DURABILITY OF SELF-COMPACTING CONCRETE WITH COAL BOTTOM ASH AS SAND REPLACEMENT MATERIAL UNDER AGGRESSIVE ENVIRONMENT

AHMAD FARHAN BIN HAMZAH

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Universiti Tun Hussein Onn Malaysia

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Special dedication to the memory of
Allahyarham Muhammad Faiz Hamzah.
May ALLAH bless his soul and forgiveness.

Al-Fatihah
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ABSTRACT

Concrete is a major building material use for numerous purposes in construction including the concrete structures that exposed to aggressive environment or seawater such as coastal berthing facilities, breakwaters, retaining walls, tidal barriers, dry docks, container terminals, off-shore floating docks and drilling platforms. As these concrete structures that usually exposed to an aggressive environment, it is expected to require a minimum level of repair or maintenance during their service life. The ingress of aggressive agents, in particular chloride and sulphate ions, can lead to corrosion of reinforcing steel bars and thus cause a reduction in strength and subsequent decrease in the service life of concrete structures. In context to resist this type of premature deterioration, concrete must be proportioned to achieve high durability in aggressive environments. The incorporation of coal bottom ash as sand replacement material was possible to help self-compacting concrete in designing mix proportion which contains extra fine particles content and fewer amounts of coarse aggregates. On the other hand, self-compacting concrete was developed to respond to the need for a self-compacting concrete with improved durability. In this study, six replacement levels were considered for self-compacting concrete: 0%, 10%, 15%, 20%, 25% and 30% by volume. The essential workability properties of the fresh self-compacting concrete containing coal bottom ash were prepared and evaluated by the test of slump flow, L-box and sieve segregation resistance. Later, the effect of coal bottom ash subjected to sodium chloride (NaCl), sodium sulphate (Na₂SO₄) and seawater through cyclic wetting and drying was also investigated. The durability performance of the coal bottom ash self-compacting concrete exposed to aggressive environment was evaluated through compressive strength, Rapid Chloride Permeability Test (RCPT), chloride penetration by Rapid Migration Test (RMT) and carbonation depth test. In addition, microstructural changes that occur in specimens due to aggressive environmental effects were identified through X-ray diffraction (XRD) techniques and Scanning Electron Microscopy (SEM). Test results show that coal bottom ash can be acceptably used as a fine aggregate replacement material in order to achieve good durability of concrete. The self-compacting concrete containing 10% coal bottom ash replacement showed excellent durability to chloride, sulphate and seawater attack. The test results also indicate that the amount of calcium hydroxide (Ca(OH)₂) in the 10% coal bottom ash concrete was slightly lower than that of control sample due to the pozzolanic reaction of coal bottom ash and cement. The combination of compressive strength, XRD and SEM analysis leads to the identification of Friedel’s salt, ettringite, gypsum, calcium hydroxide formations in specimen.
ABSTRAK

Konkrit adalah bahan binaan utama yang digunakan untuk pelbagai tujuan dalam pembinaan termasuk struktur konkrit yang terdedah kepada persekitaran yang agresif atau air laut seperti kemudahan pantai (berthing), pemecah ombak, dinding penahan (retaining wall), benteng pasang surut, dok kering, terminal kontena, dok terapung di luar pantai dan penggerudian pelantar. Oleh kerana struktur konkrit tersebut terdedah kepada persekitaran yang agresif, ia memerlukan tahap pembaikan yang minimum atau penyelenggaraan semasa hayat perkhidmatan. Pendedahan dan kemasukan agen agresif, terutamanya ion klorida dan sulfat boleh mengakibatkan kakisan bar tetulang keluli dan menjejaskan kekuatan dan seterusnya menyebabkan penurunan hayat perkhidmatan struktur konkrit. Justeru, bagi mengelakkan kemerosotan konkrit ini, konkrit perlu disesuaikan untuk mencapai ketahanan yang tinggi dalam persekitaran yang agresif. Penggunaan abu enapan arang batu (coal bottom ash) sebagai bahan pengganti pasir adalah sesuai untuk menghasilkan konkrit tanpa getar (self-compacting concrete) dalam merancang nisbah campuran yang mengandungi kandungan zarah halus dan jumlah agregat kasar yang sedikit. Sehubungan dengan itu, konkrit tanpa getar telah dibangunkan dengan ketahanan yang lebih baik. Melalui kajian ini, enam tahap penggantian berdasarkan volumetrik telah dipertimbangkan untuk konkrit tanpa getar iaitu 0%, 10%, 15%, 20%, 25% dan 30%. Sifat penting kebolehkerna dalam konkrit tanpa getar yang mengandungi abu enapan arang batu dinilai oleh ujian aliran turunan (slump flow), ujian L-box dan ujian rintangan pengasingan. Selain itu, kesan abu enapan arang batu yang tertakluk kepada natrium klorida (NaCl), natrium sulfat (Na₂SO₄) dan air laut melalui kitaran pembasahan dan pengeringan juga dikaji. Prestasi ketahanan konkrit tanpa getar mengandungi abu enapan arang batu yang terdedah kepada persekitaran yang agresif telah dinilai melalui ujian kekuatan mampatan, ujian kebolehpan ion-klorida pantas (RCPT), penemuan ion-klorida oleh ujian migrasi pantas (RMT) dan ujian karbonasi. Di samping itu, perubahan mikrostruktur yang berlaku dalam spesimen kerana kesan alam sekitar yang agresif telah dikenal pasti melalui teknik X-ray pembelauan (XRD) dan Scanning Electron Microscopy (SEM). Keputusan ujian menunjukkan bahawa abu enapan arang batu boleh digunakan sebagai bahan gantian agregat halus untuk mencapai ketahanan konkrit yang baik. Penggantian sebanyak 10% abu enapan arang batu menunjukkan ketahanan yang sangat baik untuk persekitaran klorida, sulfat dan air laut. Keputusan ujian juga menunjukkan bahawa jumlah kalsium hidroksida (Ca(OH)₂) dalam spesimen konkrit penggantian 10% abu enapan arang batu adalah lebih rendah daripada sampel kawalan yang disebabkan oleh tindakbalas pozzolanik abu enapan arang batu dan simen. Berdasarkan keputusan RCPT dan RMT, kadar penembusan ion-klorida meningkat dengan peningkatan abu enapan arang batu di dalam konkrit. Hasil ujian karbonasi bagi spesimen yang terdedah kepada air laut melalui kitaran pembasahan-pengeritingan menunjukkan
bahawa hasil dapatan adalah selari dengan spesimen yang terdedah kepada persekitaran klorida dan sulfat. Berdasarkan hasil keputusan ujian, dapat diperhatikan bahawa kedalaman pengkarbonan spesimen terdedah kepada persekitaran yang agresif pada tahap penggantian sebanyak 10% abu enapan arang batu adalah paling rendah berbanding spesimen yang lain. Ini menunjukkan bahawa rintangan pengkarbonan adalah sangat tinggi dengan penggunaan nisbah penggantian sebanyak 10%. Justeru, gabungan hasil ujian kekuatan mampatan, analisis XRD dan SEM telah membawa kepada pengenalpastian garam Friedel, ettringite, gypsum, kalsium hidroksida dan lain-lain sebatian kimia dalam spesimen.
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LIST OF ABBREVIATION

