Microscopy
Si	-	Silicon
UPMU	-	University Laboratory Management Unit
UPV	-	Ultrasonic Pulse Velocity
UTM	-	Universiti Teknologi Malaysia
XRD	-	X-ray Diffraction

LIST OF SYMBOLS

$m(\%)$	-	Percent of change in mass
$m_{initial}$	-	Cube specimen mass before first drying-wetting cycle (control specimen)
m_t	-	Cube specimen mass at t days after drying-wetting cycle
$f_c(\%)$	-	Percent of change in compressive strength
$f_{c(t=0)}$	-	Compressive strength of the concrete mixture before first drying-wetting cycle (control specimen)
$f_{c(t\ days)}$	-	Compressive strength at t days after drying-wetting cycle

CHAPTER 1

INTRODUCTION

1.1 Introduction

Concrete has become the most widely used building material in civil engineering. It is the main structural material due to its advantages of easy availability of raw materials and flexibility. Concrete is strong in compression but inherently weak in tension. It can form cracks and voids due to thermal expansion and shrinkage. As a consequence, these voids will permeate water through the concrete. Concrete manufactured using Ordinary Portland Cement (OPC) cannot be considered durable concrete unless it could maintain its original form and quality when subjected to weathering and loading effects, and chemical reactions over the intended duration. However, concrete made with OPC is not recommended to construct the structure under the aggressive environment due to the brittleness of OPC concrete and the high risk of material deterioration. The chemically aggressive environment for concrete includes alkali-aggregate reaction, carbonation, chloride attack, sulphate attack and acid attack. The durability performances of concrete will be reduced as the concrete structure starts to deteriorate due to its environment.

The concrete structures may subject to aggressive environments to such a very varied situations and the range of aggressive agents is extensive. Among the many circumstances, concrete structures in contact with soils, groundwaters and wastewaters are exposed to higher concentration of sulphate. Jetty and port facilities may be subject to the combined action of magnesium, sulphates and chlorides from the seawater. Foundation is experiencing significant degradation notably associated with organic acid in wastewater. Chemical attack is one of the major durability problems for the OPC concrete degradation. Insights into deterioration mechanism by such aggressive agents is crucial element towards the development of concrete performance in these environments and an improvement in service life of the structures.

1.2 Background of Problem

Sulphates exist in nature and can be used in commercial applications. Sulphates can penetrate into concrete or may be incorporated into cement-based materials while reacting with cement compounds to form volumetric expansive products. Thus, high concentrations of sulphates may greatly influence on concrete performance and service life. Service life of structural tends to reduce and it can lead to progressively damaged and failure.

An early effort to improve the resistance of concrete to sulphate attack was achieved by the finding on sulphates react with hydrates of the cement and trigger the growth of concrete expansion. Following by the field investigation on the sulphate attack mechanism and evaluating the durability of concrete under sulphate-rich environments. Most sulphate resistance findings have been obtained from accelerated test carried out in laboratory where the result parameters are entirely based on macroscopic properties, specifically in expansion (Chabrelie et al., 2011).

While sulphate attack on concrete has been under research for over 60 years, the mechanisms of attack are still uncertain, and the failure of concrete is still existing. There are numerous studies work over the past decades on development of the time-dependent diffusion model and degradation mechanism of concrete under external sulphate attack. It has been reported that the mechanism of degradation was primarily attributed to the formation of gypsum and ettringite (Sarkar et al., 2010; Sun et al., 2013; Cefis and Comi, 2017). Most recent studies as well as current work focus on degradation mechanism of concrete induced by internal and external combined sulphate attack and drying-wetting cycles (He et al., 2020; Li et al., 2020; Zhao et al., 2020). In most of these contexts, concrete specimens were fully immersed in sulphate-containing solutions so that the test conditions are more than just those encountered by concrete in service or under real conditions, accelerating the sulphate accumulation and deterioration of concrete.

