EFFECT OF CURING REGIMES ON MORTAR INCORPARATING MEATAKAOLIN AND SLAG AT LOW WATER/BINDER RATIO

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(Hons.) Civil Engineering

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DECLARATION

I hereby declare that this project report is based on my original work except for citations and quotations which have been duly acknowledged. I also declare that it has not been previously and concurrently submitted for any other degree or award at UTAR or other institutions.

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APPROVAL FOR SUBMISSION

I certify that this project report entitled **“EFFECT OF CURING REGIMES ON MORTAR INCORPARATING MEATAKAOLIN AND SLAG AT LOW WATER/BINDER RATIO”** was prepared by **HO LIANG CHEE** has met the required standard for submission in partial fulfilment of the requirements for the award of Bachelor of Engineering (Hons.) Civil Engineering at Universiti Tunku Abdul Rahman.

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ABSTRACT

For the past two decades, there has been an increased popularity in incorporating a variety of admixtures into concrete or mortar to modify one or more properties, either fresh or hardened, of concrete and mortar to achieve desire objectives. Among these admixtures utilized are ground granulated blast furnace slag (GGBFS) and metakaolin (MK). There have been numerous researches indicating that the incorporating GGBFS and MK exert a positive effect on mechanical as well as durability properties. Hence, this particular project works is performed in the attempt to verify that incorporation of GGBFS and MK into mortar would improve its durability. In addition, the effect of curing regimes on durability of mortars incorporated with GGBFS and MK is examined as well. Mortar of four different mix proportions was prepared: (i) control mix in which the mortar's binder is constituted of 100% OPC (Ordinary Portland Cement), (ii) mortar with 5% of OPC replaced by GGBFS, (iii) mortar with 5% of OPC is replaced by MK, and (iv) mortar with 5% of OPC replaced by 2.5% of GGBFS and 2.5% of MK respectively. Specimens of each mix proportion were cured under three different curing regimes, namely, water curing, plastic curing and air curing. In each curing regimes, the specimens were further subjected to five different curing periods of 7, 28, 56, 90 and 180 days respectively. At the end of each curing period, the specimens were taken out from their respective curing regime for three durability tests: initial surface absorption Test (ISAT), water absorption test, and sorptivity test. Each of the tests utilized different type of specimens-100mm cubes for ISAT, 50mm cubes for water absorption test and 100mm-diameter cylinders with a depth of about 50mm to 80mm for sorptivity test.

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LIST OF SYMBOLS / ABBREVIATIONS

ACI	American Concrete Institute
ASTM	American Society for Testing and Materials
C-S-H	Calcium silicate hydrate
GGBFS	ground granulated blast furnace slag
ISA	initial water absorption
ISAT	initial water absorption test
MK	metakaolin
MLS	modified lignosulfonates
MPa	mega Pascal
OPC	Ordinary Portland Cement
PC	polycarboxylate
POFA	Palm Oil Fuel Ash
SCC	self-compacting concrete/self-consolidating concrete
SMF	sulfonated melamine-formaldehyde
SNF	sulfonated naphthalene-formaldehyde
SP	Superplasticizer
w/b	water-to-binder ratio
WA	water absorption
a_d	weight of the oven dried specimen (g)
$a_s(t)$	increase in mass (g/mm^2) or expressed in mmas 1g or water is equivalent to 1cm^3
a_t	weight at time, t (g)
b_1	mass of the oven dried specimen (g)
b_2	mass of the wet specimen in air(g)

A	average of four readings in mm, minus the original inside base diameter in millimetres.
CM	mass of cementitious materials in kg per cubic metre
d	superplasticizer dosage as a percentage of mass of solids in comparison to the total mass of cementitious material
D_{base}	Original inside base diameter of the flow mould in mm
div	number of scale divisions during period of t
G_c	Specific gravity of cement of cementitious material
G_{SSD}	aggregate specific gravity in saturated surface dry condition
G_{sup}	specific gravity of the liquid superplasticizer
M_f	mass of fine aggregate
M_{sol}	mass of solids in superplasticzer
N	cross sectional area of bottom surface (g)
s	total solid content of the superplasticizer in per cent
Sor	sorptivity coefficient ($g.min^{0.5}/mm^2$)
T	time measured in minutes at which the mass is determined (min)
t	time period in seconds
V_{liq}	volume of liquid superplasticizer
V_{sol}	volume of solids in the liquid superplastcizer
V_w	volume of water in the liquid superplasticizer
W	mass of water in kg per cubic metre of concrete
w_{abs}	absorbed water in the aggregate in per cent
w_c	moisture content of the aggregate in per cent
w_{tot}	total water content of the aggregate in per cent

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Mortar is one of the most ancient construction materials created a few thousand years ago, has evolved and become a fundamental material in today's construction industry. The main purpose of mortar in construction is to fill gaps between construction blocks and attach them into larger units. Besides, mortar can be used to produce building blocks such as mortar bricks. Portland cement mortar, or often simply known as cement mortar, is produced by mixing OPC and aggregate (or sand) with water. In fact, mortar is concrete without coarse aggregates. Therefore, the properties of mortar directly affect the properties of concrete.

Due to a number of factors, researches have been carried during recent years on the effects of introducing admixtures such as GGBFS, silica fume, palm oil fuel ask (POFA) and others into cement mortar. The admixtures are used to replace part of the cement or used as fillers in mortar. Out of these admixtures, the two admixtures that will be paid attention on in this project work are GGBFS and metakaolin (MK).

The application of GGBFS and MK offers a few advantages. First, these materials are relatively cheaper and construction cost can be reduced when they are employed to partially replace OPC. Besides, extensive research discovers that addition of GGBFS and/or MK into mortar or concrete improve mechanical and durability properties of concrete. Thus, higher quality concrete or mortar can be produced with lower cost. The upgraded durability of mortar and concrete is favourable in construction in long term as the structure service time would be prolonged. This turn reduces the cost required for maintenance, demolition and re-construction of structure. The enhanced durability of mortar and concrete also enable structures to withstand chemical assault and extreme weather better.

Besides, the curing regime of mortar is crucial to produce durable concrete. Adequate and continuous supply of moisture is necessary for hydration of cement paste to take places within the mortar so gain strength and durability over time. Poor curing condition of mortar will lead to plastic shrinkage, increased permeability and increased susceptibility to sulphate and chloride attack.

1.2 Background of Study

GGBFS is generated as a by-product during the iron manufacturing industry. The use of GGBFS in Malaysia’s construction sector is rather limited due to the lack of agents that convert this by products into useful form to be applied in construction sector. Malaysia imports GGBFS which is crushed in a rolling mill and used both for the production of GBS cements and directly for concrete production. In line with the field of application, cements are produced with three differing GGBFBS contents of 30, 50 and 70% through separate grinding processes and mixtures. No additional sulphate setting is made in the production of cement. In 1999, there was some two thirds of the overall produced GGBS used in cement and about one third used in concrete (Müller, Zementwerke & Düsseldorf, 2007).

Clays have been and remain as one of most important industrial minerals today. Metakaolin is an innovative clay product developed in recent years. It is generated via controlled thermal treatment of kaolin. Due to its pozzolanic properties, metakaolin, can be used as concrete and mortar constituents by replacing part of the cement content. In recent years, there has been an intensive study on the utilization of metakaolin as a material to partially replace cement in mortar and concrete. However, there are still unknowns with the use of metakaolin. Great concerns have been placed on the strength and durability of metakaolin-concrete to be used as construction materials in the construction industries (C.H. Ong, 2006). The information on the use of MK in Malaysia's construction field is hardly available and it can be inferred that the MK is not yet popular in Malaysia. Nevertheless, a great deal of positive effects of MK when blended with concrete or mortar is believed to the reason that makes MK to gain popularity in the future.

1.3 Objectives of Study

This study is conducted to accomplish the following objectives:

1. To investigate the effect of different curing regimes on durability of mortar incorporating MK and GGBFS.
2. To evaluate the effect of incorporating MK and GGBFS on the durability of mortar in terms of initial surface absorption test, sorptivity test and water absorption test.
3. To compare the performance of GGBFS to MK on durability improvement of mortar.
4. To access the workability of mortar incorporated with slag and/or metakaolin

1.4 Scopes of Study

This project work aims to investigate the how different curing regimes influence the durability of mortars with different mix proportions in term of water absorption, initial surface water absorption and sorptivity of mortar. Four different mix proportions were prepared: (i) control mix in which the mortar's binder is constituted of 100% OPC (Ordinary Portland Cement), (ii) mortar with 5% of OPC replaced by

GGBFS, (iii) mortar with 5% of OPC is replaced by MK, and (iv) mortar with 5% of OPC replaced by 2.5% of GGBFS and 2.5% of MK respectively. Specimens of each mix proportion were subjected to three different curing regimes: water curing, plastic curing and air curing. In each curing regimes, the specimens were cured for different curing periods of 7, 28, 56 and 90 days respectively. When the desired curing period was raeach, the specimens were taken for three typed of durability tests: initial surface absorption test (ISAT), water absorption test, and sorptivity test. Each of the tests employ different type of specimens-100mm cubes for ISAT, 50mm cubes for water absorption test and 100mm-diameter cylinders with a depth of about 50mm to 80mm for sorptivity test.

Prior to all casting work, the suitable water to binder (w/b) ratio and appropriate dosage of superplasticizer (SP) was determined based on a few trial mixes. The workability of fresh mix and the time for final set to occur were observed in choosing the necessary w/b ratio and SP dosage. In order to evaluate the effect of slag and MK on mortar’s workability, flow table test was carried for each of the four different mix proportions. The cured samples were oven-dried to remove all moisture contain and were allowed to cool down in a desiccators before any test was performed. The data were than collected and the durability of the mortar was study and discussed.

1.5 Significances of Study

The significances of this study are as followed:

1. Incorporating GGBFS and MK to partially replace cement in into mortar and concrete mix to encourage the utilization of industrial by-products and natural materials as sustainable and environmental friendly construction materials.
2. Produce cement mortar and concrete of higher quality and performance, which have improved mechanical and durability properties compared to ordinary mortar and concrete.

3. Investigate the suitability and evaluate the effects of incorporating more than 1 type of pozzalans into mortar or concrete.

1.6 Layout of Report

This report comprises 5 chapters. Chapter 1 includes the introduction, background, objectives, scopes, significance of this study and finally the layout of report.

Chapter 2 reviews mortar and concrete incorporated with slag and/or metakaolin. This entails the review on materials used, for instance, sand, cement and superplasticizer (SP), slag and metakoalin. In addition, the properties of mortar and concrete incorporate with slag and metakaolin are discussed in this chapter.

Chapter 3 provides the details of methodologies used in this study. The material preparation, method to get the mix proportion and mixing procedure are discussed in this chapter. Besides, the testing methods utilized for testing of specimens are also described in this chapter.

Chapter 4 mainly presents and discusses results of testing programmes. The results of flow table test and the other three durability tests: ISAT, sorptivity and water absorption test are presented and followed by a detail analysis and discussion on the results obtained.

Chapter 5 makes a conclusion on the whole study. Few conclusions have been drawn with respective objectives listed in accordance to the results derived from this study. Other than that, a few recommendations are listed in this chapter for future studies.

CHAPTER 2

LITERATURE REVIEW

2.1 Cement

Cements can be defined as substance which sets and hardens independently, and capable together different materials (Wikidot, n.d). In 1824, Joseph Aspdin, an English mason invented and patented his product. The product was named as Portland cement as a concrete that was of similar colour to the prestigious Portland Stone in the English Channel was produced.

The raw materials for manufacturing cement are divided into two types: calcareous materials and argillaceous materials. Conventional calcareous materials comprise of chalk, limestone, marl and oyster shell while the sources of argillaceous material are clay, shale, slate and selected blast furnace slag. The mixture of raw materials, in a definite proportion, is burned to a high temperature (about 1450 ℃) in a rotary kiln. The product obtained from the combustion, called clinkers or nodules, is cooled and grinded to required fineness to get the final product which is cement (Gambhir, 2009).

The modern cement is distinct from ancient cement. Modern cement can generally be classified into hydraulic cement and non-hydraulic cement. According to ASTM C125, hydraulic cement is an inorganic material or a mixture of inorganic materials that sets and develops strength by chemical reaction with water by formation of hydrates and is capable of doing so under water (ASTM, 2004).

2.1.1 Principal Compounds of Cement

Ordinary cement contains four major compounds and several minor compounds as listed in Table 2.1.

Table 2.1: Principal Compounds of Cement (Neville, 2010)

Compound	Chemical Formula	Industry Code (Abbreviation)	Percentage Amount (range)	Rate of Reaction with water
Tricalcium Silicate	$3CaO.SiO_2$	C_3S	35-65	Medium
Dicalcium Silicate	$2CaO.SiO_2$	C_2S	15-40	Slow
Tricalcium Aluminate	$3CaO.Al_2O_3$	C_3A	0-15	Fast
Tetracalcium Aluminoferrite	$4CaO.Al_2O_3.FeO_3$	C_4AF	6-20	Medium
Others (Na_2O & K_2O)			0-9	

2.1.2 Chemical Composition of Portland Cement

Generally, the actual proportions of various compounds of Portland cement vary significantly due to different manufacturers. A general composition of Portland cement can be obtained from Table 2.2, which illustrates the oxide composition limits of Portland cements.

Table 2.2: General Composition Limits of Portland Cement (Neville, 2012)

Oxide	Content, %
CaO	60 - 67
SiO_2	17 – 25
Al_2O_3	3 – 8
Fe_2O_3	0.5 – 6.0
MgO	0.5 – 4.0

Na_2O	0.3 – 1.2
SO_3	2.0 – 3.5

2.1.3 Types of Portland Cement

The ASTM C150, Specification for Portland cement, provides for the following types of Portland cement:

Table 2.3: Types of Portland Cement (ASTM C 150)

Type I	General Portland Cement
Type II	Moderate-sulfate–resistant cement
Type III	High-early-strength cement
Type IV	Low–heat-of-hydration cement
Type V	High-sulfate-resistant cement
Type IA, Type IIA and Type IIIA	Have same composition as Type I, II and III respectively but contain air-entraining agent

Type I Portland cement is cement for general usage, and is the most conventionally used Portland cement. It is used when special properties from other cement types are considered.

Type II Portland cement is used when moderate sulate resistance or moderate hear of hydration is desired. Type II cement has better resistance to deterioration from sulfate attack than Type I cement. It is appropriate for locations where concentration of sulfate is higher than normal but not severe. The relatively low C_3A content Type II cement generates lower heat of hydration than that of Type I. Therefore, it is suitable for mass concrete works such as piers and heavy footings. Besides, its lower heat of hydration makes it to be advantageous for placing concrete in hot weather.

Type III Portland cement is applied if high early strength is demanded. Type III cement possesses more C_3A content than other type of Portland cement and are finer, causing the cement to set and harden rapidly. It is utilized when the formwork is to be removed as early and soon and the structure is to be brought into service within shorter period of time. In addition, it may be used for emergency construction and repairs as well as machine bases constructions and gate installations.

Type IV Portland cement is employed when the heat of hydration must be decreased. Among all types of cement, Type IV cement has minimum contents of C_3A and C_3S, which are the compounds that produce the most heat of hydration. It is adopted for construction of large structures, such as concrete dams and massive foundations, in which the temperature gradient within the cross-section must be carefully controlled and the utilization of Type I cement may leads to cracking.

Type V Portland cement is applied if higher sulfate resistance is required. This cement has very low C_3A content. It is used when the structure is in contact with alkali soil and groundwater. Type V Portland cement is normally chosen for construction below ground, inland lake water and seawater, and around sewage disposal sites.

2.2 Superplasticizer

Superplasticizers are also known as high-range water reducing admixtures, which cuts down the needed quantity of mixing water to gain workability while maintain the quality of concrete of mortar. In accordance to ASTM C494-Standard Specifications for Chemical Admixture for Concrete, superplasticizer is classified as Type F admixtures, and is defined as an admixture which decreases amount of mixing water necessary to produce concrete of a given consistency by 12% or greater (ASTM, 2003). Other than being water-reducing agents, many of the superplasticizers available are also retarding admixtures (ASTM Type G), which delay the setting and hardening of mix. Superplasticizers are usually added in small dosage, typically from 0.2% to 0.6% by weight of cement into fresh mortar or concrete mix.

2.2.1 Structure and Families of Superplasticizers

Superplasticizers are linear polymers consists of sulfonic acid groups, which are water soluble organic polymers, attached to the polymer backbone at regular intervals (Verbeck, 1968). The sulfonic acid groups play the role to neutralized surface charges on the cement particles. This results in dispersion, therefore releasing the water trapped in cement particle agglomerations and thereafter reducing the viscosity of the paste and concrete (Mindess and Young, 1981). Most of the commercial formulations belong to one of the four families: sulfonated melamine-formaldehyde condensates (SMF); sulfonated naphthalene-formaldehyde condensates (SNF); modified lignosulfonates (MLS); polycarboxylate derivatives (PC) (Federal Highway Association of America, 2012).

2.2.2 Water Reducing Mechanism

Excess water than required for cement hydration is necessary for workability. Water-reducing admixtures aim to improve the mobility of cement particles in plastic mix, enable workability to be achieved at lower water contents.

Cement grains develop static electric charge on their surface as a result of the cement-grinding process. Unlike charges attract, causing the cement grains to cluster or “flocculate” (Figure 2.1(a)) which in turn limits the workability. The chemicals in the water-reducing admixtures reduce the static attraction among cement particles. The molecules of water-reducing admixtures have both positive and negative charges at one end and a single charge (usually negative) on the other end, as illustrated in Figure 2.1(b). The charge surface of cement grains attracted these molecules. The water reducers neutralize the static attraction on the cement surfaces. As a result, the clusters of cement grains are broken apart. Mutual repulsion of like charges pushes the cement grains apart, achieving a better distribution of particles (Figure 2.1(c)), more uniform hydration, and a less-viscous paste (Kosmatka et al, 2002).

Water reducing admixtures absorb onto the cement surfaces via the negatives charges on the admixture onto positively charged calcium ions on the cement surface. This breaks up the flocs, and form individual cement grain which is able to move pass each other easily. Thus, the fluidity of the mix is enhanced. The water trapped within the cement flocs is released without any change on the volume of the mix. (Newman and Choo, 2003).

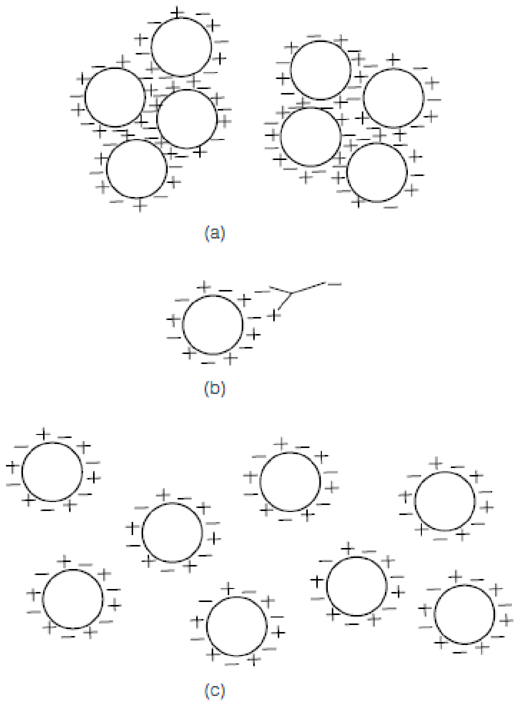


Figure 2.1: Water Reducer Mechanism: (a)Clustering of Cement Grains Witout Water Reducers, (b) Molecule of Water Reducers and (c) Better Distribution of Cement Grains Due to Use of Water Reducers

2.2.3 Mechanism of Retardation

The retarding admixtures reduce the initial rate of hydration or prolong the setting of cement in concrete. Retarders delay the setting in two ways. First, the retarding-admixture molecules forms a thin coating on cement particles therefore slow their dissolution and reaction with water. Another way of attain retardation is by increasing the intra-molecular distance of reacting silicates and aluminates from water molecules by forming certain transient compounds in the system (Gambhir, 2009). With the formation of silicates and aluminate hydrates, the influence of retardants cease and hydration process becomes normal.

In John Dransfield’s work (2003), two main processes that cause retardation were proposed. The first process is through a blocking mechanism. The admixtures strongly absorbed admixture on surface of cement disrupt the generation of silicate hydrates. Meanwhile, the calcium ions chelation in solution also prevents the calcium hydroxide leads precipitation and leads to retardation as well. One of both processes may be entailed, relying on the type of admixture chosen.

2.3 Pozzolans and Pozzolanic Reactions

Pozzolans, or known as mineral admixtures, are natural materials or industrial by products that are commonly used in concrete to replace part of cement or sand. If mineral admixtures are added as a substitute for a part of cement, they are called supplementary cementing materials they. Pozzalans are defined as siliceous or siliceous and aluminous materials which in themselves possess little or no cementitious property, but in finely divided form and in the presence of moisture, can react with calcium hydroxide of cement at ordinary temperatures to form cementitous compounds (Al Rawas and Hago, 2006). This reaction between pozzolans and calcium hydroxide is called pozzolanic reaction.

2.3.1 Classification or Pozzolans

Development of classification system for pozzolans is a precursor to the development if indicators of reactivity and verification of suitability of pozzolans for various applications. A few classification systems have been proposed, one of them is the ASTM Standard C618. It classifies pozzolans in to 3 types- Type N, Type F and Type C. This is presented in Table 2.4.

Table 2.4: Classes of Pozzolans According to ASTM C618

Type/ Class	Description	Applications
N	Raw or calcined natural pozzolan	To improve permeability and durability in low-heat applications; in hydraulic structures; to improve sulfate resistance and alkali-aggregate reaction
F	Fly ash, bituminous origin	To have high later-age strength; low hear cement, mass concrete; to improve sulphate resistance and alkali-aggregate reaction
C	Fly ash, lignite ash, or subbituminous origin	In high-strength concrete; to improve water tightness; in shotcrete
	Other pozzolanic materials, such as calcined clays, shales and volcanic tuffs	
	Silica fume	

Class N pozzolans are raw or calcined natural pozzolans that comply to the requirements as stated in ASTM C618. Some examples are opaline cherts, shales and some diatomaceous earths and volcanic ashes. It also includes some other materials such as clays and shales which require calcination to induce satisfactory properties (ASTM C618, 2003).

Class F pozzolans possess pozzolanic characteristics. The pozzolans in this class include fly ash which conventionally derived through combustion of anthracite or bituminous coal at the same time satisfy the requirements stated (ASTM C618, 2003).

Class C pozzolans consists of fly ash which normally produced from lignite or subbituminous coal. This class of fly ash, besides possessing pozzolanic properties, also exhibits some cementitious quality (ASTM C618, 2003).

Besides, Mehta (2006) has established a classification system of pozzolans according to their pozzolanic and/or cementitious characteristics. Table depicts the classification of pozzolans by Mehta. The table also contains a description of mineralogical composition and particle characteristics. Mehta has categorized pozzolans into following classes:

I Cementitious
II Cementitious and pozzolanic
III Highly active pozzolans
IV Normal pozzolans
V Weak pozzolans

Table 2.5: Classification, Composition, and Particle Characteristics of Mineral Admixtures for Concrete by Mehta

Class	Description	Example	Chemical and Mineral Composition	Particle Characteristics
I	Cementitious	Granulated blast-furnace slag	Mostly silica glass containing mainly calcium, magnesium, aluminium and silica. Crystalline compounds of melilite group may be present in small quantity	Unprocessed material is of sand size and contains 10-15% moisture. Before use it is dried and ground to particles less than 45 µm. Particle have rough texture
II	Cementitious and pozzolanic	High-calcium fly ash	Mostly silica glass containing mainly calcium, magnesium, aluminium and alkalis. The small quantity of crystalline present generally consists of quartz and C_3A; free lime and periclase maybe present; CS and C_4A_3S may be present in the case of high-sulfur coals. Unburnt carbon is usually less than 2 %	Powder corresponding to 10-15% particles larger than 45 µm. Most particles are solid spheres less than 20 µm in diameter. Particle surface is generally smooth but not as clean as low-calcium fly ashes.
III	Highly active pozzolans	Condensed silica fume	Consist principally pure silica in non-crystalline state	Extremely fine powder with solid spheres of 0.1 µm in size (about 20m^2/g surface area by nitrogen adsorption).
		Rice husk ash	Consist principally pure silica in non-crystalline form	Particles are generally less than 45 µm but vastly cellular (40-60m^2/g surface area by nitrogen adsorption).

IV	Normal pozzolans	Low-calcium fly ash	Essentially aluminium, iron and alkalis-containing glass with low amount of crystalline substance. Usually contains quartz, mullite, sillimanite, hematite and magnetite	Powder corresponding to 15-30% particles larger than 45 µm. Most particles are solid spheres with average diameter 20 µm. Cenospheres and plenospheres may be present
		Natural materials	Other than natural pozzolans that consists of mica, feldspar and quartz, aluminosilicate glass	Particles are ground to mostly under 45 µm and have uneven texture
V	Weak pozzolans	Boiler slag, slowly cooled blast-furnace slag, bottom ash, field burn rice husk ash	Consists of principally crystalline silicate materials, and only a low quantity of non-crystalline matter.	It is necessary to pulverize the material into finer size to develop some pozzolanic activity. Ground particles are rough in texture.

2.3.2 Pozzolanic Reaction

The calcium silicates (C_3S and C_2S) react with water to generate calcium silicate hydrate (C-S-H) gel and calcium hydroxide (CH). Calcium hydroxide is not a desirable hydration product as it has no cementitious properties and does not contribute to strength. CH also enhances the susceptibility of hardened concrete or mortar to chemical attacks.

$$2C_3S + 6H \rightarrow C_3H_2S_3 + 3CH \quad (2.1)$$

$$2C_2S + 4H \rightarrow C_3H_2S_3 + CH \quad (2.2)$$

where
C=CaO, calcium Oxide
S=SiO_2, silicate
H=H_2O, water
CH=$Ca(OH)_2$, calcium hydroxide

The pozzolanic reaction mainly involves reaction between amorphous siliceous materials and slaked lime (calcium hydroxide). This leads to formation of calcium silicate hydrate. The fundamental of pozzolanic reaction is a simple acid-base reaction between silicic acid, H_4SiO_4 or $Si((OH)_4$ and calcium hydroxide (Porlandite), $Ca(OH)_2$. The reaction can be represented as follow:

$$Ca(OH)_2 + H_4SiO_4 \rightarrow Ca^{2+} + H_2SiO_4{}^{2-} + H_2O \rightarrow CaH_2SiO_4 \cdot H_2O \quad (2.3)$$

where
$CaH_2SiO_4 \cdot H_2O$=Calcium silicate hydrate gel

Equation 2.3 can be summarized as abbreviated notation of cement chemist:

$$CH + SH \rightarrow CSH \quad (2.4)$$

2.4 Slag

ASTM C 989 provides the definition of slag to be equivalent to ground blast-furnace slag. The molten blast-furnace slag, when cooled down rapidly by immersion in water, generates a product which is glassy and granular in nature. It is optional whether the composition is adjusted or not when the blast-furnace slag is in molten state.

2.4.1 Production of Ground Granulated Blast Furnace Slag (GGBFS)

Ground granulated blast-furnace slag (GGBFS) is manufactured as a by-product from iron blast furnace slag. It forms as a result of the fusion of a limestone flux with ash from coke, and also siliceous and aluminous residue left after the reduction and separation of the iron from the ore (Choo and Newman, 2003). GGBFS is a non-metallic hydraulic cement which mainly constitutes of calcium silicates and calcium aluminosilicates.

One of two techniques-granulation or pelletization is adapted to process slag into an appropriate form to be used as cementitious material. In any of the technique, it is necessary that the slag should be cooled at a adequately rapid rate to produce a glassy, disorder structure. Otherwise, if slag is permitted to cool too slowly, it will lead to formation of a stable and unreactive well-ordered crystalline structure.

Using granulation technique, the steam of molten slag is forced over a weir into high pressure water jets. This causes the slag to cool rapidly into glassy granules with diameter less than 5mm. A majority of modern granulators maintains the water temperature below or around 50 ℃. The water slag ratio is generally between twenty to ten to one.

Pelletization involves pouring molten slag onto a water cooled steel rotating drum of about 1m diameter. The drum has fins projecting from it, which throw the slag through the air inside a building where water is sprayed onto it thus cause it to col rapidly. Pelletizing produces material from a bout 100mm down to dust. The larger particles tend be crystalline in nature and have little and no cementitious value. Hence, particles larger than about 6mm are screened off and used as lightweight aggregate in concrete. Only the finer section which is less than 6mm is utilized to manufacture GGBFS.

Granulation is much more efficient at producing material with high glass content. However, the capital costs of granulation are approximately six times greater than that of a pelletizer. Both materials can be used as raw feed for GGBFS and most standards do not differentiate between them.

2.4.2 Properties and Chemical Composition of GGBFS

The physical properties and chemical composition of GGBFS are varied, depending on the source of raw materials and blast furnace condition. Table 2.6 shows the physical properties of GGBFS while Table 2.7 presents the typical chemical composition of GGBFS as compared to ordinary Portland cement.

Table 2.6: Typical Physical Properties of GGBFS (Civil Marine, 2007)

Colour	Off White
Bulk Density	1000 to 1100kg/m^3 (loose) 1200 to 1300kg/m^3 (vibrated)
Relative Density (Specific Gravity)	2.85-2.95
Fineness	400-600m^2/kg

Table 2.7: Typical Chemical Composition of Portland Cement and GGBFS (Newman and Choo, 2003)

Oxide	Composition (%)	
	Portland Cement	GGBFS
CaO	64	40
SiO_2	21	36
Al_2O_3	6.0	10
Fe_2O_3	3.0	0.5
MgO	1.5	8.0
SO_3	2.0	0.2
K_2O	0.8	0.7
Na_2O	0.5	0.4

2.4.3 Classification of Slag

According to ASTM C 989, slag is classified on the basis of level of reactivity or performance in the slag activity test into three grades: Grade 80, Grade 100, and Grade 120. The slag activity is evaluated by determining the compressive strength of both Portland cement mortars and corresponding mortars made with the same mass of 50-50 mass combinations of slag and Portland cement. A reference cement complies ASTM standards is utilized. It is used to produce a reference cement mortar and a slag reference mortar which satisfied specifications stated in the standard. The mortar specimens are than tested at 7 days and 28 days for compressive strength. The slag activity index to the nearest index is calculated as followed:

$$Slag\ activity\ index, \% = \frac{SP}{P} \times 100 \tag{2.5}$$

where

SP=average compressive strength of slag-reference cement mortar cubes at designated ages, MPa

P=average compressive strength of reference cement mortar cubes at designated age, MPa

Table 2.8 provides the physical requirements for the classification of slag according to ASTM C989.

