

**SMART CONSTRUCTION MATERIALS
INCORPORATING MICROENCAPSULATED PHASE
CHANGE MATERIALS**

Mohammad Al-Shdaifat

A Thesis
in
The Department
of
Building, Civil and Environmental Engineering

Presented in Partial Fulfillment of the Requirements
for the Degree of Master of Applied Science (Civil Engineering) at
Concordia University
Montréal, Quebec, Canada

August 2019

© Mohammad Al-Shdaifat, 2019

CONCORDIA UNIVERSITY

School of Graduate Studies

This is to certify that this thesis prepared

by: Mohammad Al-Shdaifat

Entitled: SMART CONSTRUCTION MATERIALS INCORPORATING
MICROENCAPSULATED PHASED CHANGE MATERIALS

and submitted in partial fulfillment the requirements for the degree of

Master of Applied Science (Civil Engineering)

complies with the regulations of the University and meets the acceptable standards with respect to originality and quality.

Signed by the final examining Committee:

_____	Chair
_____	Supervisor
Dr. A. Soliman	
_____	Examiner
Dr. M. Nokken	
_____	Examiner External (to program)
Dr. Mehdi Hojjati	
_____	Examiner
Dr. Biao Li	

Approved by

Dr. M. Nokken, GPD
Department of Building, Civil and Environmental Engineering

Dr. Amir Asif, Dean
Faculty of Engineering and Computer Science

Date

ABSTRACT

SMART CONSTRUCTION MATERIALS INCORPORATING MICROENCAPSULATED PHASED CHANGE MATERIALS

Canada has a cold climate that experiences freezing temperatures over long periods of the year, which impedes in-situ construction activities or puts them to a halt, resulting in considerable socioeconomic losses. Hence, construction companies have shifted to precast concrete systems in order to continue work throughout this harsh weather condition. However, applying heat curing and controlling the concrete temperature during the fabrication process at a favorable temperature of 10°C to 32°C is the main challenge for the precast concrete sector. Therefore, in this dissertation, mixtures for selected construction materials related to precast industry (i.e. cement-based and alkali activated concrete) were optimized/developed to have less energy intensive curing process with the aid of phase change materials (PCMs). Microcapsulated phase change materials (MPCMs) were used in order to avoid chemical interaction between PCMs and binding materials. The experimental program focused on evaluating fresh, mechanical, thermal and durability performance for these construction materials with different types and additional rates PCMs. Mixtures without PCMs were also test for comparison purpose. Results illustrated the role of MPCMs in storing and releasing the heat either generated from the hydration process or applied during curing process. The changes in heating rates during hydration reaction induced by MPCMs had influence of hydration kinetics for used binding materials. The performance of MPCMs will be alerted based on the binding materials. Thermal properties for tested construction materials, especially heat capacity, had enhanced by the addition of MPCMs. However MPCMs addition

had induced reduction in strength, adjusting mixtures design and producing dense microstructure would help in minimize this adverse effect. It is anticipated that these results will pave the way for wider implementation for PCMs in construction materials, which in turns will result in both economic and environmental benefits on the industrial and social levels. On the industrial level, this advance will speed up the production process while saving costs associated with heat curing leading to high production-cost efficiency and maximized profits. On the social level, it will reduce the carbon footprint for the precast concrete industry through decreasing its energy demand and fossil fuel consumption leading to a better life quality for Canadians.

ACKNOWLEDGEMENT

First and foremost, praises and thanks to God, the Almighty, for the showers of blessings throughout my research work which without nothing would have been accomplished. Throughout this master's thesis, I have received a great deal of support and assistance. I am happy to express my sincere gratitude to all of them now.

I would first like to sincerely thank my supervisor, Dr. Ahmed Soliman, without whom I would not have earned the opportunity to further my education at Concordia University. His integral view of research, keen enthusiasm, patience during the whole trip, and timely help led to the successful completion of this work. I would particularly like to single out my supervisor Dr. Ahmed Soliman, I want to thank you for your excellent cooperation and for all of the opportunities I was given to conduct my research and further my dissertation. Your advice, dedication, and patience are truly appreciated and are skills I hope to assimilate in my future endeavors.

Special thanks go to my *mother, father, sisters, and brother* for their prayers, love, support, and motivation. *You are always there for me.* In addition, there are my colleagues and friends, who were of great support in deliberating over our problems and findings, as well as providing a happy distraction to rest my mind outside of my research.

I'm extending my thanks to Lafarge Canada and ENCAPSYS LLC for supplying some of the materials used in this research.

Table of Contents

List of Figures	IX
List of Tables	XVI
List of Abbreviations	XVII

CHAPTER 1 INTRODUCTION

1.1 Background	1
1.2 Objectives and Scope	3
1.3 Structure of Thesis	4
1.4 Original Contributions	5

CHAPTER 2 LITERATURE REVIEW

2.1 Industrial Issues	7
2.2 Definitions of Phase Change Materials.....	9
2.3 Theory of Phase Change Materials	9
2.4 Phase Change Materials Types	12
2.5 Phase Change Materials Containment Methods	14
2.5.1 Microencapsulation	15
2.5.2 Macro-encapsulation.....	17
2.5.3 Shape Stabilization.....	19
2.5.4 Porous Inclusion.....	21
2.6 Phase Change Materials in Construction Materials (Mortars).....	22
2.7 Applications in Construction	24

CHAPTER 3 PERFORMANCE OF CEMENT MORTAR INCORPORATING PHASE

CHANGE MATERIALS AT AMBIENT CURING CONDITIONS

3.1 Experimental Work.....	27
3.2 Materials	28

3.3	Testing and specimens preparation	31
3.4	Results and Discussion	39
3.4.1	Flowability	39
3.4.2	Density	41
3.4.3	Compressive Strength	42
3.4.4	Tensile Strength	46
3.4.5	Heat of Hydration.....	49
3.4.6	Thermal Transfer.....	52
3.4.7	RCPT.....	58
3.4.8	Sorptivity or Absorption	59
3.4.9	Volume of Permeable Voids	66

CHAPTER 4 PERFORMANCE OF CEMENT MORTAR INCORPORATING PHASE CHANGE MATERIALS CURED AT HIGH TEMPERATURE

4.1	Introduction.....	69
4.2	Experimental Work.....	70
4.2.1	Materials	70
4.2.2	Testing and specimens preparation	70
4.3	Results and Discussions	71
4.3.1	Compressive Strength	71
4.3.2	Tensile Strength	83
4.3.3	Heat of Hydration.....	85
4.3.4	Heat Profile	91

CHAPTER 5 PERFORMANCE OF ALKALI-ACTIVATED MORTAR INCORPORATING PHASE CHANGE MATERIALS

5.1	Introduction.....	98
5.2	Experimental Work.....	100
5.2.1	Materials	100
5.2.2	Testing and Specimens Preparation	101

5.3	Results and Discussions	102
5.3.1	Followability	102
5.3.2	Compressive Strength	103
5.3.3	Heat of Hydration.....	109
5.3.4	Heat Profile	110
5.3.5	Thermal Transfer.....	113
5.3.6	Sorptivity and Absorption.....	118
5.3.7	RCPT.....	122

CHAPTER 6 PERFORMANCE OF CEMENT AND ALKALI-ACTIVATED MORTAR INCORPORATING PHASE CHANGE MATERIALS IN SUBFREEZING CONDITION

6.1	Introduction.....	124
6.2	Materials	125
6.3	Testing and Specimens Preparation	126
6.4	Results and Discussions	127
6.4.1	Compressive Strength	127
6.4.2	Heat Profile	135

CHAPTER 7 CONCLUSIONS AND FUTURE WORK

7.1	Conclusions.....	141
7.2	Future Work.....	143
	References.....	144

List of Figures

Figure 2.1 Phase Diagram. [Adapted from (Kuznik, 2011)]	10
Figure 2.2 Description of Microcapsule	16
Figure 3.1 Sieve analysis of fine aggregate	30
Figure 3.2 Particles size range for used MPCMs (obtained using SEM XXX at Concordia)	31
Figure 3.3 Flow table test apparatus	32
Figure 3.4 UTM	33
Figure 3.5 Direct tensile test apparatus	34
Figure 3.6 Calmetrix I-CAL 2000 HPC	35
Figure 3.7 RCPT testing apparatus	36
Figure 3.8 Absorption test setup [Adapted from (ASTM 1585 – 13)]	37
Figure 3.9 Thermal transfer specimens setup	38
Figure 3.10 SEM test setup	38
Figure 3.11 MPCM 28	40
Figure 3.12 MPCM 43	40
Figure 3.13 Reduction in average flow table readings for incorporated mortars vs control mortar	41
Figure 3.14 Hardened density for all mixtures with and without MPCMs	42
Figure 3.15 Compressive strength development for mortars with and without MPCM 28 incorporation under ambient condition	43
Figure 3.16 Compressive strength development for mortars with and without MPCM 43 under ambient condition	44

Figure 3.17 Capsuled MPCMs inside mortar matrix	45
Figure 3.18 MPCM microcapsules breakage.....	46
Figure 3.19 Tensile strength development for mortars with and without MPCM 28 incorporation under ambient condition	47
Figure 3.20 Tensile strength development for mortars with and without MPCM 43 incorporation under ambient condition	48
Figure 3.21 Heat flow OPC samples with and without MPCM 28 incorporation under ambient condition	51
Figure 3.22 Heat flow OPC samples with and without MPCM 43 incorporation under ambient condition	52
Figure 3.23 Thermal behavior of control OPC specimen.....	54
Figure 3.24 Thermal behavior of 3% MPCM 28 OPC specimen.....	54
Figure 3.25 Thermal behavior of 6% MPCM 28 OPC specimen.....	55
Figure 3.26 Thermal behavior of 12% MPCM 28 OPC specimen.....	55
Figure 3.27 Thermal conductivity for Ch1	57
Figure 3.28 Total Charge Passed.	59
Figure 3.29 Initial Sorptivity OPC/OPC 3% MPCM.....	61
Figure 3.30 Initial Sorptivity OPC/OPC 6% MPCM.....	62
Figure 3.31 Initial Sorptivity OPC/OPC 12% MPCM.....	63
Figure 3.32 Secondary Sorptivity OPC/OPC 3% MPCM.	64
Figure 3.33 Secondary Sorptivity OPC/OPC 6% MPCM.	65
Figure 3.34 Secondary Sorptivity OPC/OPC 12% MPCM.	66
Figure 3.35 Average Volume of Permeable Voids (%).....	67

Figure 4.1 Different stages for heat curing regime	71
Figure 4.2 Compressive strength for control mortar under ambient temperature, hot temperature for 5 hours, & hot temperature for 10 hours	72
Figure 4.3 Compressive strength for mortar incorporated MPCM 28 under hot temperature for 5 hours.....	73
Figure 4.4 Compressive strength for mortar incorporated MPCM 28 under hot temperature for 10 hours.....	74
Figure 4.5 Compressive strength 6% MPCM 28 incorporating mortars under different curing regimes	75
Figure 4.6 Compressive strength for control mortars & MPCM 28 incorporated mortars at 28 days under different curing conditions.....	76
Figure 4.7 Compressive strength for mortar incorporated MPCM 43 under hot temperature for 5 hours.....	77
Figure 4.8 Compressive strength for mortar incorporated MPCM 43 under hot temperature for 10 hours.....	78
Figure 4.9 Compressive strength for control mortars & MPCM 43 incorporated mortars at 28 days under different curing conditions.....	79
Figure 4.10 Compressive strength for mortar incorporated MPCMs 43&28 under 5 hours hot temperature curing	81
Figure 4.11 Compressive strength for mortar incorporated MPCMs 43&28 under 10 hours hot temperature curing	82
Figure 4.12 Mortar incorporated MPCM 28 tensile strength under 5 hours hot temperature	83
Figure 4.13 Mortar incorporated MPCM 28 tensile strength under 10 hours hot temperature.	84

Figure 4.14 Mechanical strength development for mortar incorporating MPCM Compressive/Tensile.....	85
Figure 4.15 Heat flow for control OPC & 3% MPCM 28 OPC, both under hot temperature.....	87
Figure 4.16 Heat flow for Control OPC & 6% & 12% MPCM 28, Both Under Hot Temperature	88
Figure 4.17 Heat flow for control OPC and 3% MPCM 43 OPC, both under hot temperature. ..	89
Figure 4.18 Heat flow for 3% MPCM 43 & 12% MPCM 43 OPC, Both Under Hot Temperature	90
Figure 4.19 Heat flow for OPC 12% MPCM 43 & OPC 12% MPCM, both under hot temperature	91
Figure 4.20 Heat profile test setup.....	92
Figure 4.21 Temperature profile for control OPC samples for 5/10 hours 60 °C.....	93
Figure 4.22 Temperature profile for control OPC samples for 5/10 hours 60 °C and 12% MPCM 28 for 5 hours	95
Figure 4.23 Temperature profile for control OPC samples for 5 hours 60 °C and 12% MPCM 43 for 5 hours.....	96
Figure 4.24 Temperature profile for control OPC sample for 5 hours 60 °C and 12% MPCM Mix (MPCM 43 & MPCM 28) for 5 hours	97
Figure 5.1 Reduction in average flow table readings for incorporated AAM mortars vs control AAM mortar.....	103
Figure 5.2 Compressive strength development for AAM mortar incorporated MPCM 28 under ambient temperature.....	104
Figure 5.3 AAM bonding with MPCMs.....	105

Figure 5.4 Compressive strength development for AAM mortar under different curing conditons	106
Figure 5.5 Compressive strength development for AAM mortar incorporated MPCM 28 under 5 hours hot curing	107
Figure 5.6 Compressive strength development for Geopolymer mortar incorporated MPCM 28 under 10 hours Hot Temperature	108
Figure 5.7 Heat flow for control AAM with and without MPCM 28 incorporation under hot temperature	110
Figure 5.8 Temperature profile for control geopolymer samples for 5 hours 60 °C	111
Figure 5.9 Temperature profile for control OPC samples for 5/10 hours 60 °C and 12% MPCM 28 for 5 hours	113
Figure 5.10 Thermal behavior a) control AAM specimen and b) 3% MPCM 28 AAM specimen	115
Figure 5.11 Thermal behavior a) 6% MPCM 28 AAM specimen and b) 12% MPCM 28 AAM specimen	116
Figure 5.12 Initial Sorptivity Geo/Geo 3% MPCM.....	118
Figure 5.13 Initial Sorptivity Geo/Geo 6% MPCM.....	119
Figure 5.14 Initial Sorptivity Geo/Geo 12% MPCM.....	119
Figure 5.15 Secondary Sorptivity Geo/Geo 3% MPCM.	121
Figure 5.16 Secondary Sorptivity Geo/Geo 6% MPCM.	122
Figure 5.17 Secondary Sorptivity Geo/Geo 12% MPCM.	122
Figure 5.18 Total charge passed for AAM specimens with and without the incorporation of MPCMs.....	123

Figure 6.1 Curing Regime.....	127
Figure 6.2 Compressive strength development for OPC mortar incorporated MPCM 28 under AMB then freezing condition	128
Figure 6.3 Compressive strength development for OPC mortar incorporated MPCM 6 under AMB then freezing condition	129
Figure 6.4 Compressive strength development for OPC mortar incorporated MPCM 6 under hot temperature 10 hours then freezing condition	130
Figure 6.5 Compressive strength development for OPC mortar incorporated MPCM -10 under AMB then freezing condition	131
Figure 6.6 Compressive strength development for OPC mortar incorporated MPCM -10 under hot temperature 10 hours then freezing condition	132
Figure 6.7 Compressive strength development for AAM mortar incorporated MPCM -10 under AMB then freezing condition	133
Figure 6.8 Compressive strength development for AAM mortar incorporated MPCM -10 under hot temperature 10 hours then freezing condition	134
Figure 6.9 Temperature profile for OPC specimens incorporating MPCM -10 exposed to hot cure regime	136
Figure 6.10 Temperature profile for AAM specimens incorporating MPCM -10 exposed to hot cure regime.....	137
Figure 6.11 Temperature profile for AAM specimens incorporating MPCM -10 exposed to AMB curing regime	138
Figure 6.12 Temperature profile for OPC specimens incorporating MPCM 6 specimens exposed to AMB regime	139

Figure 6.13 Temperature profile for OPC and AAM specimens incorporating MPCM -10 exposed to hot cure regime	140
---	-----

List of Tables

Table 2.1 Comparison between Sensible and Latent Heat Storage.	9
Table 2.2 Organic PCM in literature [Adapted from (Kuznik, F. 2011)].....	12
Table 2.3 Inorganic PCM in literature [Adapted from (Kuznik, F. 2011)]	13
Table 2.4 Advantages and disadvantages of Organic and Inorganic PCMs.	14
Table 2.5 Commercially Used PCMs [Adapted from (Marani, 2019)].....	16
Table 3.1 Chemical and physical properties of cement	29
Table 3.2 MPCM 28°C & MPCM 43°C properties	29
Table 3.3 Mixture compositions for mixtures with MPCMs, volumetric ratio	30
Table 3.4 Temperature variation between Ch #1 and Ch #3 at 70 °C	57
Table 3.5 Chloride Ion Penetrability Based on Charge Passed (ASTM C 1202).....	59
Table 5.1 Chemical composition and physical properties of GBFS.....	101
Table 5.2 Temperature variation between Ch1 and Ch3 at 70 °C	117
Table 6.1 MPCMs Properties.....	126

List of Abbreviations

AA	Alkali-Activated
AAB	Alkali-Activated Binder
AAM	Alkali-Activated Mortars
PCM	Phase Change Material
MPCM	Microencapsulated Phase Change Material
OPC	Ordinary Portland Cement
C-S-H	Calcium Silicate Hydrate
GGBFS	Ground Granulated Blast Furnace Slag
LWA	Lightweight Aggregate
OPC	Ordinary Portland Cement
PC	Portland Cement
AMB	Ambient Temperature
SEM	Scanning Electron Microscopy
UTM	Universal Testing Machine

CHAPTER ONE

INTRODUCTION

1.1 Background

Energy is key to most of today's activities and the whole running economy, production, and daily life. With such a growing economy and uprising technologies, energy consumption has become one of the world's challenging problems. The needs and demands for energy are increasing, along with limited energy resources. For instance, consumption of world petroleum and other liquid fuels are expected to increase by 38% between 2010 and 2040 (Sieminski, 2014). Hence, there is an urgent need to reduce energy consumption by developing new methods for energy saving in different sectors. This can be achieved through utilizing new technologies, updating procedures and methodologies to proper energy consumption, and implement new materials with energy-saving ability.

On the other hand, energy production and consumption have their own negative and dramatically ever-increasing on the planet's environment. The raw sources of energy like fossil fuels burning induced a considerable amount of carbon dioxide (CO₂) pollution, and has the largest share of global energy-related carbon emissions (Sieminski, 2014).

The construction sector, as part of the energy consumers, has gotten the chance to contribute to the energy-saving concept. Energy consumption in buildings is mainly directed to achieve thermal comfort. Hence, preventing or minimizing energy transfer between buildings and surrounding

environments will effectively reduce energy loss (Rao, 2018). Using the thermal mass of buildings has been acknowledged as a significant energy saving technique in buildings (Ruud, 1990; Braun, 2001). Increasing the thermal mass of the building by implementing engineered materials with a high ability to store energy is getting popular. One of the promising new materials is the phase change materials.

Using phase change materials (PCMs) has become one of the main areas of study in the last decade. Phase change materials are well known for their properties of absorbing and desorbing heat. The simple principle of PCMs is changing from the solid-state to the liquid state as the temperature increases. This involves an endothermic reaction; so that the PCMs absorb heat and store it (called latent heat). As the temperature decreases, an exothermic reaction takes place and PCMs phase change from liquid to solid while desorbing heat (Jayalath, 2016). It was found that PCMs can store a significant amount of thermal energy and specific heat capacity for buildings, compared to other thermal storage techniques (Schossig, 2005; Tyagi, 2011). Recently, many researchers had focused on PCMs incorporation in different types of building materials (Lu, 2017). For instance, Alqallaf (2013) investigated PCM in concrete roofing. Shilei (2007) integrated PCM in building walls. Moreover, the research effort by Ansuini (2011) integrated PCM in the flooring of a 16m² room, pursuing to achieve thermal comfort. The main idea was to control the indoor temperature to delay the peak temperature, in consequence, conserve the used energy for indoor conditioning purposes.

Recently PCM incorporation moved towards the cementitious materials, as a significant part of the construction materials. They are taking into consideration the fact that the construction industry is a growing industry all over the world (Ortiz, 2009). The ever-growing population of the world combined with the political, social, and economical factors all together serves along with several

different reasons to drive population to live in urban areas (Cohen, 2015). This influential growth led to increasing demand for more infrastructure (Doyle, 2009). One of the world most growing construction materials consumed is concrete (Miller, 2016), and the second most consumed substance on earth after water (Roskos, 2011).

Moreover, the concrete production industry is responsible for 5 percent of the carbon dioxide emission worldwide (Müller, 2008). With this information about the concrete industry and technologies, it's important to develop new combinations and materials to tackle the energy consumption principle. As part of the growing production, precast concrete is being dominant in the market. During this process, heat and energy are used to accelerate the hydration process, and the strength gains development criteria. Phase change materials are promising materials when it comes to this portion of the production. It may help in storing some heat and releasing it back to the concrete medium during the first 24 hours curing, which will lead to extending the higher temperature and strength gain in shorter periods of heating.

1.2 Objectives and Scope

The main goal of this thesis is to investigate the effects of incorporating microencapsulated PCMs (MPCMs) in different construction materials on their performance under various curing conditions. This will be achieved through:

1. Introducing an essential knowledge of the MPCMs inclusion in cement-based and alkali-activated based mortars.
2. Inspecting of the effects of MPCMs incorporation in mortars under different curing conditions (including ambient, warm, and cold).
3. Criticizing the influence of MPCMs inclusion on the structure of the mortar.

To help to address objectives above, the scope of this research includes modifying the standard mortar mixtures to incorporate MPCMs as a partial replacement of sand by volume. Various percentages of MPCMs, namely 3%, 6%, and 12%, were investigated. Moreover, different types of dry powder paraffin core MPCMs with different phase-change temperatures, namely 28 °C, 43°C, 6°C and -10°C were tested. The first two types were used to study the effects on mortars exposed to hot-moderate climatic environments. The last two were used to develop mortars exposed to subfreezing conditions (i.e. under zero °C).

1.3 Structure of Thesis

This thesis has been prepared according to the guidelines of the Faculty of Graduate Studies at Concordia University. It comprises seven chapters, five of which display the progress in the experimental program starting by enhancing the fundamental knowledge about phase change materials and its production techniques, moving to introduce and evaluating its potential in a new application. Substantial parts of these chapters have been either published, accepted, or submitted for possible publication in peer-reviewed technical journals and national and international conferences.

Chapter 2 contains a state-of-the-art review of the current knowledge on PCMs properties, their relationships, influences of various production and implementation techniques in construction materials. Chapter 3 discusses the interaction mechanisms between MPCMs and cementitious materials under ambient curing conditions. Chapter 4 introduces a new concept of using MPCMs in extending the curing process for heat-cured cement-based mortar. The underlying phenomena and experimental results that demonstrate the concept are presented in this chapter. The performance of the new concept and its efficiency for alkali-activated materials are described in Chapter 5. The benefits of adding different MPCMs at cold conditions simulating winter weather

are presented in Chapter 6. Finally, general and specific conclusions drawn from the research study, along with recommendations for future research are included in Chapter 7.

1.4 Original Contributions

This research introduces a series of fundamental investigations related to phase change materials and their role in enhancing the performance of different construction materials under various exposure conditions. It explores the influence of a wide range of curing conditions and the efficiency of available PCMs products. It proposes a new strategy to extend curing while achieving adequate performance with a smaller environmental impact. Specific original contributions of this dissertation include:

1. Developing an extensive database on the available phase change materials and their applications in the construction sector.
2. Identifying the interaction mechanisms between different types of binding system and MPCMs. Specifically, it was revealed that: (i) the state of the phase change materials (i.e. liquid or solid) is going to control the performance; (ii) the small size of MPCMs can enhance the hydration process through providing nucleation sites; (iii) broken MPCMs will increase voids leading to lower strength; and (iv) MPCMs are stable in high alkalinity environments.
3. Evaluating the performance and efficiency of different MPCMs and their interaction with the surrounding environment. Specifically, it was found that: (i) under ambient curing condition, the heat liberated from hydration reactions is enough to activate the PCMs and its ability to store heat while this heat will be released during the cooling cycle; (ii) under heat curing, increasing the temperature will increase lead to complete transfer of PCMs

from the solid to liquid stage. Hence, the maximum heat storage is achieved. (iii) During the cooling cycle, PCMs will alternate the cooling pass by releasing heat.