ACV  Aggregate Crushing Value
AIV  Aggregate Impact Value
ASTM American Society for Testing and Materials
BS EN British Standard
CBA Coal Bottom Ash
CSH Calcium silicate hydrate
EFNARC European Federation of National Associations Representing for Concrete
RCPT Rapid Chloride-ion Permeability Test
RMT Rapid Migration Test
SCC Self-compacting concrete
SEM Scanning Electron Microscopy
XRD X-ray Diffraction
XRF X-ray Fluorescence
### LIST OF SYMBOL

<table>
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<tr>
<td>Ca(OH)$_2$</td>
<td>Calcium hydroxide</td>
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<tr>
<td>CaCO$_3$</td>
<td>Calcium carbonate</td>
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<tr>
<td>CaO</td>
<td>Calcium oxide</td>
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<tr>
<td>CO$_2$</td>
<td>Carbon dioxide</td>
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<tr>
<td>C$_{PR}$</td>
<td>Chloride penetration rate (mm/v.hr)</td>
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<tr>
<td>$f'_{ch}$</td>
<td>Average compressive strength subjected to chloride solution with wetting-drying cycles</td>
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<td>$f'_{dw}$</td>
<td>Average compressive strength cured in tap water</td>
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<tr>
<td>$f'_{sl}$</td>
<td>Average compressive strength subjected to sulphate solution with wetting-drying cycles</td>
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<td>$f'_{sw}$</td>
<td>Average compressive strength subjected to seawater with wetting-drying cycles</td>
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<tr>
<td>$f_{ct}$</td>
<td>Compressive strength (N/mm$^2$ or MPa)</td>
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<tr>
<td>$f_{fl}$</td>
<td>Flexural strength (N/mm$^2$ or MPa)</td>
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<tr>
<td>$f_{st}$</td>
<td>Split tensile strength (N/mm$^2$ or MPa)</td>
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<tr>
<td>H$_2$CO$_3$</td>
<td>Carbonic acid</td>
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<tr>
<td>H$_2$O</td>
<td>Water</td>
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<tr>
<td>m$_p$</td>
<td>Weight of pan</td>
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<tr>
<td>m$_{pl}$</td>
<td>Weight of sample</td>
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<tr>
<td>m$_s$</td>
<td>Weight of laitance</td>
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<td>Na$_2$SO$_4$</td>
<td>Sodium Sulphate</td>
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<tr>
<td>NaCl</td>
<td>Sodium Chloride</td>
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<td>$W_d$</td>
<td>Dry weight of specimen after 24 hours in oven</td>
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<td>$WL_{ch}$</td>
<td>Average initial weight of specimens subjected to chloride solution with wetting-drying cycles</td>
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<td>$WL_{sw}$</td>
<td>Average initial weight of specimens subjected to seawater with wetting-drying cycles</td>
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<tr>
<td>$W_{ssd}$</td>
<td>Weight of sample in saturated surface dry condition in air</td>
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<tr>
<td>$W_{ssw}$</td>
<td>Weight of saturated sample in water</td>
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CHAPTER 1

INTRODUCTION

1.1 Introduction

Concrete is one of major building materials used for numerous purposes in construction such as buildings, dams, foundations, highways, and others (Matthias, 2010). Often, the structures exposed to aggressive environments or seawater such as coastal berthing facilities, breakwaters, retaining walls, tidal barriers, dry docks, container terminals, off-shore floating docks, and drilling platforms. Therefore, it is expected that the concrete structures will require a minimum maintenance during their service life. The ingress of aggressive agents, particularly chloride and sulphate ions, can lead to corrosion of reinforcing steel bars, therefore will cause a reduction in strength and the service life of concrete structures. In context to resist this type of premature deterioration, concrete must be proportioned to achieve high durability in an aggressive environment.

A common method used to prevent premature deterioration of concrete structures is the application of concrete mixture in which designed to be relatively impermeable. However, deterioration on a structure can usually be attributed to poor construction practice and not from the properties of the concrete mixture itself. The poor construction practices (construction factors) are including improper casting, concrete placement, consolidation techniques, and inadequate curing regime which eventually will lead to surface cracking, poor compaction, and high concrete permeability. Of these factors will reduce the resistance of concrete for the aggressive agents to ingress and consequently led to premature corrosion of reinforcing steel and deterioration on the concrete structure.
Interestingly, the current construction specifications and codes including normal vibrated concrete exposed to marine or aggressive environments are only emphasize on the quality of the concrete mixture itself. The construction specifications are including recommendations regarding the water to cement (w/c) ratio, minimum cement content, and concrete cover thickness, however the effect of construction factors is not highlighted. For example, the American Concrete Institute (ACI) Committee 357 on Design and Construction of Fixed Concrete Offshore Structures (ACI Committee 357 1994) has recommended that concrete with a maximum w/c ratio of 0.45, a minimum cement content of 400 kg/m$^3$ and minimum cover thickness of 50 mm, can be used for marine structures. Similar requirements are also specified in other codes such as British Standards and European Codes (British Standard, BS 8110, 1985 and European Pre-Standard, ENV-206 1992). It should be noted that the in-situ quality of such concrete is not solely a function of its w/c ratio, cement content, cover thickness, but also a function of the construction factors. Essentially, the durability of concrete structure is proven by the production of a workable concrete mixture, its effective consolidation, and the application of proper curing techniques. The latter is sometimes very difficult to specify due to the lack of skilled workers and variations in construction techniques among the construction companies.