Table 2.8: Physical Requirements for Classification of Slag (ASTM C989)

<table>
<tr><td colspan="3">Item</td></tr>
<tr><td colspan="3">Fineness:</td></tr>
<tr><td colspan="2">Amount retained when wet screened on a 45-µm (No. 325) sieve, max %</td><td>20</td></tr>
<tr><td colspan="2">Specific surface by air permeability, Test Method C 204 shall be determined and reported although no limits are required</td><td>-</td></tr>
<tr><td colspan="2">Air Content of Slag Mortar, max %</td><td>12</td></tr>
<tr><td colspan="3"></td></tr>
<tr><td>Slag Activity Index, min, %</td><td>Average of Last Five Consecutive Samples</td><td>Any Individual Sample</td></tr>
<tr><td colspan="3">7-Day Index</td></tr>
<tr><td>Grade 80</td><td>-</td><td>-</td></tr>
<tr><td>Grade 100</td><td>75</td><td>70</td></tr>
<tr><td>Grade 120</td><td>95</td><td>90</td></tr>
<tr><td colspan="3">28--Day Index</td></tr>
<tr><td>Grade 80</td><td>75</td><td>70</td></tr>
<tr><td>Grade 100</td><td>95</td><td>90</td></tr>
<tr><td>Grade 120</td><td>115</td><td>110</td></tr>
</table>

2.5 Metakaolin

Metakaolin is a pozzolanic material and is a dehydroxylated form of clay mineral kaolinite. Metakaolin is refined kaolin clay, which is calcined under carefully controlled conditions to produce an amorphous aluminosilicate that is reactive in concrete and mortar. The raw material used to manufacture metakaolin is kaolin clay or kaolinite, which consist mainly of hydrated aluminium disilicate, Al_2O_3:$2SiO_2$.

$2H_2O$. When kaolinite is subjected to thermal treatment at a specific temperature regime, it lost water through dehydroxylization and is transformed into metakaolin, Al_2O_3:$2SiO_2$ or $Al_2Si_2O_7$ (Siddique and Khan, 2011).

2.5.1 Production of Metakaolin

Metakaolin is produced via calcination of kaolinite clay at a temperature between 500 ℃ to 800 ℃. When heated up to a temperature between 100 ℃ to 200 ℃, clay minerals lost most of their adsorbed water (Siddique and Khan, 2011). When the temperature is raised to 450 ℃, dehydroxylization of kaolin clay occurs and the hydrated aluminosilicates are converted to materials consist predominantly or chemically combined aluminium, silicon and oxygen. The rate at which water of crystallization is removed increases with increasing temperature and it proceeds to a completion at 600 ℃ (ECCI; Highley, as cited in Newman and Choo, 2003). Metakaolin is produced in kilns when kaolin clay is heated at a temperature between 700 ℃ to 800 ℃. Beyond the temperature of dehydroxylization, kaolinite retains two-dimensional order in the crystal structure and the product is termed metakaolin.

In order to produce metakaolin qualified for application as supplementary cementing material or pozzolan, it is critical to achieve virtually complete dehydroxylization as possible without overheating, that is, thoroughly roasted but not burnt. This generates metakaolin with amorphous, highly pozzolanic properties. Overheating leads to sintering, to form the dead burnt refractory which is non-reactive, called mullite (Advanced Cement Technologies, 2013). In addition, the calcined product should be cooled rapidly and ground to a fine powder. The metakaolin formed in this way has a highly disorganized structure (Newman and Choo, 2003).

Brindley and Nakahira (as cited in Li, Sun & Li, 2010) investigated the phase transformation of kaolinite in the process of calcinations. MK was obtained at temperature of 500 ℃ and transforms to silicon-spinel at 925 ℃. While the temperature is above 1400 ℃, mullite is generated. It should be noted that silicon-spinel and mullite both have low reactivity. The exothermal dehydroxylation reactions are represented by equation 2.6 to 2.9:

~500 ℃: $Al_2O_3 \cdot 2SiO_2 \cdot H_2O \rightarrow Al_2O_3 \cdot 2SiO_2 + H_2O$ (2.6)

Kaolinite Metakaolin

925 ℃: $2[Al_2O_3 \cdot 2SiO_2] \rightarrow 2Al_2O_3 \cdot 3SiO_2 + SiO_2$ (2.7)

Silicon-spinel

1100 ℃: $2Al_2O_3 \cdot 3SiO_2 \rightarrow 2[Al_2O_3 \cdot SiO_2] + SiO_2$ (2.8)

1:1 mullite type phase

>1400 ℃: $3[Al_2O_3 \cdot SiO_2] \rightarrow 3\ Al_2O_3 \cdot 2SiO_2 + SiO_2$ (2.9)

3:2 mullite

Shavarzman et al. (as cited in Siddque & Klaus, 2008), studied the effect of heat treatment parameters on the dehydroxylation/amorphization process of the kaolinite-based materials such as natural and artificial kaolin clays with different amounts of amorphous phase (metakaolin). At calcination temperature below 450 ℃, kaolin showed relatively low level of the dehydroxylation degree, less than 0.18. In the range from 450 to 570 ℃, the degree of dehydroxylation sharply increased to 0.95, and finally between 570 and 700 ℃, the kaolinite was fully dehydroxylated. The dehydroxylation was accompanied by kaolinite amorphization, which affected the activity of additives.

2.5.2 Physical Properties and Chemical Composition of Metakaolin

The physical properties of metakaolin are highly dependent on the properties and quality of raw materials input, the calcination process and the finishing products. The typical physical properties of metakaolin available in North America are listed below in Table 2.9 (Advance Cement Technologies, LLC, 2013):

Table 2.9: Typical Properties of Metakaolin Available in North America

Physical form	Powder
Colour	Off white
Specific Gravity	2.60
Bulk Density (g/cm^3)	0.32-0.4
Surface area (m^2/g)	15
D10	<2.0 µm
D50	<4.5 µm
D90	<25 µm

The major constituents of metakaolin are silica oxide (SiO_2) and alumina oxide (Al_2O_3). The others constituents such as iron oxide, magnesium oxide, calcium oxide, potassium oxide and others are present in a relatively lower amount. The typical chemical composition of metakaolin is listed in Table 2.10.

Table 2.10: Typical Chemical Composition of Metakaolin (Siddique & Khan, 2011)

Ingredients	Ambroise et al.	Wild and Khatib	Tefraoui et al.
SiO_2	51.52	52.1	58.10
Al_2O_3	40.18	42.0	35.14
Fe_2O_3	1.23	4.32	1.21
CaO	2.00	0.07	1.15
MgO	0.12	0.19	0.20
K_2O	0.53	0.63	1.05
SO_3	-	-	0.03
TiO_2	2.27	0.81	-
Na_2O	0.08	0.26	0.07
L.O.I.	2.01	0.60	1.85

2.5.3 Pozzolanic Reaction of Metakaolin

Metakaolinute, with a cement chemist notation of AS_2, reacts well with lime (calcium hydroxide, CH) and form hydrate compounds of Ca and Al silicates. As a type fired clay, the development of pozzolanic properties of metakaolin largely depends on the nature and abundance of clay minerals in the raw material, the calcination condition and the fineness of the products. Metakaolinite has a highly disordered structure and provides good characteristics as a mineral additive for concrete and mortar. Metakaolin reacts with calcium hydroxide, a product from cement hydration in the presence of water to form additional cementitious aluminium containing C-S-H gel, together with crystalline products, which include calcium aluminate hydrates and alumino-silicate hydrates. The crystalline product formed depends mainly on the metakaolin/calcium hydroxide (AS_2/CH) ratio. Besides, carbonate aluminates may also be produced if carbonate is freely available. The chemical reactions for different AS_2/CH ratio maybe expressed as follow (Dunster et al., as cited in Ong, 2006):

AS_2/CH=0.5: $$AS_2 + 6CH + 9H \rightarrow C_4AH_{13} + 2C - S - H \tag{2.10}$$

AS_2/CH=0.6: $$AS_2 + 5CH + 3H \rightarrow C_3AH_6 + 2C - S - H \tag{2.11}$$

AS_2/CH=1.0: $$AS_2 + 5CH + 3H \rightarrow C_2AH_8 + C - S - H \tag{2.12}$$

where
AS_2=metakaolin
CH=calcium hydroxide
H=water
C_4AH_{13}=tetracalcium aluminate hydrate
C_3AH_6= tricalcium aluminate hydrate
C_2AH_8 = hydrated gehlenite
C-S-H=calcium silicate hydrate

The type of C-S-H in the above reaction can be more specifically classified as C-S-H (I), which consists of poorly crystallized foils or platelets with a tobermorite-like structure. The exact chemical composition of the calcium silicate hydrates formed in the pozzolanic reaction varies with the water: solid ratio of the mix and the temperature; the composition also changes over the course of the reaction (Lea, 1971, as cited in Rogers, 2011).

2.6 Curing

Curing is referred to any procedure that maintains proper moisture and temperature to ensure continuous hydration of cement (Somayaji, 2001). Curing aims to maintain concrete or mortar saturated, or as nearly saturated, until the hydration of products of cement fill the space which is initially occupied by mixing water in fresh cement paste to the desired extent. Curing of concrete and mortar protect moisture to loss into surrounding and controls the temperature of concrete and mortar for a period sufficient to achieve desired strength level.

2.6.1 Importance of Curing

Hydration is the reaction between cement compounds with water. When cement grains come into contact with water, hydration proceeds until virtually all the grains are hydrated. Hydration takes a very long time, say, a few years to complete. The rate of hydration is rapid at the beginning and the rate decreases progressively for an indefinite period of time.

The hydration gives rise to hardening of concrete and mortar. The properties of concrete and mortar improve with age as long as conditions are favourable for continuous hydration. The degree of hydration is of importance as it concerns with strength development and durability properties. At a given water-cement ratio, the degree of hydration influences the porosity of a hydrated cement paste. The quality of concrete depends on the gel/space ratio of the cement paste. Greater hydration- which means more gel, will lead to higher strength and lower permeability.

Hydration of cement grain at maximum rate can proceed under saturated conditions. It will adversely reduce when the vapour pressure of water in the capillary pores of drops below 80% of the saturation humidity (Mehta and Monteiro, 2006). Since the cement hydration is able to occur solely in water-filled capillaries, it is essential to prevent the concrete or mortar from drying out so that hydration can be prolonged without interruption. Without the inadequate quantity of water, the reactions slow down and eventually come to a total stoppage.

The water loss in concrete and mortar is caused by both external internal factors. The external factors include heat and surrounding air movement which drive the moisture inside the concrete pores. The internal loss of water is by self-desiccation due to the chemical reactions of hydration of cement. In this circumstance, the water loss has to be replaced by water from outside, that is, ingress of water into concrete or mortar, must be made possible.

If concrete and mortar are allowed to dry out, premature termination of hydration happens. The implication is the desired properties, such as compressive strength, water tightness and durability will not be attained. In addition, improper curing leads to enhanced permeability, weaker resistance to abrasion and plastic shrinkage. The effect of inadequate curing is also greater at higher water-cement ratio and is greater in concretes with a lower rate of strength development.

The strength development continues for a short period of time when wet curing is interrupted. It comes to a stop when internal humidity of concrete or mortar drops below 80%. Strength development is reactivated when moisture is supplied again. However, the initial potential strength cannot be yielded. Re-saturation is practical in in laboratory yet it is difficult to be operated in the field. Thus, the optimum way is to provide continuous moist curing to concrete and mortar from the time it is place and finished until it has gained sufficient strength, impermeability, and durability.

2.6.2 Curing Conditions

The curing for concrete and mortar are affected by a few variables-time, temperature and humidity conditions. These factors combined to give effect to the strength durability development of concrete and mortar. Hence, each of the factors must be controlled carefully to ensure the desired properties of concrete and mortar can be realized.

2.6.2.1 Time

The time strength relations in concrete technology generally assume moist curing conditions and normal temperatures. For a provided water-cement ratio, the longer the moist curing period is, the higher the strength of concrete. A study done by Gonnerman and Shuman (as cited in Kosmatka, Kerkhoff, Panarese) on the most curing time on strength gain of concrete (Figure 2.2). In addition, a research conducted by Cement Concrete and Aggregates Australia (2006) how different length of wet curing affects the permeability of cement paste (Figure 2.3).

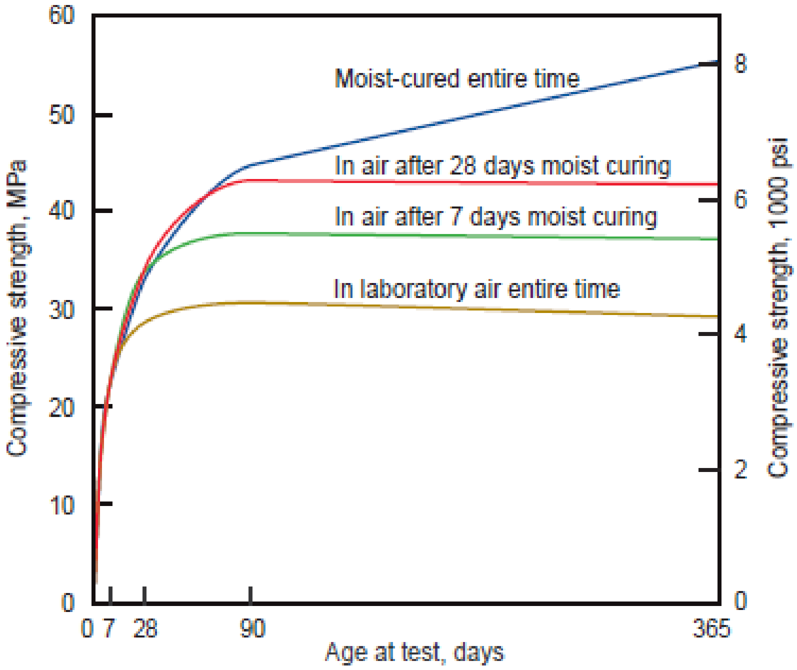


Figure 2.2: Effect of Moist Curing Time on Strength Gain of Concrete (Gonnerman and Shuman)

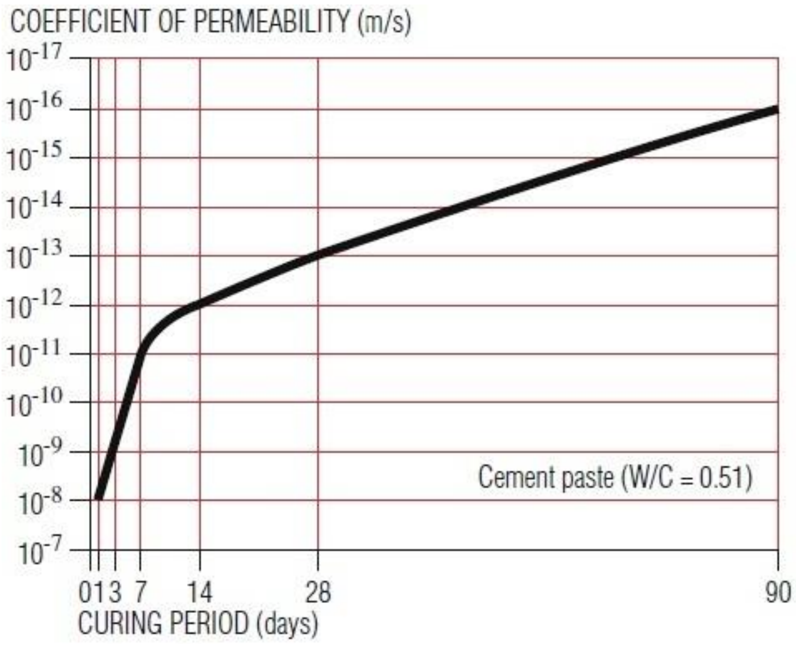


Figure 2.3: Effect of Different Periods of Water Curing on The Permeability of Cement Paste

From Figure 2.2 it can be inferred the longer the time of concrete or mortar subjected to moist or wet curing, the higher the strength. Meanwhile, in Figure 2.3, it is seen extending the water curing period lowers the permeability of cement paste.

The permeability is a parameter that is closely related to the durability of concrete and mortar. The permeability is associated to the structure's porosity, as well as the pores interconnected or discrete. Moreover, the size and number of pores determine the permeability as well. Over time, water curing promotes the formation of hydration products which fill, partially or completely, the existing pores and capillaries. This reduces the porosity of the structure and disrupts the continuity of the concrete of mortar structure. Consequently, the permeability decreases thus the durability is improved.

2.6.2.2 Temperature

Verbeck and Helmuth (as cited in Chini et al, 2003), discovered the reaction between cement and water is like other chemical reactions- the rate speeds up at higher temperature. High curing temperature leads to high initial rate of hydration, which impede later hydration of cement and uneven distribution of hydration products within the paste microstructure. A high temperature causes hydration products to have low solubility and diffusivity thus do not have sufficient time to diffuse away from cement particles. The products concentrate in the region immediately around the grain. As a consequence, there is a non-uniform precipitation of hydration products within the cement paste. Conversely, curing at normal temperature offers adequate time for relatively uniform diffusion and precipitation of hydration throughout the interstitial place within the cement paste.

The pore structure of concrete is crucial for durability as it determines the ease of with which hazardous substances such as chloride ions are transported into concrete. Elevated temperatures bring about coarse, interconnected pores. The microstructure created at high temperature substantially influences the strength and durability of concrete and mortar. Large and interconnected pores boost the permeability of concrete and mortar, thus lower their resistance to chemical attack and lower durability.

In an experimental work carried out by Chini et al (2003), when pure Portland cement concrete specimens were exposed to a controlled ascending temperature rise simulating approximately the situations of mass concrete cured in fields, a moderate reduction in 28-day compressive strength and a substantial rise in permeability were detected compared to samples cured at room temperature.

Conversely, if concrete and mortar are cured at low temperatures near freezing point, the hydration reactions is still able to proceed but with a slower rate. The hydration only cease completely at about -10℃ (Kosmatka, Kerkhoff and Panarese, 2002). However, concrete and mortar should only be exposed to such temperature when a significant amount of mixed water has been used for hydration process. This is because the expansion of free water within the capillaries and pores structure on freezing will disrupt weak and immature concrete.

2.6.2.3 Humidity

The importance of curing humidity is another critical factor as water is a basic substance required for hydration process to occur. The influence of curing humidity can be seen from the data in Figure 2.2. For a given water-cement ratio, the long term-strength of continuously moist-cured concrete was much higher than that of continuously air-cured concrete. Besides, humid environment ensure continuous supply of water for hydration and results in a better-developed pore structure in concrete and mortar. Probably due to microcracking in the interfacial transition zone caused by drying shrinkage, there is a slight retrogression of strength occurs in thin members of moist-cured when they subjected to air drying. (Mehta and Monteiro, 2006). The rate of water loss is a function of surface/ volume ratio, relative humidity, temperature and velocity of surrounding air.

2.6.3 Curing Methods

The curing methods available can be divided into three categories. The first category entails methods that maintain the presence of water (wet curing). In wet curing, the surface of concrete or mortar is continuously in contact with water for a specified length of time, commencing as soon as the surface is not liable to damage, until the desired propertied are achieved. Examples of wet curing methods include spraying or fogging, ponding or immersion, and saturated wet coverings. These methods afford some cooling through evaporation, which is beneficial in hot weather.

Another type of methods applied is by preventing the loss of mixing water (membrane curing). This can be done by applying membrane-forming curing compounds or by covering the concrete with impervious paper or plastic sheets. In membrane curing methods, the surface of concrete or mortar is covered with one or multiple layer of barrier to prevent loss of water from surface, and without the possibility of external water ingress into it.

The third type of methods involves the acceleration of hydration process by supplying heat and moisture (heat and moisture application). This is usually accomplished with live steam, heating coils, or electrically heated forms or pads. Steam curing is advantageous if strength gain during early stage in concrete is important or where additional heat is required to accomplish hydration, as in cold weather.

2.7 Durability and Deterioration of Concrete

Besides the ability to sustain loads, concrete and mortar is also required to be durable during its service life. According to American Concrete Institute (ACI) Committee 201, durability of Portland cement concrete is defined as its ability to resist weathering action, chemical attack, abrasion or any other process of deterioration. In other words, a durable concrete and mortar will maintain its original form, quality and serviceability when exposed to its intended service environment (as cited in Mehta & Monteiro, 2006).

Other than the mechanical properties as well as initial cost, the durability of concrete and mortar have gained more attention in recent years for a variety of reasons. First, the repair and replacement costs of structures due to material failure have occupied a significant portion of total construction budget. The escalation in replacement costs of structures and emerging emphasis on life-cycle costs have urged engineers to put serious attention to durability issues.

Besides, the durability of materials is closely related to ecology. Making construction materials to last longer is an important step towards conservation of natural resources.

2.7.1 Water as an Agent of Deterioration

Water is the prominent agent for the destruction of most of the materials including concrete and mortar and this is the core of most durability problems. In porous solids, water is responsible to cause various types of physical processes of degradation. As a transport agent for aggressive ions, water can be the root cause of chemical processes of degradation.

Water is undoubtedly the most abundant fluid and exists in various forms in the nature: seawater, groundwater, river water, ice, vapour and others. The small size of water molecules enable them to penetrate into very fine pores or cavities. As a solvent, water is known to be able to dissolve more substances that any other known liquids. This characteristics lead to existence of a variety ions and gases in water, which in turn imposing chemical decomposition of solid materials.

Furthermore, water possessed of the highest heat of vaporization among the common fluids. Hence, at normal temperatures, water tends to exist in liquid state in a porous material rather than vaporizing and leaving the material dry. The internal movement of moisture and structural transformation of water within porous solids are known to inflict disruptive volume changes. For instance, freezing of water into ice, development of osmotic pressure due to concentration gradient, and hydrostatic pressure build up by differential vapour pressure can lead to high internal stress.

A water molecule has a chemical formula H_2O and is covalent bonded. Due to the difference in the charge centres of hydrogen and oxygen, the hydrogen is partially positively charged while the oxygen is partially negatively charge. This leads to the polarity of water molecules and the positively charged hydrogen in a water molecule attracts the negatively charged oxygen of the neighbouring molecule. The relatively weak force of attraction, known as hydrogen bond, is responsible for the structure of water. The polarity of water molecules enable most of the ionic compounds to be dissolved. The structure of water molecules is depicted in Figure 2.4.

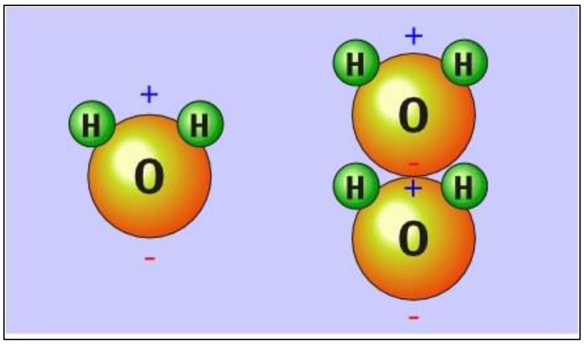


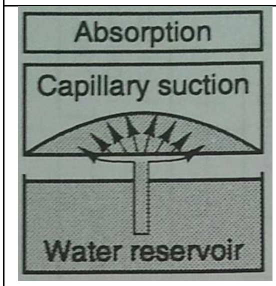
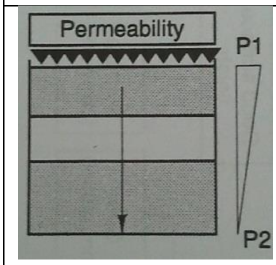
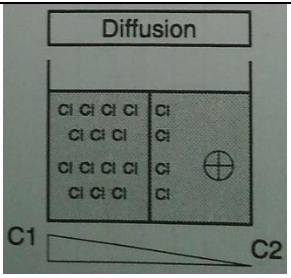
Figure 2.4: Structure of Water Molecules

Water in room temperature has about 50% of hydrogen bonds broken. This results in unsatisfied surface charges which give rise to surface energy. The surface energy in liquids brings about surface tension (the force required to pull the molecules apart), which accounts for the tendency of a large number of molecules to adhere together. The high surface tension of water prevents it from acting as an effective plasticizing agent in concrete mixtures (Mehta and Monteiro, 2006).

2.7.2 Permeation Properties of Concrete and Mortar

Many processes leading to deterioration of concrete and mortar entails the ingression of aggressive fluids into them from surrounding environment. This is followed physical or chemical processes attacking the concrete or mortar fabric. Likewise, internal damage of concrete and mortar is determined mainly by moisture movements. Assessment of the resistance concrete and mortar to potential damage can be conducted via measurement of permeation properties, which is interpreted as the ease with which agents can move in and out of concrete (Jackson & Dhir, 1996). The permeation properties comprise of three fundamental transport mechanisms which can operate in semi-permeable (porous) medium such as concrete and mortar: absorption, permeability and diffusion. This is illustrated as in Table 2.11:

Table 2.11: Permeation Properties of Concrete and Mortar

Illustration	Mechanism	Example
	Capillary suction, liquids	Cyclic wetting and drying, marine structures
	Pressure differential, liquids and gases	Water-retaining structures
	Concentration differential, liquids, gases and ions	Sulfate bearing ground waters, foundation elements

Absorption is the by which concrete takes a liquid such as water or aqueous solution by capillary attraction. The rate at which water enters is call absorptivity or sorptivity and it is dependent on the size and interconnection of the capillary pores in concrete, and the moisture gradient existing on surface.

Permeability refers to the flow property of concrete that quantitatively characterises the ease by which a fluid passes through it, under the action of pressure differential. This property relies on the pressure gradient as well as on the size and interconnectivity of the capillary pore in concrete and mortar.

Lastly, diffusion is a process by which a vapour, gas or ion can pass into concrete or mortar due to concentration gradient’s action. Diffusivity defines the rate of movement of the agent and is affected by the concentration gradient from the concrete or mortar surface, the type of ingressing agent and any reaction with the hydrating cement paste, size and interconnection of capillary pores within ((ackson & Dhir, 1996).

2.7.3 Classification of Causes of Concrete Deterioration

Generally, the causes of deterioration on concrete and mortar can be categorized into two main groups-physical causes and chemical causes. Mehta and Gerwick (as cited in Mehta and Monteiro, 2006) classified the physical causes of concrete deterioration into two categories: (i) surface wear or loss of mass due to abrasion, erosion and cavitation; (ii) cracking due to normal temperature and humidity gradients, crystallization of salts in pores, structural loading, and exposure to temperature extremes such as freezing and fire.

On the other hand, the authors divided the chemical causes of deterioration into three groups: (i) hydrolysis of cement paste components by soft waters; (ii) cation exchange reactions with aggressive fluids and the cement paste; (iii) reactions that form of expansive products, such as sulfate attack, alkali silica reaction, and corrosion of reinforcing steel in concrete. Table depicts the classification of causes of deterioration by by Mehta and Gerwick:

Table 2.12: Physical and Chemical Causes of Concrete and Mortar Deterioration

Physical Causes	Surface Wear	Abrasion, Erosion and Cavitation
	Cracking	Volume change due to: 1. Normal temperature and humidity gradient 2. Crystallization pressure of salts in pores
		Structural Loading: 1. Overloading and impact 2. Cyclic loading
		Exposure to temperature extremes: 1. Freeze thaw cycles 2. Fire
Chemical Causes	Reaction involving hydrolysis and leaching of components of hardened cement paste	Soft water attack on calcium hydroxide and C-S-H present in hydrated Portland cements
	Exchange reactions between aggressive fluids and components of hardened cement paste	Removal of Ca^{2+} ions as soluble products: Acidic solution forming soluble calcium compounds such as calcium chloride, calcium sulfate and calcium bicarbonate
		Removal of Ca^{2+} ions as non-expansive insoluble products: Solutions of oxalic acid and its salt, forming calcium oxalate
		Substitution reaction replacing Ca^{2+} in C-S-H: Long-term seawater attack weakening C-S-H by substitution of $Mg^{2+}$for Ca^{2+}

	Reaction involving formation of expansive products	Sulfate attack forming ettringite and gypsum, alkali-aggregate attack, corrosion of steel in concrete, hydration of crystalline CaO and MgO

2.7.4 Physical Causes of Concrete and Mortar Deterioration

Damage due to physical attack occurs may owe to several mechanisms. First, concretes experience expansion or contraction of elements under restraint, caused by nature of concrete itself, for instance drying shrinkage and plastic shrinkage. Damage can be inflicted on concrete via fluctuations in environmental conditions, such as cyclic freezing and thawing wetting and drying, In addition, physical deterioration occurs as a consequence of explicit physical interaction with the environment, such as fast moving water and traffic on industrial floors.

2.7.4.1 Abrasion, Erosion and Cavitation

Abrasion, erosion and cavitation cause progressive loss of mass from concrete and mortar surface. The term abrasion normally refers to dry attrition, such as in the case of wear o pavements and industrial floors by vehicular traffic. In other words, abrasion can be referred to the wearing away of the surface by rubbing and friction (Emmons & Emmons, 1993). Generally, abrasion wears away the surface uniformly including cement matrix and aggregates.

The term erosion is used to describe wear by abrasive action of fluids containing solid particles in suspension. Erosion happens in hydraulic structures such as canal linings, spillways, and concrete pipes. When a fluid containing suspended solid particles is in contact with concrete, the impinging, sliding and rolling action of particles will impose surface wear. The rate of surface erosion is a function of porosity, compressive strength, as well as the size, shape, density, hardness and velocity of the moving particles. For small sized and low quantity of solids, the erosion loss will be negligible at bottom velocities up to 1.8m/s (Mehta & Monteiro, 2006).

Cavitation erodes concrete surfaces resulting from the collapse of vapour bubbles formed by pressure changes within a high velocity water flow. When vapour bubbles form, they flow downstream with water. They collapse (implode) with great impact when enter a region of higher pressure. The formation of vapour bubbles and their subsequent collapse is known as cavitation. The collapse of vapour bubbles release considerable amount of energy and causes cavitation damage-cement matrix is eroded and harder aggregate is left in place. At higher velocities the cavitation forces may be great enough to wear away large quantities of concrete (Emmons & Emmons, 1993).

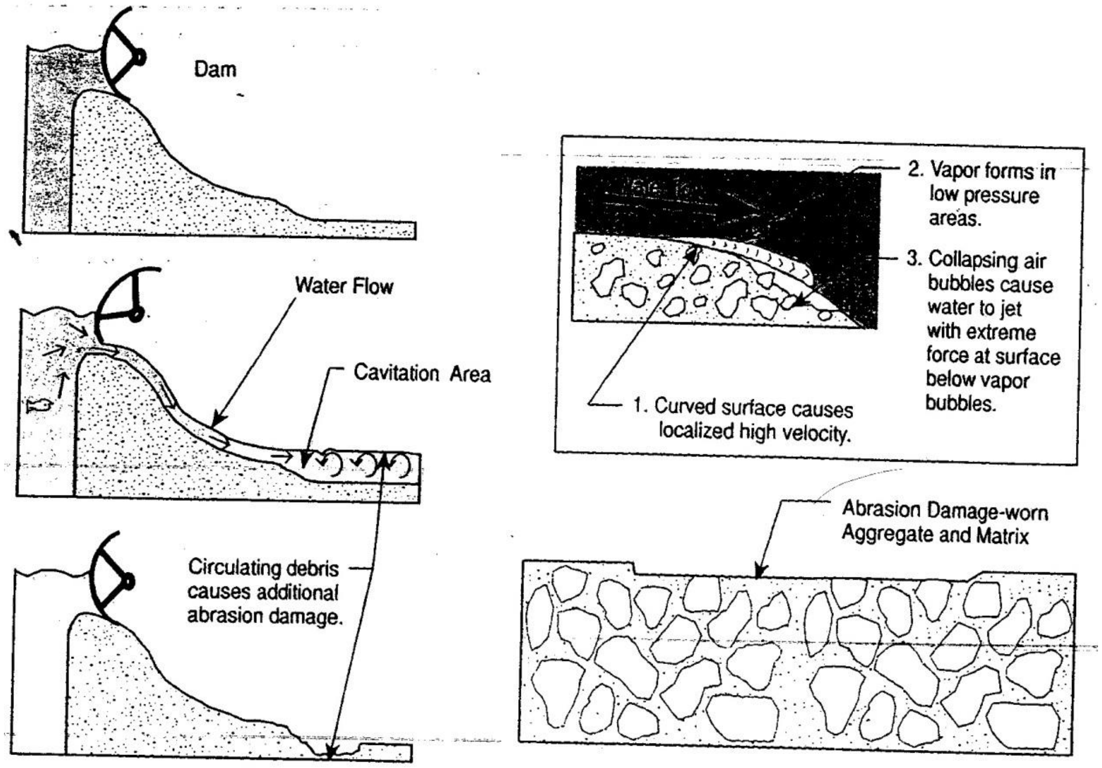


Figure 2.5: Abrasion, Erosion and Cavitation

2.7.4.2 Freeze-Thaw Disintegration

Freeze-thaw disintegration occurs when the following conditions are fulfilled: First, there is a freezing and thawing temperature cycles within the concrete or mortar; next, the porous concrete or mortar absorbs water into the pores and capillaries. Freeze thaw disintegration generally happens on horizontal surfaces that are exposed to water, or on vertical surfaces which are at the water line in submerged portion of structures. The water in pore structure expands when it is converted to ice upon freezing. The expansion creates localized tension forces that fracture the surrounding concrete matrix. The fracturing occurs in small pieces and works from outer surface inward. The rate of freeze-thaw disintegration is dependent on the porosity, moisture saturation, number of freeze-thaw cycles, air entrainment and the absorption properties of aggregate (Emmons & Emmons, 1993).