4. Developing for the first time a curing technique capable of improving the mechanical properties through extending the hydration process at cold weather along with reducing solving the economic and environmental problems associated with energy consumption. More specifically: (i) the concept of using low melting point MPCMs (i.e. 6 °C and -10 °C) was investigated; (ii) it was demonstrated for the first time that MPCMs has a high potential for extending curing process through securing adequate temperature for hydration.

CHAPTER TWO

LITERATURE REVIEW

2.1 Industrial Issues

Energy consumption is one of the world most rivet fields for studies and research. Energy consumption is ever-growing as well as the demand for energy. In parallel with the growing industries and energy demands, more significant environmental impacts are widening. Higher greenhouse gas emissions, especially carbon dioxide (CO₂) which causes global warming, climate changes, and ozone layer depletion (Jeon, 2013). The building sector is on the highest energy consumption and environmental impacts. According to the International Energy Agency, the building sector is responsible for about 30% of the energy consumption and around 40% of the CO₂ emissions. The main reason behind this large energy consumption is the changes in living standards and life comfort, in terms of heating and cooling for both hot and cold regions (Soares, 2013).

Consequently, many governmental and international policies had established standards to reach less energy consumption and fewer pollutant environs (Lu, 2017). The need for novel materials that can enhance the energy efficiency for building envelopes has gained a high interest recently. Different energy-saving tools with high energy efficiency had been developed. One of the useful tools is thermal energy storage. Thermal energy storage has three different ways: chemical heat, sensible heat, and latent heat. Chemical heat mechanism is based on breaking and forming for

molecular bonds. Sensible heat is through heating and cooling for a material. Latent heat is exchange by a material during melting and solidifying. Latent heat reflects the materials' heat storage capabilities and sensitivity for temperature variations (Kalnæs, 2015). This is the controlling mechanism for phase change materials (PCMs).

Phase change materials are operating due to a simple mechanism, changing the state according to the change in the exposed temperature. As temperature increases, it meltdown, changing the state from solid to liquid, so absorbing and storing energy. Reciprocally, when the temperature decreases, it solidifies changing the state from liquid to solid; therefore, releasing the formerly mentioned stored energy (Pomianowski, 2013). Hence, a reasonable choice of PCMs to incorporate in building materials would be with great benefits, namely, controlling temperature fluctuation, enhancing thermal comfort, as well as reducing energy consumption. Thus, utilizing such materials, especially in extremely hot or cold regions, is with enormous benefits toward a lower energy consumption level (Nkwetta, 2014). Research on PCMs started in 1970 and studies on some of these materials applications, and implications were published in 1975 in the USA. Later in 1978, the idea of using PCMs in a wall what is known as Trombe wall was discussed by Telkes (1978). Roughly, it was the start for utilizing PCMs in building structural and non-structural elements.

In the last decade, PCMs has earned lots of interest, especially for the incorporation in building materials. PCMs was integrated into walls, floors, ceilings, windows, indoor applications, outdoor applications, mainly in building components (Kalnæs, 2015). Also, it was used in heating and cooling units (Akeiber, 2016). Moreover, PCMs utilization in building materials has developed on wallboards, mortar, and concrete (Cui, 2017). Mostly, the studies focused on PCM utilization in

mortar. A wide range of investigation developed to address the thermal benefits from PCMs utilization (Rao, 2018).

2.2 Definitions of Phase Change Materials

The phase change materials are materials that can change from solid to liquid when temperature increases, and vice versa (Kuznik, 2011). As temperature increases, an endothermic reaction takes place; so that the PCMs absorb heat. As temperature decreases, an exothermic reaction occurs, so that PCM desorbs heat and materials change phase from liquid to solid. In this technology, the main characteristic is that materials temperature remains constant during the phase change (Theoretically) (Kuznik, 2011). Either sensible or latent heat storage can accomplish thermal energy storage. **Table 2.1** demonstrates the main differences between latent and sensible heat storage mediums.

Table 2.1 Comparison between Sensible and Latent Heat Storage.

	Sensible heat storage	Latent heat storage
<i>Definition</i>	Heat stored by changing the temperature of storage medium such as (water, air, oil, rock beds, bricks, concrete or sand), is stored as sensible heat.	In latent heat storage, the material stores heat while changing phase. The phase change "solid-to-liquid" is the most used, but also solid-to-solid change is of interest.
<i>Main Difference</i>	It needs a large amount of materials (mass) to store the same amount of energy for latent.	It stores larger amount of energy per unit mass.

2.3 Theory of Phase Change Materials

The phase change theory for the pure ideal body was defined by Kuznik (2011) as follows: an area in the space of the thermodynamic parameters (Temperature, pressure, and volume) of the system

composed uniquely of the pure body, in which the free energy is an analytical function. Generally, any matter can be found under several states. The most common three states are liquid, solid, and gas. The state corresponds to the phase, which led to the terms “liquid Phase,” “Solid Phase,” and “Gas-Phase.” Considering a fixed volume (V) of the system and the abscissa is the temperature (T), and the ordinate is the pressure (p), there are three phases represented on the phase diagram, as an example in **Fig. 2.1**. During heat exchange, phase change materials pass by solidification process, where materials transform from liquid to solid, nor with the fusion; where materials transform from solid to liquid.

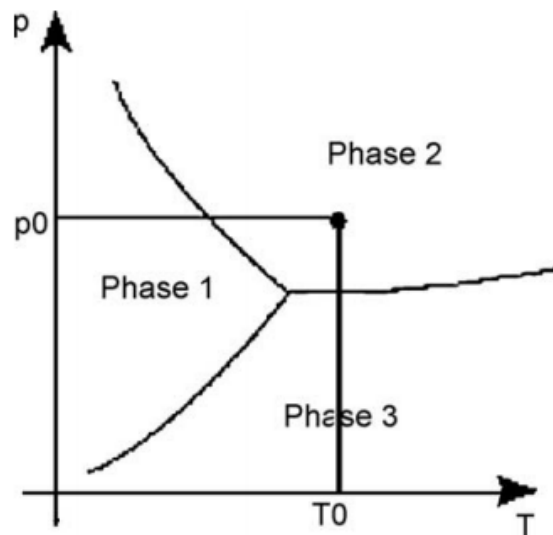


Figure 2.1 Phase Diagram. [Adapted from (Kuznik, 2011)]

Defining the solidification effect of the PCMs is essential, especially when dealing with low nucleation rate materials. For instance, solidification of PCMs begins with the nucleation by forming initial crystals, then propagating to form the solid phase. The nucleation rate is the capability of the material to produce a nucleus when the temperature reduces below the fusion temperature. Materials with a low nucleation rate can remain in the liquid phase even after the crossing of the fusion temperature, while the solidification starts later.

The phase change materials (PCMs) have been popularized lately as a way to enhance thermal properties for construction materials. Incorporation of PCMs in construction materials (i.e. concrete, mortar, or gypsum) directly contributes to their thermal mass and serve as a regulator for the buildings indoor temperature (Zhou, 2012). As any immersing material being imported to construction materials, PCMs has its pros and cons. For instance, the enhancement for temperature regulating and heat evolution to avoid thermal cracking in cementitious materials using PCMs have been explored by Fernandes (2014). The authors state that PCMs addition does not influence cementitious materials reactions to progress; however, it helps in controlling thermal fluctuations and temperature change in cementations materials. The thermal mass storage, hydration process, and thermal shrinkage for cementitious materials were also studied by Schossig (2005); Khudhair (2004). Other research had studied curling stresses in concrete pavements Sharifi (2018). Freezing-thawing resistance was investigated to increase the service life of bridge decks using PCMs by Sakulich (2011). Freezing-thawing resistance for geopolymer concrete and Portland cement concrete containing micro-encapsulated phase change materials were also investigated by Pilehvar (2019).

On the other hand, PCMs incorporation in construction materials has its imperfections and drawbacks (Marani, 2019). One of the drawbacks was reported by Hunger (2009) as a leakage from the broken capsules of the paraffin core of PCMs (Gschwander, 2005). PCMs will suffer leakage if they are added to construction materials without encapsulation (encapsulation processes and materials will be discussed later). Strong and structurally stable materials are needed to overcome the problem of leakage and to overcome the fire hazard of paraffin cores (Cui, 2017). Nevertheless, PCMs incorporation affects fresh and hardened properties of construction materials as will be discussed in more details in the following sections.

2.4 Phase Change Materials Types

There are three main categories, organic, inorganic, and eutectic mixtures or non-eutectic mixtures. Organic materials mainly from Paraffin carbohydrate, lipid-derived, and non-paraffin. Although inorganic materials from salt hydrates (MnH_2O). **Tables 2.2** and **2.3** summarize properties for a list for some organic and inorganic materials; respectively.

Table 2.2 Organic PCM in literature [Adapted from (Kuznik, F. 2011)]

<i>Materials</i>	<i>T_f</i> (°C)	<i>H_f</i> (kJ/kg)	<i>C_{ps}</i> (kJ/kg/K)	<i>C_{pi}</i> (kJ/kg/K)	<i>k_s</i> (W/ m ² /K)	<i>k_s</i> (W/ m ² /K)	<i>References</i>
GR25	23.2-24.1	45.3	1.2	1.2	n.a.	n.a.	(Ahmad, 2006: 2007)
PEG600	22	127.2	n.a.	2.49	n.a.	0.189	(Ahmad, 2006)
<i>n</i> -Octadecane	27	243.5	1.934	2.196	0.358	0.148	(Alawadhi, 2008)
<i>n</i> -Eicosane	37	241	2.01	2.04	0.15	0.15	(Alawadhi, 2008)
Butyl stearate	19	140	n.a.	n.a.	n.a.	n.a.	(Athienitis, 1997: Lee, 2000)
ERMEST2325	17-20	138	n.a.	n.a.	n.a.	n.a.	(Banu, 1998: Scalat, 1996)
RT27	26-28	179	1.8	2.4	0.2	0.2	(Borreguero, 2010: Castell, 2010)
MICRONAL26	26	110	n.a.	n.a.	n.a.	n.a.	(Castellón, 2009: Cabeza, 2007)
RT20	22	172	n.a.	n.a.	n.a.	n.a.	(Fang, 2006)
MP65%-MS35%	21.8-24.5	175	n.a.	n.a.	n.a.	n.a.	(Feldman, 1995)
MP77%-MS23%	22.4-23.8	177	n.a.	n.a.	n.a.	n.a.	(Feldman, 1995)
MP93%-MS7%	22.2-22.8	182	n.a.	n.a.	n.a.	n.a.	(Feldman, 1995)
GR41	43	63	n.a.	n.a.	0.15	0.15	(Huang, 2006)
GR27	28	72	n.a.	n.a.	0.15	0.15	(Huang, 2006)
Eutectic capric-myristic	21.7	155	n.a.	n.a.	n.a.	n.a.	(Karaipekli, 2008)
MICRONAL 5001	26	110	n.a.	n.a.	n.a.	n.a.	(Konuklu, 2009)
MICRONAL 5008	22	110	n.a.	n.a.	n.a.	n.a.	(Konuklu, 2009)
Heptadecane	22	214	n.a.	n.a.	n.a.	n.a.	(Koschenz, 2004)
MPCM28-D	28	180-195	n.a.	n.a.	n.a.	n.a.	(Lai, 2010)
UNICERE55	45-60	185	n.a.	n.a.	n.a.	n.a.	(Lee, 2000)
<i>n</i> -Nonadecane	31.8	160	n.a.	n.a.	n.a.	n.a.	(Li, 2010)
Eutectic capric-stearic	24.7	179	n.a.	n.a.	n.a.	n.a.	(Sari, 2008)
Non-eutectic capric-lauric	19.2-20.3	144-150	n.a.	n.a.	n.a.	n.a.	(Shilei, 2007)
U3	28	244	n.a.	n.a.	0.28	0.22	(Voelker, 2008)
U4	13.6-23.5	104.5-107.5	4	4.1	0.18	0.22	(Kuznik, 2009)
RT25	25	147	2.9	2.1	1.02	0.56	(Weinläder, 2005)

Table 2.3 Inorganic PCM in literature [Adapted from (Kuznik, F. 2011)]

<i>Materials</i>	<i>T_f</i> (°C)	<i>H_f</i> (kJ/kg)	<i>C_{ps}</i> (kJ/kg/K)	<i>C_{pi}</i> (kJ/kg/K)	<i>k_s</i> (W/m ² /K)	<i>k_s</i> (W/m ² /K)	<i>References</i>
<i>Eutectic salt</i>	32	216					(Carbonari, 2006)
<i>SP25A8</i>	25-26	180	2.5		0.6	0.6	(Castell, 2010)
<i>Calcium chloride Hexahydrate</i>	26.-29	175	2.3	1.4	1	1	(Evers, 2010)
<i>Sodium thiosulfate U1</i>	40-48	210	1.46	2.4			(Hadjieva, 200)
<i>U2</i>	30-32.5	131					(Medina, 2008)
<i>CaCl₂·6H₂O</i>	26-28	188	1.44	1.44	1.09	0.54	(Pasupathy, 2008)
<i>S27</i>	29.8	191					(Voelker, 2008)
<i>L30</i>	27	190	1.5	2.22	0.79	0.48	(Weinläder, 2005)
	30	270	1.23	1.79	1.02	0.56	(Weinläder, 2005)

Each group of PCMs has its properties (Baetens, 2010). Organic phase change materials have two groups, Paraffin, and Non-Paraffin (i.e., fatty acids, esters, alcohols, and glycols). The paraffin group characterized by low cost; thermal storage density of 120 kJ/kg up to 210 kJ/kg, melting temperature range from approximately 20 °C up to about 70 °C; chemically inert; low vapor pressure; no phase segregation; low thermal conductivity (0.2W/m K) and large volume changes during the phase transition. On the other hand, the non-paraffin group is around three times more expensive than the paraffin one. It is also characterized by relatively low melting point, high latent heat of fusion; small volume changes during phase transition; no super-cooling and lack of materials with phase transition around the comfort temperature of 21°C. **Table 2.4** summarizes the main advantages and disadvantages of paraffin and non-paraffin phase change materials.

Table 2.4 Advantages and disadvantages of Organic and Inorganic PCMs.

	Organic PCM	Inorganic PCM
<i>Advantages</i>	<ul style="list-style-type: none"> • Availability in a large temperature rang. • Freeze without much supercooling. <ul style="list-style-type: none"> • Ability to melt congruently Self-nucleating properties. • Compatibility with conventional material of construction. <ul style="list-style-type: none"> • No segregation. • Chemically stable. • The high heat of fusion. • Safe and non-reactive. <p>Recyclable.</p>	<ul style="list-style-type: none"> • High volumetric latent heat storage capacity. • Low cost and easy availability. <ul style="list-style-type: none"> • Sharp phase change. • High thermal conductivity. <ul style="list-style-type: none"> • Non-flammable.
<i>Disadvantages</i>	<ul style="list-style-type: none"> • Low thermal conductivity. • Low volumetric latent heat storage capacity. <p>Flammable (depending on containment).</p>	<ul style="list-style-type: none"> • High volume change. <ul style="list-style-type: none"> • Super cooling. • Segregation.

2.5 Phase Change Materials Containment Methods

Several methods for PCMs incorporation in the materials had been investigated, including impregnation; shape stabilized PCMs, micro-encapsulation, and macro-encapsulation (Marani, 2019). Considering its interaction with the surrounding medium. The Impregnation is produced by mixing PCMs and the building material (Khudhair and Farid, 2004). For instance, for building walls, PCMs is directly impregnated into gypsum, concrete, or other porous materials. Porous materials transport the heat through pores leading to changes in its mechanical properties. Although small pores occupied by PCMs lead to the prevention of solid crust loss. Distinctly a major leakage was reported by Xiao (2002). Moreover, the interaction between PCM and the

hosting material is another drawback reported by Cabeza (2007). Hence, other techniques were proposed for PCMs incorporation.

2.5.1 Microencapsulation

The Micro-encapsulation method is based on encasing the PCMs in microscopic polymer capsules. This method has been widely used as a useful technique for the incorporation of PCMs within construction materials. To avoid chemical reactions between capsules and building materials; special attention must be given to capsules materials selection. Capsules are small enough to avoid isolation of PCMs solid crust (Kuznik, 2011). Several parameters were inspected by Hawlader (2003) to evaluate the microencapsulation process, including encapsulation process duration, the quantity intervolved of PCMs in the solution for a satisfactory encapsulation. Generally, the quality of the microencapsulation is increased by the ratio of the produced capsule and the total mass of the PCMs powder (Kuznik, 2011).

Many studies had conducted to investigate the capsules process. The in situ polymerization was studied by Zhang (2005). It was reported that the strength of the beater in the manufacturing process leads to variation in the mean size of capsules. The in-homogeneities degree for capsules in the powder statistical analyzed by Sarier (2007).

Microencapsulation techniques can be divided into three groups. First, physical microencapsulation methods including; Pan Coating, Air-Suspension Coating, Spray Drying, Vibrational Nozzle, Centrifugal Extrusion, and Solvent Evaporation. Physic-chemical methods including; Ionic Gelation, Coacervation, and Sol-Gel. Lastly, Chemical methods including; Interfacial Polymerization, Suspension Polymerization, and Emulsion Polymerization (Jamekhorshid, 2014).

To conclude, **Fig. 2.2** illustrates the microcapsules which consist of a (core) solid or liquid, surrounded by continues polymeric materials the (shell) in the micrometer to millimeter range.

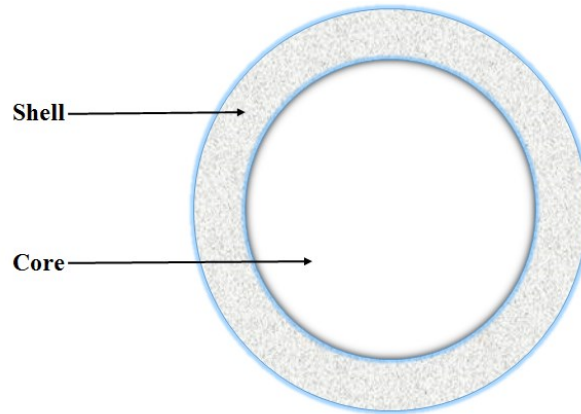


Figure 2.2 Description of Microcapsule

Wide varieties of microencapsulated PCMs have recently been developed and available in the market **Table 2.5**.

Table 2.5 Commercially Used PCMs [Adapted from (Marani, 2019)]

PCM Name	Medium	Reference
<i>Micronal DS 5001</i>	Concrete	(Franquet, 2014)
<i>Micronal DS 5001</i>	Concrete	(Figueiredo, 2016)
<i>Micronal DS 5001</i>	Concrete	(Eddhahak-Ouni, 2014)
<i>Micronal DS5040X</i>	Concrete	(Jayalath, 2016)
<i>Micronal DS5040X</i>	Concrete	(Pomianowski, 2014)
<i>Micronal, Microtek</i>	Concrete	(Wei, Z., 2017)
<i>Microtek MPCM 24</i>	Concrete	(Ricklefs, 2017)
<i>Microtek MPCM 24</i>	Concrete	(Falzone, 2016)
<i>Microtek MPCM 24</i>	Concrete	(Wei, 2017)
<i>Microtek MPCM 28</i>	Concrete	(Lecompte, 2015)
<i>Microtek MPCM 28</i>	Concrete	(Young, 2017)
<i>Micronal DS 5008</i>	Mortar	(Lucas, 2013)
<i>Micronal DS 5001</i>	Mortar	(Joulin, 2014)
<i>Micronal DS 5001</i>	Mortar	(Haurie, 2016)
<i>Inertek</i>	Mortar	(Bahrar, 2018)
<i>Devan Mikrathermic</i>	Mortar	(Kheradmand, 2014)
<i>Microtek MPCM 28</i>	Geopolymer	(Shadnia, 2015)
<i>Micronal DS 5001</i>	Plaster	(Lachheb, 2017)
<i>Micronal</i>	Gypsum	(Toppi, 2013)
<i>Micronal</i>	Sandwich Panel	(Castellón, 2010)
<i>Micronal DS5040X</i>	Concrete/LWA	(Tuncel, 2018)
<i>Micronal, Microtek</i>	Wood and Plastic	(Jamekhorshid, 2017)

During the last decade, micro-encapsulated (MPCMs) are commonly used with cementitious composites, and investigated by many researchers. Also, direct incorporation of MPCMs, particularly as a replacement for sand, were used. Some other researchers used the addition method (Meshgin, 2012). Replacement method showed better strength compared to additional methods. MPCMs in cementitious matrices may lead to several effects on the mechanical and thermal properties of such matrices, like compressive strength, tensile strength, specific heat capacity, and thermal conductivity, even to the durability performance of cementitious composites.

2.5.2 Macro-encapsulation

Macro-encapsulation is another technique similar to the micro-encapsulation. The main difference between these two methods is that in the later PCM is not mixed with the ingredients of the hosting medium like concrete and mortar, but it is added to the medium as an object or as a connecting element. This will prevent detrimental effects PCM has on the hosting medium properties along with improving the thermal performance of incorporated construction materials.

On the other hand, some studies referred the micro-encapsulation to other techniques like shape stabilization and PCM impregnation in lightweight aggregate (Memon, 2015; Cui, 2015). Other studies have used the shape stabilized PCMs and microencapsulated PCMs as their PCM prefabricated members (Zhou, 2007; Cai, 2015). As for any incorporation method, macro-encapsulation had some drawbacks. One of them is based on the location of PCM in the medium where it may solidify only at the edge, in other words, poor conductivity PCM outcome. Placing such materials in some locations may need protection against drilling, for example.

Moreover, the implementation of the technique needs to be performed in-situ (Memon, 2014). The two most common parts of construction materials which plays convincible successors are roofs and walls. Based on the places that they are located at, among construction map, as the first barriers facing the thermal influence.

Walls are mostly in direct contact with the thermal circumstances of the outdoor, as well as it is exposed to solar radiations. Thus several research efforts examined PCM micro-encapsulation with the building walls (Evola, 2013; Bastani, 2014). Noting that macro-encapsulation containers must have high thermal conductivity, parts as the panels are successful candidates for the macro-encapsulation (Shi and Memon, 2014). Steel containers for macro-encapsulation in concrete sandwich wall panels were used. Authors integrated PCM in a model room walls, contained in steel containers to evaluate the effect of such incorporation method on the indoor temperature fluctuations. They indicated that indoor room temperature was normalized, where the maximum temperature was significantly reduced, and the minimum temperature was significantly increased using such technique. Hence, the place, shape, and the nature of the hosting medium plays a pivotal part in such a technique. Also, the surrounding climatic circumstances; like sunlight and temperatures; as well as the thermal properties for hosting material will have effects. Several studies have been done on the walls brick focusing on thermal performance and temperature control (Vicente, 2014; Barzin, 2015). Other boundaries that are exposed to the thermal influences, which also provides beneficial candidates for PCM macro-encapsulation are roofs and floors.

The existence of hollow spheres, tubes, cylinders plunges the effectiveness of incorporating PCMs as a micro-encapsulation medium. Several studies have examined macro-encapsulation of PCM in hollow-core slabs (Royon, 2014; Navarro, 2015). The results of the research above concluded that PCM macro-encapsulated slabs did perform a charge and discharge for almost 70% of the summer and winter days, as well as, it was reported that such combination regulated the indoor temperature and worked as a thermal conditioner in the hot summer days. Moreover, macro-encapsulation PCMs were used for the completely outdoor application such as pavement as incorporated in

embedded metal pipes (Farnam, 2017). They were utilizing the effect of the latent heat storage to melt ice and snow.

2.5.3 Shape Stabilization

Overcoming the well-known PCMs disadvantage of its possible leakage and the interference with the hosting material ongoing reactions or properties, attracted many researchers. Various techniques were suggested. Among those techniques, shape stabilized PCM (SSPCMs) is one of the promising techniques. As for the micro-encapsulation, SSPCMs prevents the leakage of the PCM in its molten stage.

Moreover, it enhances the thermal conductivity, and cyclic thermal propriety of PCMs during melting/solidifying process (Marani, 2019). To stabilize the PCM in its molten stage, supporting materials are used. Several materials have been used as supporting material for PCMs. Such as Silica fume, Diatomite, Bentonite, Nano-silica, and expanded graphite. In the fabrication of SSPCMs, there are several methods used; namely; direct absorption or blending, vacuum impregnation as for the physical methods. Sol-gel, and graft copolymerization, as for chemical methods (Amaral, 2017). Considering the alternation of PCM during the shape stabilization method, a decisive decision should be made for choosing the right supporting material. This is coupled with an evaluation of the effects of the supporting material on the properties on the hosting medium for SSPCMs. Although with its difficulties and complexities, shape stabilization is proved to be one of the most favorable techniques (Zhai, 2013). Advantages of SSPCMS technique include large apparent specific heat attained, the suitable thermal conductivity, along with its reliability and stability while changing the phase over long periods (Akeiber, 2016; Fallahi, 2017). Another benefit for SSPCMs is the ability to incorporate as much as 80% PCMs of the total weight (Frigione, 2019).