The improper casting, placement, and consolidation of typical concrete mixtures can lead to high concrete permeability and thus impair the durability of the structures that exposed to aggressive environment. Additionally, the effect of high temperature will cause poor fluidity of typical fresh concrete and therefore will create difficulty in placing of such concrete at the field. This will need a considerable amount of vibration and consolidation. There are few cases in which normal vibration and consolidation techniques have not produced proper consolidation although a high range water reducing admixtures has been applied. Such cases can be found especially where structural members require congested reinforcement or when unusual shapes and geometries are specified. If concrete is not properly consolidated, voids, gaps, and cracks can be easily formed in the concrete structure. Furthermore, over vibration may be applied if the concrete mixture is very stiff or in case of congested reinforcement, which can result in bleeding and segregation. These defects may occur as a result of poor vibration and will therefore reduce the permeability of concrete and will impair
the ability to resist aggressive agents. Consequently, it will reduce the concrete’s serviceability.

A large number of concrete structures has been built in accordance with recent codes with exposed to aggressive environments have showed considerable deterioration before the end of their service lives due to poor construction practices. Because the specifications and codes for vibrated concrete do not account for possible effects of construction practices, it has been postulated that without adequate compaction and curing, even correctly proportioned concrete will often not be as impermeable as expected on the basis of its mixture composition.

Previous research have suggested that coal bottom ash as sand replacement material which has particles appear similar to the natural size and, more recently, self-compacting concrete has provided significant improvements in both its fresh and hardened properties (Domone, 2006; Aggarwal et al., 2008; Pathak & Siddique, 2012; Kadam & Patil, 2013). The incorporation of coal bottom ash as sand replacement material was found to help self-compacting concrete in designing mix proportion which contains extra fine particles content and fewer amounts of coarse aggregates (Dubey & Kumar, 2012). On the other hand, self-compacting concrete was developed to respond to the need for a self-compacting concrete with improved durability. Therefore, the use of self-compacting concrete can minimize or even eliminate the detrimental effects of construction factors. Additionally, the combination of the benefits of coal bottom ash and self-compacting concrete can provide possible durability advantages to the concrete structures in aggressive environments. There are many issues which are lack of knowledge in its characteristics in relation to the behaviour mainly in self-compacting concrete. Hence, further research about the self-compacting concrete expose to aggressive environment, characterization of self-compacting concrete using laboratory test methods, and construction issues related to self-compacting concrete are practical.
1.2 Problem Statement

The durability of concrete has become a major concern in the construction industry. It is also known that aggressive environments are the major factor affecting the durability of concrete (Zuquan et al., 2007). The deteriorative of concrete is the resulted from an ineffectiveness durability that might cause by physical, mechanical and chemical factors and of these can be induced by external and internal factors to the concrete structure. Physical and chemical deterioration are external influenced from climate such as weathering, variation of temperature, wetting and drying cycles and exposure to aggressive environment (Hekal et al., 2002; Mohr et al., 2005). Chemical deterioration is usually determined by the rate at which concrete is decomposed as a result of chemical reaction. The aggressive chemicals are greatly affected the concrete durability which include chlorides and sulphates with their related cations.

Degradation consists of dissolution of calcium and hydroxide ions out of the matrix when chloride dissolved in water, which causes an increase in porosity and transport properties; and subsequently, affecting the strength and permeability of concrete (Rozière et al., 2009). According to Bertolini et al. (2004), the increment of coal bottom ash content in concrete shows a decreasing trend in the porosity of concrete. As the degradation of chloride-water dissolved caused an increase in porosity, potentially the coal bottom ash will be employed in the concrete due to pore refinement. This process is vital to produce good quality of durable concrete. Calcium, sodium, magnesium and ammonium sulphates may involve forming a progressive loss of strength and loss of mass due to loss of cohesiveness in the cement hydration products. Gypsum formation leads to reduction of stiffness and strength, then by expansion and cracking. If concrete cracks, its permeability increases and the aggressive chemical solution penetrate more easily into the interior, thus accelerating the process of deterioration (Bassuoni & Nehdi, 2009; Monteiro & Kurtis, 2003).

Previous studies have suggested that coal bottom ash as sand replacement material has particles appear similar size to natural river sand and, more recently, self-compacting concrete provide concrete with significant improvements in both its fresh and hardened properties (Domone, 2006; Aggarwal et al., 2008; Pathak & Siddique, 2012; Kadam & Patil, 2013). The incorporation of coal bottom ash as sand replacement material is possible to help self-compacting concrete in designing mix
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1.3 Research Objectives

This research involves experimental study on self-compacting concrete. The aims of this research are:

i. To investigate the physical and mechanical properties of self-compacting concrete incorporating coal bottom ash with different percentage of sand replacement and optimum content of coal bottom ash sand replacement.

ii. To investigate the microstructure of self-compacting concrete incorporating coal bottom ash with different percentage of sand replacement.

iii. To evaluate the performance of coal bottom ash self-compacting concrete with different percentages of sand replacement subjected to aggressive environments (sodium chloride, sodium sulphate and seawater) by wetting and drying cycles.
1.4 Research Significant

Self-compacting concrete signify the most important progress in concrete knowledge for over decades. This type of concrete has been designed to make sure sufficient compaction process and to ease the progress of concrete placement with complex geometry areas and fully reinforced. The process is complicated or impracticable to use mechanical compaction for fresh concrete. As the durability of concrete turns out to be a crucial issue in exploring the potential cementitious materials, a good compaction by expert workers was essential to get durable concrete structures (Bouzoubaa & Lachemi, 2001; Zhu & Bartos, 2003).