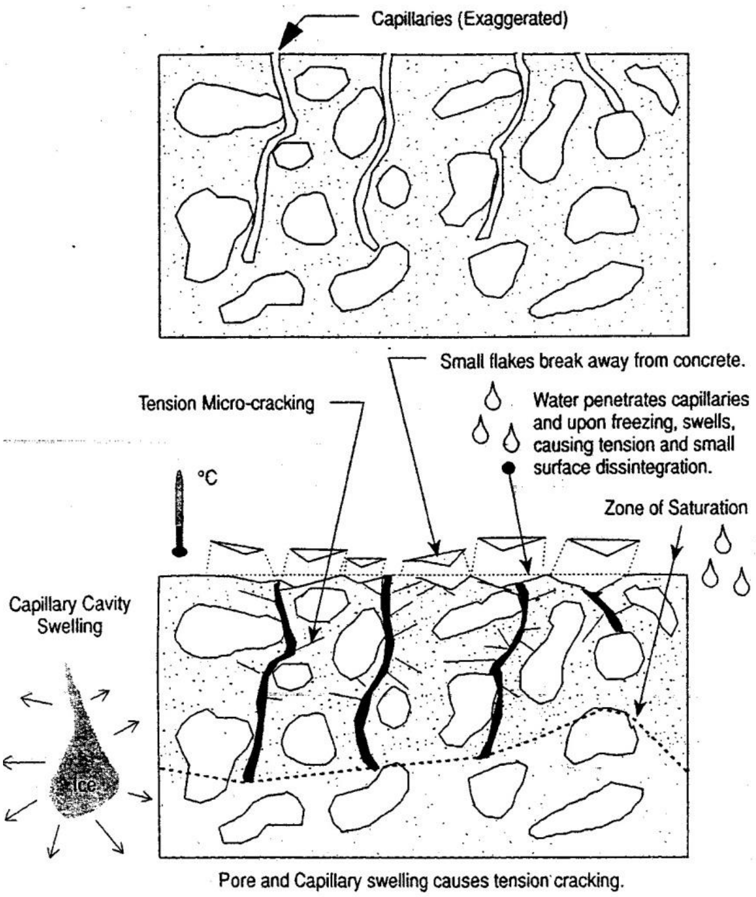


Figure 2.6: Deterioration Caused by Freeze-Thaw Action

2.7.4.3 Effect of Heat/Fire Damage

Damage to concrete when subjected to high temperatures occurs via the effect on both hydrated cement paste and aggregates. If there is any reinforcement bar embedded in the concrete, it will be affected as well under high temperature. Concrete exposed to up to 100 ℃ is considered as healthy. For temperatures up to 300 ℃, the strength of concrete is not adversely decreased. Nevertheless, repeated cycles to up to this temperature might cause the concrete to loss strength progressively (Campbell, Allen & Roper, as cited in Jackson and Dhir, 1996). When the temperature is above 500 ℃, concrete losses its compressive strength and modulus of elasticity significantly (Concrete Society, as cited in Jackson and Dhir, 1996).

Fire attacks concrete in extreme ways. First, it causes uneven volume change in affected members due to ample temperature gradient from source of fire to near surface. It results in distortion, buckling and cracking of concrete. Next, extreme heat causes spalling of rapidly expanding concrete surface near source of fire. Some aggregates expand in burst, damaging the surrounding matrix. Besides, moisture within concrete is rapidly converted to steam and causing bursting of small pieces of concrete. In addition, cement mortar is changed to lime at ℃ and seriously disrupt the concrete structure. For reinforced concrete, the steel reinforcements lose tensile capacity at elevated temperatures (Emmons& Emmons, 1993).

2.7.5 Chemical Causes of Concrete and Mortar Deterioration

In concrete and mortar, the deterioration processes initiated by chemical reactions, generally, but not necessary, entail chemical interactions between aggressive agents in surrounding environment and the constituents of cement paste. A well hydrated cement Portland cement paste has a high-pH pore fluid, ranged from 12.5 to 13.5 when in the state of stable equilibrium. Theoretically, any environment with pH of less than 12.5 may be deemed as aggressive as a reduction of alkalinity of pore fluid will eventually lead to destabilization to hydration products of cement.

2.7.5.1 Sulfate Attack

Soluble sulfates, principally those of sodium, calcium and magnesium are generally present in areas of mining operations, chemical and milling industries. Sodium and calcium are the most common sulfates in soils, water and industrial processes whilst magnesium sulfates are less common but more destructive. All sulfates are potentially harmful to concrete as they react chemically with hydrated lime in cement paste and hydrated calcium aluminate. Consequently, solid products with volume greater than the products entering the reaction are generated. Two common products generated from sulfate attack are gypsum and ettringite. These products expand, pressurize and disrupt the cement matrix. This causes surface scaling and disintegration is triggered, followed by mass deterioration.

On hydration, Portland cements with more than 5% of C_3A will contain most of the alumina in the form of monosulfate hydrate, $C_3A \cdot C\bar{S} \cdot H_{18}$. If the of C_3A content is greater than 8%, the hydration products will also contain$C_3A \cdot CH \cdot H_{18}$. Due to the presence of calcium hydroxide, when a cement paste comes in contact with sulfates ions, both the alumina containing hydrates are converted to high sulfate form (ettringite, $C_3A \cdot 3C\bar{S} \cdot H_{32}$) as depicted by the following equations (Mehta and Monteiro, 2006):

$$C_3A \cdot CH \cdot H_{18} + 2CH + 3\bar{S} + 11H \rightarrow C_3H_2S_3 + 3CH + C_3A \cdot 3C\bar{S} \cdot H_{32} \quad (2.13)$$

$$C_3A \cdot C\bar{S} \cdot H_{18} + 2CH + 2\bar{S} + 12H \rightarrow C_3H_2S_3 + 3CH + C_3A \cdot 3C\bar{S} \cdot H_{32} \quad (2.14)$$

Gypsum formation occurs as a result of cation-exchange reactions and is capable to cause expansion. A study conducted my Mehta revealed that deterioration of hardened Portland cement paste by gypsum formation first goes through a process which first reduce the pH of the system and loss in stiffness and strength, subsequently by expansion and cracking and transformation of concrete into a mushy and non-cohesive mass.

Depending on the type of cation associated with sulfate solution such as Na+, K, or Mg, both calcium hydroxide and C-S-H present in the hydrated Portland cement paste may be converted into gypsum by sulfate attack (Mehta and Monteiro, 2006):

$$Na_2SO_4 + Ca(OH)_2 + 2H_2O \rightarrow CaSO_4 \cdot 2H_2O + 2NaOH \qquad (2.15)$$

$$MgSO_4 + Ca(OH)_2 + 2H_2O \rightarrow CaSO_4 \cdot 2H_2O + Mg(OH)_2 \qquad (2.16)$$

$$\begin{aligned} &3MgSO_4 + 3CaO \cdot 2SiO_2 \cdot 3H_2O + 2H_2O \\ &\rightarrow 3\,(CaSO_4 \cdot 2H_2O) + Mg(OH)_2 + 2SiO_2 \cdot H_2O \end{aligned} \qquad (2.17)$$

When attacked by sodium sulfate, sodium hydroxide is formed as a by-product. This continuously maintain a highly alkali environment in the system, which is necessary for the stability of hydration products, C-S-H. On the other hand, when the cement paste is attacked by magnesium sulfate, calcium hydroxide is converted into gypsum is accompanied by simultaneous formation of magnesium hydroxide. Magnesium hydroxide is insoluble and its formation reduces the alkalinity in the system. In the absence of hydroxyl ions in solution, C-S-H is no longer stable. Hence, the magnesium sulfate attack is more severe on concrete (Mehta and Monteiro, 2006).

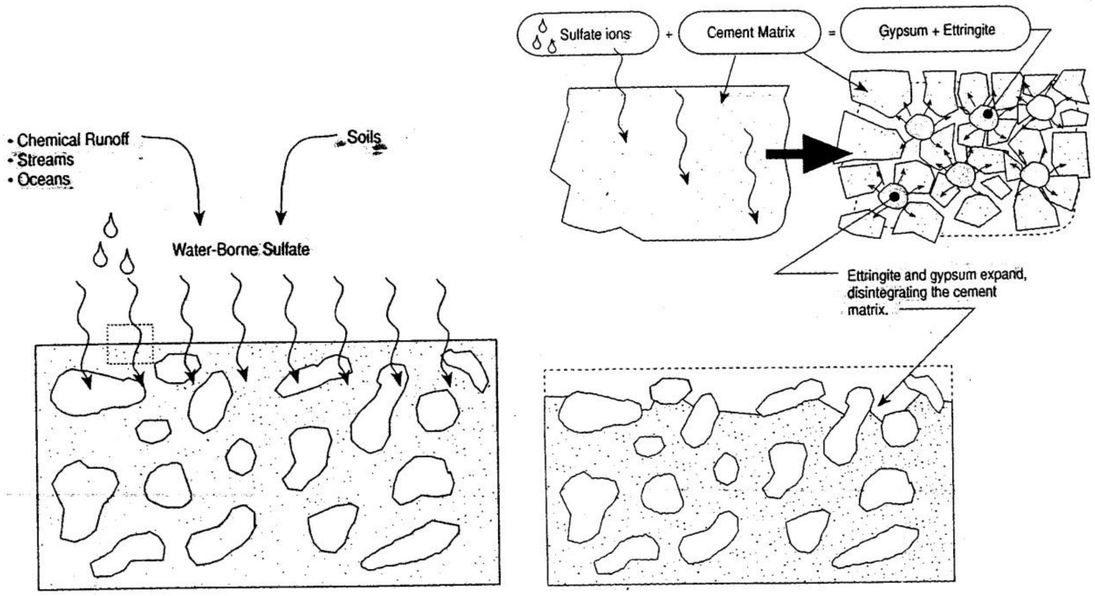


Figure 2.7: Deterioration of Concrete by Sulfate Attack

2.7.5.2 Chloride Penetration

Chlorides generally are not harmful to hydrated cement paste and aggregates. However, in reinforced concrete, chlorides are considered as an aggressive agent because they cause corrosion of reinforcement steel. Chlorides can be introduced into concrete or mortar by coming into contact with environments containing chlorides, such as sea water or de-icing salts. Penetration of chlorides commences on surface and moves inward. The rate of penetration depends on the concentration of chlorides comes in contact with the concrete, the permeability of concrete and amount of moisture present.

Eventually, the reinforcing steel is corroded by chlorides with the presence of oxygen and moisture. As the rust layers builds, expansion of the oxides creates tension forces and makes the concrete to crack and delaminate. When cracking and delamination of concrete progress, corrosion is accelerated because of easy access of corrosive salts, oxygen and moisture. Concrete’s pH is the primary factor determines the concentration of chlorides required to promote corrosion (Emmons and Emmons, 1993).

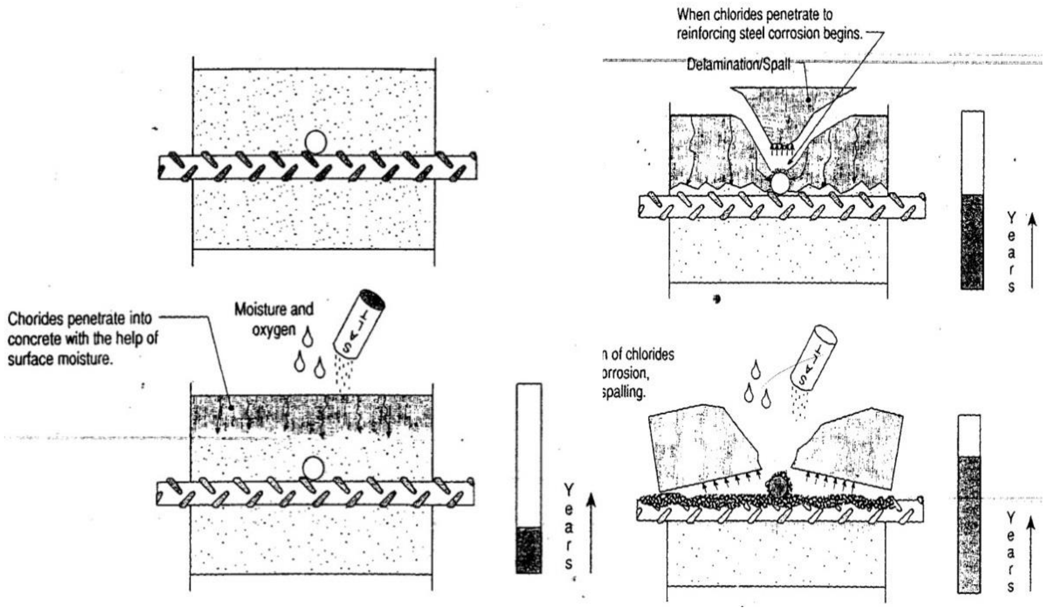


Figure 2.8: Deterioration of Concrete Due to Penetration of Chlorides into Concrete

2.7.5.3 Alkali-Aggregate Reactions (AAR)

Alkali-aggregate reactions (AAR), or alkali-silica reactions (ASR), are chemical reactions involve alkali and hydroxyl ions from Portland cement paste with certain reactive siliceous materials that are often present in the aggregate. The reactive silica reacts with potassium, sodium, and calcium hydroxide and form a gel around the reacting aggregates. When the gel around the aggregate is exposed to moisture, it expands and generates forces which cause tension cracks around the aggregate.

Alkali-silicate gel is soluble in water and able to move from the interior of aggregates to the micro-cracked regions both within the aggregate and concrete. If water continues to be available, the concrete enlarges and causes extension of microcracks which eventually reach the outer surface of concrete. The crack pattern is irregular and referred to as map cracking. Alkali-aggregate reaction may goes unnoticed for some period of time, possibly years, before related serious distress developed.

2.7.5.4 Carbonation

Carbonation is a term used for the reaction between the lime in concrete and acidic carbon dioxide from air, obtaining calcium carbonate. Conventional air contains carbon dioxide (CO_2) in relative low concentration of about 0.03%. In the contrary, the carbon dioxide in industrial area is higher.

Carbon dioxide penetrates into the pore of concrete and mortar by diffusion and reacts with calcium hydroxide dissolve in pore water. The alkalinity is reduced as a consequence of this reaction to a value of about 10. This results in downgrade of concrete quality and loss of its ability to protect reinforcement from corrosion. By other word, the passivity of the protective layer on steel is destroyed. When depassivated steel is exposed to mild alkaline or acidic environment, corrosion commences if moisture and oxygen gain into concrete.

Carbonation is most active at around 50% of relative humidity, and it virtually disappears when the relative humidity is below 25% or near saturation (Somayaji, 2001). The process needs constant change in moisture levels from dry to damp to dry. Carbon dioxide will not occur if concrete or mortar is constantly under water. Carbonation can contribute to significant weight gain and is able to cause a soft surface, dusting and colour change. Carbonation may accelerated steel corrosion if the surface is exposed to salts and moisture.

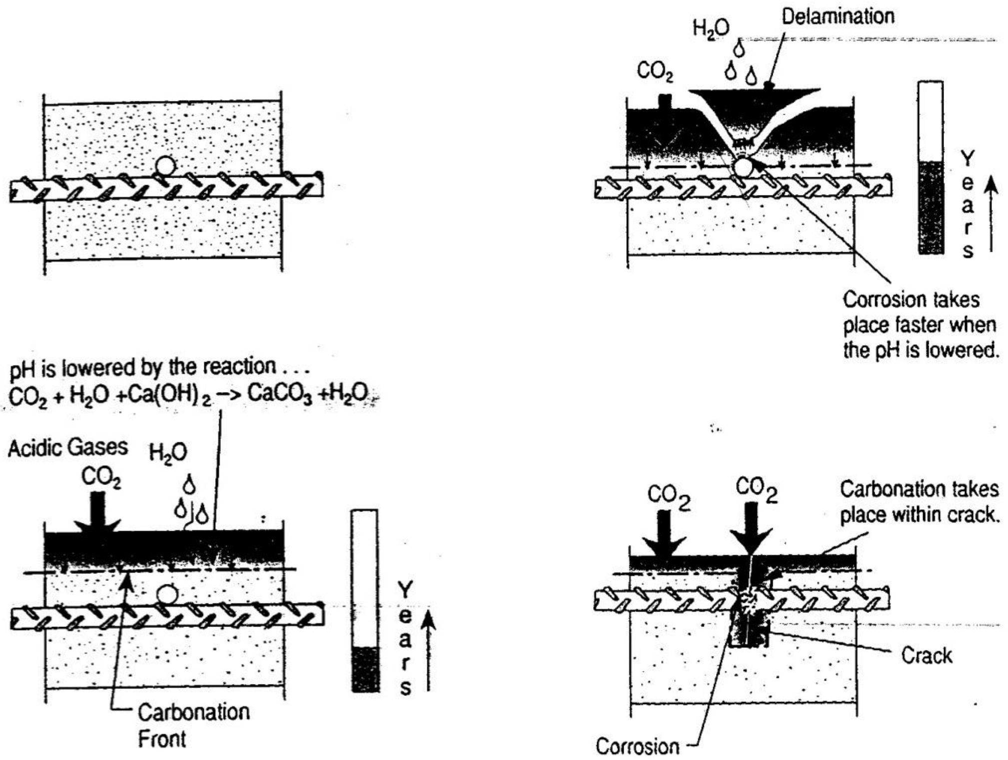


Figure 2.9: Carbonation Process on Concrete

2.8 Porosity, Pore System and Durability

Concrete and mortar are porous materials and contain numerous pores over a wide range of size, from a few nanometres to about tenth of centimetres. The porosity within concrete and mortar is generally contributed by three different types of air: gel pores, capillary pores and entrapped or entrained air.

Gel pores are referred to the space between layer in C-S-H with thickness between 0.5 and 2.5nm, including interlayer spaces, micropores and small isolated capillary pores. Gel pores can contribute up to 28% of cement paste porosity. They impose little impact on strength and permeability but can influence shrinkage and creep.

Capillary pores are formed when the water-filled space within the cement paste is not replaced by hydration products as the hydration of cement progresses.

The amount and interconnection of capillary pores depend on initial separation of cement particles, which is determined by w/c ratio. The size of capillary pores is on the order of 10 to 50 nm, although they can be as large as 3mm to 5mm for higher w/c ratio.

In addition, concrete and mortar may contain a small amount of entrapped air during mixing process. Air voids are generally spherical and unlike capillary voids which have irregular shape. For some reasons, air voids are introduced by adding air-entraining admixture into the fresh mix. Entrained air provides path for water to migrate from larger void to smaller voids. Entrapped air voids may as large as 3mm while entrained air voids normally ranged from 50 µm to 200 µm. Both entrapped and entrained air voids are much larger than capillary voids and thus give a more substantial effect on strength and durability.

The ingress of fluid into the interior of concrete occurs through the pore system and subsequently by physical or chemical changes in the in the internal structure leading to deterioration. The permeation indices of concrete are measures of durability. A permeation index is further dependent on the pore system characteristics of concrete and mortar. The most critical properties of concrete or mortar’s pore system are its porosity and pore-size distribution. The two properties mentioned have the most decisive role In deterioration processes of concrete (Garboczi; Valenta; Atzeni et al.; Da ̈rr & Ludwing; Rostasy & Wiedemann, as cited in Kumar and Bhattracharjee, 2003).Hence, the durability quality of concrete and mortar and be examined implicitly from the knowledge of pore system characteristics.

According to Meeks and Carino (1999), the distribution of larger pore sizes imposes greatest influence rather than the total porosity on durability related properties of concrete and mortar. Largest pores refer to those ranged from 0.09 µm to 0.15 µm. Justice (2005) also pointed out that it is the permeability and pore size distribution but not total porosity are critical to the performance and durability of concrete.

2.9 Effect of Curing Regimes on Durability Properties of Mortar

The curing conditions, especially during the early stage of hydration, are essentially important to the durability properties of concrete and mortar. The importance of curing was remarked by Ramezanianpour and Malhorta (as cited in Sajedi et al., 2011): “If the potential of concrete with regards to strength and durability is to be fully realized, it is mostly essential to be cured adequately. The curing becomes even more important if the concrete contains supplementary cementing materials such as fly ash or GGBFS or silica fume, and is subjected to hot and dry environments immediately after casting”. Substantially curing is important for a concrete to fully develop its potential performance (Khatri & Sirivivatnanon, as cited in Tasdemir, 2003).

2.9.1 Transport Properties and Permeability

According to Ramezanianpour and Malhorta (as cited in Khatib and Hibbert, 2011), concrete containing GGBFS is more sensitive to poor curing than normal Portland cement concrete, and this sensitivity increases with increasing amount of GGBFS. A study by Meeks and Carino (1999) reported that good curing practices can reduce the capillary porosity of a concrete specimen by as much as 80%.This is beneficial on the properties of absorptivity, corrosion resistance and scaling resistance. According to Graf and Grube (as cited in Darwish, 1995), with good curing, slag concrete is capable to achieve lower permeability than equivalent OPC concrete, but a higher permeability if cured poorly.

Kumar and Bhartacharjee (2003) investigated the initial surface absorption (ISA) of concrete exposed to two different cuing conditions, namely exposure to open air and exposure to acidic environment of pH 4-5. The results showed that concretes cured in open air have lower ISA than those cured in acidic environment. Besides, ISA decreases when concretes are cured for longer period of time. Parniani et al. (2011) suggested effective curing is absolutely essential for surface durability.

Fulton (as cited in Parniani et al, 2011) reported that concrete containing GGBFS is more susceptible to poor curing conditions than concrete without GGBFS where GGBFS is used in percentages greater than 30%.

Kasai et al. (as cited in Darwish, 1995) concluded that when early moist curing is carried out for longer period, the permeability becomes lower, as the cement paste achieves greater degree of hydration resulting in more compact matrix. This is consistent with the results reported by Lee and Chisholm (2005). The sorptivity of concretes decreases if they are subjected to longer continuous wet curing period. Futhermore, curing of concretes in water bath normally gives lower sorptivity than those cured for an equivalent length of time in a fog room.

Bouikni, Swamy and Bali (2009) studied the effect of curing regime on concrete containing 50% and 65% slag. The authors reported concretes with slag when exposed to prolonged drying, resulted in increased of pore volumes. Nevertheless, the prolonged drying exerted a less detrimental effect to the durability of 50% slag concrete compared to that of 65%. Prolonged drying only increased the number of smaller pore sizes for 50% slag concrete. Yet, for 65% slag concrete, prolonged drying enhanced the volume at all pore sizes. The worst situation occurred when there was no water curing at all. An initial 7 day water curing was found to be beneficial in reducing the pore sizes and thus permeability.

Mangat and El-Khatib (as cited in Darwish, 1995) investigate the influence of curing on pore structure of blended cement pastes and concretes. The results connote that dry curing at early ages results in higher intruded pore volume, coarser pore structure and higher absorption of surface zone as compared to initial moist curing. The effect is more adverse in slag blended cement mixes than in control mix.

Chen et al. (2012) investigated the effect of curing regime on high blast furnace slag cement (HBFS) concrete and normal Portland cement concrete. The specimens were exposed to two curing regime: (i) seawater curing and (ii) air curing in marine atmosphere. The total pore volume of concrete, measured by Mercury Intrusion Porosity (MIP), decreases with increased curing period. A lower porosity than the respective OPC was yielded. Besides, the authors reported the total porosity for seawater-cured specimens is lower than those air-cured specimens, regardless of the types of concrete mixture. The HBFS concretes have a lower porosity than the corresponding OPC concrete. The authors proposed these are due to formation of denser microstructure within cement matrix.

A research conducted by Darwish (1995) reported that for all concretes mixes, with or without mineral admixtures, have largest porosity when continuously cured in air. The continuously water cured specimens exhibit smallest porosity, subsequently by those exposed to 7 days water/air-curing. Besides, it is noticed that concrete containing slag have higher porosity if continuously cure in air as compared to control mix and lower porosity if exposed to 7 days water/air-curing or continuous moist curing. In addition, concrete with or without slag subjected to air curing produced with very coarse pore size distribution with large threshold pore diameter, compared to 7 days water/air-curing and continuous moist curing. The results imply when slag concrete is exposed to a drying environment and cured inadequately, the mircrostructure properties and thus its long term durability will be adversely affected.

Cakır and Aköz (2006) studied the effect of curing conditions on mortars with and without GGBFS. The cement content was partially replacement by 0%, 30% and 60% of GGBFS. The specimens were cured in two conditions-the first group was stored in lime-saturated water at 20 ± 2 ℃ whilst another group was put into humidity and temperature controlled cabinet at 40 ± 2 ℃ and approximately at 100% relative humidity until test age. The appearing porosity (water porosity) of water-cured specimens of all mixes is higher than their corresponding cabinet-cured specimens during early age of 7-days. The appearing porosity of mortars decreases with time for both curing conditions. At later age, water-cured specimens have lower appearing porosity than cabinet-cured specimens. The appearing porosity decrement of control and slag mortar cured in water is higher compared to those of cured in moisture cabinet. The results reflect that higher degree of hydration during early stage due to increased temperature is not continual, and the hydration products are also porous. (Neville; Zain, as cited in Cakır and Aköz, 2006).

Moreover, Cakır and Aköz (2006) also reported that the sorptivity coefficient (capillary coefficient) of specimens cured in cabinet is higher compared to specimens cured in water in the beginning, although a reversed result is obtained for porosity. It appears that higher temperature leads to fast hydration but at the same time, causes capillary cracks which boost the capillary coefficient. The capillary coefficient decreases with time for both curing conditions. At later age, all water cured specimens, with or without slag exhibit lower capillary coefficient than their respective cabinet-cured specimens.

Several experimental investigations have revealed that the capillary permeability of is adversely influenced by the curing condition (Khatri & Sirivivatnanon, as cited in Tasdemir, 2003). Curing is generally deemed to have a more substantial effect on concrete with mineral admixtures such as GGBFS, fly ash and silica fume than normal concrete (Bentur and Goldman, as cited in Tasdemir, 2003). Besides, curing impose greater effect on the permeability than on the strength of concrete. Tasdemir (2003) investigated the effect of curing conditions on the sorptivity coefficient of concrete with different mineral admixtures. The three curing regimes applied were: (i) air curing in room for 28 day, (ii) 7day curing under polyethylene sheet and wet burlap for 7day followed by 21 day of air curing in room, and (iii) water curing in water tank for 28 days. The results showed that the sorptivity coefficient for all mixtures including reference specimens without mineral admixture is lowest when cured under condition (iii), followed by curing condition (ii). Specimens cured under condition (i) possessed the highest sorptivity coefficient.

Ortega et al. (2011) approached the capillary sorptivity coefficient of OPC and slag cements mortars exposed to different environmental conditions. Four different environmental conditions were studied: (i) 20℃ and 100% relative humidity (RH); (ii) 15℃ and 85% RH; (iii) 20℃ and 65% RH and (i) 30℃ and 40% RH. The sorptivity test was performed at 7, 28, 90, 180 and 365 days of age. The authors concluded that a high RH environment improves the capillary absorption of both OPC and slag mortars. A higher temperature in environment enhances the capillary absorption at the initial stage for both OPC and slag mortars. It was also reported that the durability properties of slag mortars are more influence by the environmental temperature than by relative humidity.

Bernal et al. (2012) cured alkali-activated GGBFS/MK blends concretes at a relatively humidity of 90% at 25 ± 5 ℃ and reported that curing for 90 days reduces the porosity of all concrete mixes. Besides, a total absorption into concretes within 48 hours was conducted and it is reported longer curing period leads to lower absorption due to refinement of porosity in the specimens. Incorporating MK into alkali activated GGBFS blends were observed generally reduces the water absorption into concrete at both 28 and 90 days of curing period. The reduction is more notable for 10% than 20% of MK replacement as cementitious material. Other than that, it is observed that at the mentioned curing regime, the initial capillary absorption of both concretes solely based on GGBFS and GGBFS/MK blends is much lower at the age of 90 days than 28 days.

A study by Parrott (as cited in Meeks and Carino, 1999) reported that moist curing period reduces water absorption for concrete with OPC or blended cement. Concretes with cement partially replaced by 50% of slag require longer period to reach the same resistance to water absorption as concrete with 100% Portland cement

Alamri (1988) studied the effect of different curing environments on water absorption of concrete with 30% and 50% of GGBFS as cement replacement at w/c ratio of 0.5. The concrete specimens were subjected to four different curing conditions before they were tested at the age of 28 days: (i) 50 ℃ ± 2 ℃ + 15% ± 5% relative humidity (RH); (ii) 40 ℃ ± 2 ℃ + 60% ± 5% RH; (iii) wrapped for 2 more days at 20 ℃ after demoulded and then placed in oven at 50 ℃ + 15% RH (iv) 20 ℃ ± 2 ℃ + 100% RH. It was observed with an exception in curing condition (ii) concretes containing slag have lower water absorption than OPC mix for all curing conditions. The author pointed that slag mixes are more sensitive to lack of moisture for curing at relatively low temperatures. Both 30% and 50% slag mix showed much lower water absorption than corresponding OPC mix when cured under condition (iv).

Other than that, Alamri (1988) conducted initial water absorption test (ISAT) on slag with 30% and 50% GGBFS. The results revealed that slag mixes are very sensitive to lack of moisture during curing. The ISA of slag mixes increased by 80% to 150% when cured in condition (i) compared to condition (iii). For the first three curing conditions, the ISA of concrete containing slag is higher than that of OPC concrete. However, when cured in optimum condition (condition (iv)), slag concrete shows a lower ISA than OPC concrete. The author suggested that longer curing periods produce finer and denser pore structure as more of the pores near to surface become blocked by hydration products. The presence of high level of GGBFS results in a more sensitive behaviour than OPC mix due to slower reaction.