Selecting the supporting materials for SSPCMs is with essential importance. Several materials have been used in research, either from an organic base or inorganic base. For example, high-density polyethylene (HDPE), styrene, and butadiene were tested (Khadiran, 2015). Some other research efforts benefited from the use of products as a sustainable privilege to the technique (Frigione, 2018). Subsequently, selecting supporting materials that have a porous structure and high thermal conductivity of the supporting materials is essential. Silica fume has been used as a supporting material by Jeong (2013); Kang (2015) as it was always used as a micro filter and pozzolanic addition. However, thanks for its porous structure which has made it proper for PCMs impregnation. The findings marked the compatibility of using silica fume as supporting material for PCM, as well as; it was capable of storing heat while it is thermally and chemically stable.

Nevertheless, SSPCMs did not contribute to the mechanical properties such as compressive strength of concrete and mortar (Min, 2017). Particularly, the thermal conductivity of cementitious materials with the incorporation of SSPCMs has different and contradictory reported results. For instance, Zhang (2013) studied several fabricated mortars mixtures incorporating SSPCMs (composed of 90% PCM by mass percentage with the n-octadecane/EG) at percentages by mass ranging from 0 to 2.5%. They indicated a decrease in the thermal conductivity as the SSPCMs content increased. This was attributed to the increase in mortar porosity. On the other side, Kim (2014) reported an increase in the thermal conductivity for the cement mortar incorporating SSPCMS (composed of hexadecane/xGnP). Accordingly, more extensive efforts still needed in this area.

Commonly the SSPCMs have been used for the energy storage capacity rather than mechanical or structural purposes. For non-structural members (Wallboards, hollow blocks, bricks), the

utilization of SSPCMs has been investigated under laboratory and real scales for energy consumption and temperature regulation (Biswas, 2014).

2.5.4 Porous Inclusion

This incorporation method is close to the SSPCMs. Comparatively, it is mainly fabricating a lightweight aggregate loaded with PCMs, using absorption of the liquid PCM into the porous lightweight aggregates (LWAs). The main differences for this incorporation method compared to the SSPCMs are; the implementation in the construction materials method for the porous inclusions does not work as a partial replacement. Moreover, the supporting materials for the latter, such as porous aggregates, are more extensive and have some capabilities characteristics pertaining the mechanical strength.

Some characteristics and factors must be studied carefully to produce PCM-LWA porous inclusions; namely; type of the LWA, the LWA absorption capacity, and LWA characterization and performance (Sakulich, 2012).

The high porous nature of the LWA made it a successful candidate for the inclusion of molten PCMs. Subsequently, the characteristics of the LWAs; include size, surface area, porosity, absorption capabilities, and impregnation method should be considered. Some studies have been conducted on employing the LWA as PCM host, such pumice and expanded clay (Aguayo, 2017). Findings highlighted the importance of considering the effect of pore diameter along with the porosity. Porosity itself is not the only factor controlling the performance. Pumice had lower porosity compared to the expanded clay, however, attained better PCM absorption capability, thanks for its larger pore size. Also, removing particles smaller than 150 μm among the used LWAs is recommended. Tiny particles derange the absorption of PCM into LWAs by their surface absorption. Nevertheless, attaining an adequate compressive strength is possible through using some supporting materials and coating to prevent the leakage like epoxy resin (Cui, 2015).

The absorption of the PCM in the LWA is an essential aspect of this method. Therefore, two main impregnation methods were adapted by researchers, vacuum impregnation, and direct impregnation. The impregnation method for LWAs is similar to that in SSPCMs. In the LWAs method, it is more explicit because of the size, absorption capacity, and pore size. The large size of the pores and the distribution of pores play as an influencing factor enhancing impregnation of LWAs compared to SSPCMs. Comparatively, vacuum impregnation has better results in terms of absorption compared to the direct impregnation. The absorption capacity for direct impregnation was 18% while using the vacuum impregnation resulted in an absorption capacity increase up to 73% (Memon, 2015). However, some research addressed it as a speculative method by its complexity and prolonged-time for implementing (Aguayo, 2017).

2.6 Phase Change Materials in Construction Materials (Mortars)

Regulating and minimizing energy consumption is the backbone for creating a novel composite to be used in the construction materials. The illustrious native of phase change materials in absorbing and desorbing heat stands as the main motive behind its incorporation of such materials. As building and industries are growing, energy recourses are depleting. Thus, utilization of innovative ideas became a must, presuming the balance and practicality for less energy consumption and more efficient resources exploitation.

The latent heat can be stored from the solar energy to reduce the thermal gradient between indoor and outdoor and consequently reduce loads on heating and cooling equipment (Frigione, 2019). Surface areas with large heat exchange faces provided by a variety of construction materials have guided the efforts to implement PCMs in lots of cementitious construction elements (Bentz, 2007). Mortar and concrete as cementitious construction materials and gypsum-based materials are widely used in several applications for indoor and outdoor purposes. As a result, the research

during the recent years for PCM incorporation has been mainly focused on PCM incorporation with cement and gypsum as binders, based on their mechanical and thermal properties and the quality control possibilities with such binders.

The abilities for PCMs incorporation in adding energy efficiency to building materials were analyzed by Coppola (2016). However, relatively good thermal properties were achieved with PCM inclusion; there was a loss on the mechanical strength as reported by Lecompte (2015). Accordingly, research efforts were tilted a little more to the indoor applications, where mechanical properties limitations are not a concern. Recently more research efforts directed to use other binders such as geopolymer, ariel lime, and hydraulic lime (Pavlík, 2014). For example, Wang (2016) tested slag with impregnated PCM to regulate the temperature fluctuations between indoor and outdoor. They concluded that PCM was used successfully with slag to improve temperature fluctuation as well as the cooling and heating.

Generally, adding PCMs to cementitious mediums was reported to increase thermal conductivity and energy storage (Kim, 2014). Also, most of the research studies reported a reduction in temperature fluctuation with PCM incorporation (Vieira, 2014). The heat storage capacity of the incorporated mortar is improved due to the incorporation of PCM. Also, the process of storing and releasing heat is improved, too (Snoeck, 2016).

Generally, adding PCMs to mortar affects its fresh and hardened properties. Several research works reported that an increased amount of water is needed to maintain the workability of mixtures by the increasing of PCM amount, due to the increase in water absorption, which is the reason of increased porosity for PCM incorporated mortars (Cunha, 2017). Aside from that, adding PCMs as spherical particles may contribute to the workability of the incorporated mortars by reducing the friction forces (Vieira, 2014). Accordingly, based on the advantages and disadvantages

mentioned formerly, the selection of the right PCMs (i.e., organic or inorganic) to tackle the desired properties is with great importance for the construction materials.

Several ways of adding PCM to the mortar have been developed and examined. A common way for PCM incorporation in mortar is the using of microcapsules. It was recommended by many researchers to have relatively balanced results in terms of thermal performance, mechanical performance, and cost (Richardson, 2017; Lucas, 2013). In their study, microstructural analysis for mortar incorporated MPCMs, showed that smaller particles filler effect contributed more in reducing the microporosity leading to a lower total porosity (Pavlik, 2013; Cunha, 2017). On the other hand, Ventolà (2013) reported a slight increase in the mechanical proprieties, and was attributed to the crystallization of some minerals like aragonite, which enhances mortar resistance. Other research efforts refereed the reduction in the mechanical strength to the leakage happens, which leads to an increase in the porosity (Haurie, 2016).

Increasing PCM content was reported to increase the latent heat transfer up to a specific limit (Cunha, 2013). Exceeding this limit leads to higher water demand, low mechanical strength, and high porosity, which hinder the heat transfer capabilities due to the nanopores induced by PCM incorporation (Nepomuceno, 2014).

2.7 Applications in Construction

Several applications have been tested and adapted by some researchers for PCMs incorporation in construction materials. Amongst all the applications, enhancing the thermal properties has gained the most interest. Integrating PCMs in construction materials proven its benefits to enhance the thermal energy storage for several elements (Frigione, 2019). So, PCMs incorporated in different elements, like wallboards, roofs, floors, paste, mortar, and concrete. In order to get the most energy benefits for construction materials. Commonly PCMs integrated with the interior parts of

buildings, to utilize the thermal comfort. Thereby, a proper choice of PCM will sufficiently absorb and release heat for an extended period of the day.

Wallboards are one of the first elements incorporated PCMs. For several reasons, wallboards and plasterboards are very suitable for PCMs incorporation, thanks for the ease of developing the combination. Alongside with its being widely used and cheap for lightweight construction applications. Furthermore, it provides a large surface area (Memon, 2014; Huang, 2017). Bricks were also incorporated PCMs, conventional and alveolar bricks. They monitored the heat profile for the incorporated elements. Moreover, the energy savings and impact on reducing CO₂ were studied (Castell, 2010; Song, 2018). For the roofing application, the principle was simple. PCMs were incorporated in a predesigned cylindrical hole in the roof, or just by adding a layer of mortar or concrete incorporated the PCMs. Increasing the amount of PCM in such applications is with significant benefits, alongside with the selection of the most suitable PCM temperature (Alqallaf, 2013). They have also stated that PCM would enhance the insulation performance and recommended future studies for the insulation using PCMs. As in the roofs and walls, floors also have a big surface area that could be with significant benefits in regulating the indoor thermal fluctuations. Several ways investigated in the incorporation of PCMs in floors (i.e. overlay of PCM panels as a substitution for the floor, or in layers of concrete installed forthright the flooring). Results showed that significant thermal advantages gained from the application, by the fact that a notable amount of heat is lost to the floor because of heat transmogrify with the ground. (Kasaeian, 2017, Johra, 2017).

Some other examples for PCMs applications in different construction elements; include windows and sunshades. Where PCMs integrated as a filler in some of their parts and cavities. Silva (2016) Investigated PCM incorporation in glazing and shading materials. These building materials

boundaries are essential areas for high-end research. A satisfactory result from the incorporation of PCMs in such elements was reported in terms of thermal comfort and temperature regulating. On the other hand, some drawback associated with PCMs in such elements, like the blurry windows and the reduction of daylight transmission alongside the solar radiation (Silva, 2016).

CHAPTER THREE

PERFORMANCE OF CEMENT MORTAR INCORPORATING PHASE CHANGE MATERIALS AT AMBIENT CURING CONDITIONS

The primary purpose of this Chapter is to examine how the incorporation of MPCMs influences the mechanical properties and durability performance of cementitious mortar cured at ambient conditions (i.e. temperature (T) = $20^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and relative humidity (RH) = $50\% \pm 5\%$). Two types of MPCM with different melting points (namely 28°C and 43°C) were used. The chapter highlights the correlation between increasing heat capacity and achieving adequate performance for MPCMs mortar. The collected data also will be used as a benchmark for other curing conditions discussed in following chapters.

3.1 Experimental Work

Extensive experimental work was conducted on cement mortars with and without MPCMs to evaluate fresh, hardened and durability properties. As discussed in Chapter 2, MPCMs can be added either as a replacement for one of the mixture ingredient or as an addition on the top of the mixture. In this dissertation, the replacement method was selected due in order to avoid the excessive reduction in mechanical performance as recommended in the literature.

3.2 Materials

General use (GU) hydraulic cement, according to the CSA-3001-03, was used as the binding material. **Table 3.1** shows the chemical composition for the used cement. Natural riverside sand with a fineness modulus of 2.70 according to ASTM C136 (2014), a specific gravity of 2.51 and water absorption of 2.73% determined following ASTM C 128 (2015) was used as fine aggregate. Sieve analysis for the sand meets ASTM C33 (2018) as illustrated in **Fig. 3.1**. The sand was added at a ratio of 2.75 by weight with respect to cement according to ASTM C109 (2014). Commercial microencapsulated phase change material (MPCMs) with a melting point of 28.0 ± 0.9 °C and 43.2 ± 0.9 °C were used. The MPCM has a paraffin core and polymeric outer shell (the capsule). The particle size of the MPCMs ranged from 3 to 30 μm (**Fig. 3.2**). **Table 3.2** summarizes the properties for the used MPCMs according to the manufacturer. MPCMs were added as partial replacement of sand by volume at rates 0%, 3%, 6%, and 12%. Water to cement ratio was kept constant at 0.4 for all mixtures. **Table 3.3** shows the composition for all tested mixtures. Mixtures were named according to type and percentage of MPCMs. The code of each mixture had a character “P” referring to phase change material, followed by two numbers. The first number refers to the type of MPCMs (i.e 28 °C or 43°C) and the second reflect the added percentages. For instance, mixtures incorporating 3% of 28 °C and 43 °C MPCMs will be named “P28-3” and “P43-3”, respectively. The control mixtures without MPCMs was referred to as “Control”.

Table 3.1 Chemical and physical properties of cement

		OPC (1)
SiO ₂	(%)	19.80
Al ₂ O ₃	(%)	4.90
CaO	(%)	62.30
Fe ₂ O ₃	(%)	2.30
SO ₃	(%)	3.70
Na ₂ O	(%)	0.34
MgO	(%)	2.80
C ₃ S	(%)	57.00
C ₂ S	(%)	14.00
C ₃ A	(%)	9.00
C ₄ AF	(%)	7.00
Na ₂ O _{eq}	(%)	0.87
Loss on ignition	(%)	1.90
Specific gravity	--	3.15

Table 3.2 MPCM 28°C & MPCM 43°C properties

MPCM 28	Manufacturing Properties
Melting Point (°C)	28
Latent Heat Capacity (J/g)	185
Moisture (%)	< 1%
Appearance	White Powder
MPCM 43	
Melting Point (°C)	43
Latent Heat Capacity (J/g)	175
Moisture (%)	< 3%
Appearance	White to Slightly off White Powder

Table 3.3 Mixture compositions for mixtures with MPCMs, volumetric ratio

	Mixture Code			
	Control	P3	P6	P12
Cement/cement	1	1	1	1
Sand/cement	2.75	2.65	2.57	2.41
PCMs (%)	0	3	6	12
Water/cement	0.4	0.4	0.4	0.4

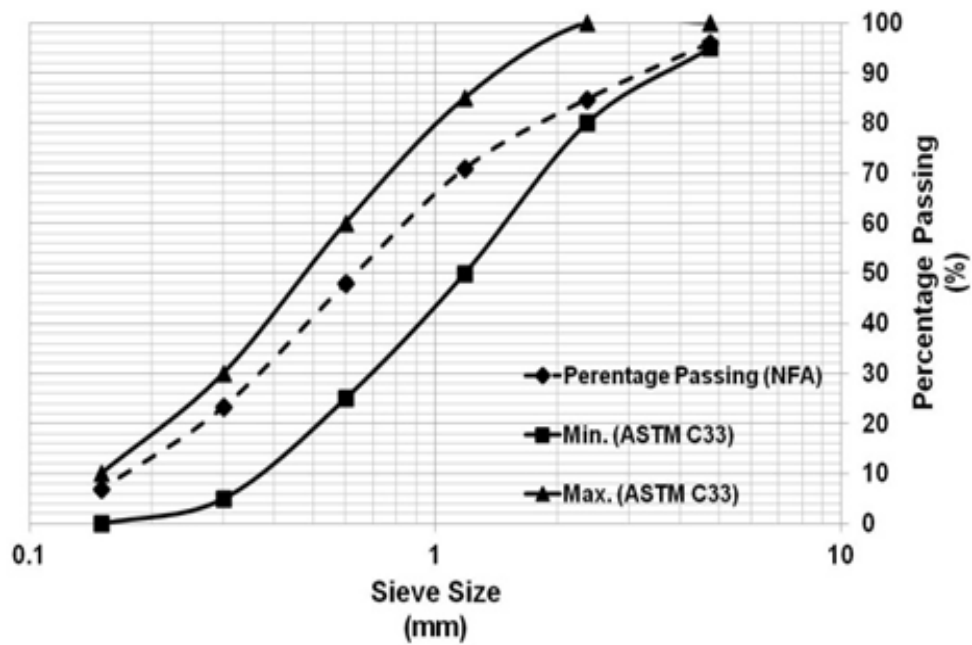


Figure 3.1 Sieve analysis of fine aggregate

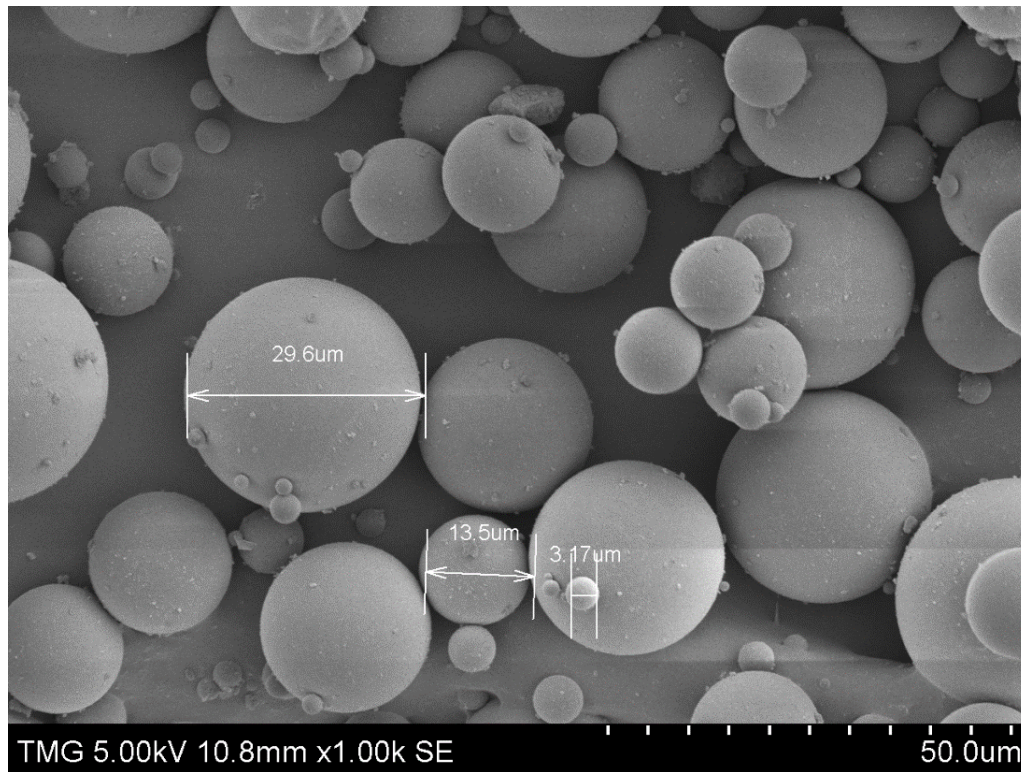


Figure 3.2 Particles size range for used MPCMs (obtained using SEM XXX at Concordia)

3.3 Testing and specimen preparation

Flowability was evaluated for all tested mixtures using the flow table as shown in **Fig. 3.4**. According to ASTM C230 (Standard Specification for Flow Table for Use in Tests of Hydraulic Cement). Fresh mortar sample was placed in the flow mold on a drop table and stroked for 25 times. Then the diameter of the collapsed mortar was measured at four different locations using the caliper. The flow reading represented the average of the four different readings.



Figure 3.3 Flow table test apparatus

Compressive strength was evaluated for various mixtures according to ASTM C109 (Standard test method for compressive strength of hydraulic cement mortars) using a 300 kN universal testing machine (UTM) as shown in **Fig. 3.4**. Cubic specimens $50 \times 50 \times 50$ mm were prepared for each mixture and tested at ages 1,3,7, 14 and 28 days. Moreover, a direct tensile strength was evaluated by a direct tension testing of a small cement mortar briquette according to AASHTO T132. The dog-bone shaped briquette has a 75 mm length, 25 mm thickness, with a 625 mm^2 cross-section at mid-length, and enlarged ends to allow a passive gripping of the specimens, as shown in **Fig. 3.5**. All mortar cubes were compacted using the vibration table and the tamping rod. All samples were demolded after 24h, then stored at temperature $=20^\circ\text{C} \pm 2^\circ\text{C}$ and relative humidity $= 90\% \pm 5 \%$.



Figure 3.4 UTM

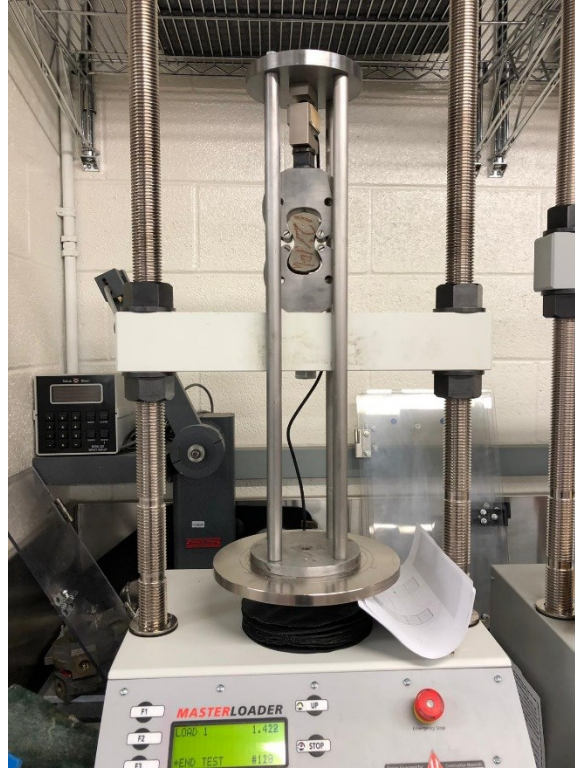


Figure 3.5 Direct tensile test apparatus

Isothermal calorimetry was used to monitor the heat liberated from different mixtures during the hydration process. The calorimeter (calmetrix I-CAL 2000 HPC) apparatus was used is shown in **Fig. 3.6**. The mass of the samples and the volume were calculated for each mixture after demolding, in order to get the density. Other cylindrical samples were prepared according to the ASTM 1202 (Standard Test Method for Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration), and ASTM 1585 (Standard Test Method for Measurement of Rate of Absorption of Water by Hydraulic-Cement Concretes), each with a diameter of 100 mm and a thickness of 50 mm to test the rapid chloride ion penetration, and the sorptivity of the mixtures respectively, as durability tests as shown in **Fig. 3.7** and **Fig.3.8**.



Figure 3.6 Calmetrix I-CAL 2000 HPC

Specimens were prepared according to ASTM 1585 as follow: Cylindrical disc specimens with dimensions of 50mm \times 100mm were cast, then demolded after 24 h and cured in plastic containers for 28 days at 23°C , then all samples were conditioned at 50C with relative humidity (RH) of 80% in an environmental chamber for three days. Thereafter, each specimen was stored in a separate sealed container for another 15 days at °C in order to allow moisture redistribution. After 15 days, all samples sides and one face were sealed with plastic sealing tape for the sides and plastic sheet for the top face, and the other side of the sample and 3mm of the side near this face left open to be exposed to water at 23°C. Change in a mass of specimens was recorded over nine days using a laboratory balance with the precision of ± 0.01 g. Mass measurements were taken over pre-seated intervals, tailoring the mass increment of samples starting from 1 min immediately after the open sample side touches the water. Following, continued for the periods of 5 min, 10 min, 20 min, 30

min, 60 min, 120 min, 180 min, 240 min, 300 min, 360 min, and then every day up to 9 days. The cumulative volume of the absorbed water per unit area I (in mm) was calculated according to (Eq. 3.1)

$$I = \frac{m_t}{a \times d} \quad \text{Eq. 3.1}$$

Where m_t the change in specimen mass (grams), at the time t , a is the exposed area of the specimen (mm^2), and d is the density of the water (g/mm^3).