Even there are advantages of utilization of self-compacting concrete; this type of high performance concrete has not attracted local attention although it has been introduced for the last 10 years especially in Malaysia. Mewah & Ehsan (2002) acknowledged that the delivery of high strength concrete by the local ready mix industry is not a current problem. There are many benefits from spectacular design and complex engineering whereby numerous high-rise structures have been constructed such as Petronas Twin Tower and Menara Telekom Malaysia. The Menara Telekom is a headquarters office that has been recognized as a noticeable project which composed of excellence concrete technology and the use of high strength concrete.

The knowledge of high strength concrete and self-compacting concrete have reached across the world, which driven by the concerned of poor compaction and durability (Ahmadi et al., 2007; Madandoust & Mousavi, 2012; Azeredo & Diniz, 2013). However, the awareness about self-compacting concrete is still unfavourable and this has showed the lack of commercial use of self-compacting concrete in the Malaysia so far. The negative response in utilization of self-compacting concrete is possibly due to limited research on locally utilizing this type of concrete. Moreover, potential problems for the production and manufacturing self-compacting concrete with local marginal aggregates due to accessibility of raw materials have an impact on the self-compacting concrete consumerism in the industry as well as for environmental concern by considering the construction cost, ecosystem and environmental aspect, various option and alternative towards the utilization of local construction materials are must be established. Mehta (2004) stated that one resolution to cut the cost of self-
compacting concrete is by using mineral admixtures or waste materials such as limestone powder, natural pozzolans, ground granulated blast furnace slag and fly ash.

Malaysia is a country that has hot and humid climate all the year, with an average temperature of 27°C (80.6 °F) and small variability in the annual temperature. The climate is characterized by high temperature and humidity with moderate fluctuations in seasonal temperature and humidity. Conventional concrete admixtures is greatly examined which subjected to aggressive environment such as sodium chloride (NaCl), sodium sulphate (Na₂SO₄) solution and seawater. However, the knowledge on exposure of fresh or harden self-compacting concrete to the harsh condition are still limited Therefore, it is necessary to carry out a study on self-compacting concrete using local admixture materials incorporating suitable superplasticizer subjected to aggressive environment with wetting and drying cycles that signify the region in Malaysia. Moreover, a preliminary standard mix design nomograph of self-compacting concrete incorporating bottom ash will be produced for future references.

1.5 Scope of Works

This study is focusing on the material performance of the self-compacting concrete. The performance analysis were involved studying the characteristic of raw material of coal bottom ash in terms of physical and mechanical properties to investigate the durability and performance of self-compacting concrete containing coal bottom ash with different proportion of fine aggregate replacement subjected to aggressive sodium chloride, sodium sulphate solution and seawater with wetting and drying cycles.

Standard strength concrete containing regular blended cement was designed and prepared through a series of trial mixes to achieve a minimum compressive strength of 40 MPa at the age of 28 days. The coal bottom ash replacement levels of 10%, 15%, 20%, 25% and 30% were used to replace sand. The experimental program involved studying the characteristics of materials in terms of their physical and mechanical properties, chemical compositions, particle morphology and phase identification. The laboratory testing were conducted at the ages of 7, 28, 60, 90 and
180 days to study the effects of sodium chloride (NaCl), sodium sulphate (Na$_2$SO$_4$) and seawater on the durability of self-compacting concrete containing coal bottom ash with repetitive wetting and drying cycles including concrete exposed with normal water. The selection of chemical is based on dominant chemicals in marine environment, as the most is chloride, sodium and sulphate.

Results from laboratory testing of different mixture of self-compacting concrete were analysed. There are 3 different mixture of self-compacting concrete with different water/cement ratio which is 0.35, 0.4 and 0.45. The data obtained from the experiment were analysed comprehensively to present a design mix nomograph of self-compacting concrete and finally to meet the objective of this study.
1.6 Structure of Thesis

The thesis consists of eight chapters:

Chapter one discusses on the background of the study with a short overview of the current situation, research and problem statement. Several research needs are also identified. The objectives and scope of study are also presented.

Chapter two presents a background of the use of pozzolanic materials, its characteristic and performance in concrete durability. The description on various chemical solutions including chloride solution, sulphate solution and aggressive seawater related to concrete were discussed.

Chapter three provides an overview of the research program and approach, describes the preparation and testing of materials, discusses the selection and testing of constituent materials, and highlights the procedures for experimental work, apparatus and mixture design.

Details of finding and data about self-compacting concrete are presented in chapter four. It has included various degrees of sand replacement on coal bottom ash and different water-cement ratios on self-compacting concrete. Fresh and hardened properties of concrete are analysed and presented in an appropriate forms. The chapter discusses the significance of these results and determines the optimum percentage of coal bottom ash incorporated in concrete. Additionally, mix design nomograph are presented base on the results.

In chapter five, the influences of different replacement levels of coal bottom ash to normal sand exposed to sodium chloride and sodium sulphate solution with wetting and drying cycles are described. All results were presented in this chapter.

Chapter six discusses the performance of self-compacting concrete with coal bottom ash subjected to seawater. Moreover, the chapter focuses on the exposure of seawater from wetting and drying cycles and highlights the importance of these results.

Finally, chapter seven provides a summary of the research findings, presents the relationships and contributions, and present some recommendations for the future study.
CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Concrete is a man-made construction material and has been extensively used in the world (Gambhir, 2006). According to Mehta & Monteiro (2006), concrete is made of the basic ingredients of Portland cement, aggregates and water. Nowadays, the rapid development in all areas of civil engineering has led to increase demand for concrete. As a result many researchers have directed their attention towards developing concrete that suits the contemporary requirements and proficient in the aggressive environment. The concrete technology has been developing along with problem arises. This chapter discusses the effect of aggressive environment and factors that influence the formation of the crystallizing salt in the concrete structures. A general literature review of research carried out on method for aggressive environment simulation and material of coal bottom ash in self-compacting concrete are also presented.