1.3 Problem Statement

Now with the development of modern science and technology, the deficiencies of the conventional concrete are being overcome. Many studies on the need to develop a sustainable built environment such as the use of alternative materials from industrial waste were carried out to improve the engineering properties of concrete, minimizing the use of normal aggregates, and adding admixtures can significantly shorten hardening and improve the mechanical properties. However, the use of alternative building materials in the concrete production are not favoured by many. Depending on the local resources available, the use of conventional concrete is gaining more favour.

However, lack of field data concerning concrete durability, specifically in the context of sulphate aggressive environments in Malaysia, makes it difficult to evaluate the performance of concrete except in reference to the established international standard on the limits for the aggressive sulphate content in water and soil. Also, there are limitations use of accelerated tests results on concrete specimens in presenting the sulphate attack mechanism in the field due to the varying levels of sulphates. The impact of gypsum and ettringite on the damage mechanism of concrete induced by external sulphate attack in short-term exposure remains uncertain and to be determined.

Another promising line of research would be the damage mechanism under external sulphate attack and drying-wetting cycles for short-term exposure has not been widely explored successfully. Malaysia experiences the tropical weather which is hot and humid all year round, frequently accompanied with tropical rain showers. Hence, to investigate the resistance of concrete under sulphate attack, drying-wetting cycles may reflect and simulate such real environment in Malaysia, instead of application of full immersion to simulate the actual conditions. The effect of sulphate-containing solution concentration, and penetration of sulphate ion into concrete has been previously assessed only to a very limited extent due to the limited studies in this area for the purpose of determination on how extent of the penetration occurs in the concrete. The modes of failure in durability will be of interest to designers and constructor in evaluation of design and construction of concrete in the sulphate-rich environments. Therefore, by examining the short-term effect of sulphate on concrete could be feasible solution to determine how extent of the penetration occur in the concrete.

1.4 Aim and Objectives of the Research

The aim of this study is to investigate the degradation of concrete caused by external sulphate attack and drying-wetting cycles for short-term exposure. The following objectives of the research are set:

(a) To evaluate the changes on properties over time in term of visual observation, change in mass, ultrasonic pulse velocity (UPV) and residual compressive strength of the concrete under different exposure conditions.

(b) To investigate the depth of penetration of sulphate ions into concrete under different exposure conditions.

(c) To investigate the reaction of aggressive agents on concrete based on microstructure analysis.

1.5 Scope of Study

The scope of study was highly focused on experimental research. Testing methods met the procedures as stated in American Society for Testing and Materials (ASTM) and British Standards (BS). The experimental research was conducted by using the availability of equipment at the Pusat Perkhidmatan Analysis (PPA) and University Laboratory Management Unit (UPMU) in Universiti Teknologi Malaysia (UTM).

The concrete was designed according to Department of the Environment (DOE) (Teychenné et al., 1997) with the compressive strength of 30 MPa at 28 days and water-cement ratio of 0.53. The specimens were subjected to immersion in concentration of sodium sulphate solutions with 5% and 10% for 6 days and placing at oven at 60^{o}C for 21 hours, followed by 3 hours at room temperature for drying purpose. The drying operation was performed at the end of 7, 14, 21, 28, 35, 42, 49, 56, 63, 70, 77 and 84 days. Tests before and after drying-wetting cycles at specified periods included visual observation on the specimens, determination of change in mass, UPV and residual compressive strength. The sulphate concentration of the specimens at different depth of penetration (5, 10, 15, 20 and 25 mm) was tested using Ion Chromatography instrument. Field-Emission Scanning Electron Microscopy (FE-SEM) with Energy Dispersive X-ray Spectroscopy (EDX) and X-ray Diffraction (XRD) are the tools used to carry out the microstructure analysis.

1.6 Significance of Study

This study is be of benefit in the following ways:

(a) As a reference tool to practitioners by providing understanding of the sulphate attack mechanism under drying-wetting cycles for short-term exposure at the early stage and how extent the penetration of sulphate ions into concrete may occur.

(b) The modes of failure in durability will be of interest to designers and constructor in evaluation of design and construction of concrete in the sulphate-rich environments at the early stage.

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