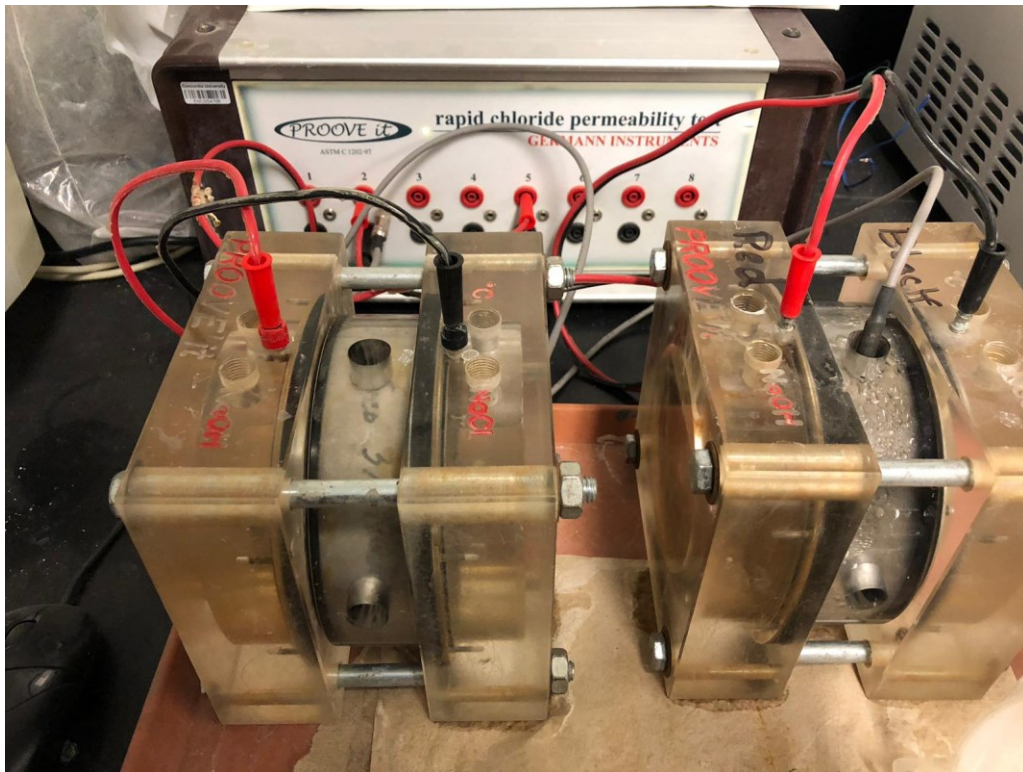


Figure 3.7 RCPT testing apparatus

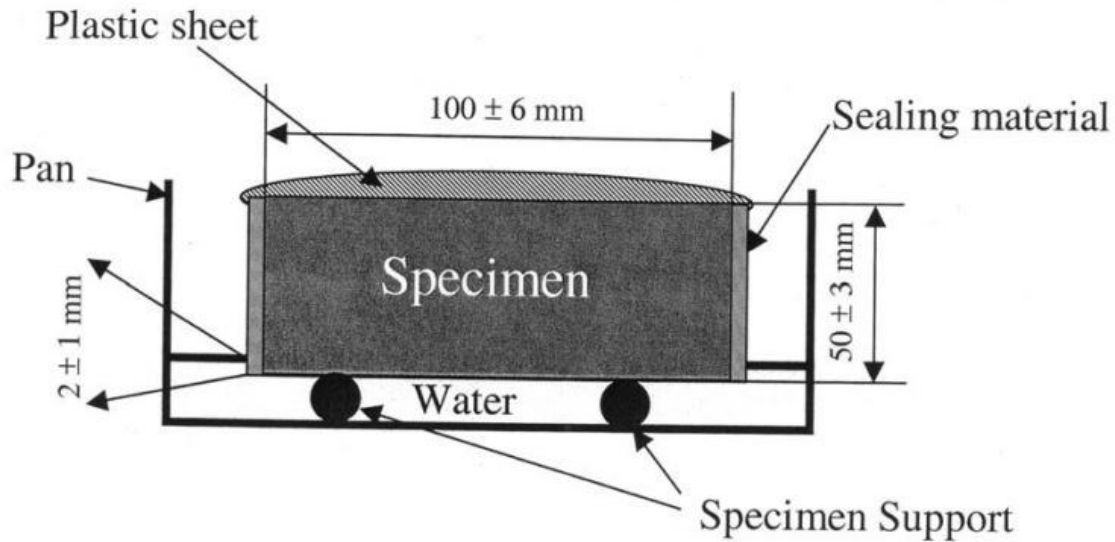


Figure 3.8 Absorption test setup [Adapted from (ASTM 1585 – 13)]

On the other hand, 50 mm thick slice specimens loaded with thermocouples were cast and used for monitoring temperature distribution and thermal response. In the vertical direction, thermocouples were pre-installed at distances 1.25 cm, 2.50 cm and 3.75 cm from the surface. At depth 3.75 cm, additional thermocouples were distributed at angles 0, 90, 180 and 270 degrees of the circular face. At the testing age, specimens were installed inside a custom built-in isolation box. In this box, specimen's sides were isolated, one face was exposed to ambient conditions, while the other face was exposed to a heat source (heat gun). Applied heat temperature was increased gradually until 100 °C, then stopped and specimens allowed to cool down. The temperature cycle was recorded, and all data were collected using a data acquisition system as shown in **Fig. 3.9**. Moreover, SEM testing were carried out on several specimens (i.e. raw MPCM, incorporated specimens). SEM machine used for microscopic image was (HITACHI S-3400N at Concordia University) and coating for the specimens was done using (Quorum Q150R ES at Concordia University) is shown in **Fig. 3.10**.

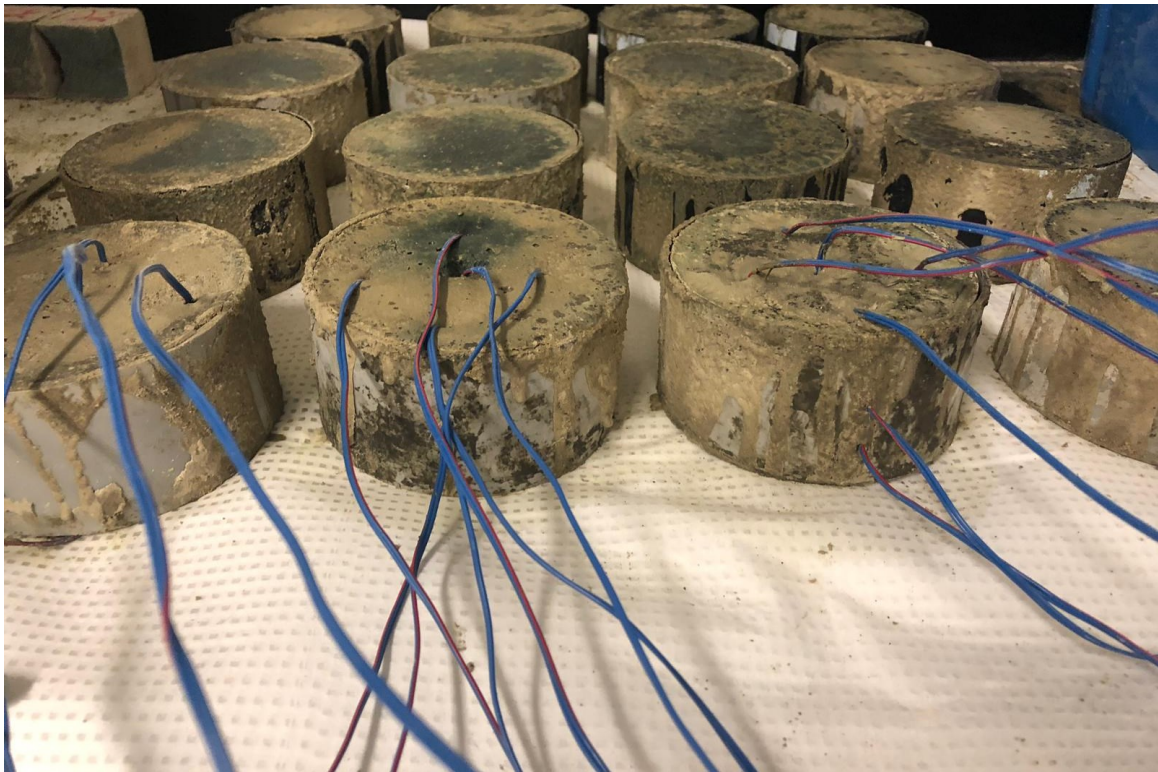


Figure 3.9 Thermal transfer specimens setup



Figure 3.10 SEM test setup

3.4 Results and Discussion

3.4.1 Flowability

Figure 3.13 shows the average reduction for flow table test readings for mixtures incorporating MPCMs compared to that of the control mortar. The reduction increased as more MPCM was added. For instance, mixtures incorporating 6% MPCM exhibited a 15% reduction in the average spread diameter compared to that of the control. Moreover, 12% MPCM induced a reduction of 23% in the average spread diameter compared to that of the control. This indicated that replacing sand by MPCMs resulted in a lower flowability.

This can be attributed to two compensating effects, the increase in friction forces and higher water demand. Examining MPCMs showed some trace elements that are expected to increase the roughness, like the surface of the MPCMs particles as shown in **Figs 3.11-3.12**. Some particles were deformed or agglomerated, especially when the temperature is beyond its phase change temperature (the core is melted) (Snoeck, 2016). According to Park (2009) cementitious materials workability depends on sizes of the microcapsules. Hence, the reduction of free water by the presence of small fine particles, which adsorb more water to its surface leading to sticky mixture (Senff, 2010).

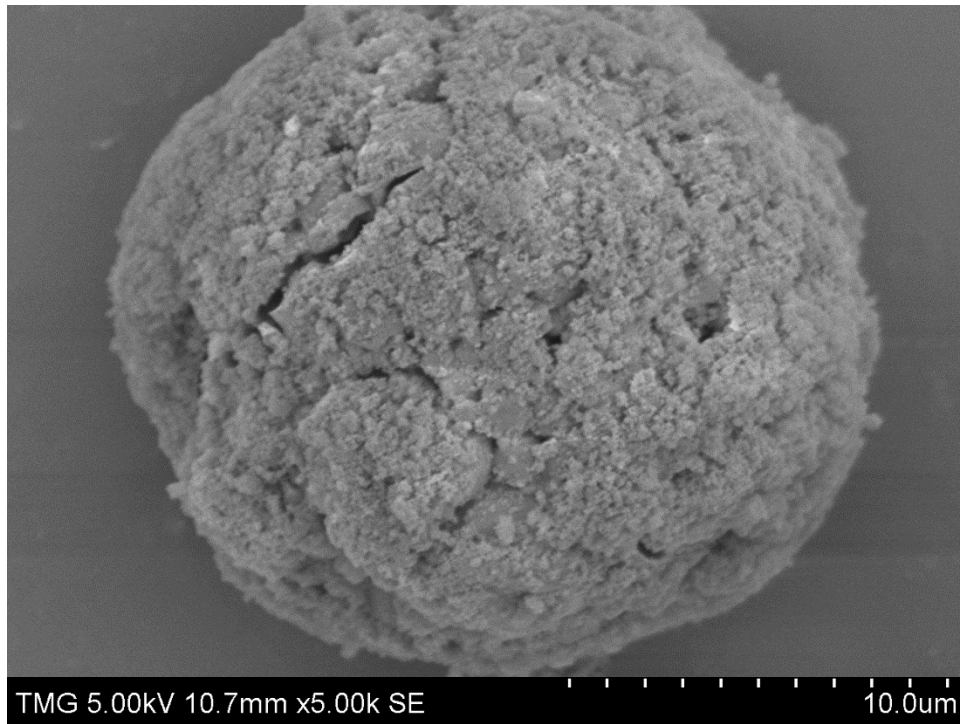


Figure 3.11 MPCM 28

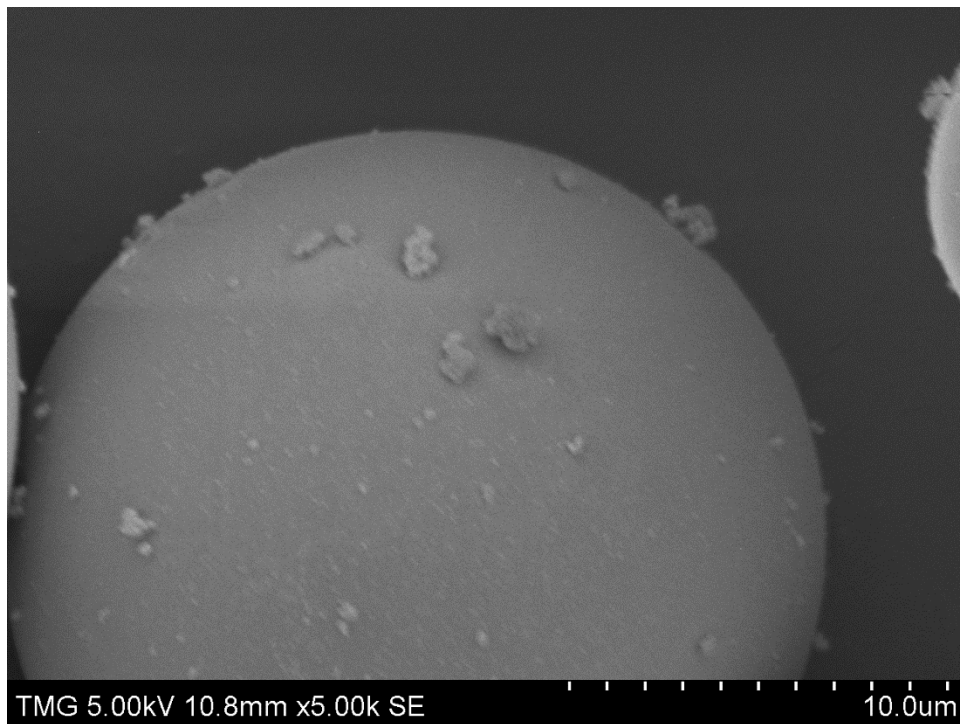


Figure 3.12 MPCM 43

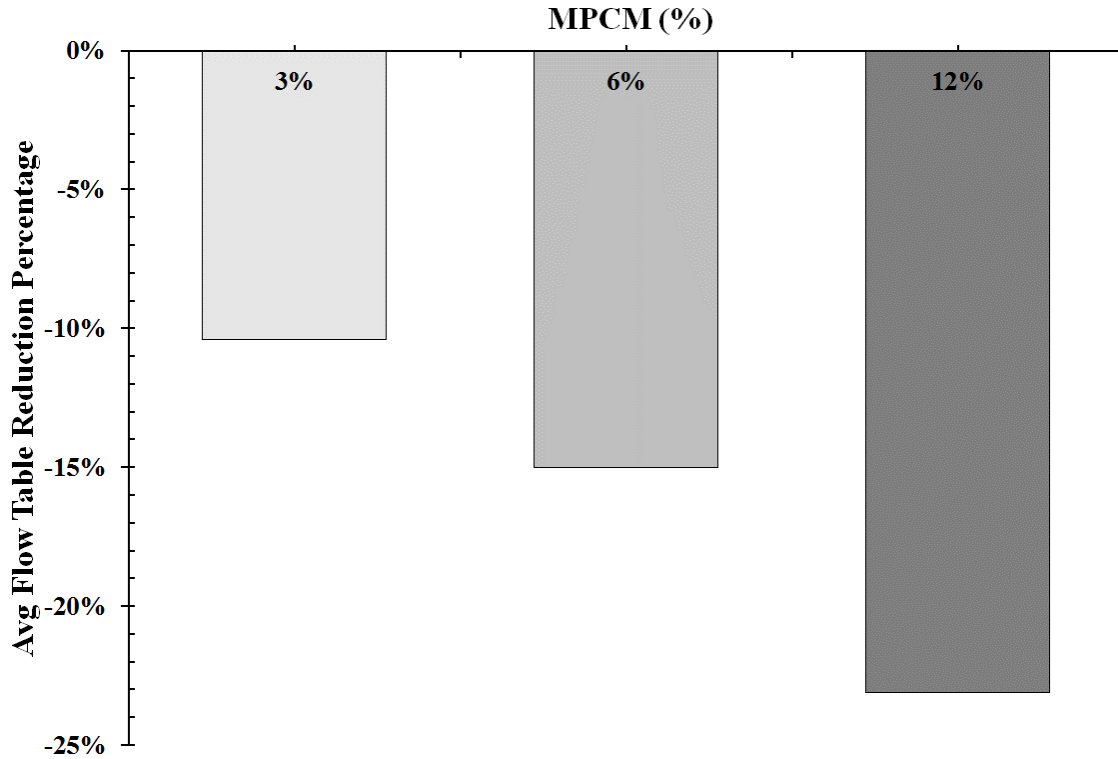


Figure 3.13 Reduction in average flow table readings for incorporated mortars vs control mortar

3.4.2 Density

The hardened density was evaluated for all mixtures at age 28 days. As shown in **Fig. 3.14**, adding MPCMs resulted in a lower density. The higher MPCMs incorporation rate, the higher the reductions in the density. For instance, comparing a mixture of 3% with the control mortar, the reduction in density observed where 6%. Although, mixtures incorporating 12% MPCMs exhibited a reduction in the density with about 14% compared to that of the control mixtures without MPCMs. This can be attributed to the lower unit weight of MPCMs compared to that of the sand, with 0.9 kg/m^3 for MPCM and 2.67 kg/m^3 for sand. Hence, as lighter materials replaced sand, the final density will reduce. Also, micro-capsules increases the porosity of the mixture as

reported by Aguayo (2016) were he used (MIP) to support his findings. Moreover, Pilehvar (2017) who used X-Ray tomography imaging in his study for cement and Geopolymer porosity. As the porosity increased at hardened ages, because of the moisture leaves the specimens with extra pores reducing the density.

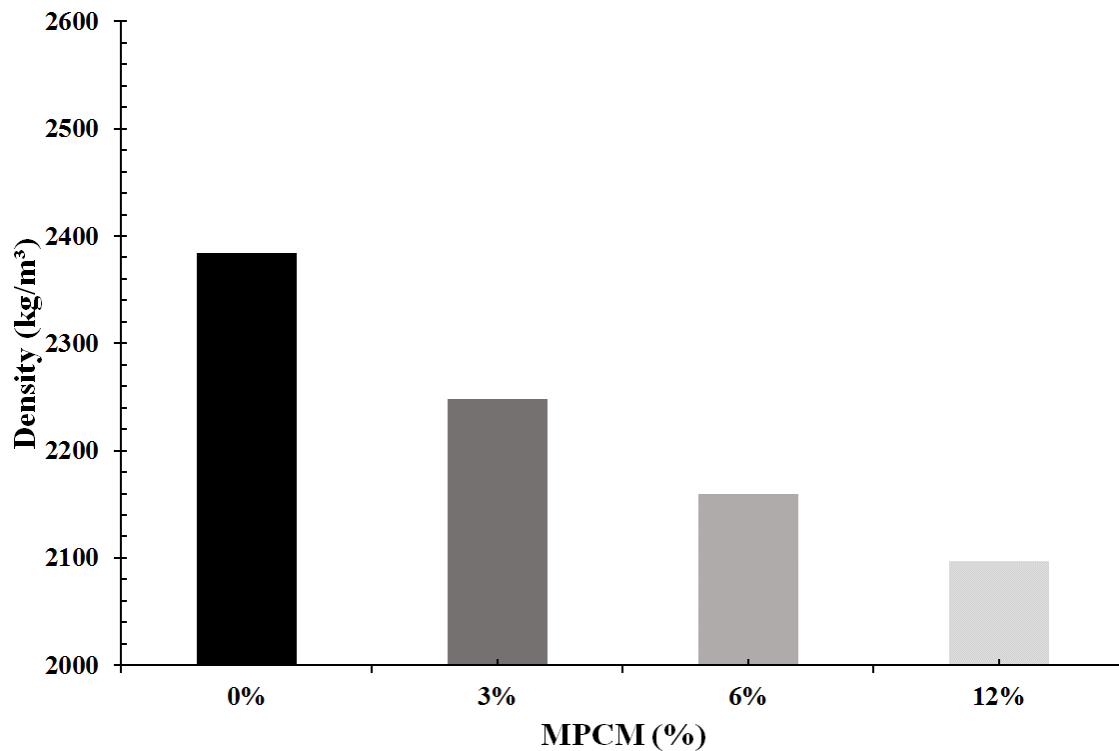


Figure 3.14 Hardened density for all mixtures with and without MPCMs

3.4.3 Compressive Strength

Compressive strength is one of the main factors judging the performance for cementitious materials incorporating MPCMs. **Figure 3.15** illustrates the compressive strength development for mixtures incorporating different percentages of MPCM 28 under the ambient curing condition. Generally, the compressive strength increased with time. The higher the MPCMs content, the lower the achieved strength. Adding 3% MPCM as a sand replacement did not induce considerable change at age 28 days (only 3%). On the other hand, increasing the amount of MPCM in the

mixture to 6% resulted in more reduction in the achieved strength over the investigated period with 7% reduction compared to the control. Moreover, adding 12% MPCMs led to about 25% reduction in the 28 days compressive strength with respect to that of the control.

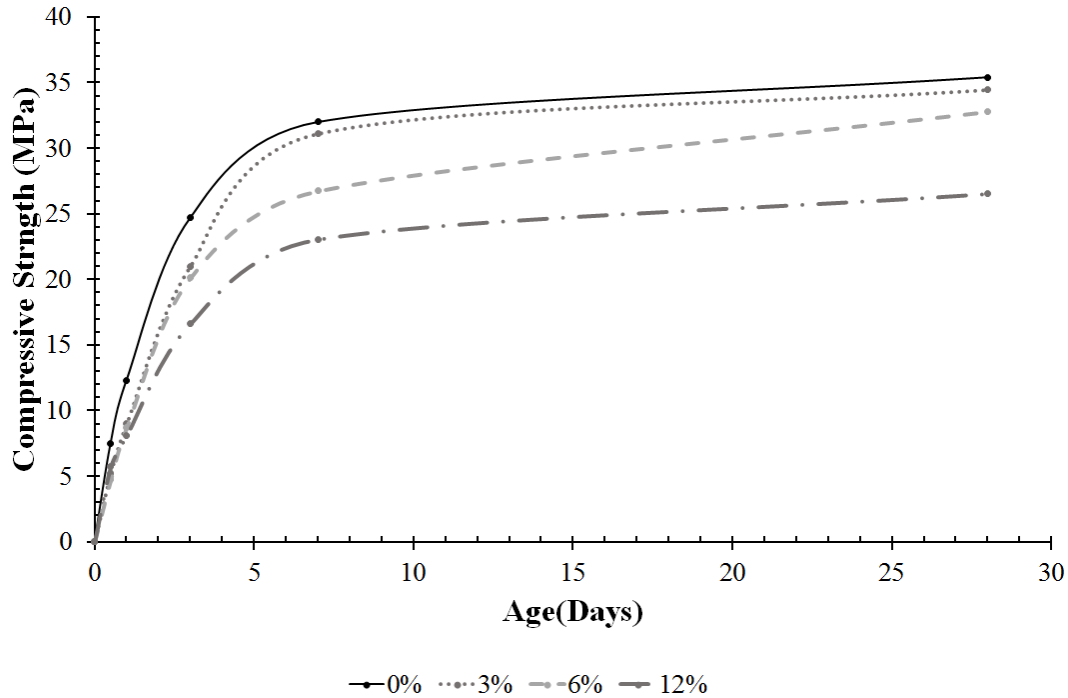


Figure 3.15 Compressive strength development for mortars with and without MPCM 28 incorporation under ambient condition

MPCM 43 was also studied under the same conditions. **Figure 3.16** displays the compressive strength development for mortars incorporated MPCM 43 under ambient curing condition. It can be noticed that MPCM 43 has the same effect as MPCM 28 under the ambient curing conditions. In addition, MPCM 43 did relatively induce more strength loss compared to MPCM 28. Adding 3% MPCM as the sand replacement at age 28 days induced a reduction of 4% which is close to the MPCM 28, proving that small dosages don't have the considerable effect on the compressive

strength. On the other hand, increasing the amount of MPCM in the mixture to 6% reduced the achieved strength over the investigated period with 13% compared to that of the control. Furthermore, adding 12% MPCMs led to about 29% reduction in the 28 days compressive strength at 28 days with respect to that of the control. Concluding that MPCM 43 has detrimental effects on the incorporated mortar as the MPCM 28, but MPCM 43 has more detrimental effects on the mechanical properties compared to the MPCM 28.