Razak, Chai and Wong (2004) studied the near surface characteristics of concrete containing 10% of MK as cement replacement. The specimens were subject to four different initial curing regimes: water curing, burlap, plastic sheet and air. Subsequently, durability tests were carried out at ages 7, 28, 56 and 90 days after initial curing of 28 days. ISAT was conducted on control concrete and MK concrete. For control concrete, the flow is highest for air-dried specimens, followed by plastic sheet, wet burlap and water cured specimens at all ages. A same trend was observed for MK mix, whereby the ISA is lowest if the specimens are water-cured.

The sorptivity test on control and MK mix indicated that water cured specimens, particularly those containing MK, have lowest sorptivity coefficient compared to other curing regimes. In the contrary, air cured specimens show the highest sorptivity. The effect of curing method is more prominent in the mixture containing MK than plain concrete. The sorptivity values of water cured specimens with MK are much lower than that of control concrete.

Other than the above, the authors conducted water absorption test by total immersion to determine the porosity of control and MK concrete. It was observed that both MK and control mixtures have low absorption characteristics (less than 10%) irrespective of their curing regimes. A similar trend to ISAT and sorptivity in which mixtures containing MK have much lower values was obtained. The absorption is highest for air curing, followed by wet burlap, plastic membrane and finally water curing. For MK mixtures, there is a considerable decrease in water absorption with age.

There is limited study and information about the effect of curing regime on the durability properties of mortar and concrete containing metakaolin. The research conducted by Razak et al. does not include replacement levels of MK below or above 10% as cement. Therefore, the variation of durability properties of mortar and concrete with curing regimes in term of ISAT, sorptivity and water absorption for MK replacement levels lower and higher than 10% replacement are still not well-known.

2.9.2 Other Durability Properties

A study by Bouikni, Swamy and Bali (2009) denoted that moist curing is essential in slowing down the process of carbonation. The concrete specimens without any water curing suffered from most severe penetration of carbonation. A 7-day initial moist curing period and continuous curing significantly retarded the carbonation process.

Lynsdale and Sit (as cited in Darwish, 1995) reported that air curing substantially affects the air permeability of concrete and the effect is more pronounced for OPC/slag concrete than OPC concrete. Lawrence, (as cited in Darwish, 1995) stated that relative humidity and degree of curing of concrete are the most important parameters controlling gas permeability. Besides, it was discovered that the oxygen permeability increases with drying age for OPC and blended cement concretes.

Other than ISA, sorptivity, and water absorption, there is rather limited study on the effect of curing regime on other durability properties, such as chloride penetration, carbonation and sulfate attack, of mortars and concretes with GGBFS and MK. Therefore, it is not clear about the influence of different curing regimes on these durability properties on mortar and concrete with slag and metakaolin.

2.10 Effect of Incorporating Slag and MK on Durability of Mortar

There have been a variety of researches over the past decade on the utilization slag and metakaolin on the durability properties of concrete and mortar. The researchers confirm that partial replacement of these supplementary cementitious materials (SCMs), either as part of cement of fin aggregate is beneficial in improving the durability of concretes and mortars. This is attributed to the filling effect as well as the pozzolanic reaction of these SCMs with calcium hydroxide in cement paste to achieve better pore structure. Some of the properties improved are capillary sorptivity of water, chloride resistance, sulfate resistance and abrasion resistance.

2.10.1 Transport Properties and Permeability

Asbridge et al. (as cited in Khatib and Hibbert, 2011), suggested incorporating both MK and GGBS creates a more refined pore structure, promotes elimination of Portlandite and suppresses the chloride diffusion rate. The durability of concrete containing MK and GGBS is expected to improve (Khatib & Hibbert, 2004). The use of GGBFS diminishes the permeability of the mature concrete as the products from pozzolanic reaction fill pore spaces in the paste and result in a denser microstructure (Neveille, as cited in Meeks and Carino, 1999).

Al-Jabri et al. (2009) studied the effect of copper slag on ISA when substituted as sand in high performance concrete. The authors reported that among different level of replacement from 20% to 100%, concrete mix with 100% copper slag shows the highest initial surface absorption (ISA) as compared to other mixes. On the other hand, 20% and 40% replacement level exhibits similar and lowest absorption rate for entire test duration at 10, 30, 60 and 120 minutes. According to the authors, there is a general decrease in ISA with the increase of copper slag content up to 40% replacement level. Above that, the ISA rapidly increases. Substitution of 40% sand by copper slag obtained lowest ISA. In another research on effect of copper slag as fine aggregate on the properties of normal mortars and concrete by Al-Jabri, Al-Saidy and Taha (2010), the ISA decreases with increase in copper slag replacement as fine aggregates up to 40%. Mixture with 40% copper slag shoed the lowest ISA for the entire testing time. Beyond 40% replacement, the ISA increases with higher amount of replacement. Mixture with 100% copper slag replacement depicts the highest ISA.

Al-Jabri et al. also conducted another research on the utilization of copper slag as fine aggregate in high performance concrete (HPC). Copper slag was substituted as fine aggregates in HPC from 30% to 100% and initial surface absorption was determined at 28 days age. The water absorption was discovered to reduce slightly as copper slag content increased up to 50% of replacement level. Beyond that, ISA increased slightly as copper slag content was higher.

A study by Hadj-sadok et al. (2010) determined the water porosity using water absorption method and showed that presence of slag increase the total water porosity level at any age of testing (28, 90 and 360 days). The effect is more significant especially at 50% slag mortar, subsequently 30% slag mortar. Mortar without slag has the lowest porosity. The authors also reported that the pore size distribution of slag mortar is coarser than mortar without slag at relatively early age of 28 days. Nevertheless, the pore structure of slag mortars seems to evolve to smaller finer pores until comparable with reference mortar at the age of 90 days. Observation made at the age of 360 days reported that slag mortars are clearly finer than that of reference mortar, irrespective of a higher total volume of pore than that of reference mortar.

A number of researchers reported that incorporation of slag produces a apparently refined structure than OPC concrete, regardless the curing regime and the adverse effect of drying on pore structure at high replacement level (Bouikni, Swamy and Bali, 2009). The scanning electron microscopy illustrated that the concrete cured continuously in a wet environment developed a compact and dense structure with time and this is irrespective of slag replacement level. Conversely, when subjected to prolonged drying, presence of pores and cracks were invariably detected in concrete.

The inclusion of GGBFS in mortar reduces the capillary water absorption per unit area (Hadjsadok et al., 2010). The authors obtained a low capillary absorption for slag mortars at the age of 90 and 360 days as compared to reference mortars without slag. The presence of slag, particularly at 50% dosage, leads to lower values of sorptivity coefficient. At 90 days old, as a comparison to reference mortar, there is a decrease of sorptivity coefficient of 16% and 32% for mortars incorporated with 30% and 50% of slag correspondingly. The improvement of sorptivity characteristics can be explained by the more refined pore structure of slag mortars.

Hadjsadok et al. (2012) examined the effect of different GGBFS content on the sorptivity coefficient of concrete for two different w/c ratios of 0.65 and 0.42 at 28 and 90 days. The cement content was partially replaced by 15%, 30% and 50% GGBFS and a normal Portland cement mixture was used as reference. The increase of GGBFS content was found to increase the sorptivity coefficient of for concrete mixtures with w/c ratio of 0.65 at 28 and 90 days of curing. As for w/c ratio of 0.42, the sorptivity coefficient at the age 28 days was similar for all level of replacement. Meanwhile, concretes containing GGBFS at the age of 90 days showed a slight decrease in the sorptivity coefficient.

A study by Cakır and Aköz (2006) examined the effect of incorporating 30% and 60% of GGBFS on appearing porosity and water sorptivity of mortars cured in two different conditions. The authors reported that the porosity of mortars decreases with slag replacement compared to reference mortars. 60% of GGBFS results in lowest porosity. Besides, the sorptivity coefficients of slag mortars are lower compared to control mortar for both curing conditions applied. The filling effect and pozzalanic reaction of slag explain the results obtained. This connotes that replacement of slag improves the durability properties of mortar.

Elahi et al. (2009) studied the durability of high performance concrete (HPC) containing supplementary cementitious materials. The sorptivity of mixes with 50% and 70% GGBFS was tested at 44 and 91 days. The mix with 50% GGBFS gives lower sorptivity at 44 days as compared to that of 91 days. At higher replacement of

70%, the sorptivity at 44 days substantially increased and greater than that of control concrete, but considerably reduced at 91 days.

Yip et al., Bernal, and Buchwald (as cited in Bernal et al., 2012) observed that addition of GGBFS to metakaolin-based binder improves microstructure of concrete. In a study carried out by Bernal et al. (2012) revealed that inclusion of small quantity of MK in GGBFS rich alkali silicate binders enhances the performance of concretes on terms of permeability and durability. The concretes with incorporated with MK report a pore volume significant lower than the slag based-concretes formulated with similar silicate/aluminate (S/A) ratio. At 28days, the porosity is lower when 10% of MK is added than 20%MK. A reversed result was observed at greater age of 90 days.

Nilam and Tambekar (2005) investigated the effect of various supplementary cementitious materials on the initial surface absorption of concrete. The ISA of a mix containing 60% GGBFS as cement replacement has lower ISA which is 81% compared to control mix without slag. The addition of MK into the previous mix leads to further and drastic reduction of ISA. The mix with 60% GGBFS and 3% MK has an ISA which is only approximately 40% of that of control mix. The mix with 7.5% of MK replacement exhibits a very low ISA compared to control mix and is the lowest among all mixes.

Siddique and Kadri (2011) studied the effect of metakaolin on the near surface characteristics of concrete. Different replacement level of MK for cement from 5% to 15% was used. The initial water absorption (ISA) of specimens was tested at 7, 28 and 56 days after they had been subjected to initial water curing of 28 days. Therefore the testing age was 35, 56 and 84 days. The concretes containing MK generally exhibit lower ISA than control mix for all replacement levels at all ages. The ISA decreases with higher amount of MK incorporated. The lowest value of ISA was obtained at 15% replacement level of MK. Furthermore, it was observed that the ISA of concrete, either with or without MK, drops when tested at greater age.

According to Siddque and Kadri (2011), partial replacement of cement by MK generally leads to lower sorptivity of concrete compared to the control concrete. As the dosage of MK increases from 5% to 15%, the sorptivity of concrete decreases till 10% replacement. However, an increase in sorptivity occurs at 15% MK replacement. The authors derived that even though higher replacement level of MK at 15% improve the surface flow characteristics of concrete, it is not helpful in improving the inner durability of concrete. This result is in agreement with the research by Ramezanianpour and Jovein (2012) who reported incorporating 10% MK offers the most substantial improvement on sorptivity when compared to other replacement levels, irrespective of w/c ratio and testing age. Increasing MK replacement level from 10% to 15% was found to adversely affect the inner permeability.

Water absorption by total immersion was conducted by the authors to evaluate the effect of MK on porosity. The results showed that the water absorption varied between 7.3% and 8.2% at all ages. All mixtures were noticed to have low absorption characteristic (less than 10%). In comparison to control concrete, there is an increase of 2.7% to 4.2% in water absorption at 15% of MK replacement level. This again supports that higher MK replacement of 15% is not helpful in improving inner core durability characteristics. Incorporation of MK up to 15% is determined to have slight effect on porosity.

Courard et al. tested mortars incorporating metakaolin at the age of 28 days and 14 months for water absorption by total immersion and water capillary absorption. Generally, the water absorption was found to increase with increasing amount of MK added. This indicates the pores volume of mortar increases with when more MK. The water capillary absorption was measured at 3, 6, 9 and 72 hours or 144 hours during the test. MK mortar with MK replacement level from to 5% to 20%, determined at the age of 28 days showed relatively insignificant variation in water capillary absorption up to 72 hours. However, when tested at the age of 14 months, the water capillary absorption of MK mortars generally shows a decreasing trend with increasing amount of MK at the early stage of test up to 72 hours. When the capillary absorption was registered after 144 hour, with an exception for 20% MK replacement, mortars with 5% to 15% of replacement had a higher capillary absorption value than reference mortar.

Goncalves et al. (2009) study the durability performance of mortar incorporated with two different types of MK and ground calcined clay brick. The authors reported that replacement of cement by MK leads to reduction of porosity and more refined pore structure while a reverse was determined for ground brick. The optimum replacement level of MK used is dependent mainly on its purity and chemical composition. Inclusion of MK reduces the proportion of macropores and increases the proportion of mesopores. The study also depicted that replacement of cement by MK generally leads to lower values of sorptivity compared to reference mortar. This improvement is associated to the effect of combined packing density, reduced porosity and refinement of the porous structure of probably unconnected pores, which obstruct the flow of water. During the initial period of sorptivity test (≤2.5 hours), there is a general reduction in sorptivity for different MK replacement level. However, the sorptivity was found to increase in the case for 30% and 40% replacement of lab-produced MK.

Vejmelkaova et al. (2010) investigated the sorptivity coefficient of self-compacting mixture (SCC) with metakaolin and blast furnace slag. One of mixture used blended cement containing 56% of blast furnace slag by weight as binder while another mixture’s binder was made up of 60% of Portland cement and 5% MK. It was reported the water sorptivity coefficient for MK mixture is very low and 40% lower than that of slag mixture. This is a consequence of a more refine prore structure with numerous of pores small than 50nm and very low amount of capillary pores in MK mix.

Güneyisi et al. (2012) studied the sorptivity coefficients of concrete at 5% and 15% cement replacement by MK at w/c ratio 0.25 and 0.35. The sorptivity coefficients determined at 28-day age revealed that MK concrete is better than the control concrete and there are notable improvements in terms of lower rate of water penetration via capillary suction. The sorptivity drops when higher replacement of MK is used. An explanation for this is the refinement of pore through filling and secondary hydration reaction of the mineral admixtures, thus results in lower capillarity.

Khatib and Clay (2003) reported that partial replacement of MK as cement reduces the water sorptivity coefficient of mortar. Different replacement level of MK from 0% to 20% was used. The sorptivity of concrete was reported to decrease when the percentage of MK in the mix increases and decrease as the curing time is longer. It was observed that the sorptivity coefficient of all MK mixes drop with the increase in curing time up to 28 days and an increase in sorptivity coefficient occurs beyond 28 days. Nonetheless, the sorptivity coefficient is still lower for mixes containing MK regardless this increase. It was suggested that partial replacement of cement by MK causes discontinuity of pores thus produces a more refine pore structure.

Joorabchian (as cited in Mikhailenko, 2012) performed sorptivity test on concrete with MK at 15% and 20% replacement level. The test conducted at the age of 28 days indicates lower sorptivity for concrete containing MK compared to control mix. The addition of MK as partial replacement to cement notably reduces the primary sorptivity (absorption) of concrete.

Madandoust & Mousavi (2012) reported that the initial and final water sorptivity of SCC decreases with the inclusion of MK, regardless of W/B ratio. The beneficial effect of MK on the reduction of water sorptivity is more notable at lower w/b ratio. For different replacement level from 5% to 20%, the water sorptivity decreases with higher proportion of MK at w/b ratio of 0.32. On the other hand, replacement level of 5% and 20% at w/b ratio of 0.38 and 0.45 respectively was determined to give the lowest initial water sorptivity. Whilst for final sorptivity, 20% MK replacement gives lowest final water sorptivity for both w/b ratio 0.38 and 0.45. The authors suggested more notable beneficial effect of MK at lower w/b ratio is due to a lower total porosity and finer pore-size distribution of MK blended matrix. A similar result was reported by Shekarchi et al. (2010). The Portland cement was substituted by MK at different dosage up to 15% and a w/b ratio of 0.38 was used. Tested at 28 days old, it was depicted concretes with MK have lower water absorption than reference concrete. The water absorption is lower with higher dosage of MK. The water absorption is 30% lower for 15% MK replacement level, compared to reference mortar.

Karahan et al. (2012) studied the effect of MK on sorptivity of self-consolidating lightweight concrete (SCLC). It was reported that the initial and secondary sorptivity of SCLC tested at 28 days decreases slightly with increasing MK content. Addition of MK causes a reduction on sorptivity compared to control mix. In addition, the authors investigated the effect of MK on water absorption of SCLC. It was observed that the water absorption plunge slightly at greater MK content.

Tsivilis and Badogiannis (2008) utilized two type of MK: commercial MK and MK obtained from thermal treatment to poor Greek kaolin. The effect of both types of MK as partial replacement for cement or sand at 10% and 20% was evaluated. It was reported that both types of MK, when incorporated as sand or cement replacement, causes a relative decrease of concrete sorptivity compared to control mix. Concrete with commercial MK performed the best than Greek MK concrete. With commercial MK as 20% replacement of sand gives the lowest sorptivity.

Other than that, Khatib and Clay (2003) tested the water absorption by total immersion on MK concrete. The water absorption is higher for greater replacement level of MK. A decline in water absorption with curing period was obtained for all MK mixes up to 14 days. This was followed by slight increase in water absorption between 14 and 28 days of curing. The change in water absorption is relatively low beyond the age of 28 days. The increase was possibly attributed to the change in phase composition during this period. This result is in agreement with Joorabchian’s study (as cited in Mikhailenko, 2012) which showed that addition of MK increases the porosity of concrete.

A similar test was conducted by Güneyisi and Gesoğlu (2007) with 10% and 20% MK as replacement of cement at w/c ratio 0.35 and 0.55. Water absorption by total immersion was carried out at 28, 90 and 120 days. The authors reported that water absorption characteristics of concrete decreases with increasing MK replacement, regardless of w/c ratio and testing age. The difference in water absorption became more adverse at later ages and significantly lower for MK concrete than plain concrete.

2.10.2 Other Durability Properties

Hogan & Meusel, Osborne, and Mehta (as cited in Khatib and Hibbert., 2011) reported that partial replacement of Portland cement with GGBFS generally improves the sulphate resistance. However, the effectiveness relies on the properties such as chemical composition and fineness. A study conducted by Nabil (2006) reported that utilization of metakaolin as partial replacement of cement was effective in improving the resistance of concrete to sulfate attack. An increase in MK replacement level increases the sulfate resistance of MK concrete. Concrete with 10% and 15% MK replacement exhibited excellent duability to sulfate attack.

Chen et al. (2012) studied the effect of high blast furnace slag cement (HBFS) on chloride resistance of concrete. It is reported except the outermost zone from 0 to 15mm, the chloride concentration determined at 90, 180 and 360 days after curing is much lower than that for OPC concrete. The improve chloride resistance is a consequence of reduced amount if inter-connected pores by pozzolanic reaction of HBFS. (Hassan et al. (2012) reported that partial replacement of cement by MK substantially reduces the chloride permeability of self-compacting concrete. The chloride permeability was cut down by 89% at 20% of MK replacement level, compared to control mixture. In another experiment by Kim et al. (2006) reported that chloride permeability of concrete decreases as replacement ratio and curing time increases.

Bilim and Atis (2011) studied the carbonation of mortars containing different replacement of GGBFS in alkali activated condition. The results imply that with an exception at 7 days, the carbonation depth of mortars containing slag is higher than that of control mortar at longer term ages. Kim et al. (2006) indicated that partial replacement of cement by MK accelerates the carbonation process. The carbonation depth of MK concrete is larger than the control mix at any age of the concrete. Higher replacement level of MK results in greater carbonation depth. However, this result is in contrast with the research by Sharif (2005). The author reported that the presence of metakaolin reduces the carbonation depth. Best resistance to carbonation was achieved by 20% and 15% replacement of cement by MK for binder content of 300kg/m^3 and 400kg/m^3 respectively.

2.11 Effect of Slag and MK on Workability

According to American Concrete Institute (as cited in Meeks & Carino, 1999), use of GGBFS enhances the workability of fresh concrete. It is believed that the smooth and dense surface of slag particles lead to low water absorption process thus decrease the water demand for the mix. Besides, cement pastes with GGBFS achieves better particle dispersion and higher fluidity, result in improved workability.

Courard et al. (2003) reported that the consistency of concrete with metakaolin decreases as compared to cement mortar reference. The decrease of consistency is higher when a higher amount of metakaolin is added into concrete mixture.

Madandoust & Mousavi (2012) observed that higher dosage of high-range water reducer is required to maintain the slump flow of self-compacting concrete (SCC) at a given w/b ratio when the MK replacement proportion is higher. The flowability of the mixtures was determined to be reduced with higher proportion of MK replacement. This could be explained by higher surface area of the MK particles compared to Portland cement.

The incorporation of MK results in decrease of workability is found to be consistent among many researchers. Wild et al. reported that the slump of concrete decreases as higher dosage of MK is included, indicating there is a decrease in workability. Li and Ding reported that the fluidity of MK-blended cement got poorer than that of Portland cement, provided the dosage of superplasticizer and w/b ratio are identical. The authors concluded that MK-blended cements demand substantially greater amount of water than relatively pure cement (as cited in Siddique & Klaus, 2009).

Li and Ding (2003) reported partial substitution of MK as cement leads to lower fluidity at the same superplasticizer dosage. After the MK mix was modified by adding ultra-fine slag, the fluidity of the mix increased.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter describes the materials used for this project work, the mixing methods and the experimental procedures for various test programs. The effects of curing regimes as well as addition of slag and metakaolin on durability properties of mortars incorporating slag and metakaolin are the major scope of study. First, raw materials and their preparations are presented followed by mix proportion design. After that, the mixing procedures and test procedures for mortars are described in details.

3.2 Materials Used

The raw materials used in this project work include Ordinary Portland Cement (OPC), sand, water, superplasticizer (SP), ground granulated blast furnace slag (GGBFS) and metakaolin (MK).

3.2.1 Ordinary Portland Cement

“Orang Kuat” ordinary Portland cement (OPC) branded from YTL Cement Sdn. Bhd was utilized throughout the project work. This OPC fulfilled the requirements for

Type I Portland Cement in accordance with ASTM C150 (2003). The chemical composition of OPC was depicted in Table 3.1. The OPC was sieved through 600 µm sieve and kept in an air-tight container to avoid it from contacting air moisture. Air moisture would hydrate cement particles and affect the formation of calcium silicate hydrate gel.

Table 3.1: Chemical composition of OPC, GGBFS and Metakaolin

	Percentage (%)		
Chemical Composition	**OPC**	**GGBFS**	**Metakaolin**
Silicon dioxiode (SiO_2)	20.99	38.25	57.40
Aluminum oxide (Al_2O_3)	6.19	7.56	35.26
Calcium oxide (CaO)	65.96	42.89	0.02
Ferric oxide (Fe_2O_3)	3.86	0.68	0.94
Magnesium oxide (MgO)	0.20	6.82	0.18
Sodium oxide (Na_2O)	0.17	0.99	<0.01
Potassium oxide (K_2O)	0.60	0.38	3.17
Phosphorus Oxide (P_2O_5)	0.05	0.01	0.09
Titanium Oxide (TiO_2)	0.40	0.52	0.43
Manganese oxide (MnO)	0.06	0.72	<0.01
Loss of ignition , L.O.I.	1.52	1.18	2.51

3.2.2 Sand

In this project work, only fine sand was used to produce mortars. The sand was first air dried to remove the moisture. The duration of drying is dependent on the surrounding temperature and humidity. After that, the dried sand was sieved through 600 µm sieve. The sand that passed through 600 µm sieve was again sieved through 300 µm sieve. The sand which retained on and passed 300 µm has particle size from 300 µm to 600 µm and less than 300 µm respectively. The sand with particle size between 300 µm and 600 µm composed 90% of the fine aggregates (by weight) used for production of mortars, while another remaining 10% was made up of sand with particle size less than 300 µm.

(a) (b)

Figure 3.1: Sand with (a) Particle Size Between 300 to 600 µm; (b) Particle Size Less Than 300 µm

3.2.3 Water

The water used to produce mortar should not contain any deleterious substance such as chloride and sulfate as the presence of these substances can be harmful to hydration process of cement and durability of mortar. In this project work, conventional tap water was used to cast mortars.

3.2.4 Superplaticizer

Superplasticizer (SP) was added in small amount to improve the workability and flowability of fresh mortar. The SP used was DARACEM 19 supplied by Grace Construction Products. This SP belongs to the family of sulfonated naphthalene-formaldehyde condensates (SNF). Before it was used, the SP was shook vigorously to ensure uniform dispersion of solid content throughout the solution. When not used, the SP must be kept in the container to avoid exposure to the atmosphere.

3.2.5 Ground Granulated Blast Furnace Slag (GGBFS)

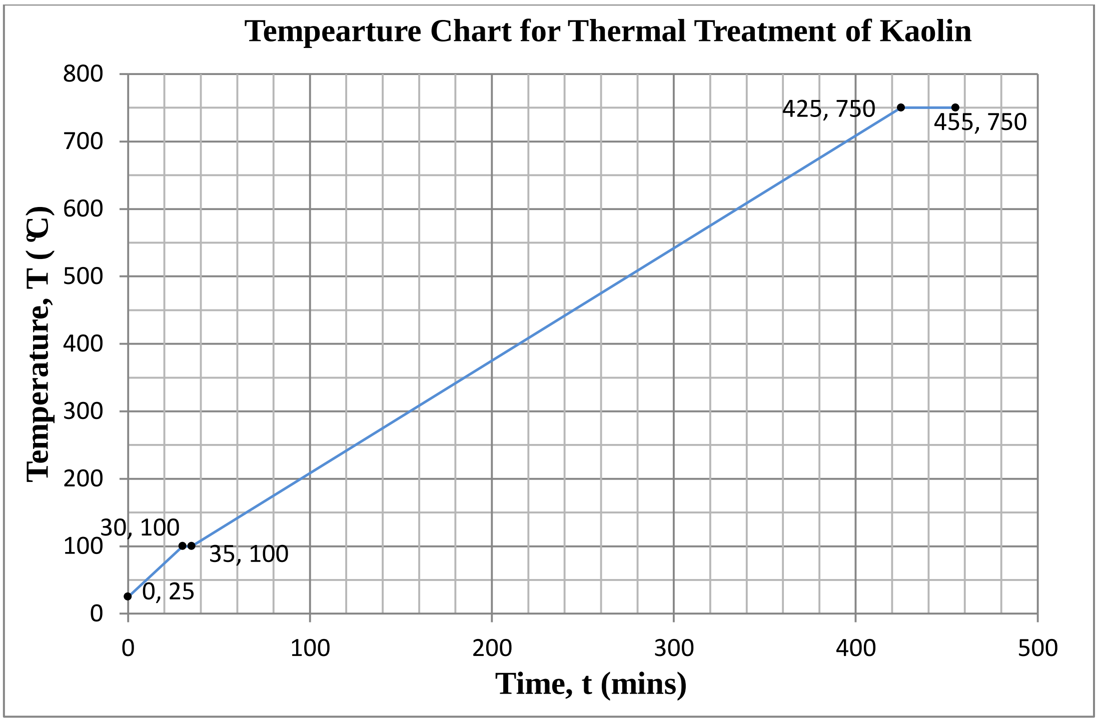
As mentioned previously, GGBFS is produced as by product in blast-furnace during manufacturing of iron. It is a non-metallic product that consists of mainly silicates and aluminates of calcium bases. Sulphate resistance cement, or also known as high slag blast furnace cement was used in this study. The chemical composition of GGBFS used was presented in Table 3.1.

(a) **(b)**

Figure 3.2 (a) and (b): Ground Granulated Blast-Furnace Slag (GGBFS)

3.2.6 Metakaolin

The metakaolin (MK) used in this project was produced through thermal treatment on raw kaolin. The kaolin was supplied by Kaolin (Malaysia) Sdn. Bhd. Thermal treatment on kaolin transforms raw kaolin into MK and the process is known as calcination. As mentioned before, when heated to specific temperature range, the chemically bonded water of raw kaolin is lost via dehydroxylation and the crystalline structure is destroyed, producing metakaolin which is a reactive pozzolanic material. The temperature regime for the thermal treatment was set as followed:



The chemical composition of MK is listed in Table 3.1.

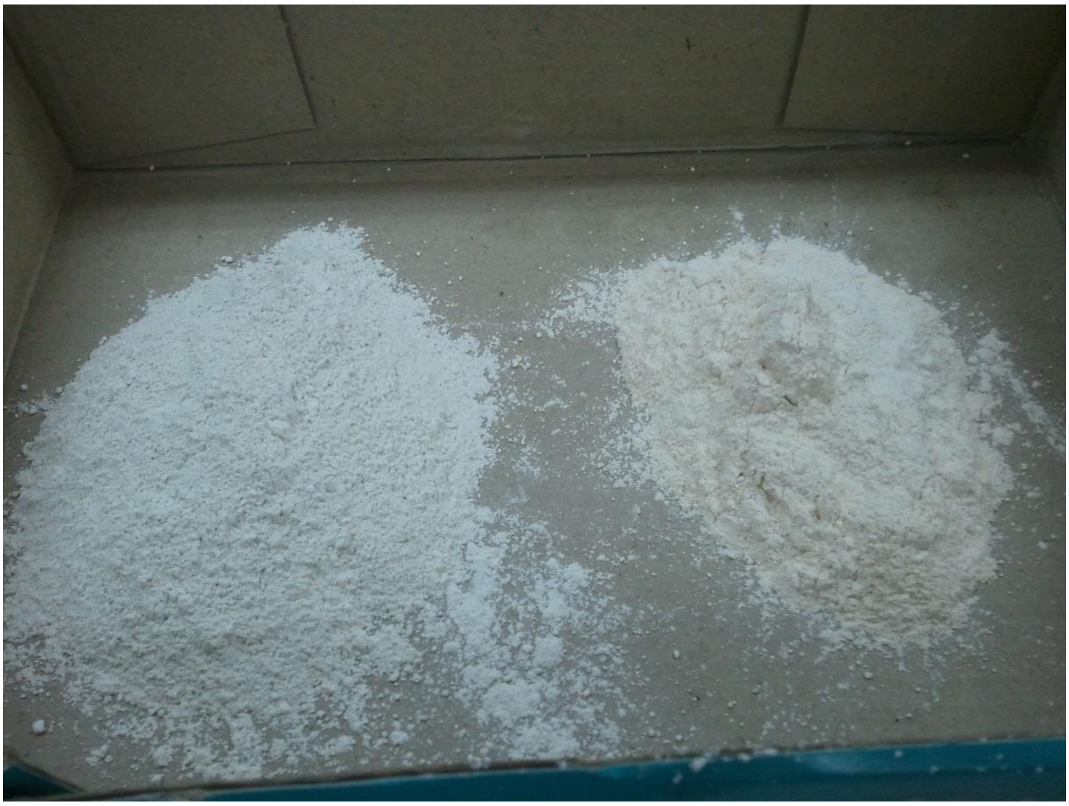


Figure 3.3: The Raw Kaolin (Left) and Metakaolin (Right) after Thermal Treatment

3.3 Mixture Proportioning and Trial Mix

A series of concrete with water ratio of 0.45 was designed. The series consisted of four different mixtures including one control mixture which contained OPC as binder only. The other three mixtures had 5% GGBFS, 5% metakaolin, and 2.5% slag + 2.5% metakaolin to partially replace cement by weight. All the mixture proportions ware designed based on Sheerbooke Mix Design Method. However, some necessary modifications were made to this design method so that it was appropriate to design mix proportions of mortar. The mortar did not contain any coarse aggregate thus any calculation associated with coarse aggregate in the original mix design method was eliminated.