2.2 Salt attack in concrete material

Salt attack is a controlled phenomenon to make the material more durable. The exposure of salt attack for a longer period where the salt residue crystallised in the pores of materials such as concrete and other masonry materials which can cause the disintegration of concrete matrix and structure. The salt is transferred through water
that may be carried in the various surrounds of the material. When the water is evaporated through dryer area, the salt residuals are deposited in the pores of the concrete material. This reoccurrence of wetting and drying of the pores will eventually cause a slow rise of salt in the pores and capillaries of the material resulting in a more concentrated salt remnant. At this time, the crystals have formed. As the crystals grow, micro-particles from the mortar will expand, thus exerting internal forces and stresses. The internal forces exerted by the salt crystals exert may lead to the eventual damage to the concrete material.

2.3 Factors affecting salt attack on concrete

The deterioration of concrete due to salt attack is considered as a complex problem and it depends on many parameters related to materials and condition of exposure. This can be found from Table 2.1 below.

<table>
<thead>
<tr>
<th>Dependence</th>
<th>Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material properties</td>
<td>Cement types and composition, mineral admixture type, water binder ratio, degree of hydration and curing conditions</td>
</tr>
<tr>
<td>Hydrated concrete properties</td>
<td>Pore structure, permeability, diffusivity, mechanical properties</td>
</tr>
<tr>
<td>Exposure conditions</td>
<td>Temperature, type of ions and concentration</td>
</tr>
</tbody>
</table>

2.3.1 Cement type and composition

The salt attack caused by sulphate is depends on availability of Ca(OH)$_2$ and C$_3$A in the hydrated concrete, hence chemical composition of binder have an important role in the salt attack resistance. Cement that contains low C$_3$A has good sulphate resistance. Sulphate Resisting Portland Cement (SRPC) has less content of C$_3$A, therefore it is protected under sulphate attack. Too high cement content in the concrete may cause increasein the sulphate resistance (Rodriguez-Camacho & Uribe-Afif, 2002). Modern cement is made for rapid development of strength in which results to an increase of tricalcium silicate (C$_3$S) contents in the cement. The susceptibility to sulphate attack will be enhanced from the increase of calcium hydroxide content in
the hardened cement which resulted from the increased of $C_3S$ (Sancak, & Özkan, 2015).

Blending of mineral admixtures such as ground granulated blast furnace slag (GGBFS), fly ash (FA), silica fume (SF) in cement increases resistance of concrete to sulphate attack. The superior performance of blended cement over plain cement concrete is attributed by the pozzolanic reactions that consumed the calcium hydroxide and the reduction of the plain cement quantity in total binder will dilute the calcium aluminate hydrates phase. Hekal et al. (2002) reported that the hardened cement paste that contained 40% GGBFS shows a significant improvement in the sulphate resistance.

### 2.3.2 Water binder ratio

Water to binder ratio is the key factor of the mechanism strength in the development of concrete. The concrete with lower water/binder ratio is commonly denser and have a higher strength. However, Prasad et al. (2006) stated that the assumption is not always true for the concrete with low water/binder ratio to have a better sulphate resistance. Al-Amaudi et al. (1995) investigated this effect and found that the lower water/binder ratio may enhance the strength reduction. The possible reasons for such behaviour could be due to sulphate attack that is not physically in nature hence it is independent from permeability of mix. Furthermore, the expensive product formed from the reaction between the cement and sulphate salts are not well accommodated in the finer pore structure of low water/binder ratio mix. Thus dense concrete mixtures have aggravated the deterioration process which attributed from the sulphate attack.

### 2.3.3 Curing condition

Curing of freshly placed concrete is an important requirement to achieve optimum performance (Turk et al., 2007). The curing condition of the concrete has greatly affected it’s hydration process. The complete hydration of binder and the influence factor of strength development in the concrete are involved with the continuous and moist curing process. Initial curing condition could affect the sulphate resistance of
concrete. Osborne (1999) reported that the beneficial effects of short initial air curing are including continuous moist curing, a long-term sulphate resistance of plain and blended concrete. The possible reason for such behaviour may be due to the initial dry curing that has reduced the calcium hydroxide concentration in the concrete surface zone. In addition, the initial dry curing have caused minor carbonation and reduced the availability of calcium hydroxide for the salt attack.

2.3.4 Type of sulphate and concentration

Deterioration rate of concrete under salt attack also depends on the type of sulphate. Al-Amaudi et al. (2002) presented the results of sodium, magnesium, and mixed sulphate solution attack on mortar and concrete specimen. The strength reduction was almost comparable in both environments up to about 100 days of exposure and subsequently the reduction is spread in the magnesium sulphate for all types of specimens. The expansion data indicates that the sodium sulphate environment has caused more expansion on all mortar specimens. Results showed a superior performance of blended cement in the sodium sulphate solution. In mixed sulphate environment, the mode of sulphate attack is mainly controlled by magnesium sulphate due to the generation of magnesium hydroxide (brucite). The solubility of magnesium hydroxide (its solubility is 0.01 g/litre compared to 1.37 g/litre for calcium hydroxide) and it’s saturated solution with the ph about 10.5 will cause destabilization of both ettringite and calcium silicate hydrate. Therefore, the formation of ettringite is significantly hindered in such environment and the deterioration of concrete is attributed mainly to the formation of gypsum.

Deterioration of concrete in the sulphate attack also depends on the Sulphate concentration in the exposure solution. Higher concentration of sulphate will leads to a quick deterioration. Al-Dulaijan et al. (2003) studied the performance of plain and blended cement mortar cubes which exposed to sodium sulphate solution of varying concentration for up to 24 months. The degree of deterioration was evaluated by strength reduction and visual inspection. Result from their study has indicated that the rate of deterioration increased with increase in sulphate concentration for both plain and blended cement. The sample prepared by fly ash blended cement shows less deterioration compared to other concentrations and at all time of exposure.
2.3.5 Presence of chloride

The chloride ions are inadvertently associated with the sulphate attack in the ground water or marine environment on the concrete. It is commonly known that the chloride ion reacts with the hydrates of cement and will form the Freidel’s salt that does not have any harmful effects on the concrete. However, when chloride contents in the concrete has reach more than the threshold value, the protective alkaline layer of steel reinforcement in the concrete structure will be broken. Later, the presence of oxygen and humidity will cause corrosion of the steel reinforcement. The presence of chloride in the sulphate solution will produce effects to the deterioration of concrete under the sulphate attack. Al-Amoudi et al. (2002) studied this effect on plain and blended cement mortar specimens, through the specimens exposure to 2.1% SO$_4$ solution and 2.1% SO$_4$ + 15.7% Cl solutions for 365 days. Deterioration was measured on the basis of the strength reduction and expansion of specimens. Result shows that the deterioration was relatively more intense in the specimen that exposed to pure sulphate solution compared to those that exposed to sulphate-chloride solution.