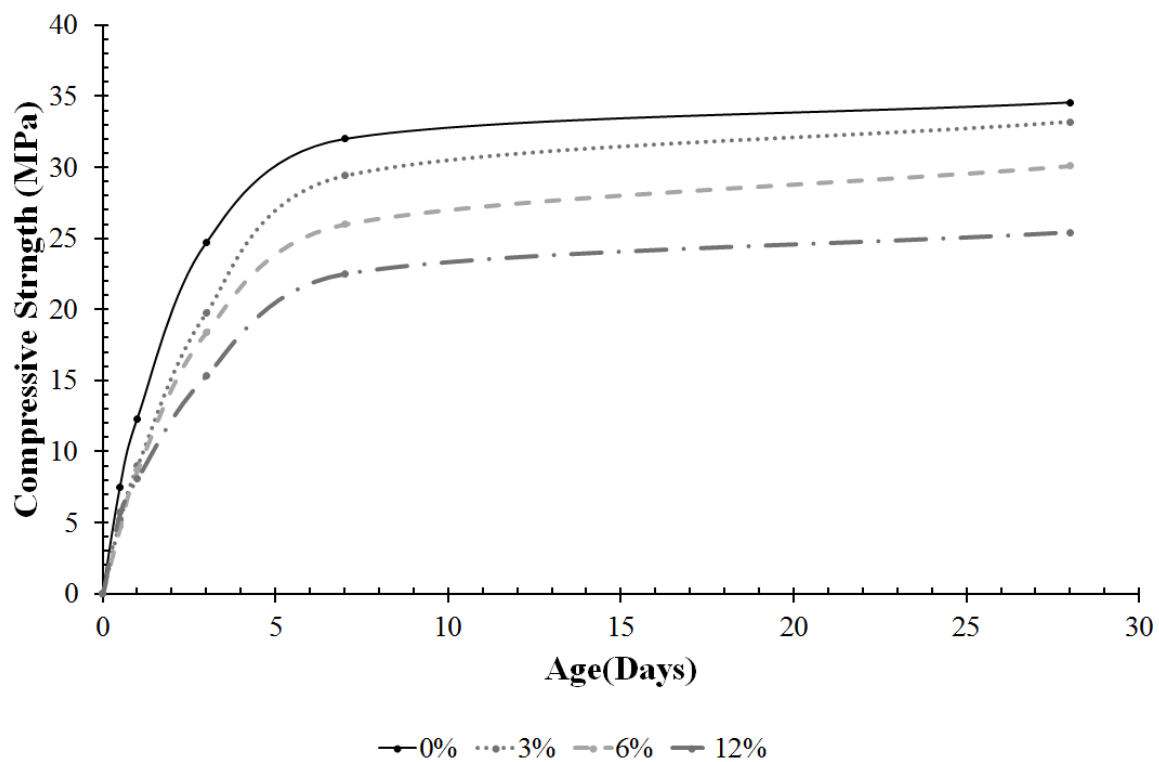


Figure 3.16 Compressive strength development for mortars with and without MPCM 43 under ambient condition

It was reported that the addition of MPCMs to cementitious materials will detrimentally affect the compressive strength (Marani, 2019). This can be ascribed to several reasons. One of them is the

softness nature of the MPCMs as an additive to the mixture, MPCMs cannot sustain loads (Eddhahak-Ouni, 2014).

Also, the breakage that happens to the microcapsules and leaks out the paraffin oil during mixing, which hinders the reaction between the binder and water. Moreover, capsules act like voids, which means that they will not contribute much to the strength. This was confirmed as shown in **Fig 3.17** and **Fig 3.18**. Furthermore, microcapsules were reported to absorb water. And this water absorption was one of the reasons behind the strength reduction (Lecompte, 2015). Nonetheless, MPCMs hinders the contact between cement and water particles, and it was reported to be the reason for lower strength at early ages (Haurie, 2016). In mixtures proportions for our research, in order to minimize such effects, the water to cement ratio was kept constant at 40%, noticing that the solid particles like sand is decreasing as the MPCM replacement is increasing.

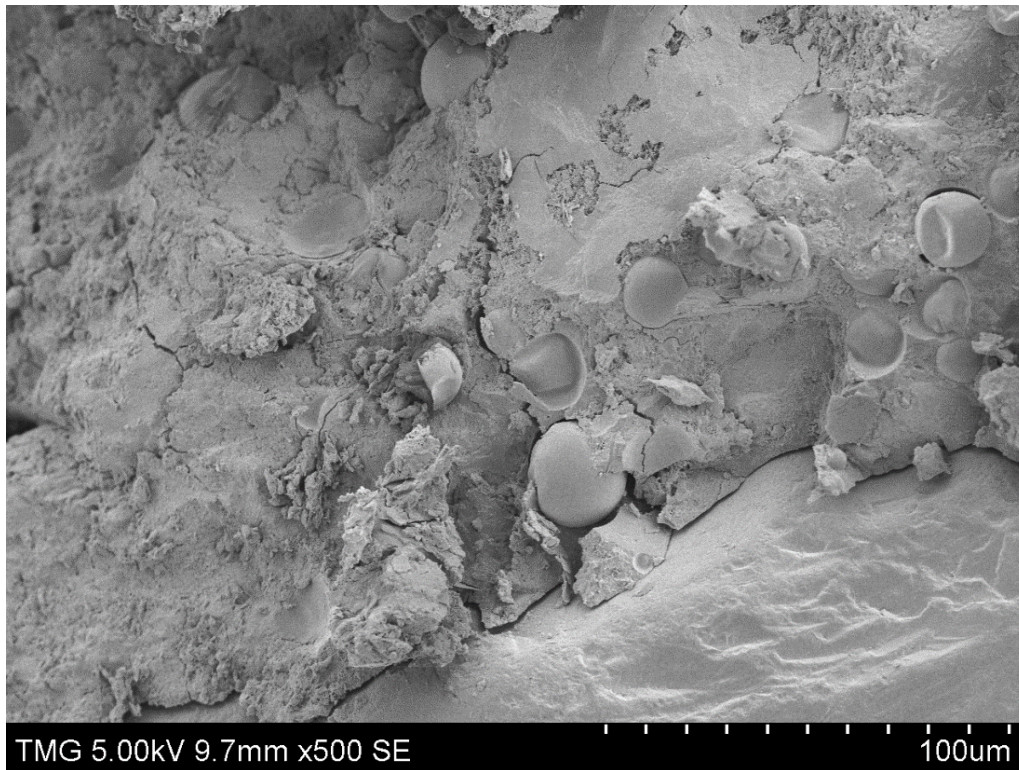


Figure 3.17 Capsuled MPCMs inside mortar matrix



Figure 3.18 MPCM microcapsules breakage

3.4.4 Tensile Strength

One of the properties of the cementitious material that is always disregarded is the tensile strength. It is disregarded due to the properties of the inherently brittle material that cementitious materials have low overall tensile capacity. A number of tests have been developed to directly and indirectly measure the tensile strength. In this research, the direct tensile method for mortar briquette was used.

Figure 3.19 shows the tensile strength development for several mortar mixtures with and without the incorporation of MPCM 28. Mostly, tensile strength slightly changed over time for all mixtures. The higher the MPCMs content, the lower the tensile strength was. For instance, for mixtures with 6%, a reduction in the tensile strength was 11%, compared to that of the control mixture at age 28 days. For the 12%, the reduction was 17% compared to that of the control. The

same trend conquered for mortar in the compressive strength, with a detrimental effect on the tensile strength development pattern for MPCM incorporated mortars, under ambient curing condition.

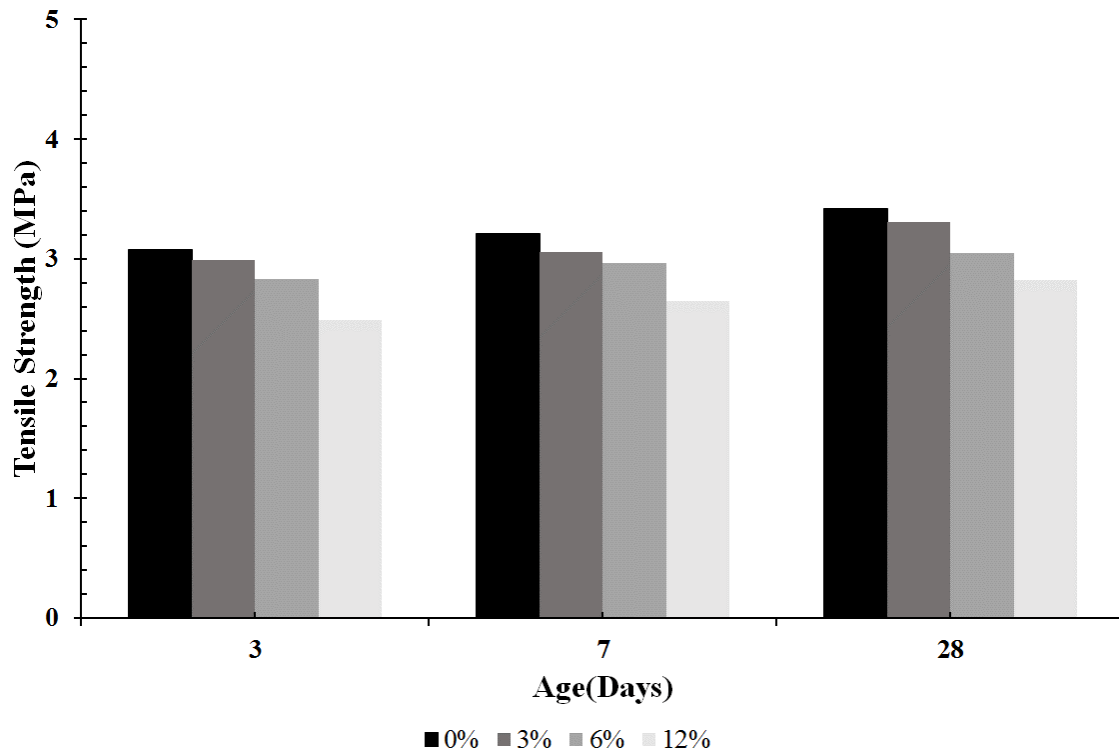


Figure 3.19 Tensile strength development for mortars with and without MPCM 28 incorporation under ambient condition

Figure 3.20 shows the tensile strength development for several mortar mixtures with and without the incorporation of MPCM 43. Mostly, tensile strength increased over time for all mixtures. For instance, mixtures incorporating 6% and 12% MPCMs exhibited reductions of 15% and 25% compared to that of the control at the age 28 days, respectively. The same trend conquered for mortar in the compressive strength, with a detrimental effect on the tensile strength development pattern for MPCM incorporated mortars, under ambient curing condition. Also, MPCM 43 has a

more detrimental effect on the tensile strength compared to MPCM 28, which confirms the findings in the compressive strength for both MPCMs incorporated mortars.

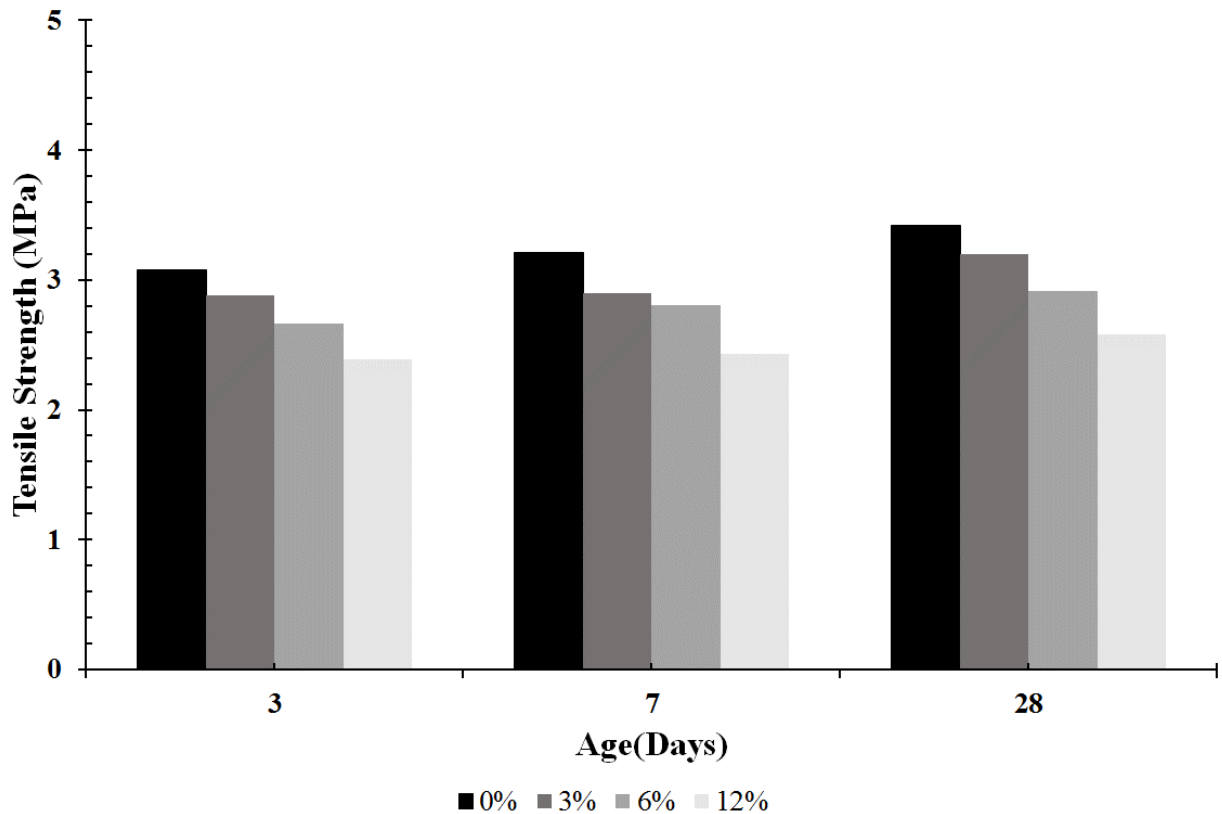


Figure 3.20 Tensile strength development for mortars with and without MPCM 43 incorporation under ambient condition

This phenomenon can be ascribed to the effect of MPCMs incorporation in cementitious mixtures on mechanical properties. For this test particle will suffer a tensile load, which is more dependable on the developed connection inside the matrix. As mentioned before, breakage of the microcapsules with leaked paraffin that plays a part in hindering the interaction between cement particles and water. Also, the softness nature of PCM microcapsules. Which weakness against load resistance (Eddhahak-Ouni, 2014).

3.4.5 Heat of Hydration

Hydration process starts from the first second of mixing. The hydration itself is the chemical reaction that happens between the binder and the water. With the presence of MPCMs as inert materials that have a thermal capacity ability, some hydration kinetics got affected (Baetens, 2010). Hydration may be delayed or interrupted (Frigione, 2019). Although, hydration peaks may also increase (Jalayath, 2016).

Mortar samples were studied at 23°C. The test was carried out for 25 hours to capture the peaks of the heat flow produced from the hydration process. Generally, a short exothermic peak appeared immediately after mixing. This initial peak can be designated to the rapid initial chemical reaction between Tri calcium silicate and water, which begins immediately upon wetting of the cement particles and water (Costoya Fernández, 2008). The early exothermic peak can be also referred to as the dissolution of the C₃S (Bullard, 2011).

Then a decrease in heat flow can be noticed in the curve lasts for a couple of hours. This low heat evolution period, is called the induction period. This induction is carried out for 1-2 hours. It was explained by many researchers, and the two main dominant explanations are fall down in the growth and the nucleation of calcium silicate hydrate (CSH). Alternatively, a protective layer formed from the initial C₃S and water reaction, which will be destroyed later by the proceeding growth mentioned above and nucleation effects (Costoya Fernández, 2008). Afterward, the heat evolution continues and reach its maximum in the acceleration period. It can be ascribed to the ongoing reaction for the C-S-H and Calcium hydrate, which reaches its maximum around 10-12 hours later. It depend on the particle size and associated with the rapid formation of hydrates that lead to setting and solidification (Scrivener, 1984). Thereafter, the hydration reaction starts to slow down entering the deceleration period. Also, a secondary shoulder peak could be seen in the curves which is associated with the formation of ettringite, where the hydration accelerates during the

aluminate phase that may be referred to the sulfate previously absorbed in the C-S-H phase (Gallucci, 2010). On the other hand, the difference in the maximum heat evolution can be noticed for 3% incorporation. The curves show that MPCM incorporation at 3% caused a higher maximum peak of hydration. The explanation for this phenomena is mostly dependable on the size of the MPCM particles. Which falls in the small group of the cement particles with a mean size around $10\mu\text{m}$. This small particle work as a physical catalyst for the chemical reaction with their higher surface area (Luo, 2013). Therefore, it may enhance the space filling effect and favorably stimulate cement hydration (Yang, 2015).

Moreover, the particle size distribution is crucial for the initial peak and after as well. Since during the first ten hours particles less than $3\mu\text{m}$ is entirely consumed and particles less than $7\mu\text{m}$ up to 24 hours (Scrivener, 1984). Hence, MPCMs might play a conquer part of the stimulation and retarding of the hydration as a mineral additive under the ambient conditions.

Figure 3.21 illustrates the hydration curves for the MPCM 28 incorporated mortars combined together under ambient temperature to more elucidate and capture the difference in peaks and delay caused. The same trend for all curves could be noticed in the figure. Starting from the reduction in the flow, then the maxium heat evolution, and the second sholduer peak. But it is observed that by increasing the MPCM percentage, the heat flow is stimulated and peaks are shifted.

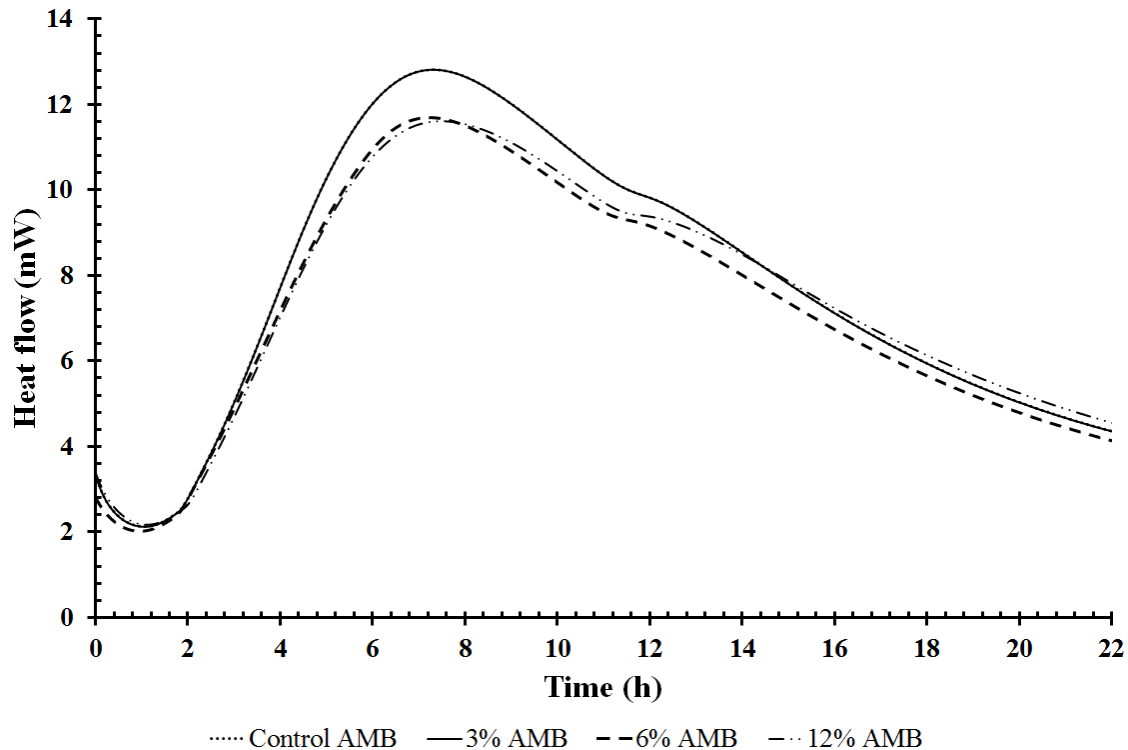


Figure 3.21 Heat flow OPC samples with and without MPCM 28 incorporation under ambient condition

Figure 3.22 displays the heat of hydration curves for MPCM 43 incorporated mortars under ambient curing conditions. The test was carried out for 22 hours to capture the peaks of the heat flow produced from the hydration process. The same behavior as MPCM 28 was captured.

The general behavior can be explained as follow: There are two compensating effects for MPCMs: nucleation and heat storage. The small size of the particles acts as nucleation sites and consequently accelerate the hydration. However, as hydration reaction progress, heat is liberated. The heat is expected to fuel the hydration reaction. The presence of MPCMs will affect this process as it will start storing/absorbing heat once the temperature exceeded it melting point. Hence it seems at low percentages of MPCMs, the nucleation effect is dominant, and however, at higher percentages the heat storage is more effective.

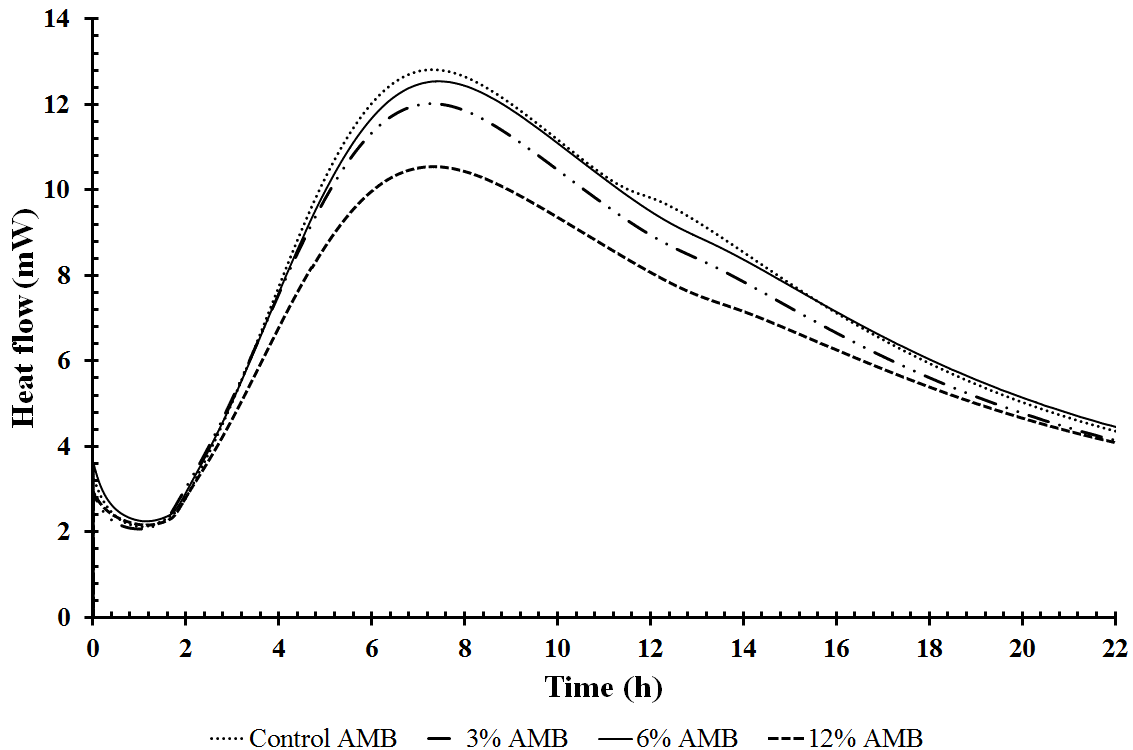


Figure 3.22 Heat flow OPC samples with and without MPCM 43 incorporation under ambient condition

3.4.6 Thermal Transfer

Thermal properties of cementitious materials is one of the areas to be tackled while studying MPCMs. Phase change materials increase the heat transfer due to its large surface area per unit volume (Riffat, 2015). It is important to mention that the microencapsulation shells had an effect on the innate heat transmission of the raw phase change materials (Whiffen, 2012). MPCMs shells affect the heat transfer for cementitious mediums differently from the core (i.e. phase change materials). Microcapsules have a relatively poor thermal conductivity, lower than that of the cement paste (Zhang, 2016). This significantly affects core phase change materials thermal conductivity.

Figures 3.23-3.26 show the rate of the heat change through an OPC disc specimen with and without MPCM 28 incorporation, versus time using two channels located as follows (Ch1 1.5 cm

from the face, Ch3 4.5 cm from the face). Exposed to dry heat gun with at a distance of 5 cm, with a temperature of 100 °C from one of its faces. Generally, the temperature increased with time for all specimens. And temperature variation between both thermocouples is observable. It can be observed from **Fig. 3.27**, that Channel #1 (1.5 cm from the exposed surface) reached 70 °C in 23 min, 29 min, 33 min, and 39 min for mixtures incorporating 0%, 3% 6% and 12% MPCMs. This indicates that the addition of MPCMs had decreased the thermal conductivity.