The appropriate mix proportion for all the specimens to be cast was obtained by trial and error method on water to binder (w/b) ratio and superplasticizer dosage. The ratio of cement to fine aggregate was fixed to be 1:2. The objectives of trial mix were to determine the suitable w/b ratio to produce workable fresh mortar mix besides to ensure that the SP dosage was not excessive. The initial w/b ratio was set to be 0.45 while the superplasticizer dosage was set to be 1.5%. DARACEM, which was the superplasticizer employed in this project is known to have retardant effect, would delay the setting time of mortar mix.

The trial mix was considered as fail if it produced a workable mix but the setting time for the mix was too long. Besides, a mix proportion was rejected if it produced a mix which too workable, that is, highly flowable and possessed of excessive degree of fluidity. A mix proportion which led to a very sticky mix with low workability would be rejected as well. A successful trial mix was a mix which was workable but the setting time is not excessively long, say, within 24 hours. The w/b ratio and SP dosage of the successful mix was used in all subsequent mix preparation of this project work.

The final w/c ratio and SP dosage to be used for the castings in this project work were determined to be 0.45 and 1.1% respectively. presents the mixture proportions of mortars in this project work.

Table 3.2: Proportions of All Mixtures

Mixture	Cement (kg/m^3)	GGBFS (kg/m^3)	MK (kg/m^3)	Water (kg/m^3)	Sand (kg/m^3)	SP (l/m^3)
w/c=0.45						
Control (CTR)	400	0	0	180	800	19
S5	380	20	0	180	800	19
M5	380	0	20	180	800	19
MS2.5	380	10	10	180	800	19

3.4 Mix Proportions Design

This section discussed about the method and associated procedures in determining and calculating the proportion of each of the ingredients used to produce the mortar. The mix design method used was based on Sherbrooke method.

3.4.1 Mix Design Sheet and Procedures

The quantity of each raw materials required for the mixture was calculated using Sherbrooke Method. The calculation was performed using a mix-design sheet as shown in
Table 3.3. The mix design sheet was created as a spread sheet using Microsoft Excel to ease the calculation for different batches of mix. The maximum amount of each batch of mortar can be produced was limited to $0.01m^3$ to minimize the deviation of designed properties from desired state.

The mix design sheet was divided into two parts. In the upper part (Part A), the characteristics of all ingredients that will be used for the mix were reported. This part of the mix design sheet must be completed before any proceeding calculations are made as these data are important for calculations followed. If some of the properties needed to perform calculation are unknown, it is necessary to assume reasonable values for them, founded on the best information available.

The following symbols and abbreviations will be applied:

G_c	Specific gravity of cement of cementitious material
G_{SSD}	aggregate specific gravity in saturated surface dry condition
w_{abs}	absorbed water in the aggregate in per cent
w_{tot}	total water content of the aggregate in per cent
w_c	moisture content of the aggregate in per cent
G_{sup}	specific gravity of the liquid superplasticizer
s	total solid content of the superplasticizer in per cent
M_{sol}	mass of solids in superplasticzer
d	superplasticizer dosage as a percentage of mass of solids in comparison to the total mass of cementitious material
V_{liq}	volume of liquid superplasticizer
V_w	volume of water in the liquid superplasticizer
V_{sol}	volume of solids in the liquid superplastcizer
W	mass of water in kg per cubic metre of concrete
CM	mass of cementitious materials in kg per cubic metre
M_f	mass of fine aggregate

Correction had to be made to the water dosage in order to take into water contained in the superplasticizer. The related expressions ere depicted in a sepearte section at the bottom of Part A, with spaces to be filled in after calculation in Part B. The moisture content of fine aggregates was also included for water correction. Besides, any calculation relevant to course aggregate was ignored as mortar contains no course aggregate.

The lower part (Part B) of the mix design sheet was a form a table in which all boxes were numbered in accordance to their sequence to be filled in. The table were divided into six columns. In the first column, initial data and calculations were reported. The volume of each ingredient was calculated in column 2. In column 3, the SSD proportions of the mix were computed. In column 4, different water corrections necessary to be made were calculated. The proportions of the mix using the actual raw materials were presented. Finally, the proportions of trial batch can be calculated in column 6.

Table 3.3: Mix Design Sheet Using Sherbrooke Method

Mixture:	5% Metakaolin	Date	

Part A : Properties of Materials

	G_c	%
OPC		
Slag		
Metakaolin		

Aggregate	G_{SSD}	W_{tot} (%)	W_{abs} (%)	W_c (%)	
Coarse					$w_c = w_{tot} - w_{abs}$ (3.1)
Fine					
Superplasticizer:	DARACEM				

G_{sup}	Solid content, s (%)	M_{sol} (kg/m^3)	V_{liq} (l/m^3)	V_w (l/m^3)	V_{sol} (l/m^3)
		16	23	20	13

Part B: Design

W/CM		1
Total cementitious, CM (kg/m^3)		2
Sand to Cement Ratio		3
Superplasticizer dosage, d (%)		4
Volume of concrete batch, (m^3)		5

	1	2	3	4	5	6
Material	Content (kg/m^3)	Volume (l/m^3)	SSD condition (kg/m^3)	Water correction (l/m^3)	Composition (kg/m^3)	
					per 1m^3	Trial Batch
Water	6	6	6		22	24
Cement	7.1	10.1	7.1		7.1	25.1
Slag	7.2	10.2	7.2		7.2	25.2
Metakaolin	7.3	10.3	7.3		7.3	25.3
Coarse aggregate						
Fine aggregate	8	11	15	19	18	26
Air	9	12				
Superplasticizer	4	13	16	20	23	27
Total		14	17	21		28

* The highlighted boxes are required to be filled in by the user

* The box numbers are labelled in the red box containing number in red-colour font

The mix design calculations were as followed and a sample calculation is shown in Appendix A:

Box 1	The water/cement ratio was written down.
Box 2	The total cementitious material required per cubic metre was written down.
Box 3	The designed sand to cement ratio was written down.
Box 4	The amount of superplasticizer needed was recorded.
Box 5	The volume of batch to be produced was entered.
Box 6	Determine the amount of water required by multiplying water/cement ratio by total cementitious material per cubic metre (box 2).
Box 7.1, 7.2, 7.3	Calculate the mass of each of the cementitious materials according to the cementitious composition selected appearing in Part A on the mix design sheet and report it in columns 1, 3, and 5 where these boxes are found.
Box 8	The mass of fine aggregate is calculated by multiplying the total cementitous by the sand to cement ratio.
Box 9	Record the assumed air content.
Box 10	The volumes of different cementitious materials are calculated by dividing their masses, as shown in boxes 6.1, 6.2 and 6.3, by their respective specific gravities that have been reported in Part A of the mix design sheet.
Box 11	The volume of the fine aggregate in l/m^3 is obtained by dividing the mass of aggregate per cubic metre appearing in box 8 by its SSD specific gravity.
Box 12	The volume of entrapped air, in l/m^3 is calculated by multiplying the air content (box 9) with 10.
Box 13	Calculate the volume of solids contained in the superplasticizer, V_{sol} using the formula: $V_{sol} = V_{liq} - V_w = V_{liq} = \left[\left(1 - \frac{100-s}{100}\right) \times G_{sup}\right]$ (3.2)
Box 14	The total mix volume (1000 l/m^3) appears here.

Box 15	The mass of fine aggregate is calculated by multiplying its volume appeared in box 11 by its SSD specific gravity.
Box 16	The mass of solid in the superplasticizer, M_{sol} is calculated: $M_{sol} = CM \times \frac{d}{100}$ (3.3)
Box 17	All the masses appearing in column 3 are summed here to give the unit mass of the concrete.
Box 18	The mass of the fine aggregate, M_f was calculated: $M_f = M\ (in\ box15) \times \left(1 + \frac{w_c}{100}\right)$ (3.4)
Box 19	Subtract the value in box 18 from that in box 15 and enter the result here.
Box 20	Calculate the amount of water brought to the mix by the superplasticizer using the formula: $V_w = V_{liq} \times G_{sup} \times \left(\frac{100-s}{100}\right)$ (3.5) A negative sign is assigned to the value in the box in Part B.
Box 21	Add algebraically all the water corrections.
Box 22	Add (taking note of the sign) the water correction appearing in box 21 to volume of water appearing in box 6.
Box 23	Calculate the superplasticizer dosage, V_{liq} using the formula: $V_{liq} = \frac{M_{sol}}{s \times G_{sup}} \times 100$ (3.6)
Boxes 24 to 27	These are calculated by the values in the adjacent box in column 5 by the desired volume of trail batch appeared in box 5.
Box 28	The mass of trial batch is calculated by summing the masses of all different ingredients of mortar appear in boxes 24 to 27. To check the calculation, the value in box 17 is multiplied by the volume of concrete batch as appearing in box 5.

3.5 Batching, Mixing and Casting

The calculated amount of materials required-sand, cement and water, were weighted. Then, the volume of SP needed is measured using a cylinder and subsequently mixed thoroughly with water. Next, the cement and sand were placed in a mixing pot and the mixed using a mixing pot for about 90 seconds. After that, the fixer was stopped and the SP-containing water was added into the dry mix. Finally, the mix was blended thoroughly by the mixer for about 90 seconds. Ultimately, the mortar mix was poured into the moulds.

The 100 x 100 x 100mm cube samples were cast for initial surface absorption test (ISAT) while 50 x 50 x 50mm cubes were cast for water absorption test. Meanwhile, cylinders with 100mm diameter and height ranged from 40 to 70mm (approximately 1/3 of the full height of a 200mm cylinder mould) were casted for sorptivity test.

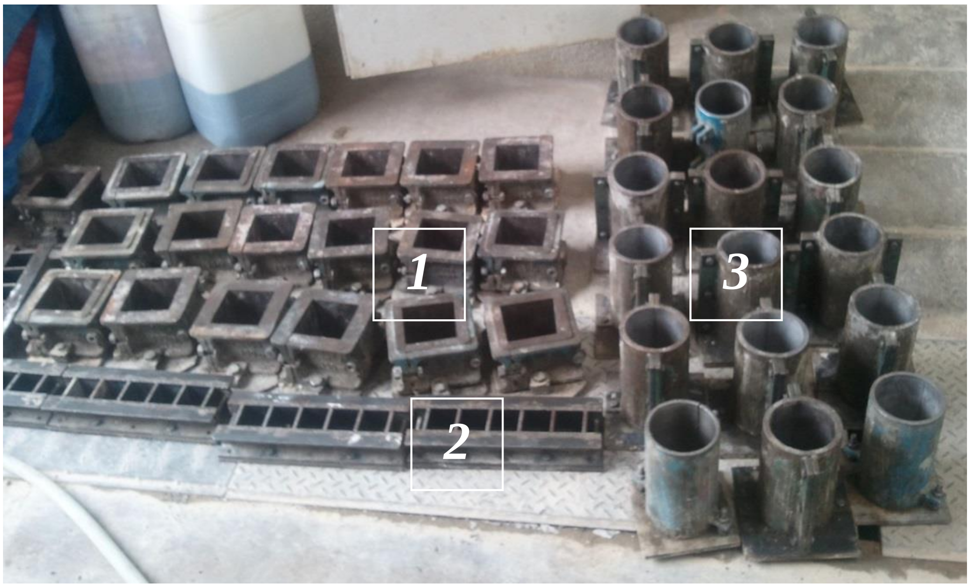
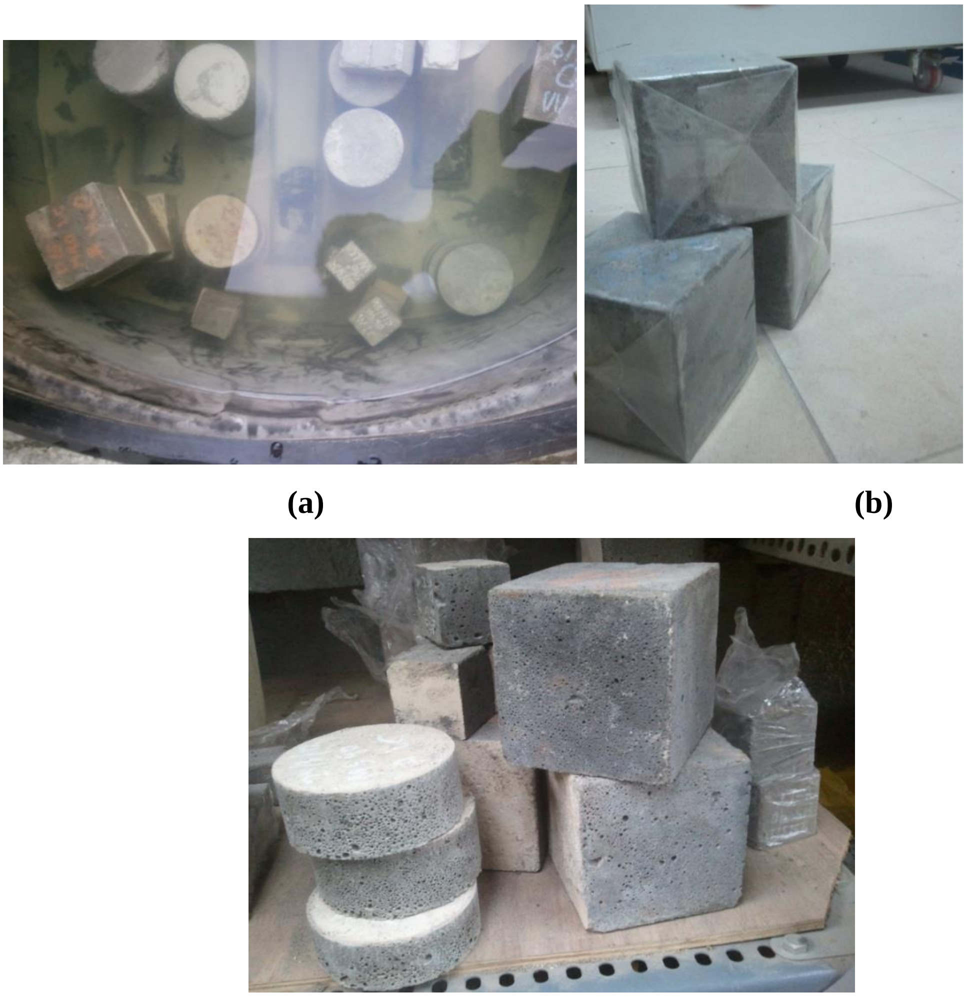


Figure 3.4: Moulds used to cast mortar specimens: (1) 100mm cube moulds; (2) 50mm cube moulds; and (3) 100mm x 200mm cylinder moulds

The fresh mortars were allowed to set and harden in moulds before they were demoulded after about 24 hours. The demoulded specimens were subsequently labelled and placed into their respective curing regimes as soon as they were demoulded.

3.6 Curing

The effect of curing regimes on the durability of mortar was investigated in this study. Therefore, different specimens were cured under three different curing regimes-water curing, plastic curing and air curing. For water curing, the demoulded samples were immersed in water tank with relatively steady temperature kept at 5 ± 2 ℃. The specimens were wrapped with plastic material and all openings were seal to ensure airtight condition for plastic curing. Meanwhile, the air cured specimens were left inside the laboratory with ambient temperature of 27 ± 3 ℃ after they had been demoulded. The specimens of each curing regimes were cured for 7, 28, 56, 90 and 180 days respectively before they were taken out for any test programmes.



(a) **(b)**

(c)

Figure 3.5: Curing of Specimens Under (a) Water Curing; (b) Plastic Curing and (c) Air Curing

3.7 Mortar Testing Programmes

Other than the durability, fresh mortar mix was tested to determine the effect of two mineral admixtures-slag and metakolin on the workability of fresh mortar. The test method employed tested the flow of cement mortar and this complied with standard ASTM C 1437.

There are three testing programmes adopted in this project work to assess the durability of mortar:

1. Initial Surface Absorption Test (ISAT)
2. Sorptivity Test
3. Water Absorption Test

3.7.1 Workability Test for Mortar

The method used to test the workability of fresh mortar originated from ASTM Standard C 1437-Standard Test Method for Flow of Hydraulic Cement Mortar. The objective of this test method is to determine the flow of hydraulic cement mortar, which can be adopted as an indication for the workability. Generally, the greater the ability of cement mortar to flow, the better the workability it is.

First, the flow table was wiped clean and dry, and the flow mould was located at the centre. A layer of mortar of about 25mm in thickness was placed in the mould and was tamped 20 times with the tamper. The tamping pressure shall be just sufficient to ensure uniform filling of the mould. Then, the mould was filled with mortar and was tamped as specified for the first layer. The mortar was cut off to a plane surface flush with the top of mould by drawing the straightedge of the edge of trowel with a sawing motion across the top of the mould. The table top was wiped clean and dry, being especially careful to remove any water from around the edge of flow mould.

The mould was lifted away from the mortar 1 minute after finishing the mixing operation. Immediately drop the table 25 times in 15 seconds, unless otherwise specified. Using a ruler, the diameter of the mortar along the four line scribed in the table top was measured and recorded to the nearest millimetre. The flow was the resulting increase in average base diameter of mortar mass, expressed as a percentage of the original base diameter. The flow in per cent was calculated using the equation below and reported to nearest 1%:

$$Flow = \frac{A}{D_{base}} \times 100\% \qquad (3.7)$$

where

A=average of four readings in mm, minus the original inside base diameter in millimetres.

D_{base}=Original inside base diameter of the flow mould in mm

Figure 3.6: Flow Table Test

3.7.2 Initial Surface Absorption Test (ISAT)

The ISAT was conducted as prescribed in BS 1881: Part 5: 1970. The test for initial surface absorption of concrete is a simple test to obtain data, which indicates the flow of water into a dry, flat concrete surface. The test is time dependent and comparative to indicate the quality of concrete to resist absorption of water. The principle of this test is to determine the time taken for a quantity of water to flow through a calibrated glass tube onto a fixed area of the concrete surface. The limitation of this test is the viscosity of water may affect the rate of flow via the tube when considering very porous concrete, result in high flow rates.

Prior to testing, specimens were oven dried at 105 ℃ for 72 hours and allowed to cool in a desiccator for 1 day. Although the drying method may be questioned for its possible adverse effect on the microstructure of concrete, this method has been used for a certain degree of success. The valve connected to the test cap was opend and filled up with distilled water or de-ionized water. It is essential that the water level in the reservoir should be maintained at 200mm from the top surface of concrete. The measurement was recorded at intervals of 10 minutes, 30 minutes, 1 hour and 2 hours after the beginning of the test. The time in units of seconds for the meniscus to travel 86 divisions was recorded. After each interval, the reservoir valve was opened and the water level was maintained at 200mm above the concrete.

The division on the scale are marked to indicate the rate of flow of water into the concrete at the rate of $ml/m^2.s$ based on the test duration of 1minute. Therefore, the data, obtained for each interval namely, 10 minutes, 30 minutes, 60 minutes, 120 minutes from the start of the experiment is calculated as equation below:

$$\mathrm{ISA} = \frac{60}{\mathrm{t}} \times \mathrm{div} \times 0.01 \tag{3.8}$$

where,

t=time period in seconds (sec)

div=number of scale divisions during period of t

ISA=flow in millimetre/meter^3/second ($ml/m^2/sec$)

Figure 3.7: Initial Surface Absorption Test (ISAT) Setup

3.7.3 Sorptivity Test

Concrete comprises aggregates filled with cement paste containing micropores in the size range of 10 to 10000 A. The fineness of the capillary pores causes absorption of water into concrete by capillary attraction, hence a measure of the rate of absorption provides a useful indication on the pore structure of concrete. If water is absorbed rapidly, it indicates that the capillary size in the concrete is large; if the absorption is slow, the capillaries are small.

The sorptivity test is considered to be a useful method in assessing the durability of concrete in relative to the porosity of concrete. In the sorptivity test, the gravitational and capillary force are in opposite direction; while for the ISAT, the water permeates from the top of the specimen, thus gravity and capillary forces are in the same direction. Thus, sorptivity test is a more suitable test in evaluating the pore structure.

Prior to testing, the specimens were oven dried for 73hours and allowed to cool in a desiccators for further 24 hours. The diameter and weight of the oven dried specimens were recorded to the nearest 0.01mm and 0.01g respectively. The cast surface that is the bottom of the specimen were immersed in a tray of water at 20 ℃ to a maximum depth of 1-2mm by resting on steel rods to permit free water movement. The uptake of water by capillary absorption was measured through mass of the specimen at interval of 5, 10, 15, 30, 60, 90, 120, and 150 minutes from the start of the test. Before weighing, the bottom surface in contact with water was wiped with paper towel to remove any excess water. After weighing, the specimen was returned to the tray immediately until the end of the experiment. The average sorptivity of three specimens was determined.

The water absorption per unit area at each time interval was calculated according to the equation below:

$$a_s(t) = \frac{a_t - a_d}{N} \times 10^3 mm^3/mm^2 \qquad (3.9)$$

where

a_t=weight at time, t (g)

a_d=weight of the oven dried specimen (g)

N=cross sectional area of bottom surface (g)

The data $a_s(t)$, was plotted versus the square root of time ($\sqrt{t}$). The sorptivity coefficient is taken as the slope of $w_s(t)$ versus $\sqrt{t}$ which might not necessary pass through the origin. The relationship between $w_s(t)$ and t is summarized in the equation below:

$$a_s(t) = Sor\sqrt{t} \qquad (3.10)$$

where

$a_s(t)$=increase in mass (g/mm^2) or expressed in mmas 1g or water is equivalent to 1cm^3

Sor= sorptivity coefficient (g.min$^{0.5}$/mm^2)

t=time measured in minutes at which the mass is determined (min)

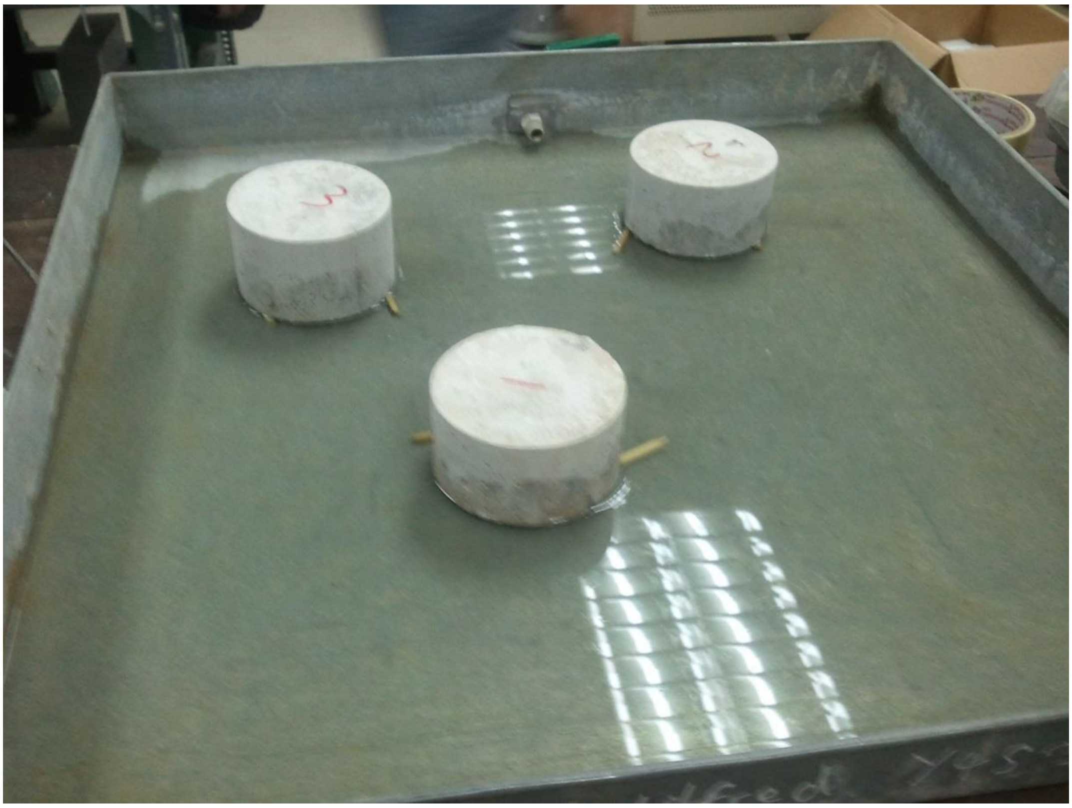


Figure 3.8: Sorptivity Test

3.7.4 Water Absorption Test

It is essential to know that the “effective porosity” of concrete structure, since water will penetrate into the concrete first by capillary suction and then ingress into disconnected pores and air voids. Water absorption is a method of measuring “effective porosity” of concrete structure. Although this is not an accurate method of determining the actual porosity, it is practical in indicating the relative porosity.

Prior to testing, the specimens were oven-dried at 105 ℃ until a constant mass by ensuring that two successive weighing do not exceed 0.1% of the initial mass. The specimens were cooled to room temperature for further 24 hours. The plastic container was filled with distilled water and allowed to reach equilibrium. The specimens were kept completely immersed in distilled water for 2 days. After the period of immersion, the mass of wet specimens in air was measured. Subsequently, the specimen was placed in a metal wire basket which immersed in water tank and the mass of wet specimen under water was recorded.

The water absorption of the specimen is calculated from the equation below:

$$\text{Water absopstion (\%)} = \frac{b_2 - b_1}{b_1} \times 100\% \qquad (3.11)$$

where

b_1=mass of the oven dried specimen (g)

b_2=mass of the wet specimen in air(g)

The above equation is also expressed in (volume/volume) %. The average water absorption of three specimens was calculated.

Figure 3.9: Water Absoprtion Test

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Introduction

This chapter first discusses the workability based on the result of flow table test for the four different mix proportions and the effect of incorporating slag and MK on workability of mortar is discussed. Each of the mix proportions is labelled as followed: (i) control mix: CTR; (ii) 5% cement replacement with slag: S5; (iii) 5% cement replacement with MK: M5; and (iv) 5% cement replacement with 2.5% of slag and MK respectively: MS2.5.

After that, the results of durability tests performed on mortars incorporated with slag and metakaolin are briefly presented and discussed. There were three different test carried out in this project work: (i) Initial surface absorption test (ISAT) on 100mm cube samples; (ii) Water sorptivity test on 1/3 cylinder and (iii) Water absorption test on 50mm cubes. The specimens were cured under three curing regimes-water cured, cured under plastic membrane and air-cured for 7, 28, 56 and 90 days before undergoing test sessions. The effect of curing regime on the durability of mortar, as well as the effect of incorporating slag and MK in mortar was discussed as well.

Finally, the correlations between the durability test results are determined using linear regression model and presented.

4.2 Workability

The result of flow table test performed in accordance to ASTM C1437 and C230 is tabulated as shown in Table 4.1. The images of the mortar spread for each mix proportion upon completion of flow table is presented in respectively. The result of flow table test is used as an indicator to the workability of mortar.

Table 4.1: Result of Flow Table Test for All Mix Proportions

Mix Proportion	**Spread Diameter of Mortar (mm)**					**Flow (%)**
	1	**2**	**3**	**4**	**Average**	
CTR	241	242	242	240	241.25	141%
S5	-	-	-	-	*Overflow	-
M5	213	214	218	213	214.5	114%
MS2.5	242	243	242	242	242	142%

** Base diameter of mould=100mm

Figure 4.1: Spread of Mortar Upon Completion of Flow Table Test-(a)CTR; (b)S5; (c)M5; and (d)MS2.5

The workability of the four different mix proportions utilized is arranged in an ascending order as followed: M5, MS2.5, CTR and S5. Incorporating 5% MK decreases the workability compared to control mix. The flow is 27% lower than the control mix. In contrast, 5% cement replacement offers a more workable mix than control mortar. The S5 mortar mix overflows the metal plate when it was dropped 25 times within 15 seconds. S5 is the only mix which results in overflowing. Therefore, it is inferred that S5 mix has highest fluidity and flowabilty hence workability than the other three proportions. As for MS2.5 mix, the workability is approximately same as that of control mix. The difference in flow (1%) is rather insignificant.

It is believed that the improvement of workability by addition of GGBFS is because of the smooth, dense surfaces of slag particles which lead to low water absorption during mixing (Meeks & Carino, 1999). Thus, more water is available as lubricant to aid the movements of particles over each other. In addition, a better particle dispersion and higher fluidity is achieved when GGBFS is incorporated into cement paste, resulting in enhance workability (Meeks and Carino, 1999). This is supported by the finding of other researchers who propose that improvement of workability via utilization of slag is owed to lower water absorption by slag particles, better particle dispersion and lubrication effect of slag particles ((Beushausen, Alexander, & Balliim, 2011).

The decrease in the workability by incorporation of MK is due to the fineness of MK particles. Small-sized MK grain possesses greater surface area, which increase the water demand to produce a mix with same workability to control mix. Besides, higher reactivity of MK also leads to decrease in workability. The MS2.5 mix which is a binary mix has almost same workability as control mix is because the effect of slag and MK cancel out each other.

4.3 Initial Surface Absorption (ISA)

The initial surface absorption (ISA) for each of the four mix proportions-control (CTR), 5% slag (S5), 5% metakaolin (M5) and 2.5% slag + 2.5% metakaolin (MS2.5) subjected to three different curing regimes (air, plastic, water), and curing period from 7 day to 90 day are illustrated in Table 4.2 to Table 4.5.