The increase of the sulphate resistance in the cement with the presence of chlorides is attributed to (i) increased solubility of calcium aluminate hydrate phase leading to calcium sulpho-aluminate crystallization i.e. ettringite formed in a non-expensive form, (ii) a decrease in the lime concentration in the pure solution leading conversion of the high insolubility of the basic aluminate hydrate phase to soluble low basic compounds thus producing ettringite of liquid phase in the non-expensive forms, (iii) a transformation of aluminate hydrate phase into chloro-aluminate phase, thereby reducing the quantity of ettringite formed, (iv) the rate of diffusion of chloride ions is much higher than the sulphate ions which allows the chloride to react with C$_3$A to form calcium chloro-aluminate hydrate, this will result the quantity of C$_3$A to be available for sulphate ions to react and the form ettringite will be reduced.

2.3.6 Temperature of exposure

The deterioration of mixes is influenced by the physical condition of sulphate solution which related to its temperature and also by the submersed level of specimens. Hekal et al. (2002) observed the sulphate exposure at 60°C temperature in the ambient
environment with drying-immersion cycle. The increase in temperature of sulphate solution has accelerated the sulphate attack on all types of specimens of mortars. The drying-immersion from the cyclic process at 60°C has accelerated the rate of sulphate attack and this can be considered as an accelerated method to evaluate sulphate resistance. Changes in compressive strength of ordinary Portland cement mortar cubes over time at different exposure conditions is shown in Figure 2.1. The curve shows more reduction in strength at higher temperature. Hence sulphate attack is more aggressive in the summer or in a hot climatic area.

![Figure 2.1: Effect of physical condition of exposure (Hekal et al., 2002)](image)

2.4 Mechanism of salt attack

Over recent decades, several mechanisms have been proposed to explain the phenomenon of damage due to crystallization in porous brittle materials. The most widely accepted is the theory of salt attack by supersaturation proposed by Scherer (2004). Salt attack is a crystal-forming process due to the presence of soluble salt in moisture or water after evaporation. The salt concentration in the solution gradually increases as the material dries. The presence of soluble salts can deteriorate the porous brittle materials either directly or indirectly. Under this sub-section, mechanism of crystallization and salt attack in the concrete material exposed to soluble salts are discussed in details.
2.4.1 Salt crystallization

Porous and brittle materials such as stone, masonry or cement-based materials can be deteriorated due to pressure induced by salt crystallization taken place in their pores. The cracking and fracture can be formed as a result of the production of pressure which has exceeded the tensile capacity of the material (Scherer, 2004). The advanced damage can compromise the integrity and performance with cyclic exposure and weathering of the affected material during a longer period. A phenomenon called as “salt weathering”, “salt scaling”, “physical salt attack”, or “salt crystallization” when the salt has crystallized in the absence of degrading chemical interaction has caused damage.

Researchers examining the salt crystallization damage on porous materials have always focused on salt crystallization by sodium sulphate, sodium chloride, and sodium carbonate (Lee & Kurtis, 2017; Haynes et al., 2010; Thaulow & Sahu, 2004). For example, Scherer (2004) experimentally investigated the stress caused by crystallization of sodium sulphate salt (thenardite and mirabilite) in the stone. This phenomenon can be explained by precipitation of salt crystals from the liquid in pore structure of a material and also related to the phase change from thenardite to mirabilite, which contains 10 molecules of water. Chatterji & Thaulow (1997) also experimentally demonstrated that the crystal growth pressure exerted by sodium thiosulphate pentahydrate could break glass test tubes without confinement. Thaulow & Sahu (2004) reviewed the mechanisms of surface scaling of concrete due to sodium sulphate salt. There were three existing proposed damage mechanisms have been compared- (i) solid volume change hypothesis, (ii) salt hydration pressure hypothesis, and (iii) crystallization pressure hypothesis - concluding that damage can only be explained by salt crystallization pressure theory. The damage mechanisms associated with salt crystallization in porous materials have been reviewed by Valenza & Scherer (2007).

Although not examined as often, other salts including nitrates, oxalates, and acetates, are also known to damage porous materials (Lee & Kurtis, 2017). For example, acetates is known to damage tiles of glazed ceramics by salt crystallization (Linnow et al., 2007), and nitrate (NO₃) salts, since they are derived from fertilizer which known to damage masonry near agricultural regions. Conservationists have
found crystallization of calcium nitrate salts to be particularly damaging to brittle materials such as plasters. In addition to their practical relevance in this application, study of crystallization of various salts is desirable because it is essential to develop accelerated laboratory tests to assess potential for damage in natural and engineered porous materials. Salts are particularly damaging at a faster rate and it is also potentially good candidate to be applied in the accelerated test methods. Cement-based materials are brittle similar to stone and masonry and have a porous microstructure which can exhibit salt crystallization damage in practice (Haynes et al., 2010). However, the microstructure and hardened properties of cement-based materials are different to the natural materials, thus the extent of damage, can be controlled or tailored to enhance durability.