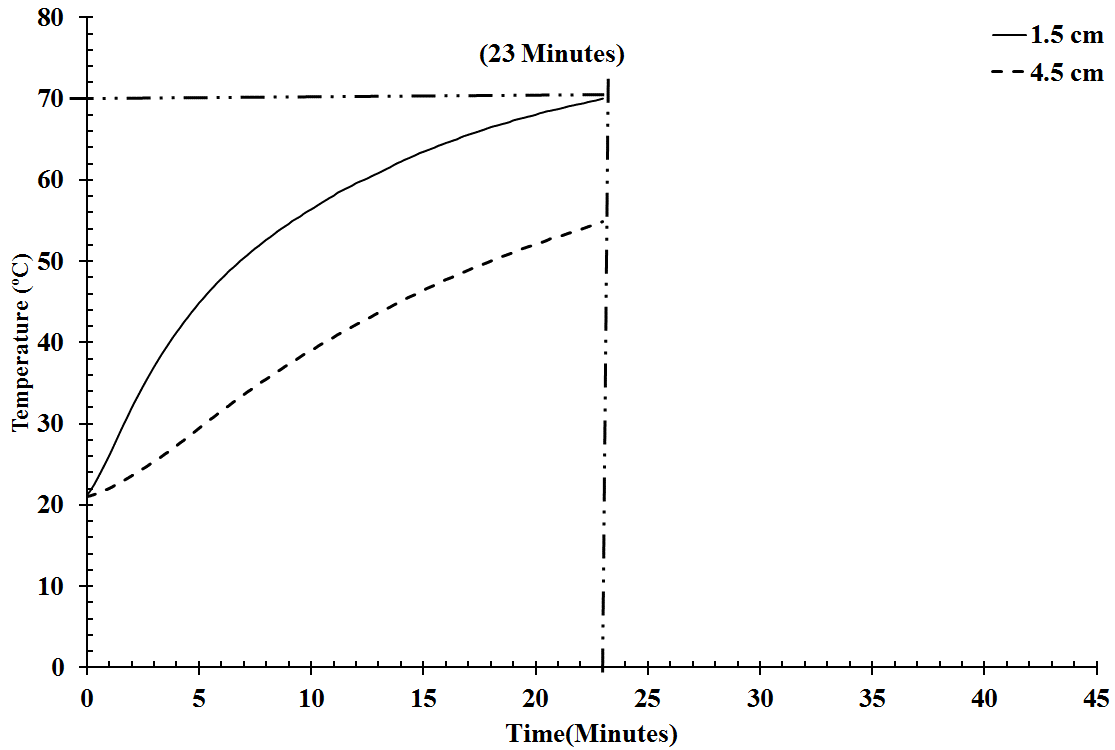


Figure 3.23 Thermal behavior of control OPC specimen

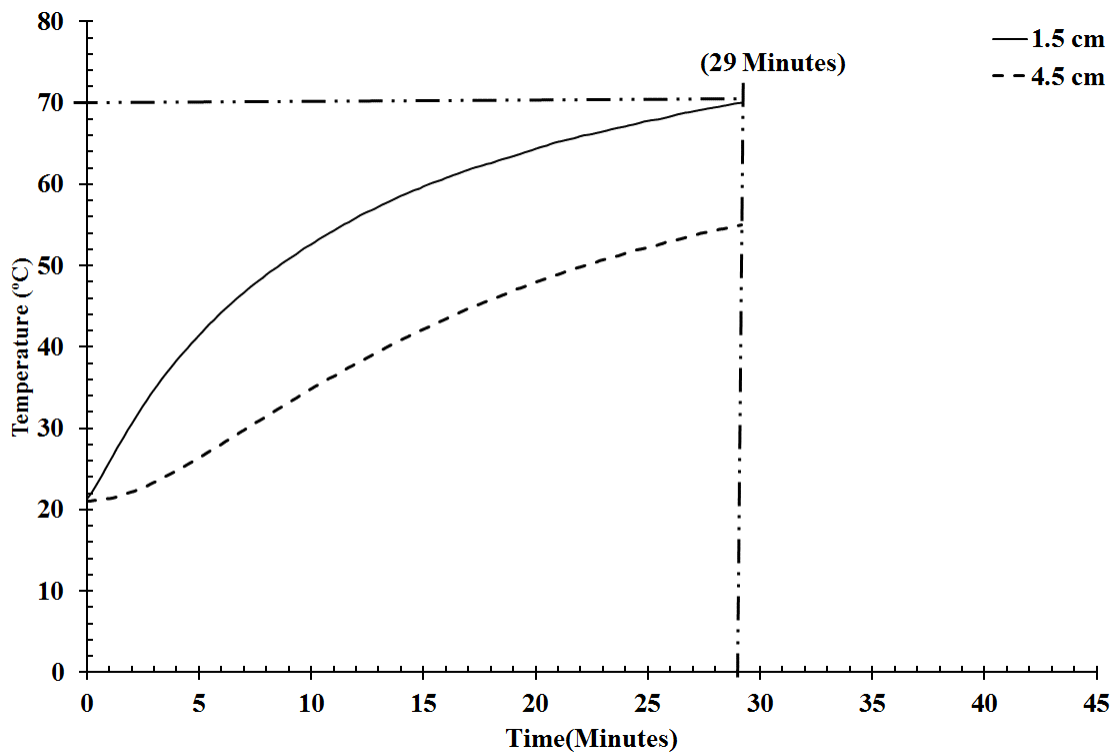


Figure 3.24 Thermal behavior of 3% MPCM 28 OPC specimen

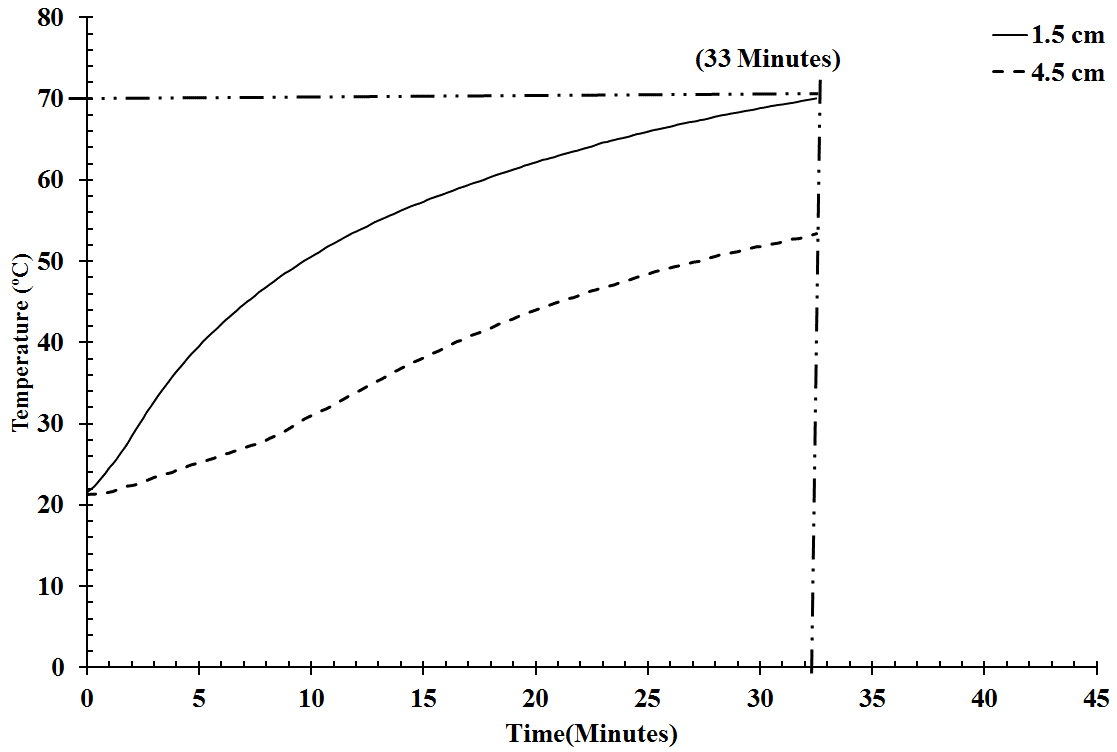


Figure 3.25 Thermal behavior of 6% MPCM 28 OPC specimen

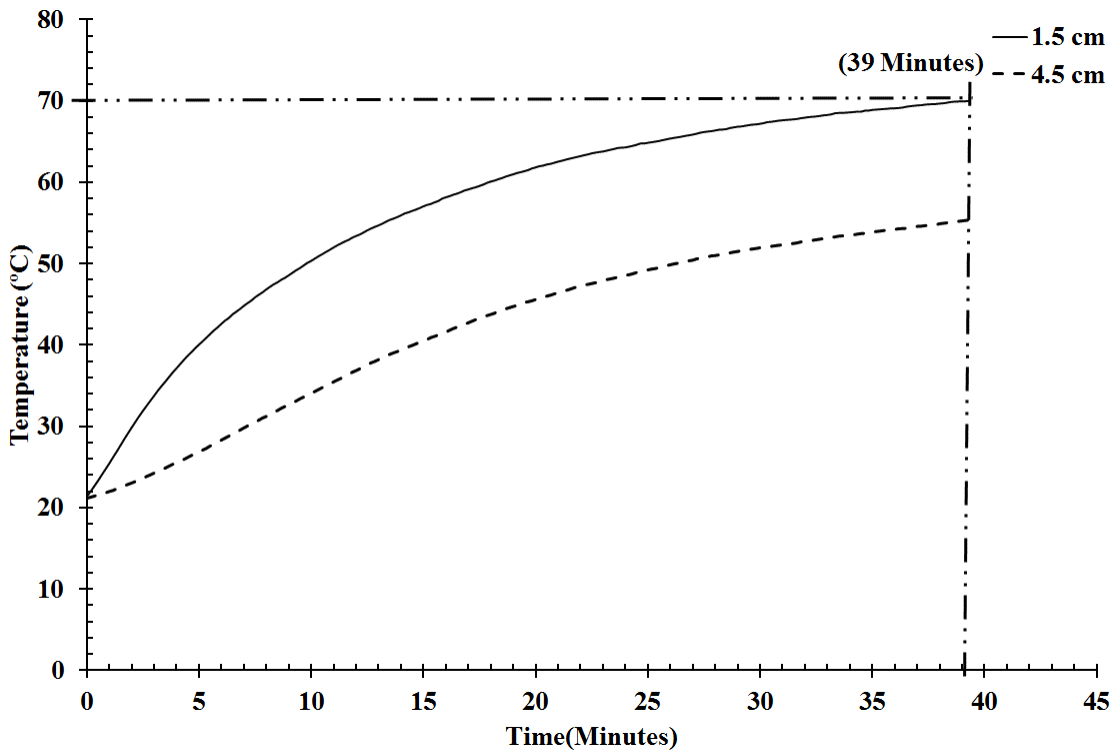


Figure 3.26 Thermal behavior of 12% MPCM 28 OPC specimen

Table 3.4 shows the time required for all mixtures to reach 70 °C at Channel #1 along with the difference between Ch #1 & Ch #3. Comparing the 3% MPCM mixture with the control mixture, Ch #1 reached 70 °C with about 5 more minutes. This is caused by the MPCM heat storage capacity and resulted in an overall delay for the heat transfer (Bahrar, 2018). The temperature variance is the same in the 3% compared to control. Moreover, increasing MPCM dosage up to 6% resulted in slower heat transfer with 10 minutes more, although with increasing temperature variance between both channels of 16 °C. This could be ascribed to the decreased thermal conductivity with increasing MPCM dosages (Jayalath, 2016). Furthermore, mixtures with 12% reached 70 °C in 40 minutes with 5 minutes more compared to the 6% MPCM mixture and 15 more minutes compared to the control, and with a temperature variance of 19 °C. Temperature variance could be ascribed to the increased MPCM dosage and its effect of decreasing thermal conductivity. MPCMs addition is reported to increase the amount of entrapped air (Hunger, 2009) which made this material with a good insulation enhancements to cementitious mixtures. Hence, created entrapped air is responsible of causing a delay in the natural convection of heat. On the other hand by replacing sand with MPCM, knowing that sand's thermal conductivity is higher compared to MPCMs it will

result in less thermal conductivity, thus lower heat transfer rate (Jayalath, 2016). Also, broken microcapsules will increase voids.

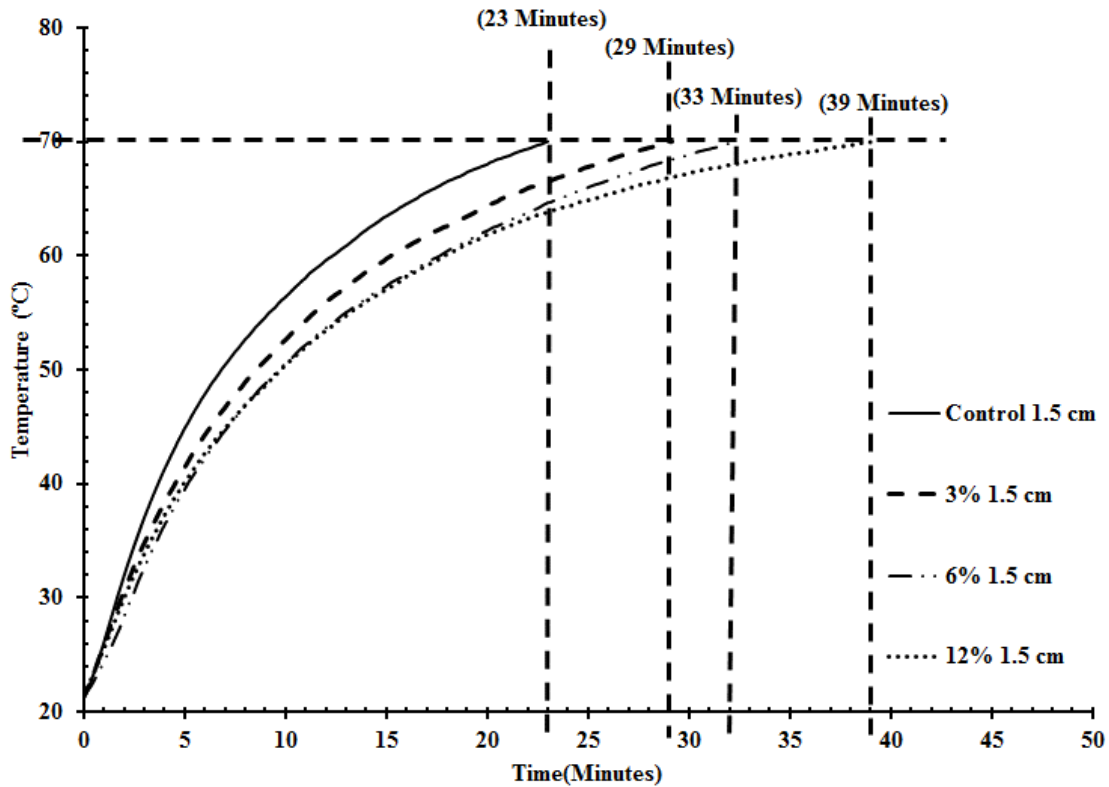


Figure 3.27 Thermal conductivity for Ch1

Table 3.4 Temperature variation between Ch #1 and Ch #3 at 70 °C

<i>Specimen</i>	$\Delta t(\text{Minutes})$	$\Delta T (^\circ\text{C})$
<i>Control OPC</i>	23	13
<i>3% MPCM 28 OPC</i>	29	13
<i>6% MPCM 28 OPC</i>	33	16
<i>12% MPCM 28 OPC</i>	39	19

3.4.7 RCPT

Rapid Ion Chloride Penetration in accordance with ASTM C1202 was conducted in order to evaluate the durability performance. Adding MPCM is known to increase the porosity of the cementitious materials (Jalayath, 2016). Hence, the penetration of the ion chloride will provide a clear vision about the permeability of the final cementitious products.

Permeability of cementitious materials can be defined as the ability of the fluids to pass through saturated porous material under pressure difference (Tam, 2012). Diffusion is defined as the process through which an agent can pass the medium due to a concentration gradient. It happens normally through the movement of ions from high to low concentration zones in the cementitious materials (Basheer, 2001). In this research, the electrical migration method was used to stimulate the accelerated movement of chloride ion using electrical potential gradient.

Figure 3.28 shows percentage of charge passed through MPCM incorporated OPC specimens at 28 days compared to that of the control. Generally, increasing MPCMs contents resulted in a higher chloride ion penetration. According to ASTM C 1202 Limits shown in **Table 3.5**, all specimens readings fall over the high chloride ion penetrability. For instance, a small increment of the total charge passed can be observed after adding 3% MPCM to the mixture compared to that of the control with 7%. Moreover, by increasing the dosage of MPCM in the mixtures, a much higher increment up to 75% is observed for mixture incorporating 6% compared to that of the control. Lastly, adding 12% MPCM doubled the total charge passed with an increment of 98% compared to that of the control. This could be ascribed to the entrapped air that was reported to be increased by MPCM incorporation as well as increasing the voids ratio (Hunger, 2009)

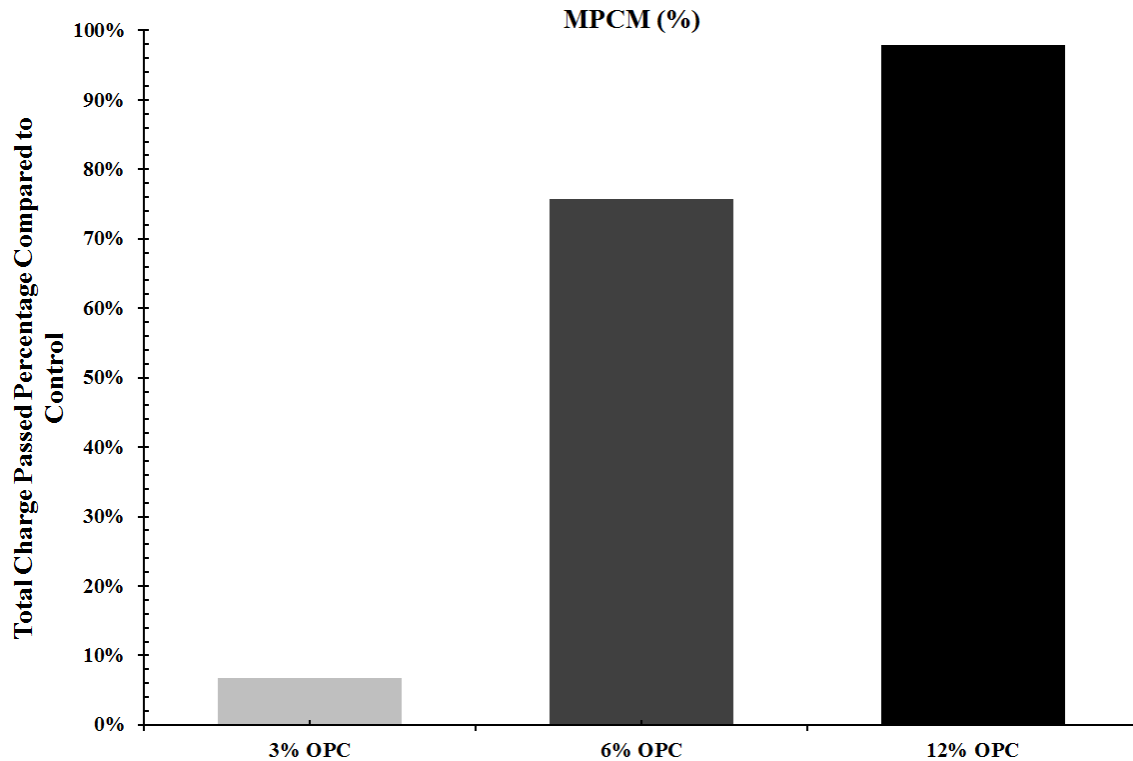


Figure 3.28 Total Charge Passed.

Table 3.5 Chloride Ion Penetrability Based on Charge Passed (ASTM C 1202)

Charge Passed (coulombs)	Chloride Ion Penetrability
>4,000	High
2,000–4,000	Moderate
1,000–2,000	Low
100–1,000	Very Low
<100	Negligible

3.4.8 Sorptivity or Absorption

Adding MPCMs to the cementitious materials associated with enhancements and drawbacks mentioned earlier. Predicting the capabilities for MPCM incorporating cementitious materials is crucial. Since it should be in good serviceable conditions along all of its service life. The rate at which the water is getting absorbed by the cementitious materials throughout the capillary suction

will provide a good indication of the pores structure (Parrott, 1992). The porous materials absorb water by the capillary action in the absence of external pressure. Hence, the water absorption for the MPCMs mortars was evaluated as per ASTM C1585 under laboratory conditions.

Figures 3.29-3.31 display the initial absorption for OPC compared to 3%, 6%, and 12% MPCM mixtures. Each point in these graphs is an average of two readings. A linear relationship between cumulative absorption and the square root of time is obtained with a correlation coefficient around 0.96 for all samples. The sorptivity was calculated as the slope of this line. For instance, after 2 hours from running the test (specimens touches the water), the difference in the absorption between OPC and 6% MPCMs specimen reaches approximately 4mm more absorbed by the OPC sample. It's was observed that initial sorptivity is decreasing by the increasing of the MPCM dosage. This could be referred to the densification action happens by adding MPCMs, and the ability of MPCM particles to obstruct and interrupt the capillary network (Kheradmand, 2018). This results in a reduction of the total permeable pores of the cement matrix. Therefore, the more you increase the inclusion of MPCMs, the lower the slope of the line and the rate of capillary suction in diminishing.

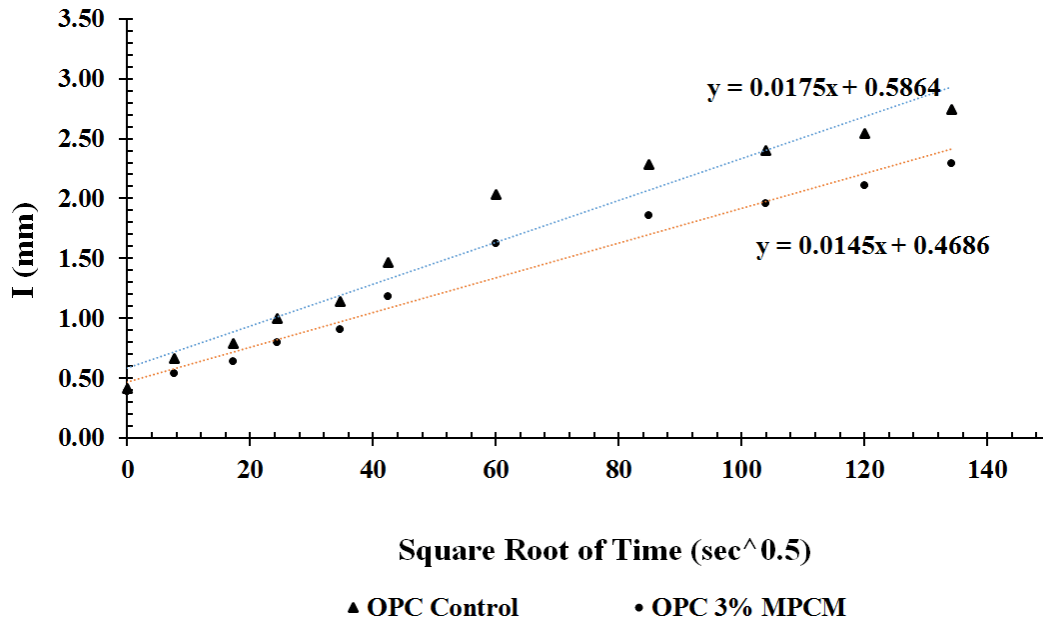


Figure 3.29 Initial Sorptivity OPC/OPC 3% MPCM.

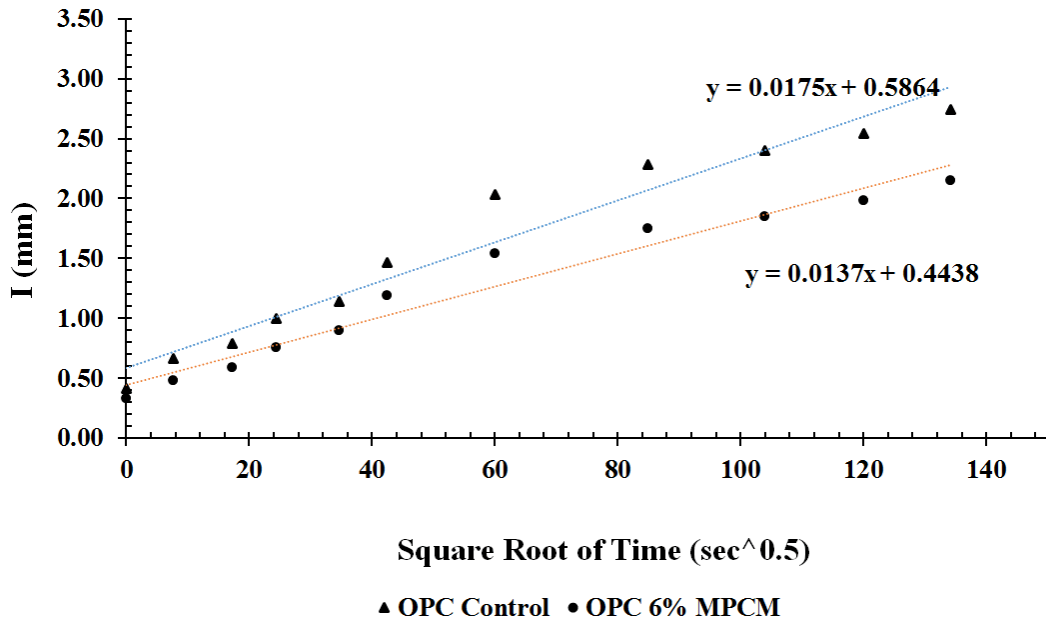


Figure 3.30 Initial Sorptivity OPC/OPC 6% MPCM.