Table 4.2: ISA at 10, 30, 60 and 120 minutes for Control Mix Subjected to Different Curing Regime and Curing Period

Mix Proportion: Control (CTR)					
Age	**Curing Regime**	**Flow (ml/m^2/s)**			
		Time (minutes)			
		10	**30**	**60**	**120**
7	**Air**	2.72	1.37	0.98	0.74
	Plastic	2.08	1.33	0.89	0.62
	Water	1.49	0.91	0.66	0.45
28	**Air**	2.42	1.41	0.99	0.77
	Plastic	1.95	1.19	0.82	0.64
	Water	1.22	0.81	0.63	0.50
56	**Air**	2.33	1.32	0.75	0.54
	Plastic	1.33	0.68	0.46	0.36
	Water	0.82	0.59	0.44	0.35
90	**Air**	1.71	0.80	0.59	0.45
	Plastic	1.11	0.52	0.35	0.28
	Water	0.80	0.38	0.26	0.20

Table 4.3: ISA at 10, 30, 60 and 120 minutes for 5% Slag Replacement Subjected to Different Curing Regime and Curing Period

Mix Proportion: 5% Slag (S5)					
Age	**Curing Regime**	**Flow (ml/m^2/s)**			
		Time (minutes)			
		10	**30**	**60**	**120**
7	**Air**	3.73	2.63	1.80	0.94
	Plastic	2.24	1.39	0.99	0.79
	Water	1.49	0.91	0.66	0.45
28	**Air**	2.19	1.22	0.78	0.61
	Plastic	1.79	0.93	0.69	0.54
	Water	1.25	0.59	0.38	0.29
56	**Air**	1.74	1.13	0.91	0.69
	Plastic	1.45	0.62	0.44	0.33
	Water	0.79	0.62	0.48	0.37
90	**Air**	1.09	0.67	0.51	0.42
	Plastic	0.86	0.62	0.48	0.40
	Water	0.68	0.40	0.27	0.22

Table 4.4: ISA at 10, 30, 60 and 120 minutes for 5% MK Replacement Subjected to Different Curing Regime and Curing Period

Mix Proportion: 5% Metakaolin (M5)					
Age	**Curing Regime**	**Flow (ml/m^2/s)**			
		Time (minutes)			
		10	**30**	**60**	**120**
7	**Air**	1.67	0.91	0.66	0.52
	Plastic	1.10	0.59	0.46	0.35
	Water	0.78	0.51	0.34	0.28
28	**Air**	1.35	0.78	0.56	0.42
	Plastic	0.94	0.73	0.52	0.41
	Water	0.73	0.58	0.38	0.29
56	**Air**	1.25	0.75	0.59	0.40
	Plastic	0.91	0.64	0.42	0.33
	Water	0.67	0.43	0.32	0.26
90	**Air**	1.01	0.67	0.51	0.42
	Plastic	0.84	0.46	0.32	0.28
	Water	0.57	0.41	0.33	0.28

Table 4.5: ISA at 10, 30, 60 and 120 minutes for 2.5% Slag+2.5% MK Replacement Subjected to Different Curing Regime and Curing Period

Mix Proportion: 2.5% Slag+2.5% Metakaolin (MS2.5)					
Age	**Curing Regime**	**Flow (ml/m^2/s)**			
		Time (minutes)			
		10	**30**	**60**	**120**
7	**Air**	2.84	1.20	0.80	0.61
	Plastic	1.21	0.68	0.50	0.38
	Water	0.93	0.56	0.46	0.39
28	**Air**	2.02	1.13	0.83	0.61
	Plastic	0.93	0.56	0.46	0.39
	Water	0.77	0.53	0.37	0.27
56	**Air**	1.47	0.95	0.61	0.45
	Plastic	0.71	0.55	0.41	0.32
	Water	0.51	0.36	0.29	0.24
90	**Air**	1.34	0.80	0.61	0.46
	Plastic	0.73	0.46	0.36	0.30
	Water	0.33	0.24	0.20	0.17

4.3.1 Effect of Curing Regime on ISA-10

Each of the following figures (Figure 4.2 to Figure 4.5) depicts the 10-minutes ISA (known as ISA-10) of specimens for four different mix proportion subjected to air, plastic and water curing at age of 7, 28, 56 and 90 day respectively. Although ISAT is perform at 10, 30, and 60 and 120 minutes, ISAT-10 is considered as the most important result as it reflects the permeability of near surface region, which adversely affects the durability. Besides, ISAT assumes a unidirectional downward flow of water through the concrete pores. This assumption generally holds true at the beginning of ISAT when the water penetrates a relatively smaller depth from concrete surface. At later time, the flow of water usually changes and no longer unidirectional. Therefore, ISA-10 is conventionally used as an indicator to measure the durability of the concrete mortar.

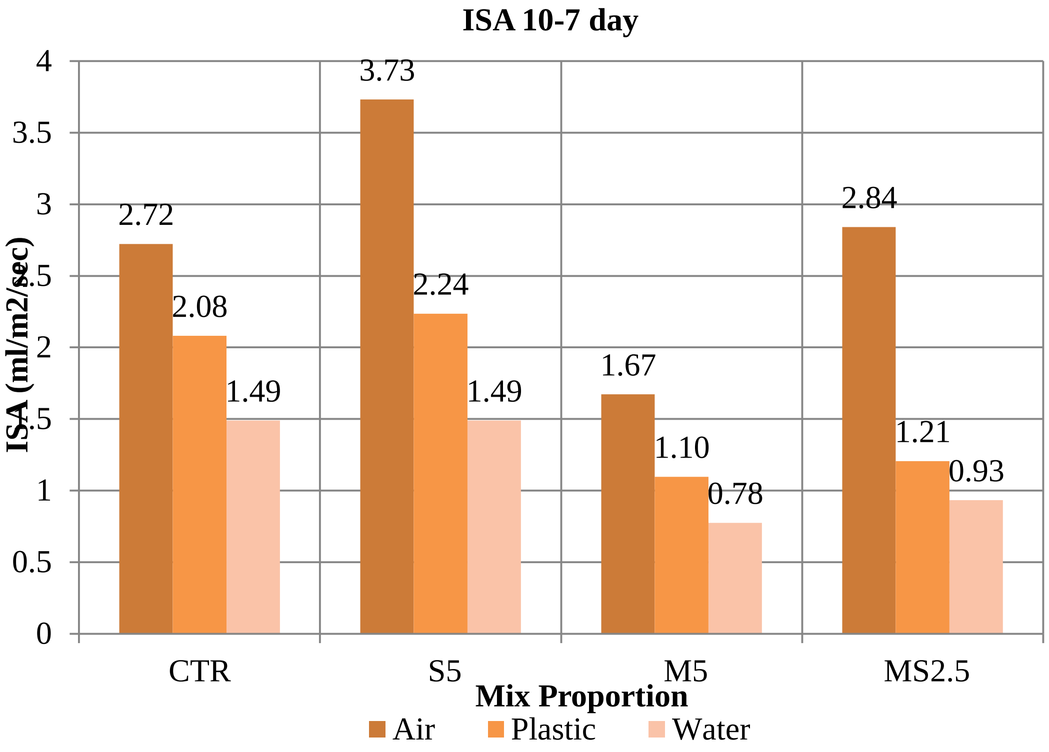


Figure 4.2: ISA-10 at 7 Days Curing Age

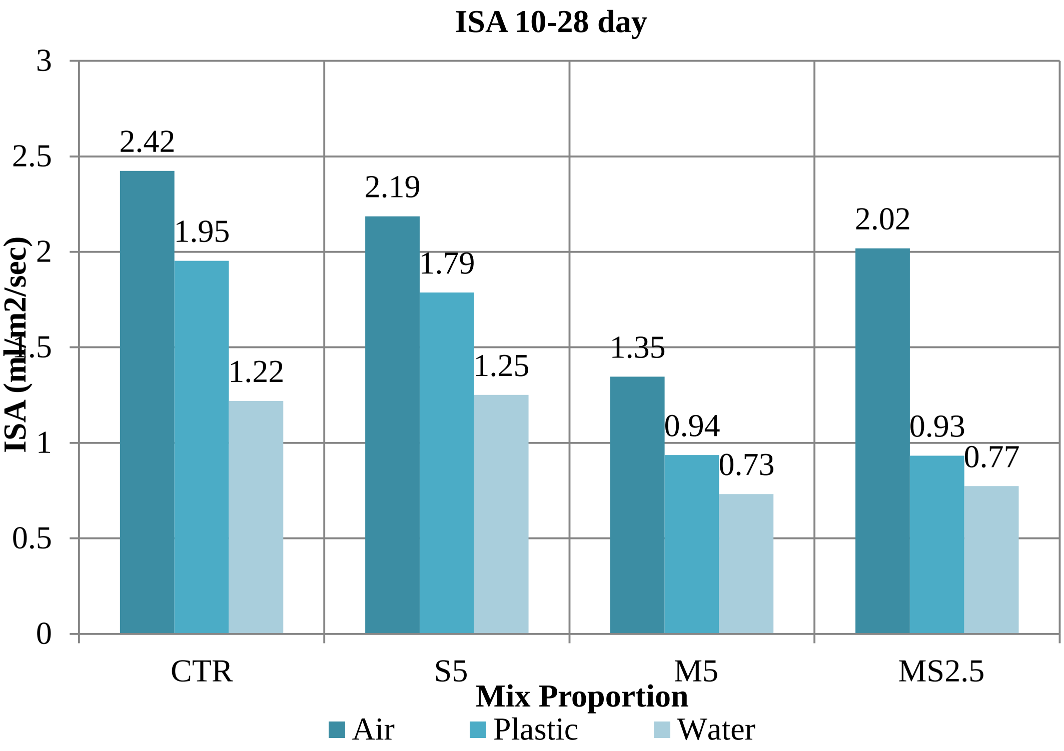


Figure 4.3: ISA-10 at 28 Days Curing Age

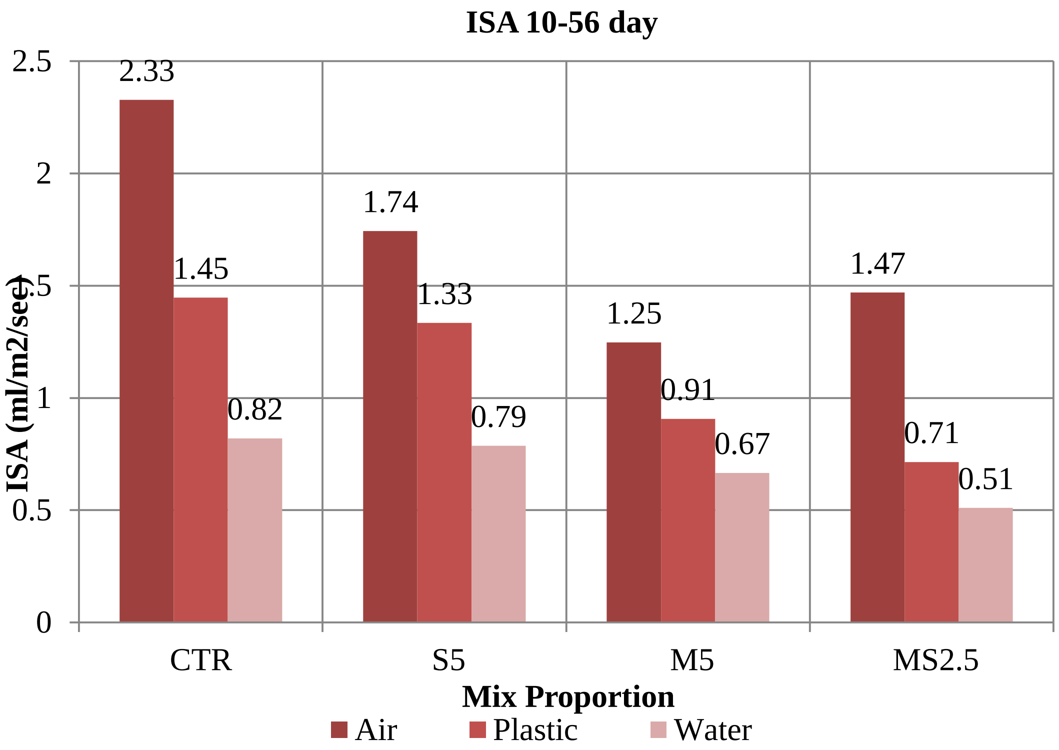


Figure 4.4: ISA-10 at 56 Days Curing Age

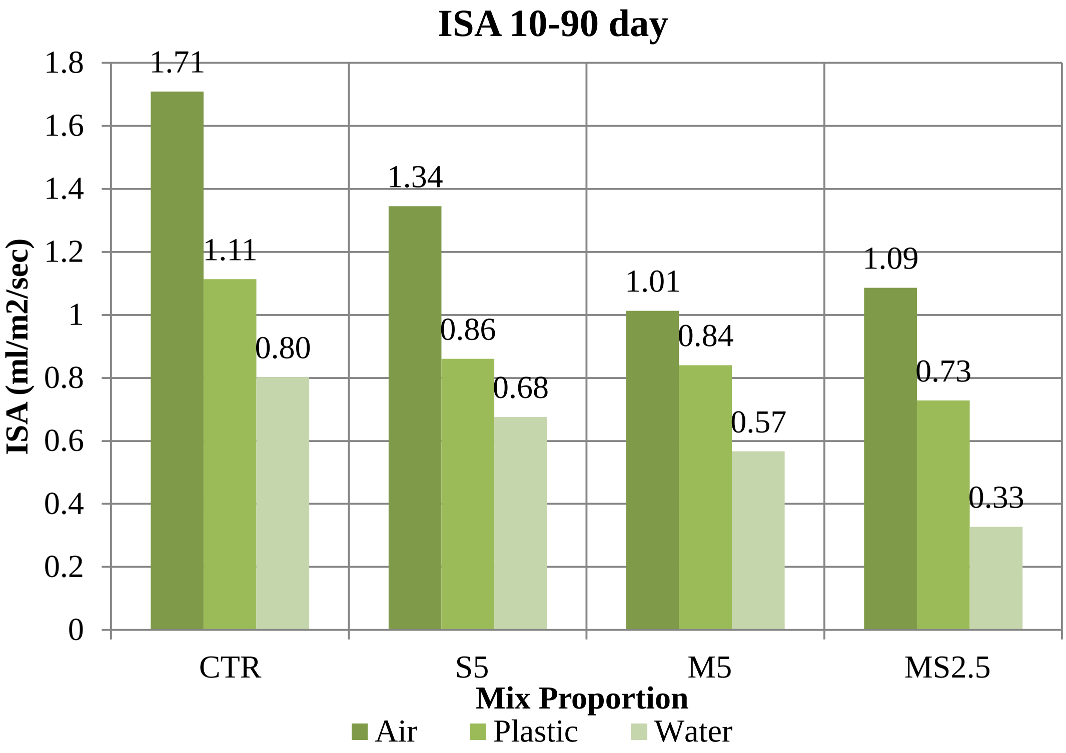


Figure 4.5: ISA-10 at 90 Days Curing Age

From Figure 4.2 to Figure 4.5, it is observed that air-cured specimens generally results in highest ISA-10 than that of plastic-cured and water cured specimens. Mortar specimens for each of the four mix proportions offer lowest value ISA-10 when subjected to water curing than other two curing regimes at all testing ages. At the age 7 days, air-cured S5 specimens have the worst surface absorption with an ISA-10 of 3.73 ml/m^2/s which is the maximum out of all data collected. Conversely, water-cured MS2.5 specimens at 90-day age give a minimum ISA-10 of 0.33 ml/m^2/s.

Cement requires water for hydration whilst supplementary cement materials (SCM) demand water for pozzolanic reactions to additional hydration products. These reactions occur within the pores inside mortar and eventually lead to pore refinement. Furthermore, hydration of cement and SCM should be continuous in order to improve the surface durability of mortar. When mortar specimens are subjected to air curing, the mixing water was allowed to evaporate into the surrounding, leading to insufficient moisture at near-surface region. Consequently, the hydration process eventually slows down or halts, leaving a coarser pore structure at this region. Thus, the water permeability of poorly cured mortar specimens is higher this in turn offers higher ISA.

As for plastic curing, the initial mixing water is prevented from escaping into the surrounding by providing an impermeable barrier. This ensures a continuous supply of moisture for prolonged hydration of cement and SCMs during the early age. Hydration products are created and they fill in the void which initially occupied by mixing water, yielding a finer pore structure in mortar. In this circumstance, the porosity is decreased and the pores become discontinuous, thus improve the surface absorption of mortar as compared to air curing.

Among the three curing regimes, water curing results in lowest ISA for mortar specimens. This method ensures adequate supply of moisture to specimens throughout the whole curing period. As a comparison, the moisture available using water curing is more abundant than plastic curing. Hence, the rate and completeness of hydration of water-cured specimens are better. The porosity and interconnectivity of pores are reduced by a greater extent and this contributes to a greater improvement on surface absorption than plastic curing.

Besides, sufficient moisture during early age is important to reduce the drying shrinkage of mortar. Drying shrinkage is unwanted as the volumetric contraction of mortar upon drying creates tension force within the cement matrix in mortar. Micro-cracks develop as a consequence and the pore structure eventually become coarser. Development of micro-cracks increases the water permeability of mortar as well. The influence of drying shrinkage is more adverse at the near surface region where moisture is more readily to escape into the atmosphere. When mortar specimens undergo air-curing, the rapid drying at near surface region tends to form micro-cracks which increases the ISA of mortar. In the contrary, adequate moisture at early stage by plastic curing and water curing reduce the drying shrinkage of mortar. Hence, the ISA was deducted as well.

4.3.2 Effect of Slag and Metakaolin on ISA-10

Each of the following figures (Figure 4.6 to Figure 4.8) depicts the variation of ISA-10 over age for four different mix proportions cured under different curing regimes.

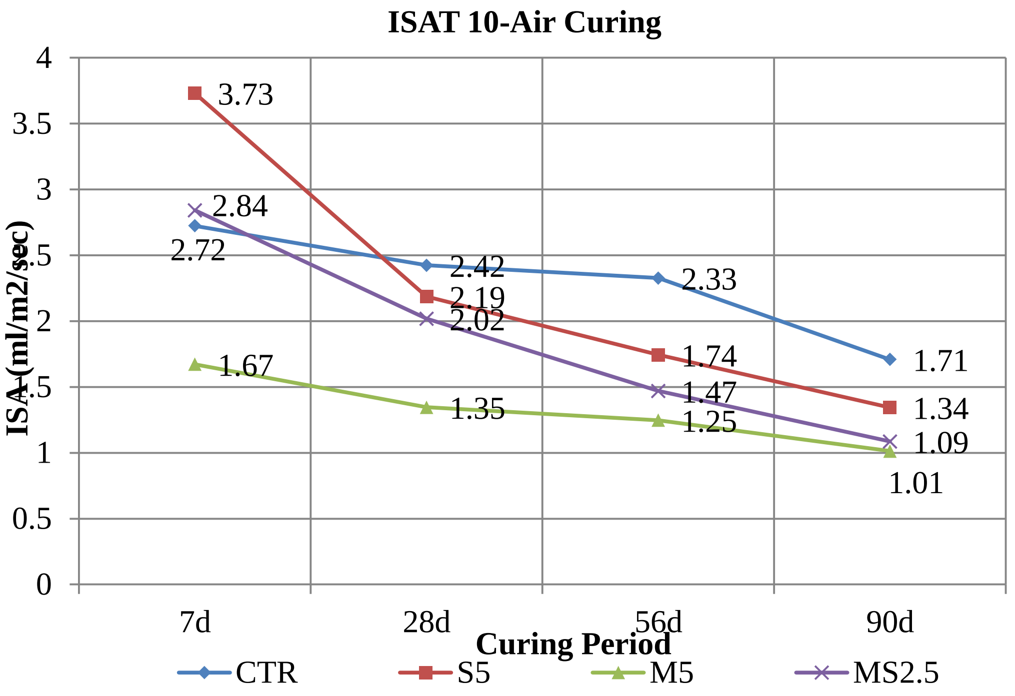


Figure 4.6: ISA-10 for All Mix Proportions under Air Curing

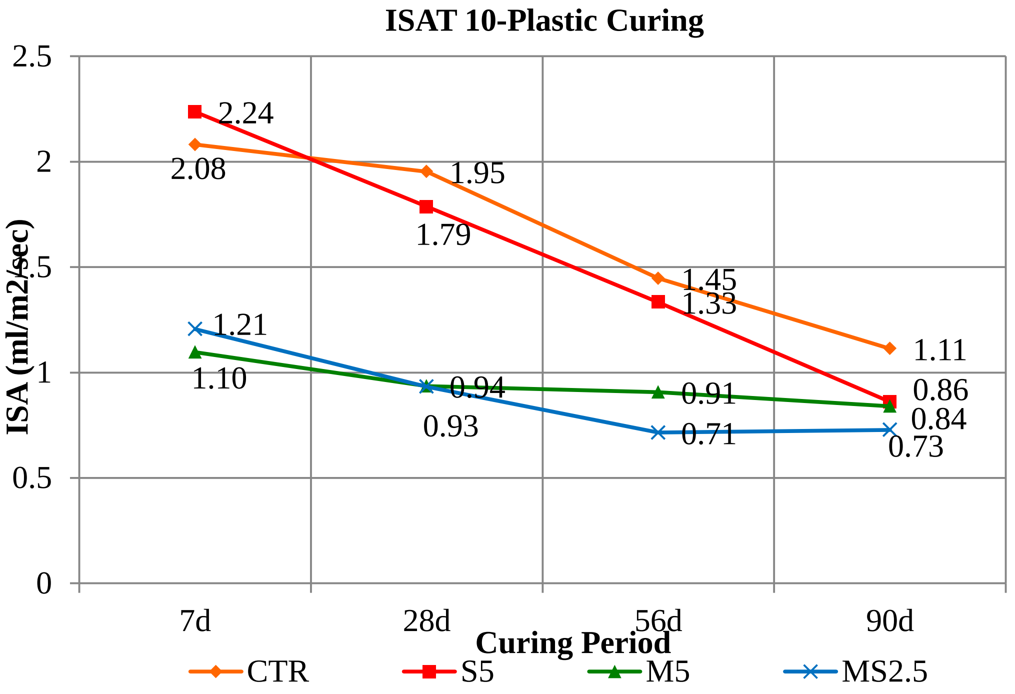


Figure 4.7: ISA-10 for All Mix Proportions under Plastic Curing

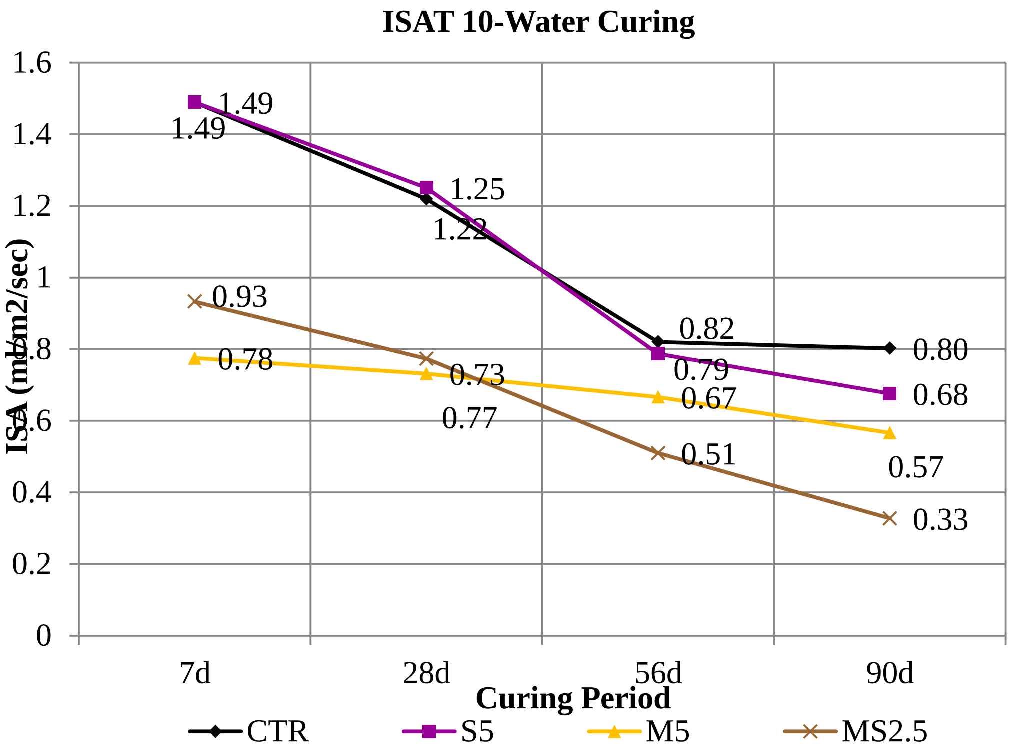


Figure 4.8: ISA-10 for All Mix Proportions under Water Curing

Overall, the ISA-10 of mortar is reduced with increasing curing period for all curing regimes and mix proportions. The reduction is the most substantial for air-cured S5 specimens, especially during early age of curing. Increasing the curing period cause the ISA-10 decreases by nearly 40% from 3.73 to 2.19. Generally, the reduction of ISA-10 is larger at early age and gradually diminishes at later age. An appropriate explanation is that of the cement and SCMs in mortar mix are abundant during early stage and reacts faster with water. When proceed to later age, only a relatively small portion of cement and SCMs are unreacted. As a result, the improvement of surface absorption over time is less significant at later age.

From the results, it is noticed that mortar incorporated with slag and metakaolin generally performed better than control mortar in all the three curing regimes. According to Figure 4.6, the M5 mix exhibits the lowest ISA, followed by MS2.5 and S5 mix. The control mix has the worst performance under air curing. Nevertheless, the ISA of S5 (3.73ml/m^2/s) mix is significantly higher than of control mix (2.72ml/m^2/s) at early age of 7 day. Similarly, the ISA of MS2.5 mix (2.84ml/m^2/s) is slightly higher than that of control mix at 7 day age. When proceed to later ages, both S5 and MS2.5 mix have lower ISA than control.

S5 mortar in plastic curing shows a higher ISA than that of control mix at early age of 7day. The ISA becomes lower than that of control at later ages of 28, 56 and 90 days. From Figure 4.7, mortar specimens incorporated with MK (M5 and MS2.5) offers substantially lower ISA than control mix throughout when subjected to plastic curing. At 7day, ISA of M5 mix is higher than MS.2.5. The gap is closed at the age 28 day in which both mix has nearly same ISA (0.94 for M5 and 0.94 for MS2.5). At greater age of 56 and 90 days, MS2.5 mix gives lower ISA than M5 mix.

In water curing, the trend of ISA over age is discovered to be similar to that of plastic curing. ISA of S5 mix is observed to be identical as that of control at 7 day. Mortar incorporated with MK shows better surface absorption properties than control.

It can be deduced that mortar incorporating with slag is more sensitive to poor curing during early age. Absence of moisture during early stage leads to poor surface quality which gives higher ISA. In accordance to Figure 4.6 and Figure 4.7, the ISA of poorly cured (air-cured) slag-incorporated (S5) specimens show even higher ISA when compared to control mix. This is possibly due to a slower reaction rate of slag with water. Slag particles possess of a complex crystalline structure which causes them hydrate slower than cement and MK particles. When the moisture in near surface region of mortar specimens lost into surrounding during early stage, the portion of hydrated slag particles within this region is relatively low. This forms a coarser pore structure and leads to high surface absorption. At later age, the unreacted slag particles in near surface region reacts with water molecules that diffuse to this region from the inner core of a mortar specimen.

The improved performance by addition of slag and MK into mortar is due to several reasons. First, both slag and MK are pozzalanic materials and able to react with calcium hydroxide (CH) in cement paste to produce additional calcium-silicate-hydrate (C-S-H) gel. C-S-H gel is the main source of strength and durability. Hence, formation of additional C-S-H gel from pozzolanic reaction is expected to improve the cement matrix and produce a denser pore structure. The deposition of extra C-S-H gel within the pores reduces the size of the pores. A denser pore structure is, definitely, less permeable to water and hence a lower surface absorption is obtained.

The slag particles are smaller than cement particles. This characteristic enables the voids between larger-size cement and sand particles to be filled. This effect is known as filling effect. The filling effect by incorporating smaller slag particles into mortar mix also creates a denser pore structure with lower amount of porosity. Therefore, the surface absorption is enhanced compared to control mix. Meanwhile, MK particles exert filling effect as well. However, MK particles are much finer than cement and slag particles and this effect is expected to be more substantial than that of slag. The very small-sized MK particles are able to create a very dense pore structure and leads to excellent surface absorption characteristics.

Furthermore, incorporation of slag and MK also impose a blocking effect. The smaller particle sizes of slag and MK, together with the additional C-S-H gel produced disrupt the interconnectivity of pores within matrix structure. The pores become discontinuous and difficult for water to pass through. The size of the pores is reduced as well due to blocking effect. As a result, the surface absorption characteristic is improved when slag and MK are added. Once again, MK particles inflict a more considerable blocking effect than slag particles due to their finer size. Therefore, MK performs better than slag in term of surface absorption.

4.4 Sorptivity

The sorptivity of mortar specimens subjected to air, plastic and water curing is tabulated as shown in Table 4.6 to Table 4.9. The sorptivity provides an indication related to the pore structure, as well as size and interconnectivity of capillaries in mortar specimens.

Table 4.6: Sorptivity for Control Mix Subjected to Air, Plastic and Water Curing at 7, 28, 56 and 90 Day Age

Mix Proportion: Control (CTR)			
Curing Period (day)	**Sorptivity (mm/min$^{0.5}$)**		
	Curing Regime		
	Air	**Plastic**	**Water**
7	65.52	63.44	50.09
28	60.67	42.56	30.76
56	42.92	30.81	22.92
90	37.70	25.76	16.59

Table 4.7: Sorptivity for 5% Slag Replacement Subjected to Air, Plastic and Water Curing at 7, 28, 56 and 90 Day Age

Mix Proportion: 5% Slag (S5)			
Curing Period (day)	**Sorptivity (mm/min$^{0.5}$)**		
	Curing Regime		
	Air	**Plastic**	**Water**
7	60.13	42.60	36.86
28	34.77	28.53	27.69
56	25.76	22.92	21.30
90	18.39	15.85	13.94

Table 4.8: Sorptivity for 5% MK Replacement Mix Subjected to Air, Plastic and Water Curing at 7, 28, 56 and 90 Day Age

Mix Proportion: 5% Metakaolin (M5)			
Curing Period (day)	**Sorptivity (mm/min$^{0.5}$)**		
	Curing Regime		
	Air	**Plastic**	**Water**
7	37.70	27.26	24.91
28	27.26	22.06	17.04
56	22.06	16.59	10.06
90	9.79	8.13	6.75

Table 4.9: Sorptivity for 2.5% Slag+2.5% MK Replacement Subjected to Air, Plastic and Water Curing at 7, 28, 56 and 90 Day Age

Mix Proportion: 2.5% Slag+2.5% Metakaolin (MS2.5)			
Curing Period (day)	**Sorptivity (mm/min$^{0.5}$)**		
	Curing Regime		
	Air	**Plastic**	**Water**
7	55.32	37.68	30.76
28	30.60	24.91	22.66
56	22.92	21.30	19.00
90	11.82	9.20	7.17

4.4.1 Effect of Curing Regime on Sorptivity

Figure 4.9 to Figure 4.12 illustrate the sorptivity of mortar specimens of all four mix proportions subjected to air, plastic and water curing at age of 7, 28, 56 and 90 day respectively..