2.4.2 Chloride attack

The durability of concrete is depends to its capability to resist quality degradation when exposed to aggressive environments which can cause deleterious effects to the concrete (Glasser et al., 2008). It is known that the ingress of chloride ions in the concrete is the most severe problem affecting the durability of the concrete construction. Chloride ions may cause adverse effect on hardened concrete in various ways. Generally, the effect is usually attributed to the formation of calcium chloroaluminate or Friedel’s salt (Islam et al., 2010). This salt crystallization has a low-medium expansion property and may increase concrete permeability by leaching due to the formation of excessive calcium chloride. The process of chloride attack on concrete may be explained by the following chemical reaction below.

\[
\text{Ca(OH)}_2 + 2\text{NaCl} \rightarrow \text{CaCl}_2 + 2\text{NaOH} \quad \text{Equation 2.1}
\]

\[
\text{CaCl}_2 + 3\text{CaO} \cdot \text{Al}_2\text{O}_3 + 10\text{H}_2\text{O} \rightarrow 3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaCl}_2 \cdot 10\text{H}_2\text{O} \quad \text{Equation 2.2}
\]

The penetration of chloride ions may attack concrete in different ways and it is usually associated with the corrosion process in the reinforced concrete. According to Montemor et al. (2003), the common causes of corrosion are (i) an ingress of carbon dioxide from the atmosphere decreasing the alkalinity of the pore solution and (ii) the
local depassivation of steel due to the presence of chlorides at the reinforcement level; this has shown in Figure 2.2. Furthermore, chloride may also infiltrate into concrete from the environment such as deicing salt and seawater (Xu et al., 2009).

![Diagram of Chloride attack on reinforced concrete](image)

**Figure 2.2**: Chloride attack on reinforced concrete (Kung, 2014)

### 2.4.3 Sulphate attack

Sulphate attack is a major problem which related to the durability of concrete structures (Chatveera & Lertwattanaruk, 2009). The attack by solutions containing sulphate can have different effects on concrete. Given that the influence of other chemicals can affect the mechanism of the reaction, therefore the nature of the sulphate solution is significant. Figure 2.3 shows the mechanism of sulphate attack. This is particularly factual in the case of seawater, where the presence of a higher concentration of chlorides can have a bearing on the action of the sulphates. Chlorides have a high tendency to bind tricalcium aluminate ($\text{C}_3\text{A}$) in the cement to produce chloroaluminate compounds, such as Friedel's salt, which do not cause any expansion (Ragab et al., 2016). The lowered accessibility of $\text{C}_3\text{A}$ can decrease the deterioration by sulphate attack due to a direct decrease in the production of ettringite. Furthermore, Mehta (1991) has stated that the ettringite formation in chloride-rich environments is not related with expansion and cracking.

The extent of sulphate attack in concrete is influence by several factors including type and concentration of sulphate solution, cement type and content, presence of mineral admixture and permeability (Mehta & Monteiro, 2006; Neville, 2002). It was reported that the deterioration caused by sulphate attack may be due to transformation of mono-sulphate to ettringite which resulted in ettringite formation,
formation of calcium sulphate upon reaction of calcium hydroxide with sulphate ions lead to gypsum formation and mass or strength loss due to sulphate attack. Both ettringite and gypsum formations are accompanied by large value expansions (Mehta & Monteiro, 2006; Alexander et al., 2013; Mardani-Aghabaglou et al., 2015). The reactions can be formulated (Santhanam et al., 2003) as follow:

\[
\text{Ca(OH)}_2 + \text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O} \rightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O} + 2\text{NaOH} + 8\text{H}_2\text{O} \quad \text{Equation 2.3}
\]

\[
3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 12\text{H}_2\text{O} + \text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O} \rightarrow 3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 31\text{H}_2\text{O} + 2\text{Al(OH)}_3 + 6\text{NaOH} + 17\text{H}_2\text{O} \quad \text{Equation 2.4}
\]

**Figure 2.3:** Schematic diagram for sulphate attack (China Microsilica Union, 2011)

### 2.4.4 Seawater attack

Marine environments are very aggressive, since seawater mainly consists of chlorides and sulphates. Both ions can be very harmful to the durability of concrete structures which can affect the long-term durability and may cause a huge economic loss. Furthermore, concretes are exposed to repeated drying–wetting cycles, such as in the marine environment of splash and tidal zone which can accelerate the deterioration of structures (Chen et al., 2016).
The main attack mechanism in marine environments is external sulphate attack. This occurs when water contaminated with sulphates penetrates into the concrete by means of diffusion or capillary suction. Sulphates are mostly found in the form of sodium sulphate (Na$_2$SO$_4$) or magnesium sulphate (MgSO$_4$). The cation associated with SO$_4^{2-}$ has an influence on the attack mechanism and the consequence of deterioration (Maes & Belie, 2014). Sodium sulphate attack will result in expansive reaction products while magnesium sulphate attack will result in reduction in strength. Al-Amoudi (2002) describes the sequence of attack by magnesium sulphate in seawater as follows:

\[ \text{Ca(OH)}_2 + \text{MgSO}_4 + 2\text{H}_2\text{O} \rightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O} + \text{Mg(OH)}_2 \quad \text{Equation 2.5} \]

\[ \text{C-S-H} + \text{MgSO}_4 + \text{H}_2\text{O} \rightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O} + \text{Mg(OH)}_2 + \text{SiO}_2 \cdot \text{H}_2\text{O} \quad \text{Equation 2.6} \]

\[ 4\text{Mg(OH)}_2 + \text{SiO}_2 \cdot n\text{H}_2\text{O} \rightarrow \text{MgO} \cdot \text{SiO}_2 \cdot 8 \cdot 5\text{H}_2\text{O} + \text{H}_2\text{O} \quad \text{Equation 2.7} \]

Zacarias (2007) stated that calcium chloroaluminate hydrate or Fridel’s salt is an important phase formed during exposure of concrete to seawater. Generally, chloride and sulphate ion from seawater penetrate into concrete forming Fridel’s salt and ettringite. However, magnesium sulphate also attacks calcium hydroxide and calcium-silicate-hydrate (C-S-H) phases. According to Leng et al. (2000), magnesium sulphate attacks calcium hydroxide and C-S-H, reacts with C-H to form secondary gypsum and brucite. Apart from that, Islam et al. (2010) found that chloride reacting with C-H of hydrated cement has led to weight loss and weakening the concrete structures. Figure 2.4 illustrates the mechanism of deterioration act on concrete exposed to seawater.
2.5 Aggressive exposure condition

The most serious problem in concrete technology is the premature deterioration of concrete structures that subjected to harsh environments. There is overwhelming evidence from the field experience that many problems related to durability, such as sulphate attack, reinforcement corrosion, carbonation and alkali-silica reaction in concrete would not have occurred if the concrete is vulnerable to the environment and during the intended service life at the time of exposure (Neville, 2004). The related issue of durability, the selection of materials, aggregate mix proportions and construction practices using concrete must be addressed. The industry must develop a new comprehensive model of concrete deterioration and quantify the environmental influences on the permeability of concrete.