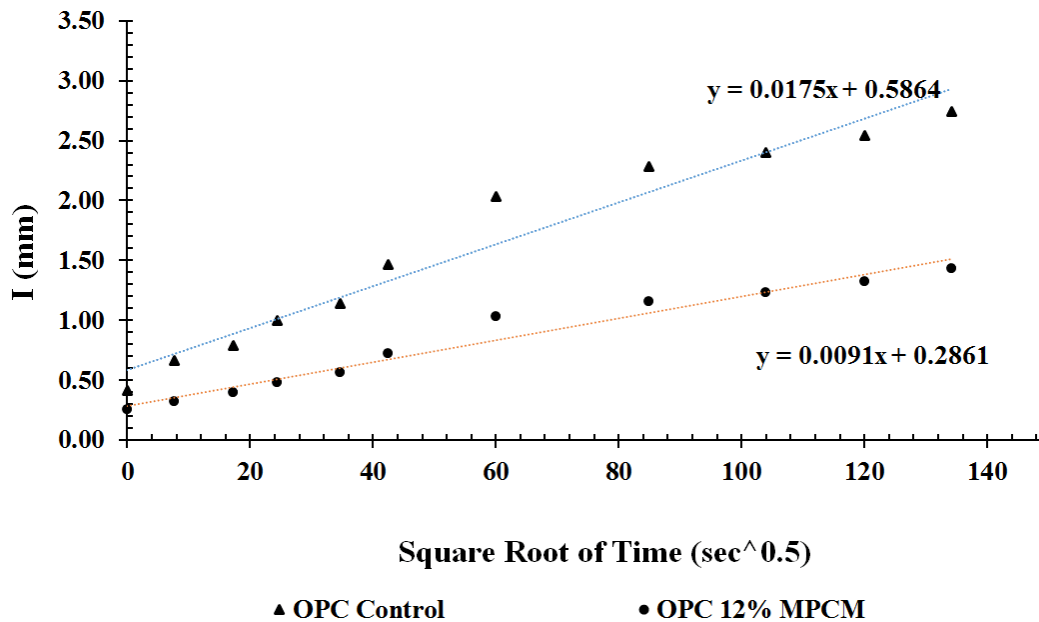


Figure 3.31 Initial Sorptivity OPC/OPC 12% MPCM.

Figures 3.32-3.34 show the secondary sorptivity results for OPC samples with and without the MPCM incorporation. Generally, all the specimens have reached a level of saturation at a certain point. This is called the saturation point for the specimen. At this point the weight of the specimen is constant and will not increase. For instance, the slope of the line at 3% MPCM inclusion is 0.0019, while it was 0.003 for the 6% inclusion. Thus, a different phenomenon is observed for the secondary sorptivity compared to the initial sorptivity. The rate of change for the water sorption decreased faster for the initial sorptivity compared to the secondary, while the rate of change in the secondary sorptivity is relatively slow and mutual. This can be attributed to several factors, like the nature of the MPCMs as a non-sorptive inclusion (Hall, 1993). Therefore, the interface of the MPCMs incorporated redirected the flow around its parameter, and played an intensifying role in the sinuosity of the transport path for the capillary (Wei, 2017).

Generally, MPCMs act as nucleation sites and accelerate the hydration leading to a denser microstructure. Besides acting as filler materials (especially for small particles in the range from $< 3\mu\text{m}$ and up to $10\mu\text{m}$). At the same time, broken MPCMs induce higher voids. The role of this voids will depend on two factors: location and connectivity. Isolated voids away from the surface will mainly have effect the mechanical performance. However, surface connected voids will affect the capillary void structure and water absorption.

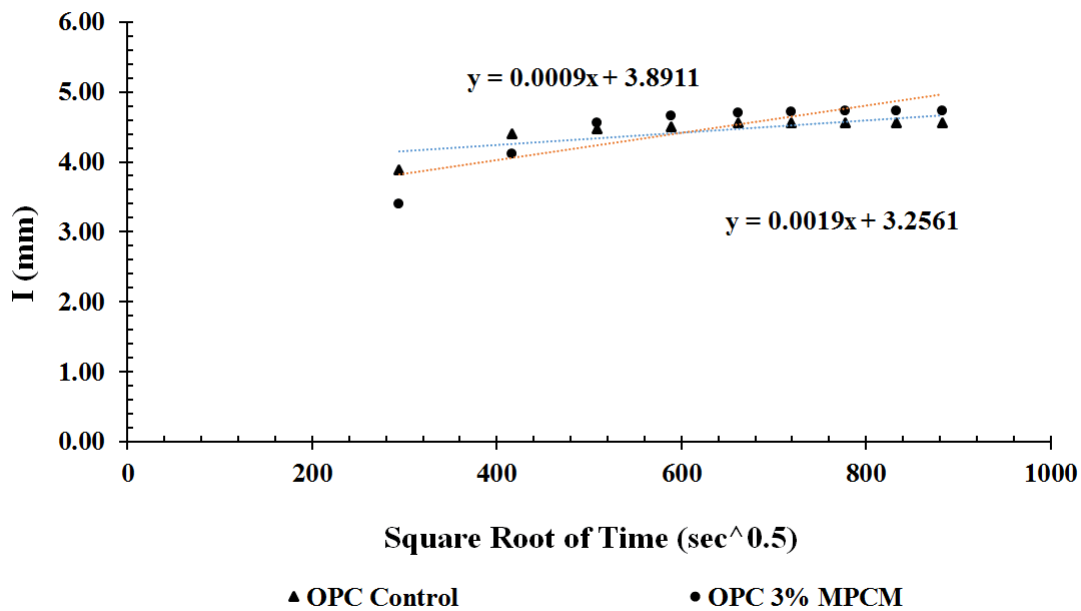


Figure 3.32 Secondary Sorptivity OPC/OPC 3% MPCM.

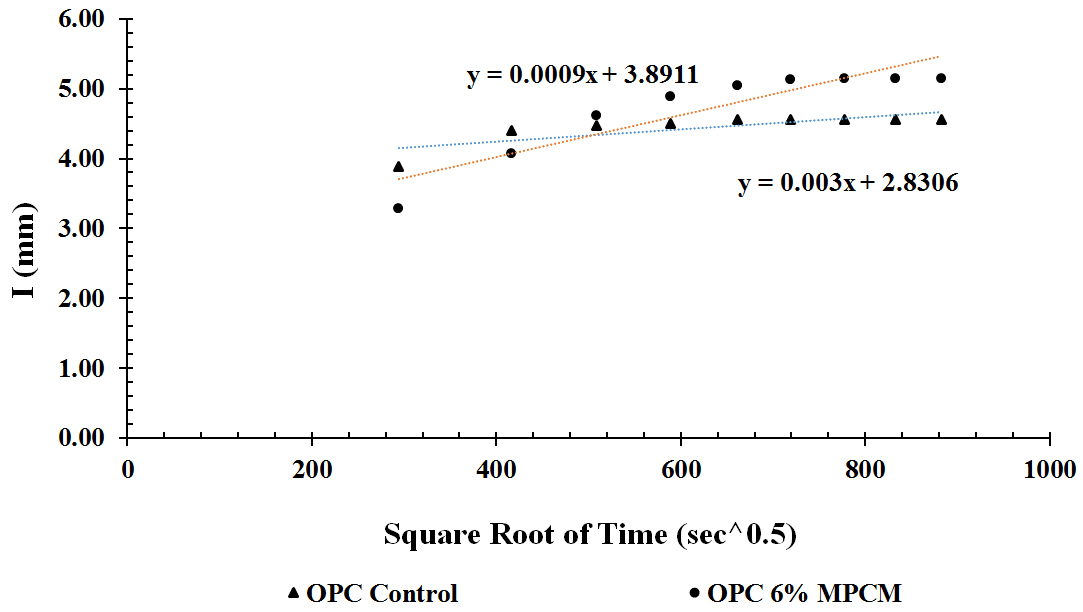


Figure 3.33 Secondary Sorptivity OPC/OPC 6% MPCM.

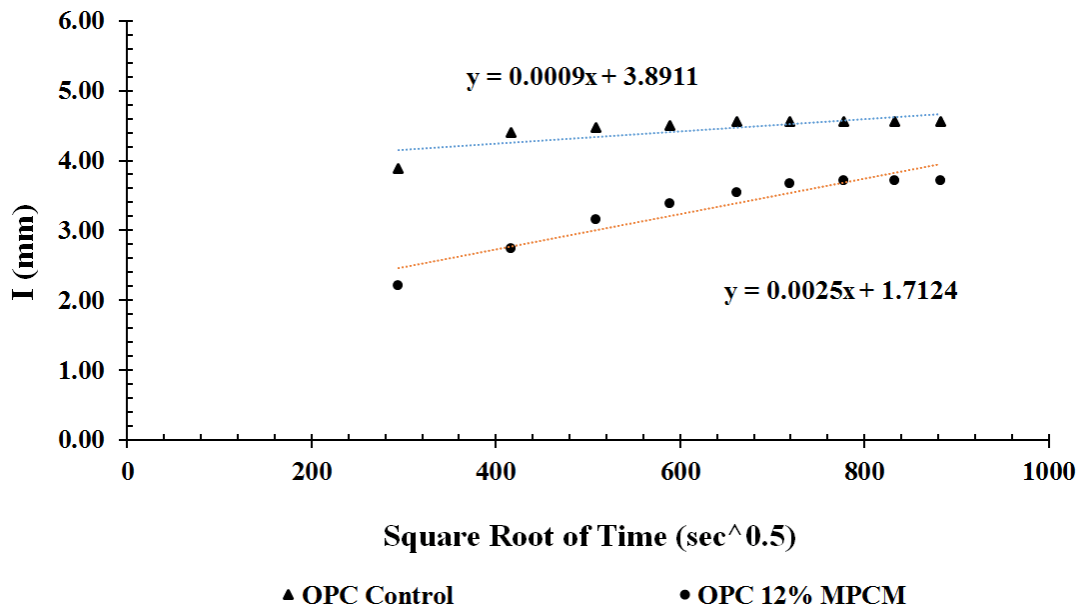


Figure 3.34 Secondary Sorptivity OPC/OPC 12% MPCM.

3.4.9 Volume of Permeable Voids

One more aspect to cover is the volume of permeable voids for mortar mixtures after adding MPCM. To better understand the voids volume, ASTM C642 test was carried out on several samples. **Figure 3.35** shows an average volume of permeable voids for samples with and without MPCM incorporation. Generally, adding MPCM did not induce a significant change to the volume of permeable voids %. While increasing the incorporation percentage showed an increasing of the volume of permeable voids. For instance, a slight increment approximately 1.5% of the volume of permeable voids can be observed for 12% compared to the control. On the other hand, comparing the 0% to 3%, a slight decrease of around 6% in the average total volume of permeable voids can be noticed. This can be ascribed to the densification action that happens from adding the microcapsules to the cementitious construction materials (Kheradmand, 2018). Leading to a

reduction in the total number of permeable voids. Moreover, the breakage of the capsules from the mechanical loading during the mixing, which can lead to increase voids and spaces inside the matrix. Keeping in mind that the breakage of the capsules is suspected to be severe or not, the microcapsules are inert non-sorptive materials act more as voids in cementitious materials (Lecompte, 2015).

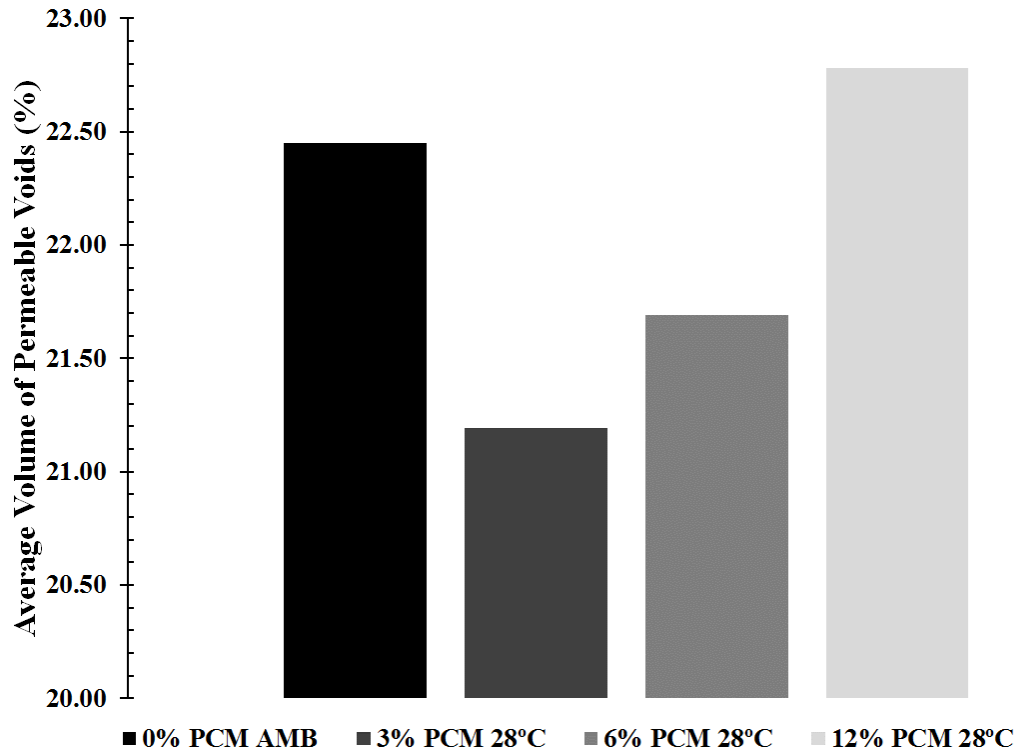


Figure 3.35 Average Volume of Permeable Voids (%)

Concluding, it was noticed that by increasing the MPCM incorporation, sorptivity is decreasing while the RCPT results showed an increase in the charged passed, and at the same time the compressive strength results are decreasing. This can be ascribed to the nature of MPCM incorporation and its effects. As mentioned, increasing MPCMs will lead to an increment of the porosity; either by the connection between MPCMs particles or by the voids that MPCMs leaves if it was broken. Nonetheless, increased porosity may not be a connected voids, which contributed

to decreasing the capillary action. On the other hand, these voids and interfacial zones, when it comes to loading resistance alongside with the brittle nature of microcapsules shells will affect the strength negatively.

CHAPTER FOUR

PERFORMANCE OF CEMENT MORTAR INCORPORATING PHASE CHANGE MATERIALS CURED AT HIGH TEMPERATURE

4.1 Introduction

In some construction projects, some of the cementitious elements have to be produced in a short time. This can be always the case for project carried in busy/crowded places, or tight scheduled projects (Maltais & Marchand 1997). However, curing time for cementitious elements is lengthy process in the production sequence. Accordingly, in precast concrete production and in winter placement conditions, applying heat curing to accelerate the cementitious materials microstructural development is a common solution. That will allow faster demolding/reuse of the molds to recast again and accelerate the production process. Some researchers have discussed the curing regime at low temperature and its capabilities to increase the strength development of mortars (Maltais, & Marchand, 1997). Some other researchers have discussed the curing temperature for heat/steam curing. It was recommended to be 60 °C. Moreover, it was stated that increasing the pre-curing time may increase strength development and the durability by promoting the pozzolanic activity (Yang, 2003 & Zhimin, 2012). On the other hand, MPCMs have the ability to store and release heat at some rate depending on the surrounding temperature. Therefore, this chapter examines, for

the first time, the potential of using MPCMs to shorten heat curing time. The effects of adding MPCMs as a volume replacement for sand on the performance of mortar cured at high temperatures, (i.e. temperature (T) = $60^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and relative humidity (RH) = $80\% \pm 5\%$) were investigated.

4.2 Experimental Work

4.2.1 Materials

Materials and testing procedures were the same as previous chapter (Chapter # 3). The only difference is the applied curing regime as will be discussed in the following section.

4.2.2 Testing and specimen preparation

Using the aid of thermocouples impeded in fresh mixtures were used to draw the heat evolution profile inside mixtures. Cylindrical samples with the dimensions of 50 mm \times 100 mm were prepared, and thermocouples type (T) were used to monitor the temperature change inside of the samples to follow temperature evolution in the samples from casting until at least 24 h.

The curing regime in this chapter consists of several stages as shown in **Fig. 4.1**. A pre-set period of 3 hours starting from mixing at ambient temperature. (23°C) is followed by a ramping period or charging, where the temperature is raised on a rate of $17.5 - 20^{\circ}\text{C}/\text{Hour}$ up to 60°C . Then, holding period where the temperature is kept at 60°C for 5 or 10 hours. Finally, the cooling period, during which temperature decreased by the same increasing rate until it reaches ambient temperature again. The choice for the 10 hours under hot temperature treatment was based on the previous recommendation for heat curing for cementitious materials (Ramezaniapour, 2013, Ramezaniapour, 2014). The 5 hours curing regime was selected to examine the effect of shorten curing time (i.e. energy saving) for mixtures incorporating MPCMs. This process was stimulated using and environmental chamber with a controlled heating, cooling, and humidity.

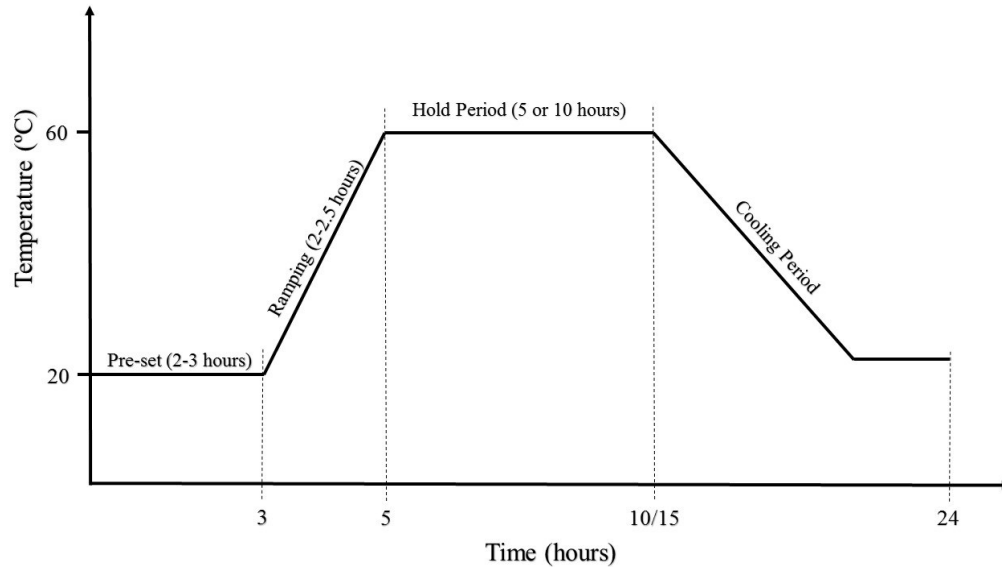


Figure 4.1 Different stages for heat curing regime

4.3 Results and Discussions

4.3.1 Compressive Strength

Compressive strength is one of the main factors judging the performance for cementitious materials incorporating MPCMs. A comparison between the elevated curing temperature and the ambient curing temperature (from the previous chapter) will be illustrated. This will be helping illustrating the benefits induced to heat cured mortars with incorporating MPCMs.

Figure 4.2 represents the compressive strength development for the control mixture under different curing regimes. Generally, the compressive strength increased over time. For instance, at the age of 7 days, control mortar cured under hot temperature for 10 hours has more strength compared to the same mortar under ambient and 5 hours curing, with about 12% and 9%, respectively. Moreover, at the age of 28 days control mortar cured under 10 hours hot temperature, compared to the mortar under ambient and 5 hours hot temperature curing, achieved more strength with 22% and 11%, respectively. Hence, the longer the heat curing, the higher the compressive strength. Besides, longer hot temperature curing time for control mortar exerted better-developed

strength over time. This is confirming with the findings from previous research Ramezaniapour (2013). Furthermore, the increased compressive strength could be referred to as the accelerated hydration reaction and the promoted Calcium silicate hydrate formation (Richardson, 2004).

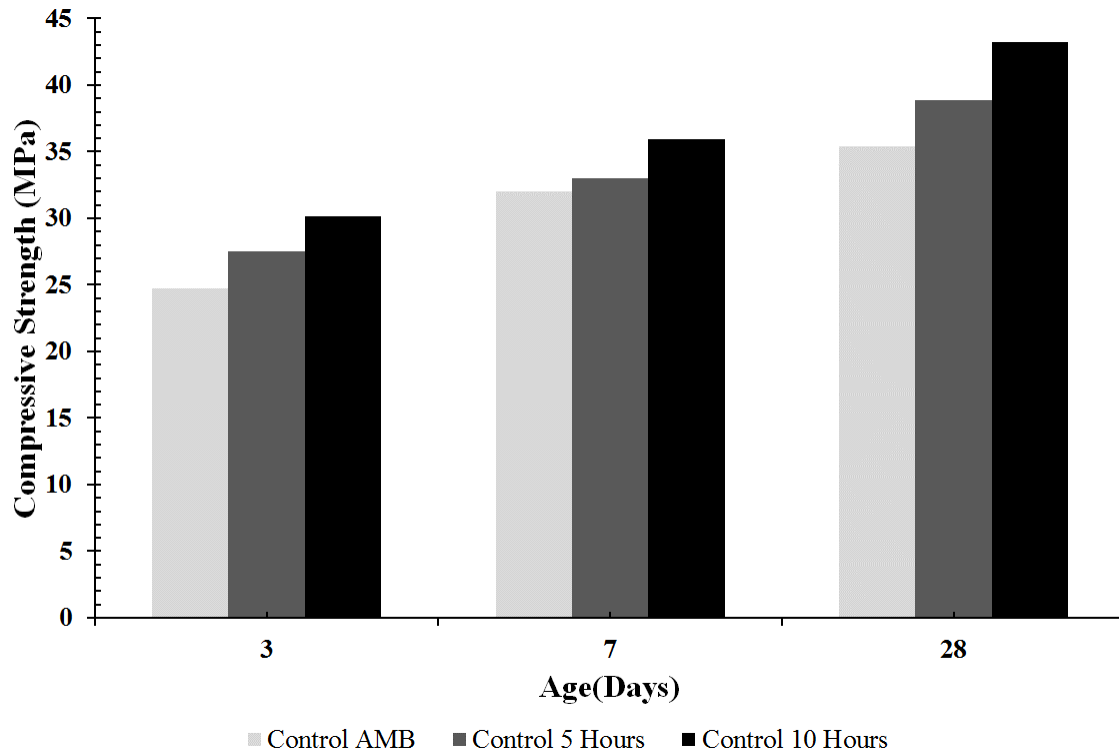


Figure 4.2 Compressive strength for control mortar under ambient temperature, hot temperature for 5 hours, & hot temperature for 10 hours

Figure 4.3 displays strength development for specimens incorporating MPCM 28 under hot temperature for 5 hours. Generally, the compressive strength increased with time. However, the achieved compressive strength for all MPCMs mortars were lower than that of the control mortar with 0% MPCMs. For instance, adding 3% MPCM resulted in a reduction in the compressive strength at the age of 28 days with 4% compared to that of the control mixture. Also, adding 12% MPCM induced more reduction in the compressive strength around 19% compared to that of the control.