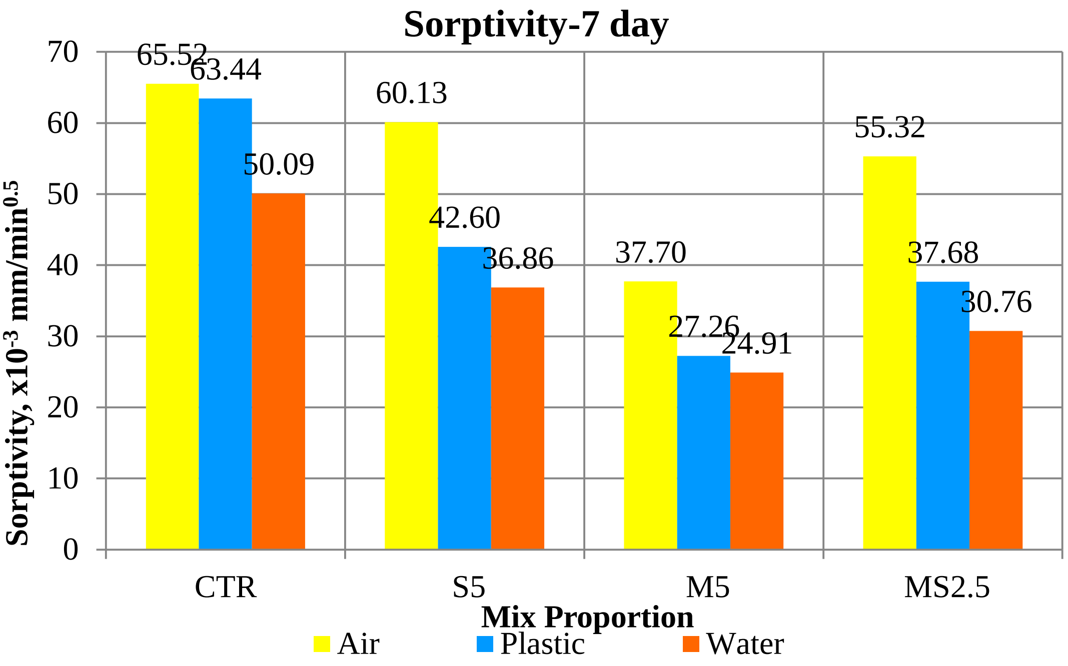


Figure 4.9: Sorptivity at 7 Days Curing Age

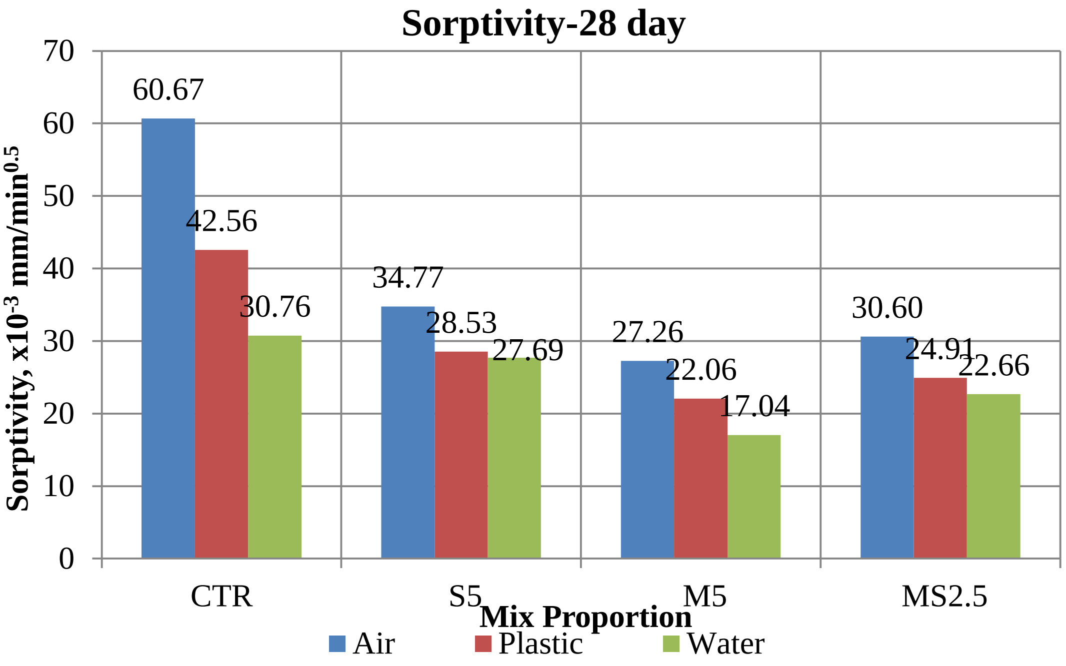


Figure 4.10: Sorptivity at 28 Days of Curing Age

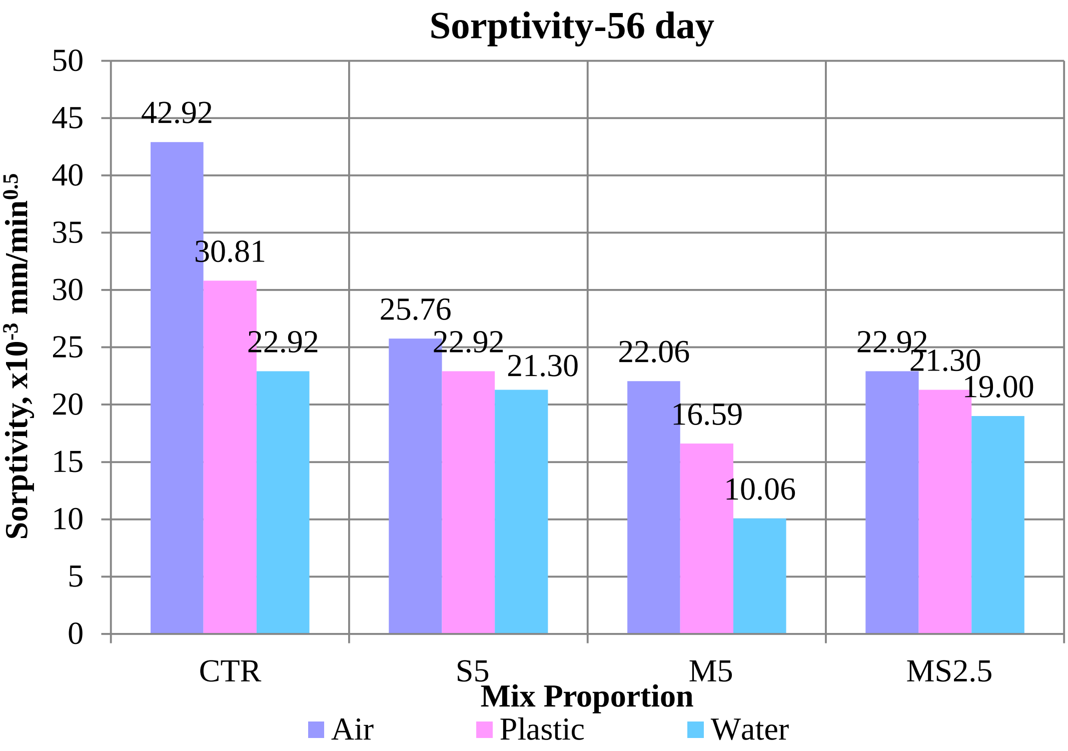


Figure 4.11: Sorptivity at 56 Days Curing Age

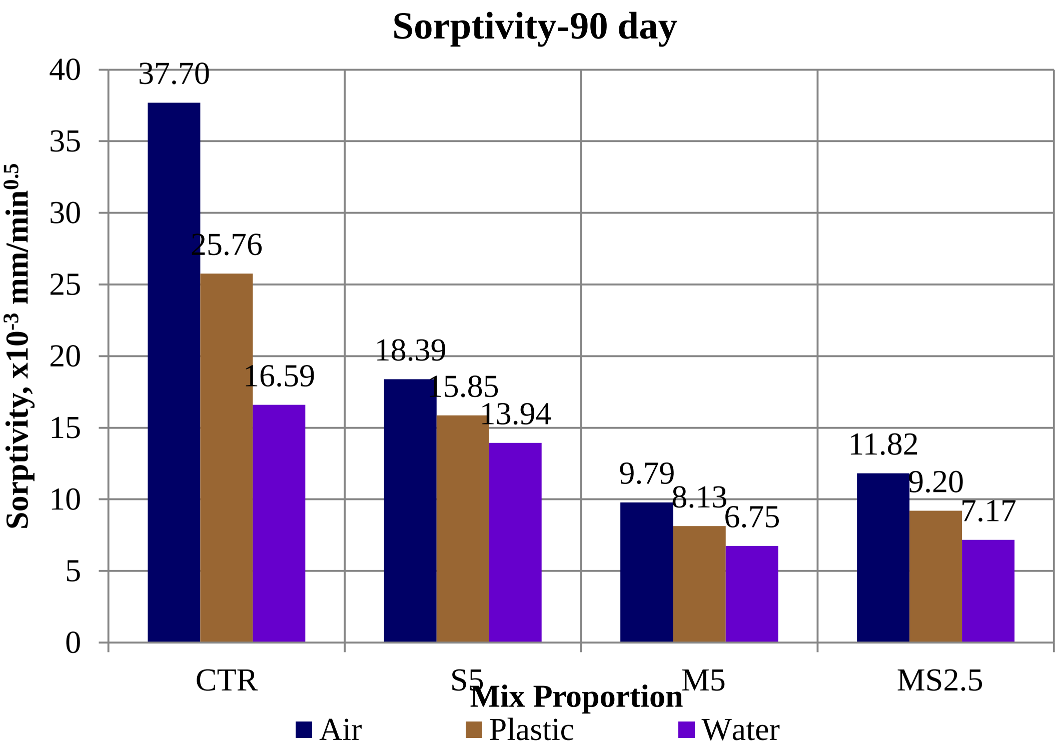


Figure 4.12: Sorptivity at 90 Days of Curing Age

A careful observation reveals that the sorptivity of mortar follow a trend similar to ISA-10. Generally, mortar spcecimens undergo air-curing give the highest sorptivity, subsequently by plastic curing. Water-cured specimens have the minimum sorptivity among three curing regimes.

At 7-day age, the soprtivity of plastic-cured control mix is only slightly higher than air-cured specimens while water-cured specimens' sorptivity is significantly lower than other specimens exposed to the other two curing regimes. At other ages, the effect of different curing regimes on sorptivity of control mix becomes distinctive. Among the four different mix proportions, M5 mix is relatively insensitive to the different curing regimes introduced. As for S5 and MS2.5 mix, plastic curing and water curing give sorptivity which is obviously lower than air curing.

As later ages (28, 56 and 90 days) the sorptivity of mortar with SCMs is relatively insensitive to curing regimes than control mix. The difference of specimens' sorptivity due to different curing method decreases compared to 7-day sorptivity. The soprtivity of M5 mix is least affected by curing regimes.

The presence of moisture is essential for the hydration of cement and also pozzolanic reaction of slag and MK. Lack of moisture in air curing lead to high sorptivity of mortar specimens. These necessary reactions for pore refinement are slowed down and even halted due to insufficient supply of moisture. Hence, the capillaries of air-cured specimens possess larger size and higher interconnectivity. As a result, the water penetrates the pore into mortar by capillary action more easily.

On the other hand, plastic curing and water curing promise adequate moisture for cement hydration and pozzolanic reaction of slag and MK. The filling effect and blocking effect of the products formed refine the pore structure. This results in a denser pore structure. The size and interconnectivity of capillaries decreases therefore a lower sorptivity is obtained. Moreover, the presence of moisture mitigates the drying shrinkage of mortar especially during the early age of curing. Thus, the development of microcracks is restrained and this produces a finer pore structure.

4.4.2 Effect of Slag and Metakaolin on Sorptivity

Figure 4.13 to Figure 4.15 display the sorptivity for all four different mix proportions over ages from 7 day to 90 day under air, plastic and water curing respectively.

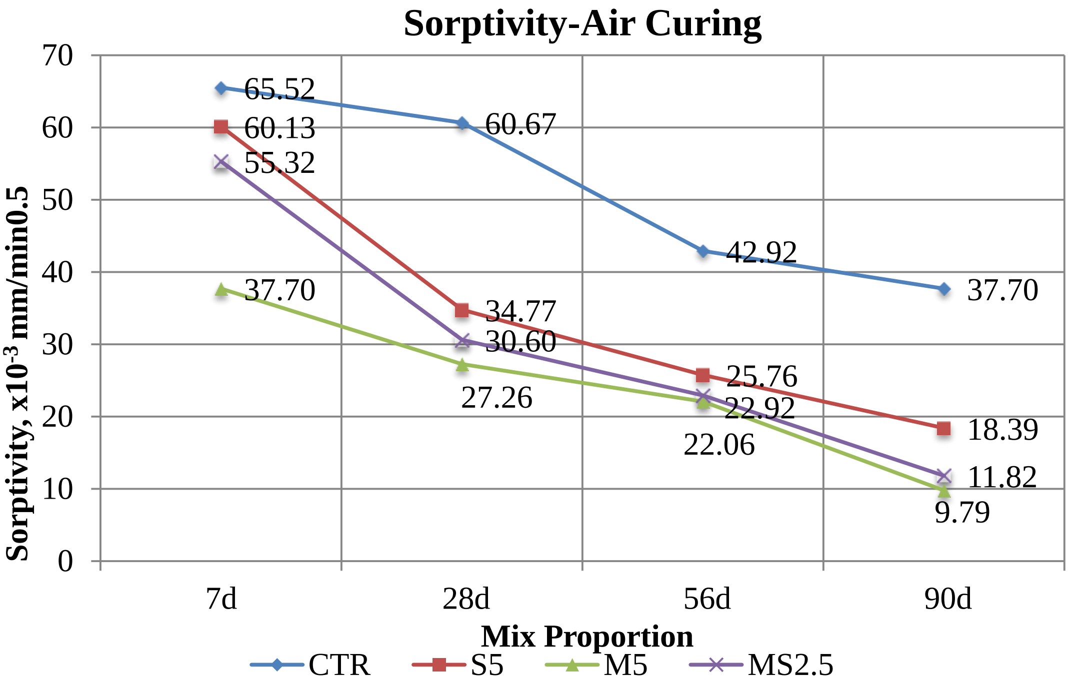


Figure 4.13: Sorptivity for All Mix Proportions under Air Curing

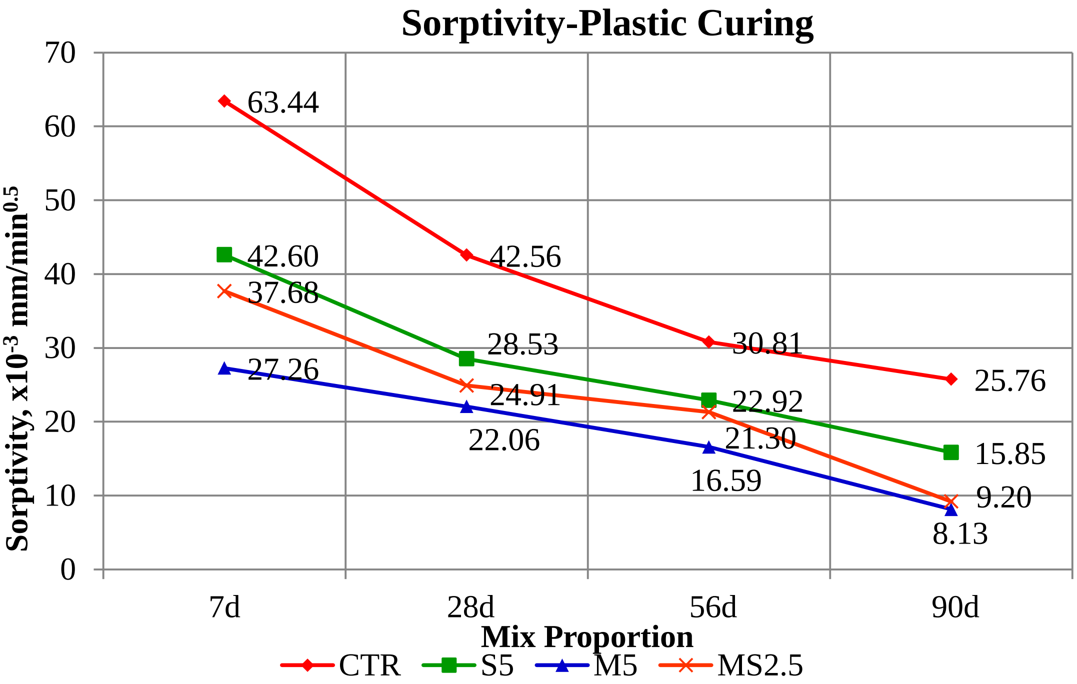


Figure 4.14: Sorptivity for All Mix Proportions under Plastic Curing

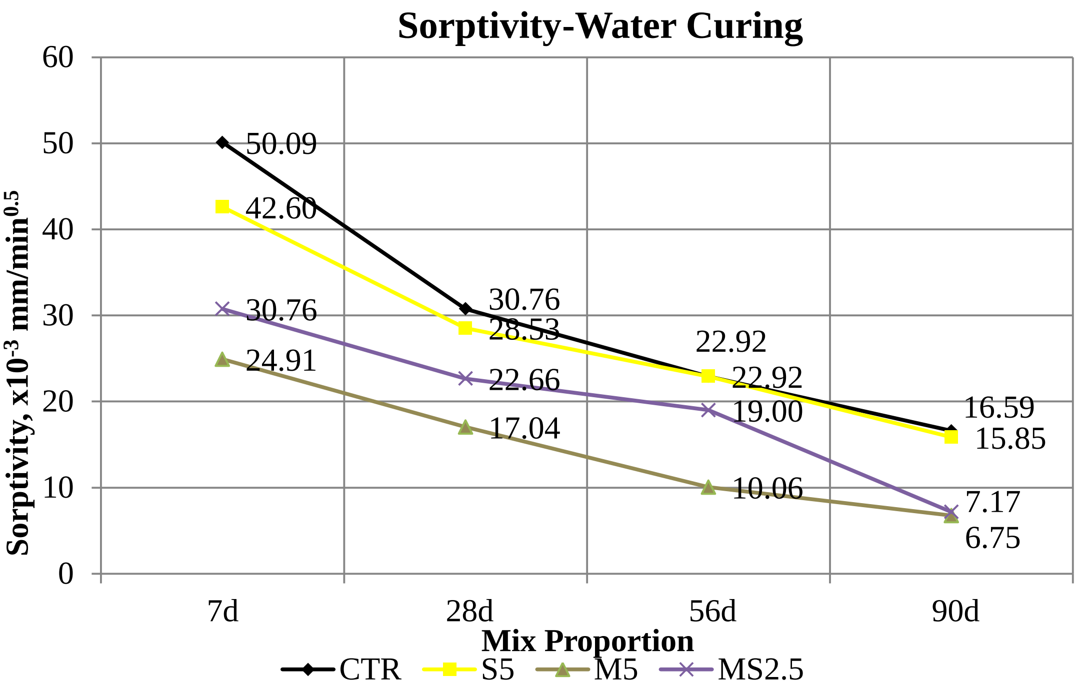


Figure 4.15: Sorptivity for All Mix Proportions under Water Curing

The results denote that mixes with slag and MK (S5, M5 and MS2.5) has better quality in term of sorptivity under all curing regimes. Among the four mix proportions employed, M5 specimens have the lowest sorptivity throughout. A minimum value of $6.75x10^{-3}$ $mm/min^{0.5}$ is achieved when M5 is subjected to water curing and tested at age of 90 day. M5 mix exhibits constantly lower sorptivity than the other three mix proportions for each corresponding age under all three different curing regimes. The performance of MS2.5 mix is ranked after M5 mix.

The control mix gives the worse sorptivity properties and the values obtained are constantly higher than other mixes at every respective age. Except at early age of 7 days, the gap between the sorptivity of control mix with other mixes is the greatest when subject to air curing. While for plastic curing, a similar trend to air curing is detected for ages from 28 to 90 days, however, the difference in sorptivity between control mix and other mixes diminishes. At 7 day age, the sorptivity of control mix is significant higher than other mixes when subjected to plastic curing. For water curing, the gap is even made closer.

The pozzolanic reaction by slag and MK is responsible for the lower values of soprtivity than control mix. Slag and MK are pozzolans and are able to reaction with calcium hydroxide which is one of the major products from cement hydration to generate additional C-S-H gel. The additional gel imposes blocking effect and filling effect, lead to a denser and less interconnected pore structure. The refined pore structure disturbs the water uptake by capillary action, resulting in a lower sorptivity.

In addition, MK deemed to perform better than slag in improving durability in term of sorptivity. The results in Figure 4.13 to Figure 4.15 depict that 5% cement replacement by MK overall obtains lower sorptivity than 5% slag replacement and 2.5% slag + 2.5% MK. The sorptivity of MS2.5 mix falls between M5 and S5 mix for every curing regime at all ages. It is also noted that the MS2.5 has relatively significant lower sorptvity than M5 mix at the early ages for plastic and water curing. Nevertheless, at later age of 90 days, the sorptivity of MS2.5 mortar is reduced to a value approaching that of M5 mix.

MK which has a smaller particle size distribution than slag provides a better filling effect and blocking effect than slag does. The pore structure created by MK is denser and discontinuous that of slag thus a lower sorptivity is achieved. Besides, smaller size of MK particles also means a greater surface area is available for reaction with calcium hydroxide. In other words, MK is more reactive than slag the reaction progress at a more rapid rate. Therefore, the pore refinement process is faster and this contributes to lower sorptivity at the early stage. In contrast, the more complex and larger particle size makes it react at a slower rate. The improvement of durability in term of sorptivity hence is not as excellent as MK does.

For all three curing regimes introduced, the sorptivity of mortar gradually decreases with increasing curing age. This is due to the greater degree of hydration of cement, together with greater extent of pozzolanic reaction by slag and MK. Over longer period of time, mortar specimens develop finer and denser pore structure thus results in lower sorptivity.

4.5 Water Absorption

Table 4.10 to Table 4.13 present the water absorption of mortar specimens of all four mix proportions subjected to air, plastic and water curing at age of 7, 28, 56 and 90 day respectively. The water absorption test provides a pathway to access the total porosity of mortar specimens. Nonetheless, it is not able to offer accurate information on the continuity of pore structure of mortar specimens tested.

Table 4.10: Water Absorption for Control Mix Subjected to Air, Plastic and Water Curing at 7, 28, 56 and 90 Day Age

Mix Proportion: Control (CTR)			
Curing Period (day)	**Water Absorption (%)**		
	Curing Regime		
	Air	**Plastic**	**Water**
7	9.96	9.69	9.11
28	9.77	7.95	7.71
56	8.81	7.32	6.94
90	7.14	6.84	6.40

Table 4.11: Water Absorption for 5% Slag Replacement Subjected to Air, Plastic and Water Curing at 7, 28, 56 and 90 Day Age

Mix Proportion: 5% Slag (S5)			
Curing Period (day)	**Sorptivity (mm/min$^{0.5}$)**		
	Curing Regime		
	Air	**Plastic**	**Water**
7	10.83	9.29	8.05
28	8.77	6.96	6.64
56	8.18	6.30	6.29
90	7.30	5.90	5.10

Table 4.12: Water Absorption for 5% MK Replacement Mix Subjected to Air, Plastic and Water Curing at 7, 28, 56 and 90 Day Age

Mix Proportion: 5% Metakaolin (M5)			
Curing Period (day)	**Sorptivity (mm/min$^{0.5}$)**		
	Curing Regime		
	Air	**Plastic**	**Water**
7	8.85	8.58	7.06
28	6.52	6.44	5.42
56	5.65	5.34	4.30
90	5.11	3.62	2.78

Table 4.13: Water Absorption for 2.5% Slag+2.5% MK Replacement Subjected to Air, Plastic and Water Curing at 7, 28, 56 and 90 Day Age

Mix Proportion: 2.5% Slag+2.5% Metakaolin (MS2.5)			
Curing Period (day)	**Sorptivity (mm/min$^{0.5}$)**		
	Curing Regime		
	Air	**Plastic**	**Water**
7	6.91	6.57	6.27
28	6.16	5.49	5.22
56	5.05	4.39	4.24
90	3.62	2.94	2.47

4.5.1 Effect of Curing Regime on Water Absorption

Figure 4.9 to Figure 4.12 show the water absorption of mortar specimens of all four mix proportions subjected to air, plastic and water curing at age of 7, 28, 56 and 90 day respectively. Water absorption test can be employed to measure the porosity of mortar specimens. However, this method is only practical to measure the effective porosity but not the actually porosity.

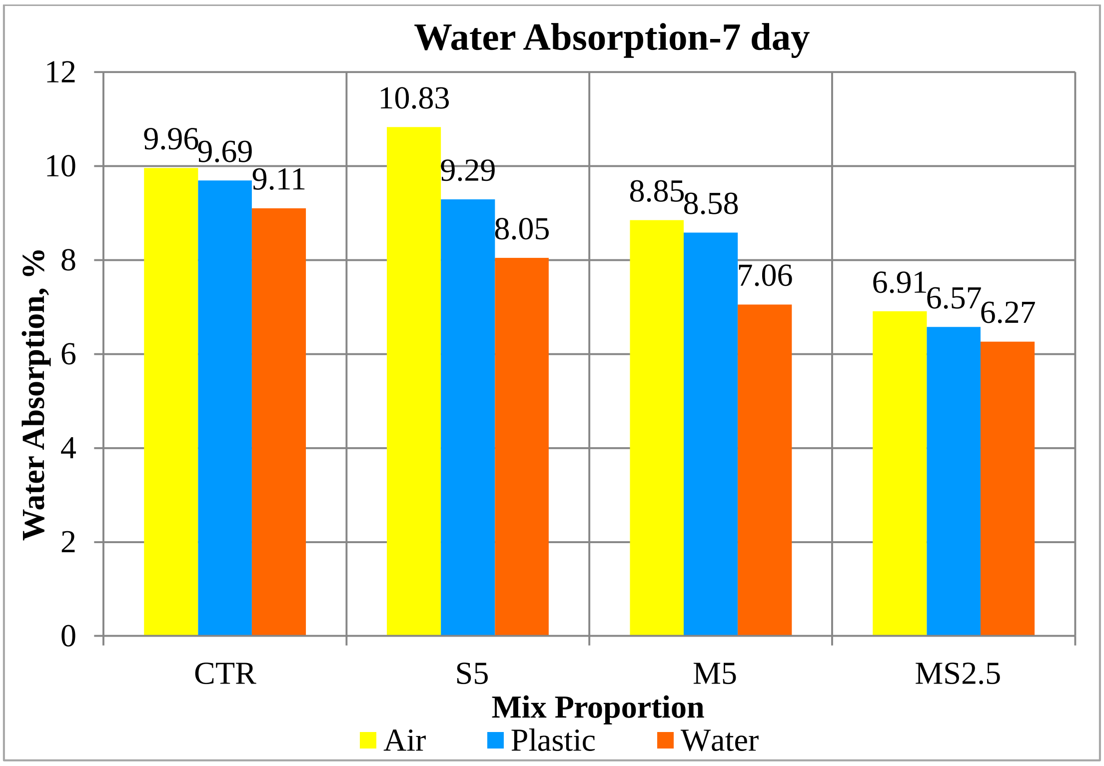


Figure 4.16: Water Absorption at 7 Days Curing Age

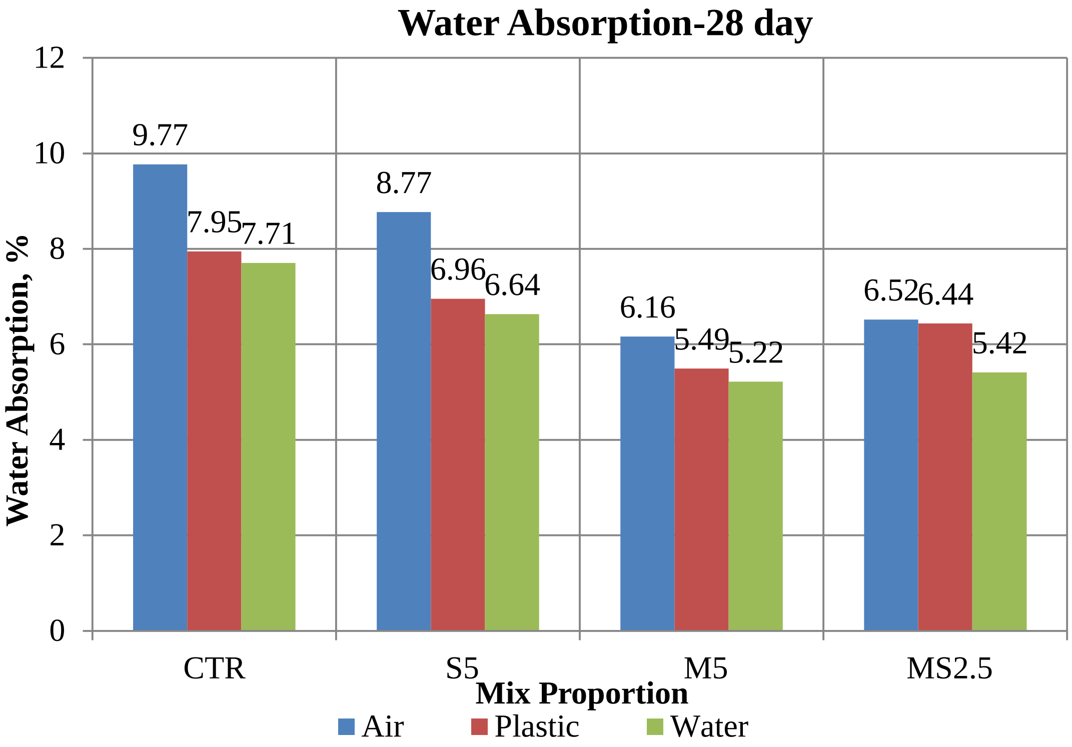


Figure 4.17: Water Absorption at 28 Days Curing Age

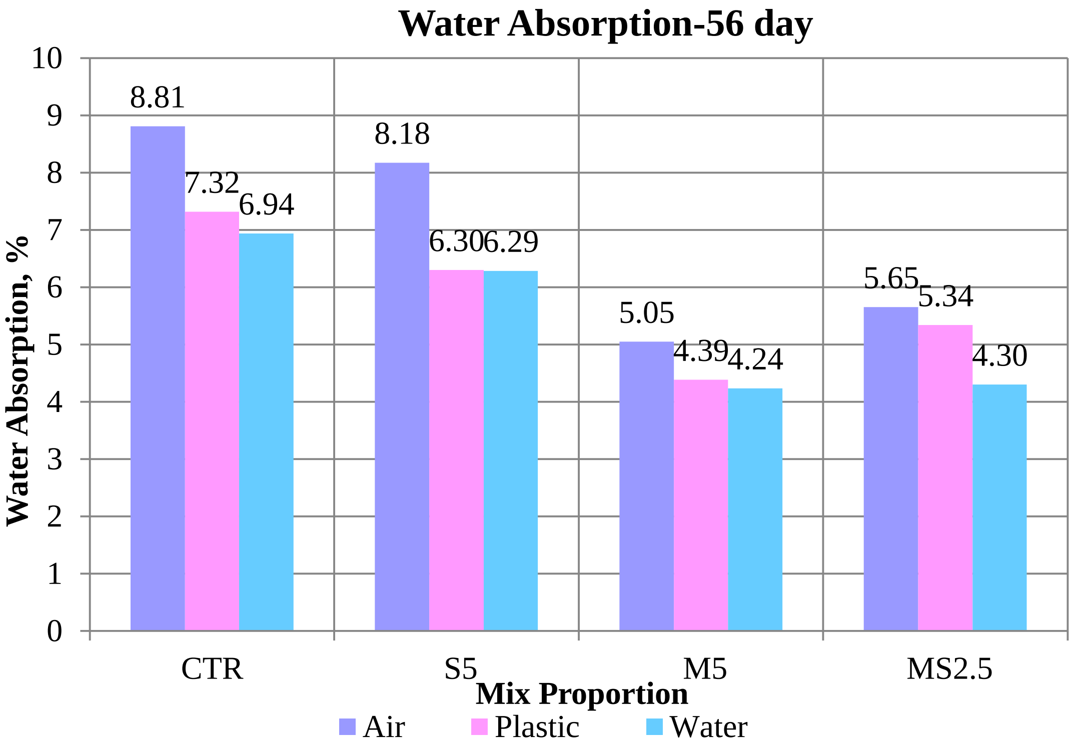


Figure 4.18: Water Absorption at 56 Days Curing Age

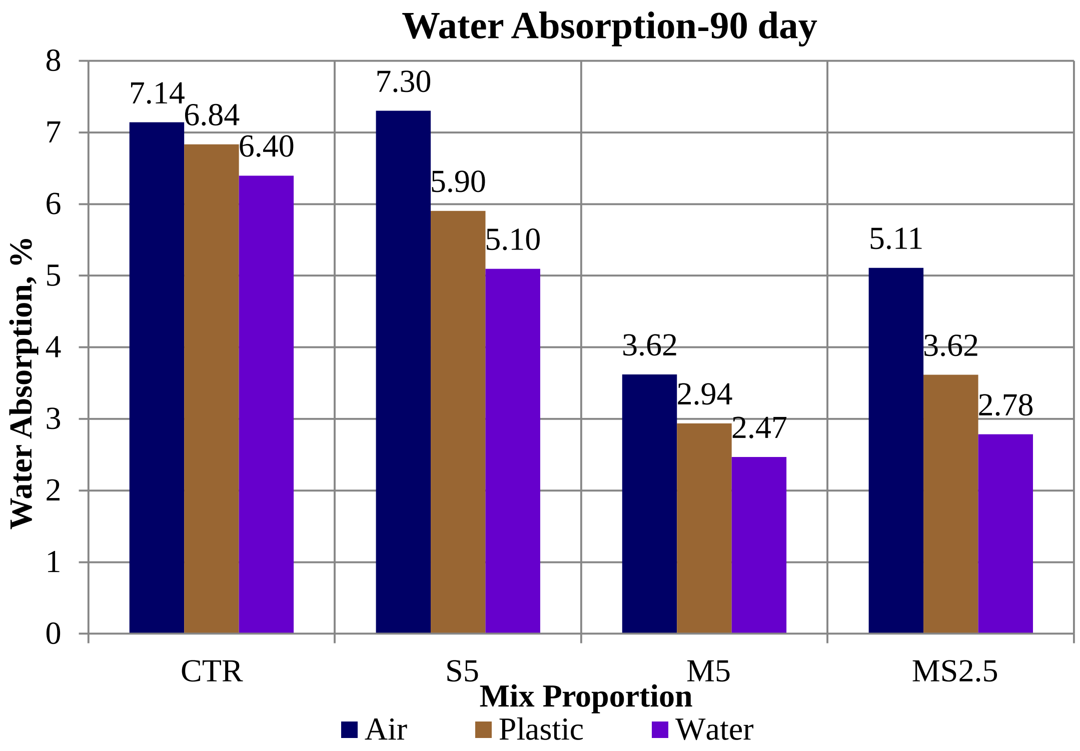


Figure 4.19: Water Absorption at 90 Days Curing Age

The above figures indicate that air curing results in highest water absorption for all the four mix proportions at all ages. Plastic cured mortar specimens, on the other hand, offers lower water absorption than corresponding air-cured specimens. Specimens subjected to water curing have lowest absorption among all three curing regimes. S5 exposed to air curing at 7-day age obtained maximum water absorption of 10.83% and this is higher than the respective air-cured control mix specimens (9.96%). Minimum water absorption occurs for water-cured M5 mix specimens, with a value of 2.47%.