Concrete bridge decks and parking structures are typically consist of concrete deck, reinforced with steel bars, post or pre-tensioning tendons, which serve as the finished wearing surface and can resist the abrasion of tires on surfaces. Some of these
decks are exposed to rain and snow, and others are subjected indirectly to moisture carried on the undersides of cars. Moisture may also be associated with deck cleaning and maintenance activities. De-icing salt is also brought from undersides of cars or applied directly to the deck to melt ice and snow for improved traction. Virtually, all de-icing salt used in the current road and highway maintenance contains chlorides (Fortin et al., 2014). The water stands on the deck surface may worsen from the exposure of chlorides. In offshore and marine areas, salty sand, salt-water spray, seawater, and prevalent high moisture conditions can also lead to serious corrosion problems.

The temperature follows seasonal and daily temperature cycles, although a temperature lag always occurs in the structure materials. Differences in temperature along cross-sections of a reinforced concrete structure result in volume changes in the members, whereby changes that are greater than those within enclosed structures. Such changes are frequently smaller in the plan dimensions and exist in an environment with more constant temperature, humidity, and moisture. Restraint of volume change can cause racking of decks and floor slabs, beams and columns, and if unprotected it may allow rapid ingress of water and chlorides leading to further deterioration (Merritt & Ricketts, 2001). When de-icing salts are used, some of these salts will be absorbed by the cover of the concrete. A high osmotic and hydraulic pressure with a consequent movement of water toward the coldest zone which causes freezing will be occurred under severe wetting, drying, freezing and thawing cycles. Damage can occur when the tensile strain exceeds the capacity of the concrete, which varies with the age of the concrete and the rate of application of strain. The extent of damage varies from surface scaling to complete disintegration as the layers of ice is formed, starting at the exposed surface of the concrete and progressing through its depth (Yuan et al., 2003).

2.5.1 Wetting and drying

Wetting and drying cycles can cause constant moisture movement through concrete pores (Hong & Hooton, 1999; Sahmaran et al., 2007; Jaya et al., 2014; Ye et al., 2016). It is well-known that wetting and drying cycles may speed up concrete degradation and durability problems because this has subjected the concrete to the motion and
accumulation of destructive elements, such as sulphates, alkalis, acids, and chlorides. According to Hong & Hooton (1999), there are several mechanisms governing chloride ingress into concrete such as absorption, diffusion, chloride binding, permeation, wicking and dispersion. Absorption and diffusion are the most significant mechanism of chloride ingress for the structures exposed to cyclic wetting-drying. Chloride-induced corrosion of steel reinforcement is a major cause affecting the service life of reinforced concrete structure exposed to aggressive environments. Chloride ions ingress by pore diffusion is a rare occasion when the concrete structure is exposed to continuous wetting–drying cycles by tidal and splash action (Maekawa et al., 2003). Various model and analysis technology have been proposed including the condition of cyclic wetting–drying for chloride ions transport in sound concrete. Most of analyses were based on the condition of chloride penetration into saturated concrete, however in reality, concrete was often found in an unsaturated condition rather than a saturation condition especially when subjected to cyclic wetting–drying (Ye et al., 2012).

Ye et al. (2016) have conducted an experiment on chloride penetration process in concrete exposed to a cyclic drying-wetting and carbonation environment. Based on the findings, the chloride penetration profile in concrete was produced by multiple interactive deteriorating mechanisms, and was dependent on the properties and stress status of the concrete itself. Their study also claimed that the incorporation of supplementary cementitious materials have made concrete more vulnerable to chloride attack under a combined deterioration of cyclic drying-wetting and carbonation, since the deficiency of portlandite dominates the positive effects such as pore refinement.

### 2.5.2 Freeze and thaw

Freeze and thaw damages may be caused by internal frost damage or salt-scaling. Resistance to internal frost damage is enhanced by providing an adequate air void system—including proper total air void volume as well as proper air void size and spacing. It can also be enhanced by using low water-cementitious materials ratios and supplementary cementitious materials to reduce both permeability and, in particular,
the number of large pores. Increasing the concrete strength also enhances the resistance to internal frost damage. Salt-scaling can be prevented by providing an adequate entrained air void system, reducing the water-cementitious materials ratio, and providing proper finishing and curing practices. There is some evidence that the use of fly ash or slag may reduce salt-scaling resistance.

The freeze-thaw durability of self-compacting concrete is frequently comparable to or better than conventionally placed concrete. The low water-cementitious materials ratios and with adequate entrain air can enhance the freeze-thaw resistance. The use of fly ash and slag, however, may reduce salt-scaling resistance. Persson (2003) found the internal frost resistance of self-compacting concrete to be better compared to conventionally placed concrete and it also found to be similar to the salt-scaling resistance. Heirman and Vandewalle (2003) found that the freeze-thaw durability was similar however the salt-scaling resistance decreased in relative to conventionally placed concrete when a variety of fillers were used and the water-cement ratio was held constant. Audenaert, Boel, and De Schutter (2002) found that the reduction of water-cement and water-powder ratios in self-compacting concrete mixtures have improved internal frost resistance. The use of some high range water reducing admixtures under certain conditions may detrimentally affect the air content and characteristics of the air void system. Khayat and Assaad (2002), however, found that the air void characteristics of self-compacting concrete were similar to those of conventionally placed concrete and the air void stability could be improved by increasing the cementitious materials content. These will reduce the water-cementitious materials ratio or adding a viscosity modifying admixtures in mixtures with low cementitious materials contents and high water-cementitious materials ratios.

2.5.3 Laboratory simulation of wetting and drying cycles

Generally, the mechanism of cyclic wetting and drying allow for deeper penetration of aggressive ions. For example, the aggressive ions can cause corrosion at rate 20 times higher than the rate achieved by exposure to continuous immersion (McCarter & Watson, 1997). Obviously, the rate of the ions penetration to the concrete is depends on the duration of the wetting-drying periods. The simulation of wetting and drying
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