The results of water absorption test further fortify that the presence of adequate moisture is important to produce durable mortar. This is particularly important at early ages. Abundance of moisture promotes the hydration of cement and pozzolanic reaction between SCMs (slag and MK) with calcium hydroxide. The enhanced rate of reaction causes greater magnitude of filling effect and reduces the porosity. Ultimately the formation of a denser structure reduces the water absorption of mortar specimens. The lower water absorption of plastic-cured and air-cured specimens is also related to reduced drying shrinkage due to presence of moisture.

It is observed that at 56 days age, the water absorption of plastic-cured specimens is virtually same as water-cured specimens with a difference of only 0.01%. This is probably due to a higher volume of entrapped air within the water cured specimens.

4.5.2 Effect of Slag and Metakaolin on Water Absorption

Figure 4.13 to present the water absorption for all four different mix proportions over ages from 7 day to 90 day under air, plastic and water curing respectively.

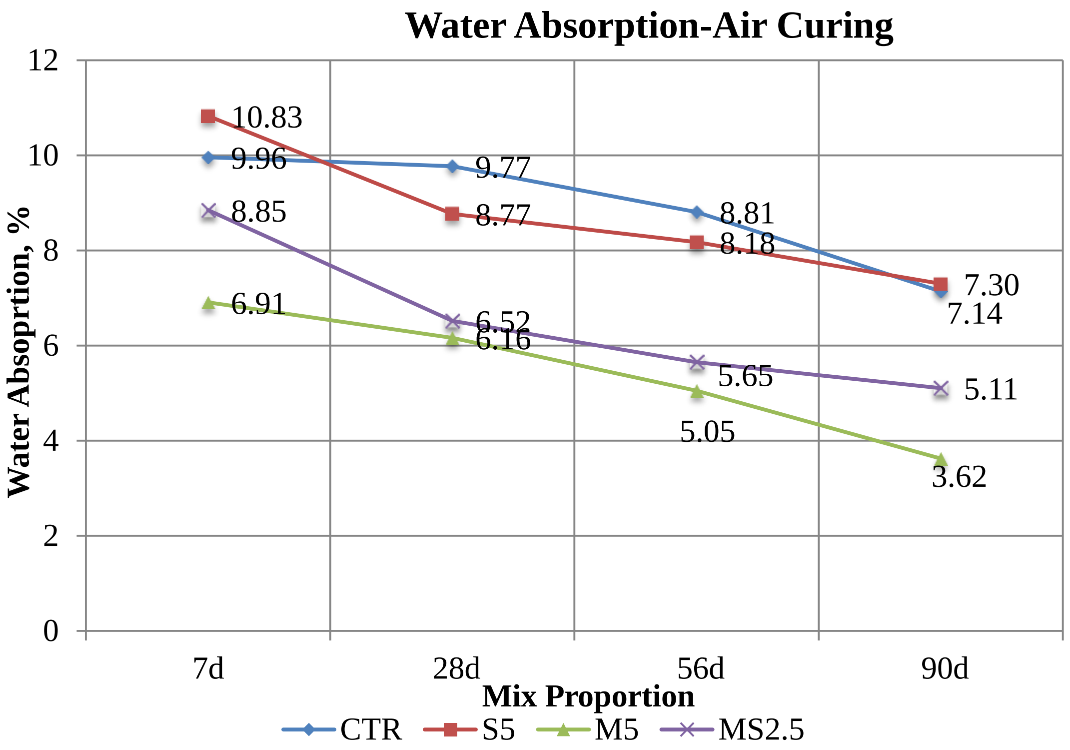


Figure 4.20: Water Absorption for All Mix Proportions under Air Curing

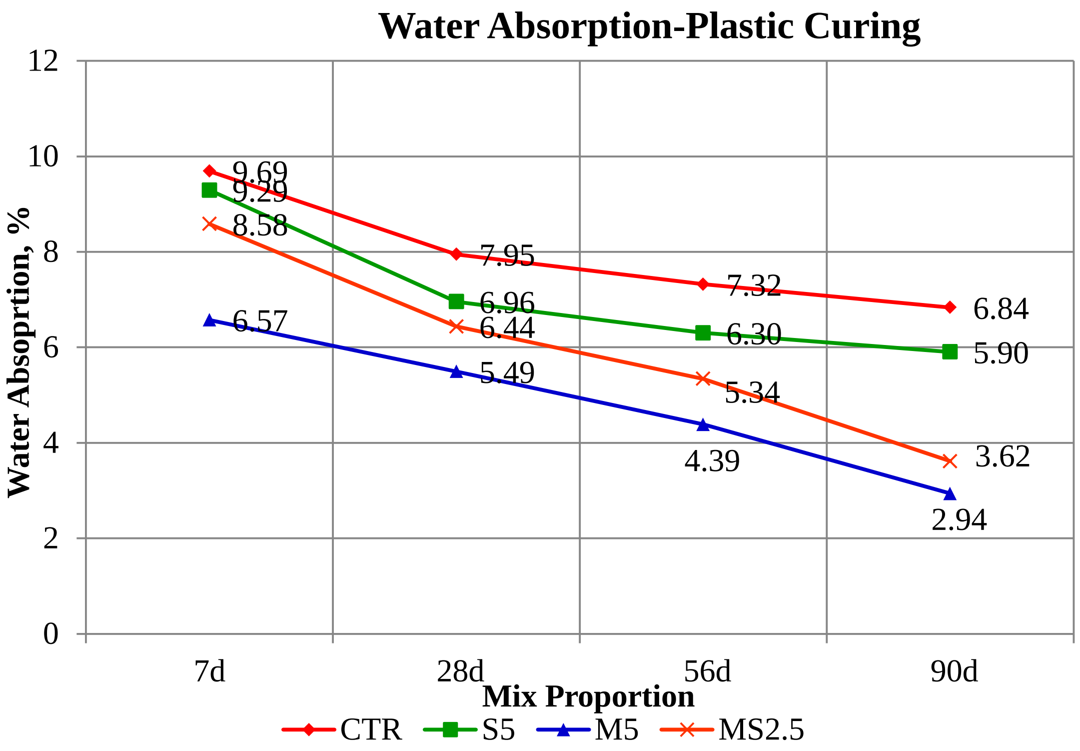


Figure 4.21: Water Absorption for All Mix Proportions under Plastic Curing

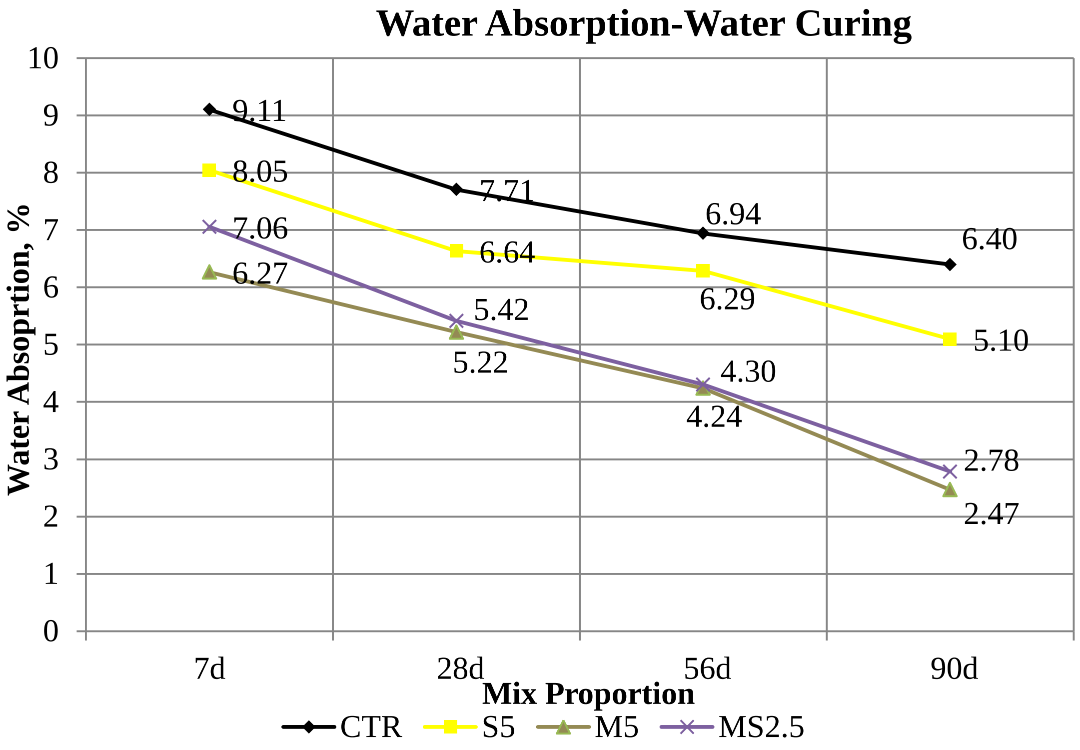


Figure 4.22: Water Absorption for All Mix Proportions under Water Curing

Generally, the water absorption of mortar specimens decreases with increasing curing age for all types of mix proportions under all curing regimes. This is because the hydration of cement together with pozzalanic reaction occurs over time and their products gradually fill the pores which have been initially occupied by mixing water. The filling effect decreases the amount of void within the mortar matrix, thus leads to lower water absorption.

Similar to the results of ISAT and sorptivity test, mortar incorporated with slag and MK offers better quality than control mix in term of water absorption. However, exceptions are detected for air-cured specimens. S5 mix exhibits higher water absorption (10.83%) than control mix (9.96%) at early age of 7 day. At 90 day, water absorption of S5 mix is also 0.16% higher than the control mix. Overall, M5 mix produces most durable mortar in term of water absorption, followed by MS2.5 mix and S5 mix. Control mix has the lowest durability among the four mix proportion used.

The filling effect of slag and MK is the main contributor to lower water absorption. Both slag and MK particles are smaller than cement grains so they are able to fill in the void between larger particles to reduce the porosity. Hence, the pore structure becomes denser and amount of water-penetrable void decrease. Once again, MK with smaller particle than slag provides better filling effect and reduces the porosity at greater extent than slag does. Besides, smaller and less complex MK particles offer ample surface area which allows pozzolanic reaction to occur at a more rapid and greater rate. Thus, more secondary C-S-H gel is produced to fill the voids so lower water absorption is yielded.

4.6 Correlation between Durability Test Results

A simple linear regression modelling approach is used to determine the correlations between the results of three different tests. This model attempts the relationship of two variables by fitting a linear equation to the data. The best fitting line is calculated and fitted using the method of least squares. A value, known as correlation coefficient, measures the strength of the relationships between two variables. The correlation coefficient has a value from -1 to 1. Positive and negative value indicates positive and negative relationship between two variables respectively. A correlation coefficient with absolute value closer to 1 represents there's a stronger relationship between two variables.

The linear regression model is produced using Microsoft Excel in which the best-fit linear regression line and correlation coefficient (R^2) is computed automatically and presented. The linear regression model is carried out for three sets of variables as followed: (i) ISA-10 against water absorption; (ii)Sorptivity against water absorption; (ii) ISA-10 against sorptivity.

4.6.1 ISA-10 against Water Absorption

The linear regression model of ISA-10 against Water Absorption is presented in Figure 4.23 .

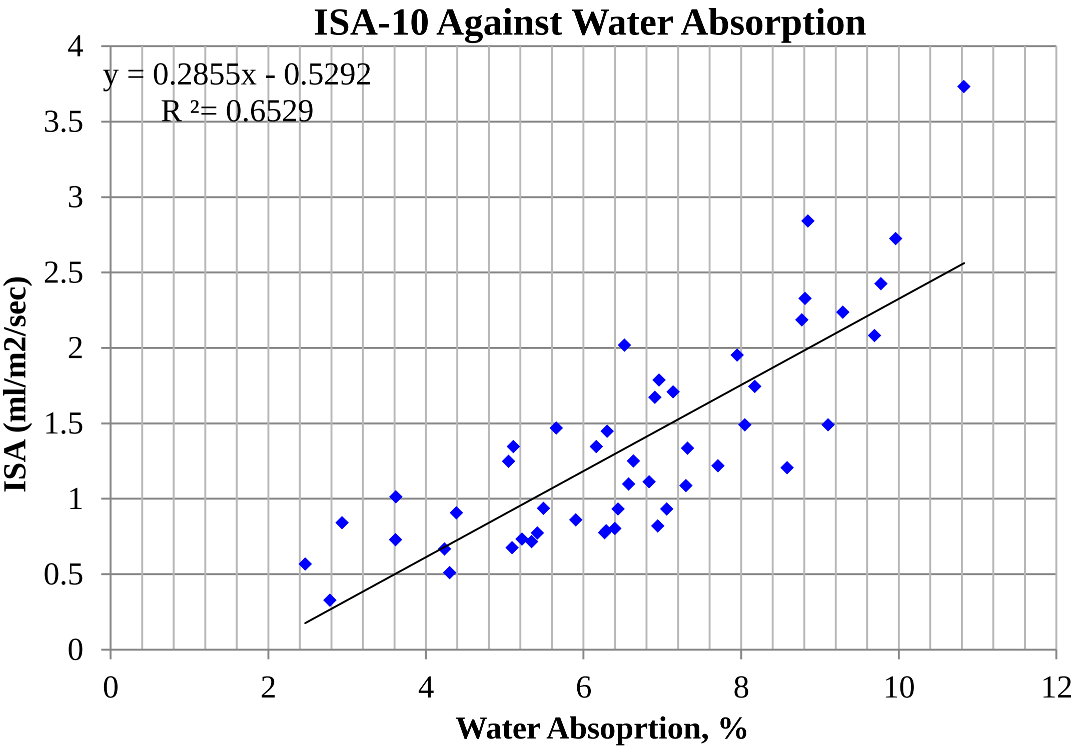


Figure 4.23: Variation of ISA-10 with Water Absorption

The ISA-10 of mortar has a medium strength of relation with water absorption with R^2=0.6529. The water absorption is an indicator to the porosity of the mortar specimens. Hence, it can be inferred that the initial surface absorption of mortar is not strongly affected by its porosity. This is because ISA greatly depends on the surface permeability rather than the overall porosity of mortar.

4.6.2 Sorptivity against Water Absorption

The linear regression model of Sorptivity against Water Absorption is presented in Figure 4.23 .

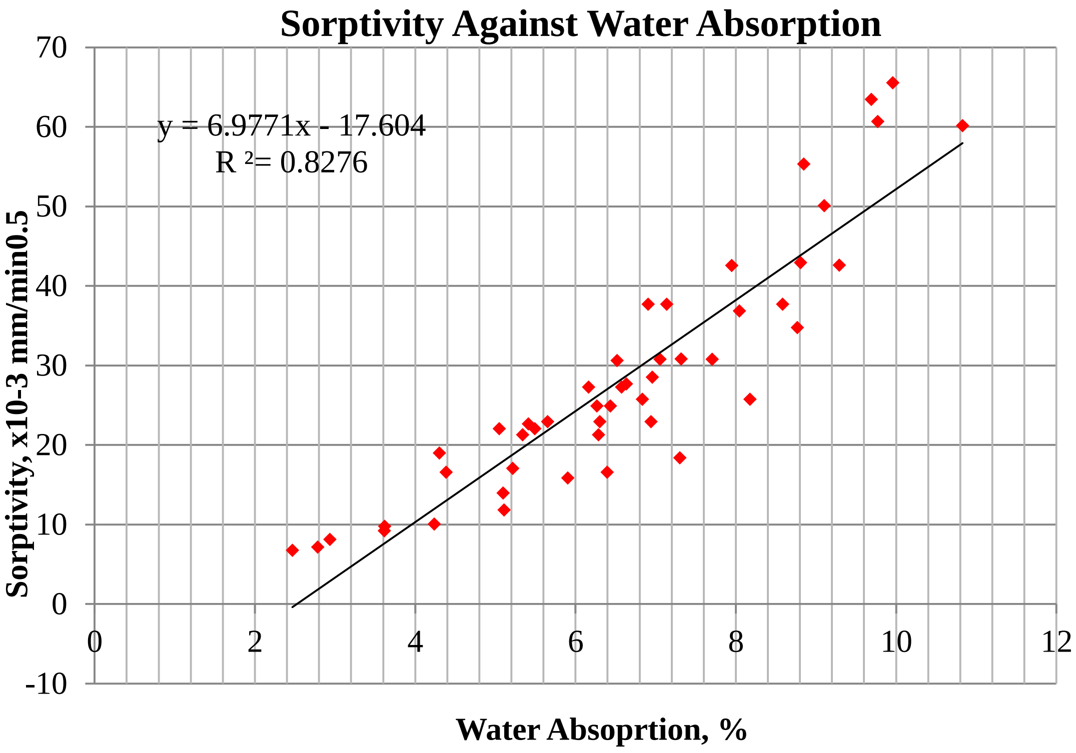


Figure 4.24: Variation of Sorptivity with Water Absorption

The above linear regression reveals that sorptivity of mortar has a relatively strong relationship with water absorption, with R^2 greater than 0.8.

4.6.3 Sorptovity against Water Absorption

The linear regression model of sorptivity against Water Absorption is presented in Figure 4.23 .

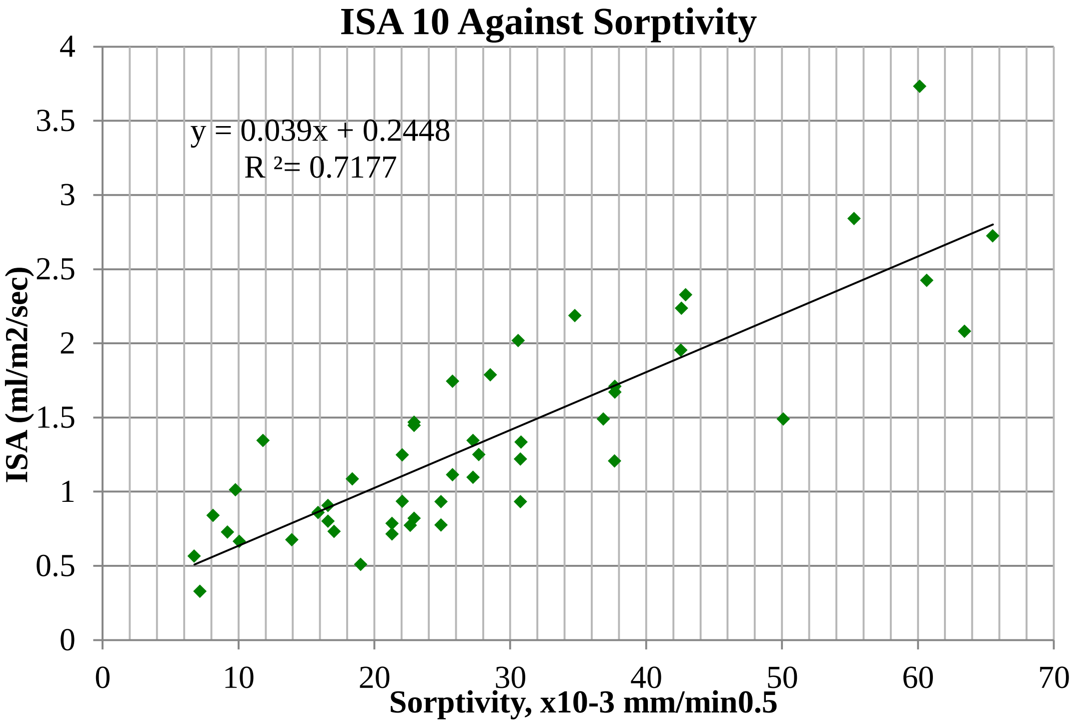


Figure 4.25: Variation of ISA-10 with Sorptivity

Based on hat sorptivity of mortar has a moderate relationship with water absorption, with R^2 =0.7177.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusion

Based on the laboratory results and the objectives established at the beginning of the study, a few conclusions can be derived.

The first objective aims to determine the effect of different curing regimes on the durability of mortar. Water curing is determined to be the best curing method, followed by plastic curing. Air curing produce least durable mortars

In accordance to second objective which investigate the effect of MK and GGBFS on durability of mortar, the inclusion of GGBFS and MK generally improves the durability of mortar in terms of ISAT, sorptivity and water absorption.

From the results of three durability tests performed, MK is discovered to perform better than GGBFS to produce higher quality and more durable mortar. This accomplishes the third objective aims to compare the effect of GGBFS on durability against MK.

Finally, the results obtained denote that GGBFS improves the workability while MK decreases the workability of fresh mortar as compared to control mix. The last objective to study and compare the effect of GGBFS and MK on workability is satisfied.

5.2 Recommendations

The scope of this study is not sufficiently broad to fully understand the effect of curing regimes as well as the effect of incorporating slag and MK on the durability of mortar. Therefore, a few recommendations to advance the associated investigation are as followed:

1. Cure mortar specimens in a closed room and control the surrounding humidity to study its effect to mortars' durability.
2. Perform curing of mortar specimens under different temperature to access the effect of curing temperature.
3. Adopt more and different replacement levels of cement by GGBFS and MK in order to determine the optimum dosage to produce the most durable mortar.
4. Perform other types of durability tests, for instance, rapid chloride permeability, sulphate resistance test and carbonation test with phenolphthalein to determine other durability properties of mortar.
5. Carry out scanning electron microscopy (SCM) to provide a microscopic view on pore structure of mortar to provide a better understand on the effect of curing regime and admixture on pore structure of mortar.
6. Use different grading for sand, say, use 80% of sand with particle size between 300 µm and 600 µm and sand with particle size less than 300 µm for the remaining 20%.

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APPENDICES

APPENDIX A: Sample Calculation Using Mix Design Sheet (Sherbrooke Method)

The properties of raw materials and designed properties of the mix were as followed:

1. Cement Used: Type I Portland Cement
2. Superplasticizer: Total solid contents=30% and specific gravity=1.05
3. Fine aggregate: A natural siliceous sand with G_{SSD}=2.63, w_{abs}=1.7%, w_a=0%
4. Metakaolin: G_c =2.52, used at 5% replacement level of total cementitious material
5. Dosage of superplasticizer: 1.1%
6. Water cement ratio: 0.45
7. Total cementitious material: 400kg per cubic metre of mix
8. Sand to cement ratio=2.0
9. Volume of mortar batch to produce: 0.01m^3
10. Assumed air content=1.0%

Part A of the mix design sheet was filled. All the characterizing values of materials to be used were reported in Part A of the mix design sheet and the moisture content of fine aggregate was calculated. The design properties of the mix were reported in box 1 to box 5 in Part B of the design sheet.

Mixture:	5% Metakaolin	Date	

Part A : Properties of Materials

	G_c	%
OPC	3.14	95
Slag	3.10	0
Metakaolin	2.52	5

Aggregate	G_{SSD}	W_{tot} (%)	W_{abs} (%)	W_c (%)	
Coarse	2.80	0.00	0.80	-0.8	$w_c = w_{tot} - w_{abs}$
Fine	2.63	0.00	1.70	-1.7	
Superplasticizer:	DARACEM				

G_{sup}	Solid content, s (%)	M_{sol} (kg/m^3)	V_{liq} (l/m^3)	V_w (l/m^3)	V_{sol} (l/m^3)
1.05	30	4 16	14 23	10 20	4 13

Part B: Design

W/CM	0.45	1
Total cementitious, CM (kg/m^3)	400	2
Sand to Cement Ratio	2.00	3
Superplasticizer dosage, d (%)	1.10	4
Volume of concrete batch, (m^3)	0.010	5

	1	2	3	4	5	6
Material	Content (kg/m^3)	Volume (l/m^3)	SSD condition (kg/m^3)	Water correction (l/m^3)	Composition (kg/m^3)	
					per 1m^3	Trial Batch
Water	180 6	180 6	180 6		183 22	1.83 24
Cement	380 7.1	121 10.1	380 7.1		380 7.1	3.80 25.1
Slag	0 7.2	0 10.2	0 7.2		0 7.2	0 25.2
Metakaolin	20 7.3	8 10.3	20 7.3		0 7.3	0.20 25.3
Coarse aggregate	0	0	0	0	0	0
Fine aggregate	800 8	304 11	800 15	14 19	786 18	7.86 26
Air	1.00 9	10 12	0			
Superplasticizer	1.10 4	4 13	4 16	-10 20	14 l 23	0.140 l 27
Total		627 14	1384 17	3 21		14 28

Box 6: The water content per cubic metre of mix was calculated:

$$water\ content = water/cement\ ratio \times CM$$

$$water\ content = 0.45 \times 400 = 180kg$$

Box 7.1-7.3: The mass of each cementitious material was calculated by multiplying the total cementitious, CM with the its respective percentage appeared in part A.

OPC = 400 x 95/100 = 380kg

Metakaolin (MK) = 400 x 5/100 = 20kg

Box 8: The mass of fine aggregate is calculated:

M_f = CM x sand to cement ratio = 400 x 2.0 = 800kg

Box 9: A volume of 1.0% entrapped air was assumed.

Now, the column 1 had been filled. Before column 2 was filled, the mass and volumes related to the superplasticizer in box 16, 23, 20 and 13 were calculated and recorded in Part A of the mix design sheet.

Box 16: $M_{sol} = CM \times \frac{d}{100} = 400 \times \frac{1.1}{100} = 4.4kg$

This value is rounded to 4.0 when filling the design sheet.

Box 23: $V_{liq} = \frac{M_{sol}}{s \times G_{sup}} \times 100 = \frac{4.4}{30 \times 1.05} \times 100 = 13.96 l/m^3$

This value is rounded to 14.0 when filling the design sheet.

Box 20: $V_w = V_{liq} \times G_{sup} \times \left(\frac{100-s}{100}\right) = 13.96 \times 1.05 \times \left(\frac{100-30}{100}\right) = 10.26 l/m^3$

This value is rounded to 10.0 when filling the design sheet.

A negative sign was added to this value when the box in Part B was filled.

Box 13: $V_{sol} = V_{liq} - V_w = 13.96 - 10.26 = 3.70 l/m^3$

This value is rounded to 4.0 when filling the design sheet.

Now, proceed to the Part B of the mix design sheer and the missing values in column 2 can be filled in.

Box 10: The volume of each cementitious material was calculated:

$$OPC = \frac{M_{OPC}}{G_{c,OPC}} = \frac{380}{3.14} = 121.02 l/m^3$$

“121” was written in box 10.1.

$$Metakaolin = \frac{M_{MK}}{G_{c,MK}} = \frac{20}{2.52} = 7.94 l/m^3$$

“8” was written in box 10.3.

Box 11: Volume of fine aggregate:

$$Vol. of\ fine\ aggregate = \frac{M_f}{G_{SSD}} = \frac{800}{2.63} = 304.18 l/m^3$$

“304” was written in box 11.

Box 12: The volume of entrapped air was:
1.5 x 10 = 15 l/m^3

“15” was written in box 12.

Box 14: The sum of all numbers appearing in column 2 was:

180 + 121 + 8 + 304 +10 + 4 = 627 l/m^3

Box 15: The SSD mass of the sand is:

304.18 x 2.63 = 799.99kg/m^3

This value is rounded to 800 when filling the design sheet.

Box 17: The unit mass of this mortar was calculated by adding all values appearing in column 3.

180 + 380 + 20 + 800 + 4 = 1384kg/m^3

“1384” was written in box 17.

Box 18: As the fine aggregate was dry and would absorb mixing water, a smaller mass less than 800kg must be weighed to compensate this lost from mixing water. For w_c = -1.7%, the corrected weight of fine aggregate was:

$$M_f = M\ (in\ box 15) \times \left(1 + \frac{w_c}{100}\right) = 800 \times \left(1 + \frac{-1.7}{100}\right) = 786.4 kg$$

“786” was written in box 18.

Box 19: The water correction for fine aggregate was:

box 15 – box 18 = 799.99 – 786.4 = 13.59kg

“14” was written in box 19.

Box 21: The correction to be applied to water was determined by adding algebraically the numbers appearing in column 4:

$13.59 + (-10.26) = 3.33$ l/m^3

“3” was written in box 19.

Box 22: The necessary weight of mixing water per m^3 to be measured is:

$180 + 3.33 = 183.33$kg (Note: 1l of water has a weight of approximately 1kg)

“183” was written in box 22.

Now, all the numbers in column 5 have to be multiplied by the volume of batch to be produced (as appearing in box 5) to obtain the mass of each material to be weighed to make the trial batch. For a mortar batch with volume of $0.01m^3$:

Mixing water:	183.33 x 0.01 = 1.833 → 1.83	box 24
OPC:	380 x 0.01 = 3.80	box 25.1
Metakaolin:	20 x 0.01 = 0.20	box 25.3
Fine aggregate:	786.4 x 0.01 = 7.864 → 7.86	box 26
Superplasticizer:	13.96 x 0.01 = 0.1396 → 0.14	box 27

Box 28: The values in box 24 to 27 were added:

$1.83 + 3.80 + 0.20 + 7.86 + 0.14 = 13.83$kg

“14” was written in box 30.

To check the calculation:

1384 (box 17) x 0.01(box 5) = 13.84kg

Since these two values were very close to each other, the calculation was verified