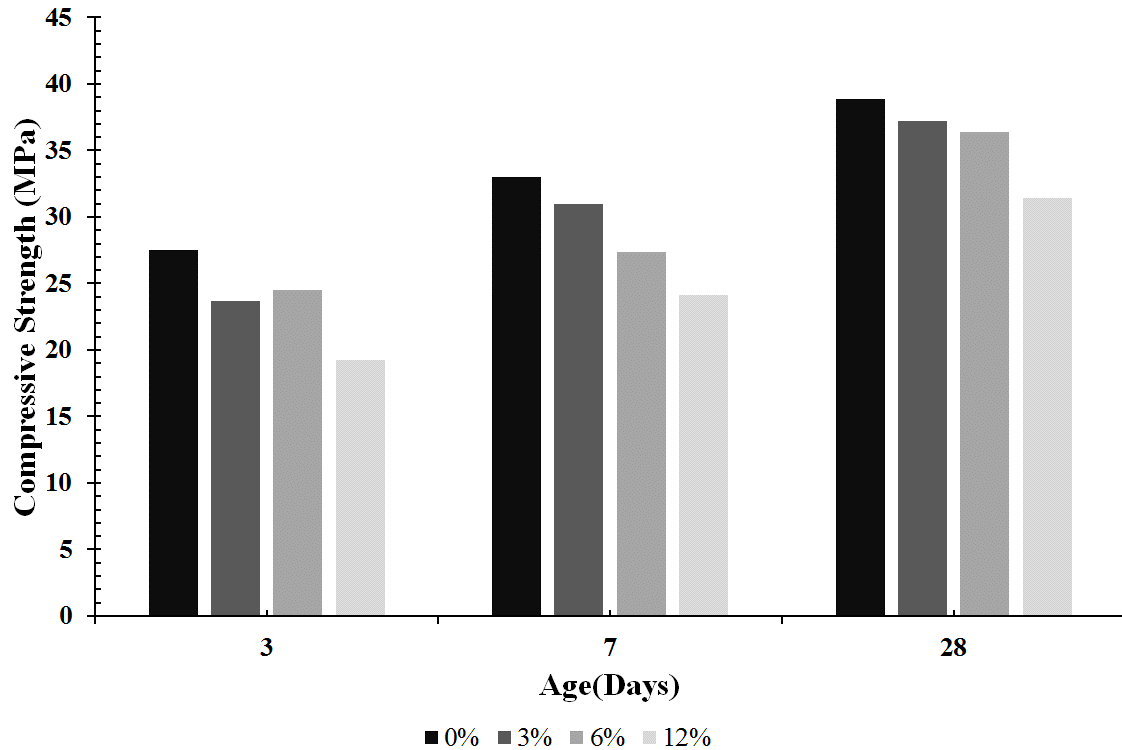


Figure 4.3 Compressive strength for mortar incorporated MPCM 28 under hot temperature for 5 hours.

Nevertheless, adding 6% MPCM did not produce a considerable reduction in the compressive strength compared to 3%. Moreover, at early ages, the strength development rate for MPCM mortars was higher compared to the later ages. Finally, MPCM incorporation induced a reduction in the compressive strength under 5 hours hot temperature curing condition, compared to the control.

The second curing regime used in this research was the 10 hours hot temperature curing condition. **Figure 4.4** shows the development of compressive strength for MPCM 28 incorporated mortar under 10 hours hot temperature curing regime.

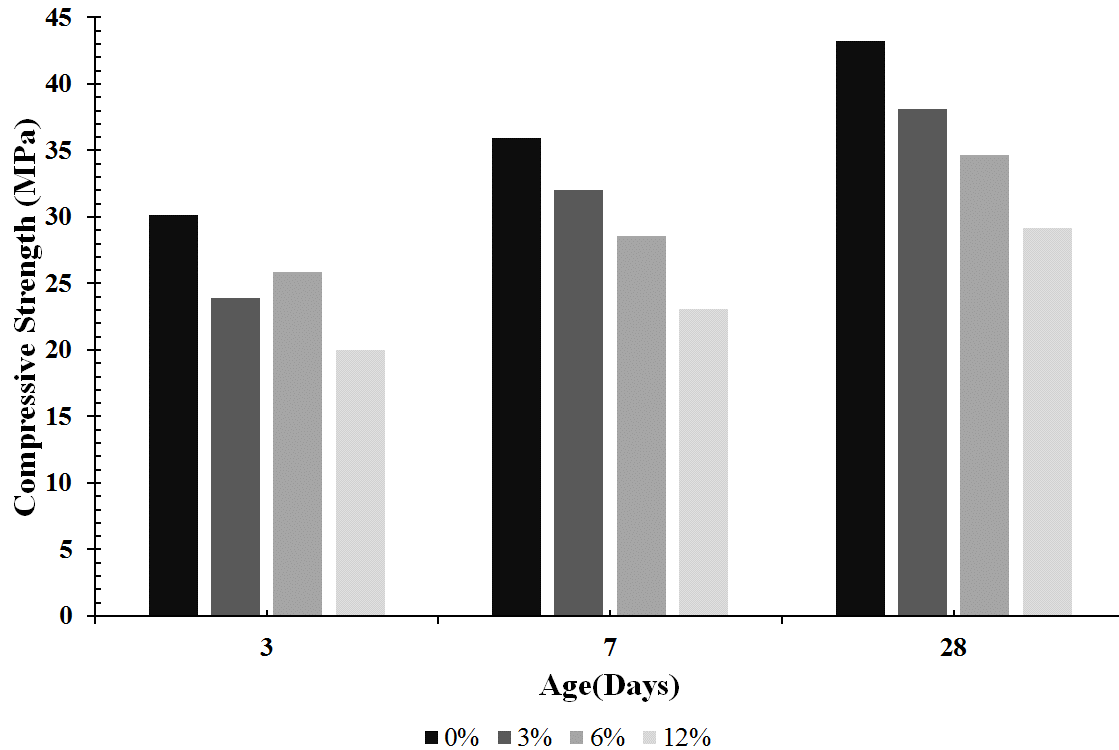


Figure 4.4 Compressive strength for mortar incorporated MPCM 28 under hot temperature for 10 hours

Generally, the compressive strength increased with time. Similar to 5 hours hot temperature curing, the compressive strength for all mortars with MPCMs were lower than that of the control mortar. For instance, adding 3%, and 12% MPCMs resulted in 12% and 33% reduction in the 28 days compressive strength, respectively. Besides, 6% of incorporation has achieved more strength under 10 hours of curing condition at early ages compared to 3% and 12% with 8% and 29% respectively. The main observation was for hot temperature curing regimes, 6% incorporation has exerted the highest compressive strength amongst the used MPCM incorporation percentages at early ages. **Figure 4.5** illustrates the compressive strength development for 6% MPCM 28 incorporated mortar under several curing regimes. For instance, at age of 28 days, specimens cured under 5

hours hot temperature, achieved more strength with 11% and 7%, compared to specimens cured under ambient condition and 10 hours hot temperature, respectively.

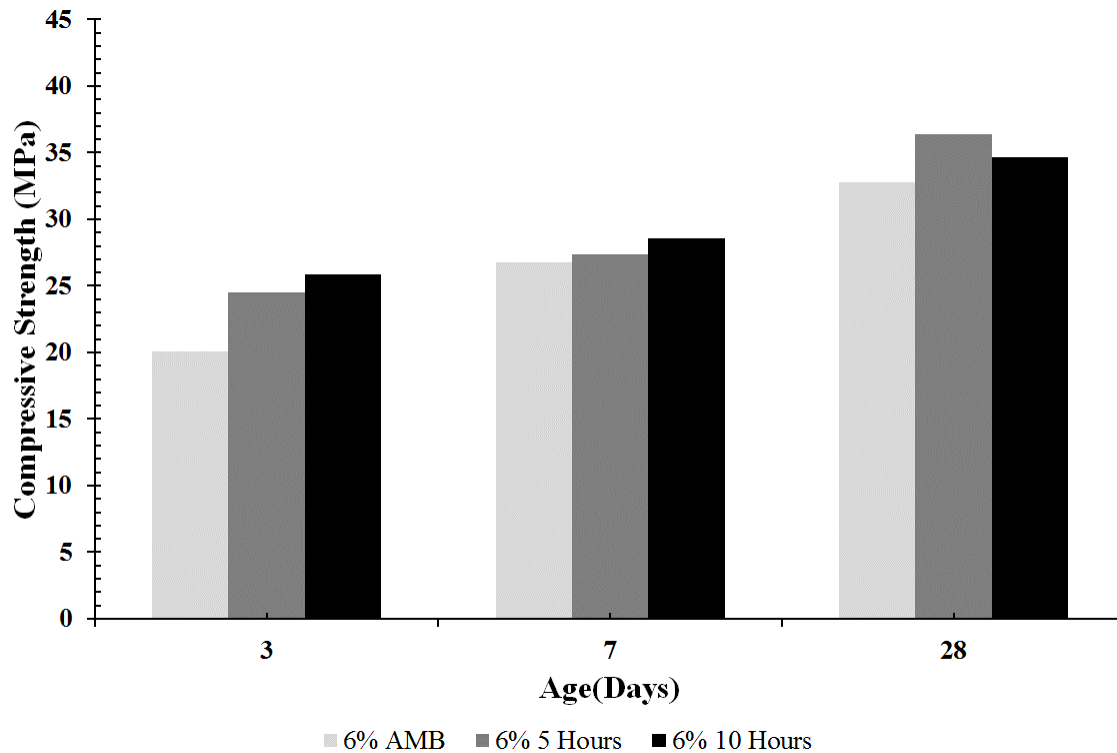


Figure 4.5 Compressive strength 6% MPCM 28 incorporating mortars under different curing regimes

Figure 4.6 compares compressive strength differences for specimens with and without the incorporating of MPCM 28 undergoes the hot curing regimes, and ambient temperature curing regime at age 28 days. For instance, control specimens have showed an increasing strength trend with increasing temperature and curing duration. For 3% MPCMs specimens, it follows the same trend as the control but with an overall reduction caused by MPCM incorporation. Nevertheless, for the 6% and 12% specimens, undergoes 5 hours hot curing regime compared to the ambient curing and 10 hours curing, achieved more strength at the 28 days, around 5% and 8%, compared to 10 hours, respectively.

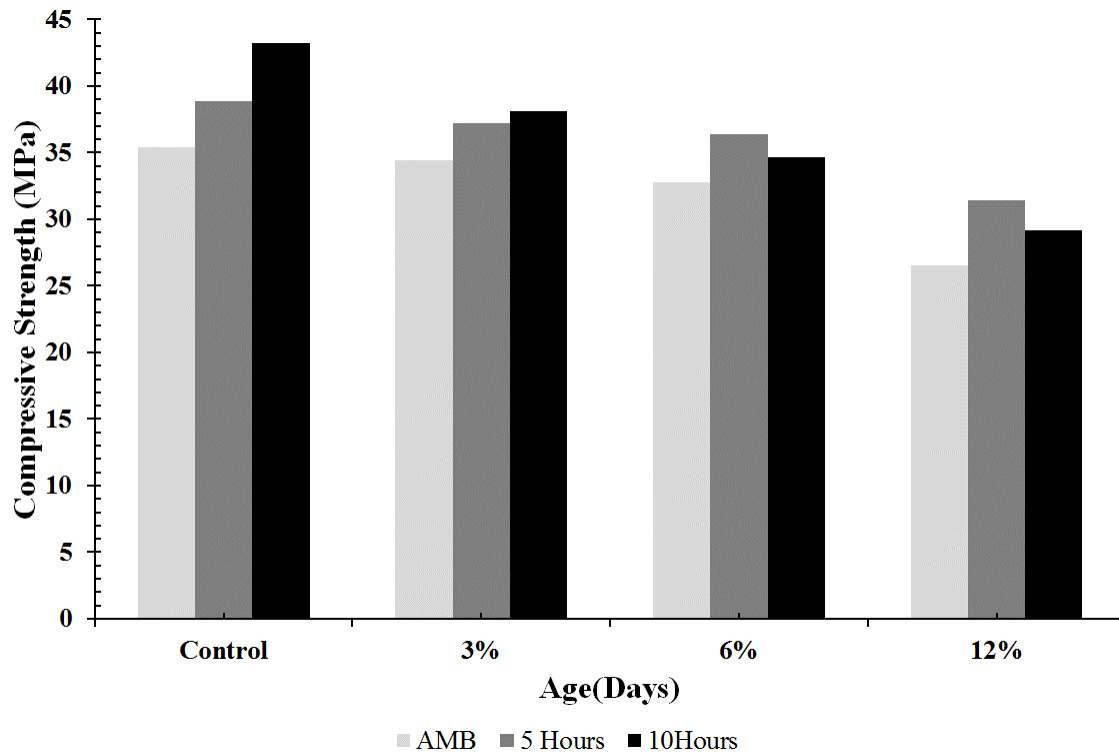


Figure 4.6 Compressive strength for control mortars & MPCM 28 incorporated mortars at 28 days under different curing conditions

Figure 4.7 displays the compressive strength results for MPCM 43 mortar mixtures under hot curing conditions for 5 hours. Generally, an increase in the strength can be observed among all the tested mixtures. However, the strength development overtime was not as high as the case of mixtures incorporating MPCM 28. For instance, 6% achieved less compressive strength at the age of 28 days compared to the control with about 30%. Furthermore, mixtures with 3% and 12% MPCMs achieved compressive strength at the age of 28 days with about 22% and 39% lower than the control, respectively. Concluding, MPCM 43 incorporation under hot temperature for 5 hours exerted a loss in the compressive strength much more than MPCM 28.

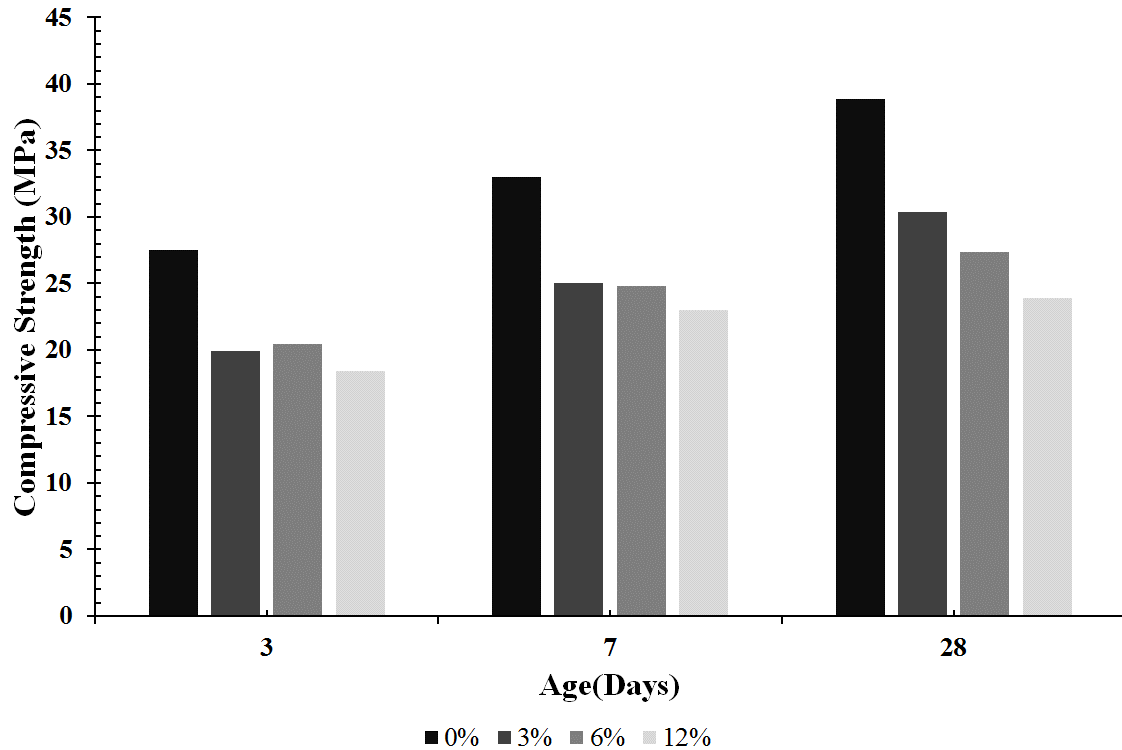


Figure 4.7 Compressive strength for mortar incorporated MPCM 43 under hot temperature for 5 hours

Figure 4.8 illustrates the compressive strength development for mortar incorporated MPCM 43 under 10 hours hot temperature curing. Generally, the compressive strength increased over the studied period. Moreover, control mortar mixtures without MPCMs attained more strength than all mixtures at all ages. For instance, adding 3% MPCM resulted in a reduction in the compressive strength at the age of 28 days, with about 24% compared to that of the control. Moreover, adding 6% and 12% MPCMs resulted in 34% and 37% reduction in the 28 days compressive strength, respectively. Finally, MPCM 43 incorporation induced a reduction in the compressive strength under 10 hours hot temperature curing condition, compared to the control.

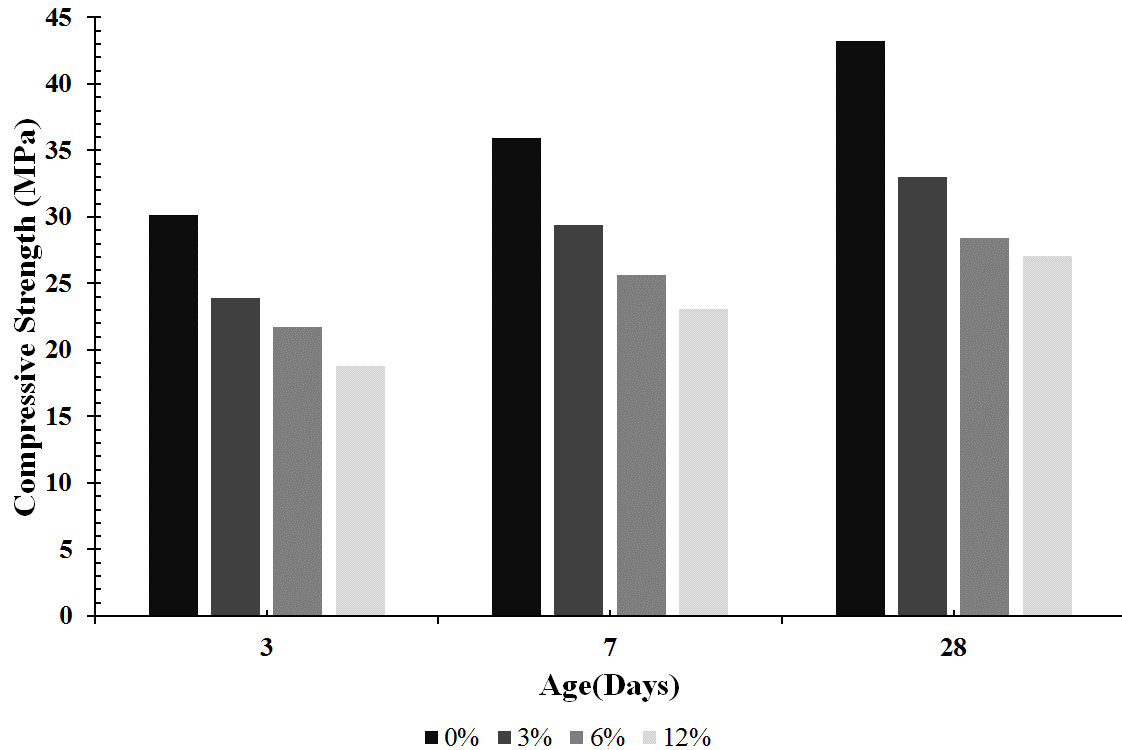


Figure 4.8 Compressive strength for mortar incorporated MPCM 43 under hot temperature for 10 hours

Figure 4.9 compares compressive strength for specimens with and without MPCM 43 undergoes the hot curing regimes, and ambient temperature curing regime at age 28 days. Control specimens have shown an increasing strength trend with increasing temperature and curing duration. For 3% specimens, 10 hours of hot temperature have gained more strength compared to 5 hours with 9%, which is the same strength as specimens exposed to ambient curing conditions. This trend indicates that MPCM 43 has more detrimental effects with an overall reduction caused by MPCM incorporation. Nevertheless, the same pattern with different percentages was captured with 6% and 12% specimens.

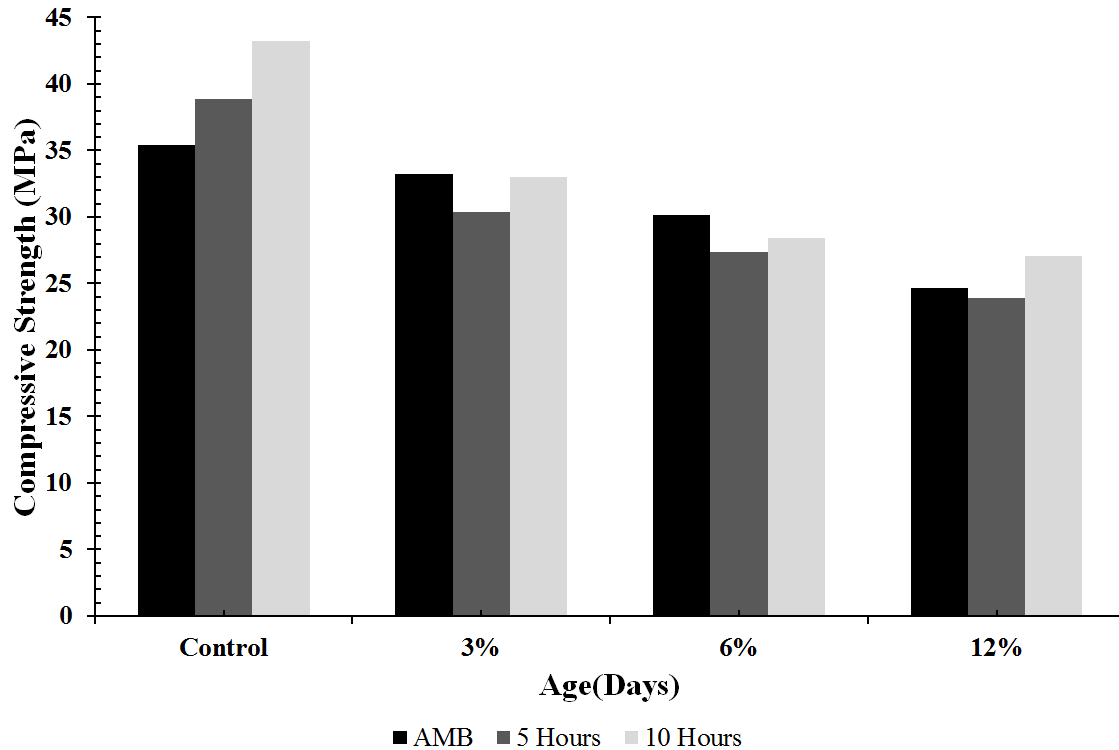


Figure 4.9 Compressive strength for control mortars & MPCM 43 incorporated mortars at 28 days under different curing conditions

Previously mentioned trends and captured behaviors can be explained by the dosage amount and its rational effect on the properties. On the other hand, if any breakage happened to the small amount of MPCM during the mechanical mixing, it detrimentally affects the heat capacity properties of the incorporated MPCMs, and the compressive strength development as well (Eddhahak-Ouni, 2014). The increased strength for specimens exposed to hot curing has become evident as the MPCM dosage increased compared to the same specimens dosages under the ambient condition. This can be attributed to the heating effect. Since increasing the heat curing up to 50°C - 70°C, at such moist environment (RH \pm 80%) is reported to increase the strength development (Neville, 1995). On the other hand, MPCMs could extend the hydration process by controlling the storage and release of such heat. Moreover, the compressive strength for

incorporated mortars showed less compressive strength development under 10 hours of hot temperature curing. It was mainly caused by the thermal expansion microcapsules may suffer by temperature, which leads to less resistance against loading (Zhang, 2004).

MPCM 28 worked better under hot curing conditions compared to the MPCM 43. Moreover, incorporation of 6% has exerted the most effective strength development for MPCM 28 under hot curing conditions. In addition, MPCM 43 exerted better strength under 10 hours hot temperature curing regime compared to the 5 hours hot temperature. Under the ambient curing, MPCM28 and MPCM 43 did not change the phase or contribute to the progression of the reaction, since the temperature gradient did not exceed their phase change temperature, which supports the implication of their presence as soft, inert material.

4.3.1.1 Compressive Strength under MPCM 28 and MPCM 43 Combination

One more trial has been made for the incorporation of MCPMs. In this trail both types of MCPMs, 28 and 43 were incorporated 50:50. This hybrid mortar with two distinct MCPMs, reported improving the thermal behavior of the mortar composite (Kheradmand, 2014). Results were following the same pattern of the most effective achieved strength from the previous trails. Mainly compressive strength of 6% incorporation may be expected to exert better strength compared to 3% and 12% MCPMs. On the other hand, MPCM 28 is supposed to contribute more in the combination.

Figure 4.10 represents the compressive strength development for mortar specimens incorporating both MCPMs under 5 hours hot temperature curing regime. Generally, the compressive strength increased over the studied period. Also, all incorporated combinations had shown approximately the same rate of strength development. For instance, at the age of 28 days, both 6% and 12% have achieved a lower strength than that of the control with about 31% and 38%, respectively. On the

other hand, 3% MPCMs at age 28 days has developed better strength compared to 6% and 12% MPCMs with about 8% and 21%, respectively.

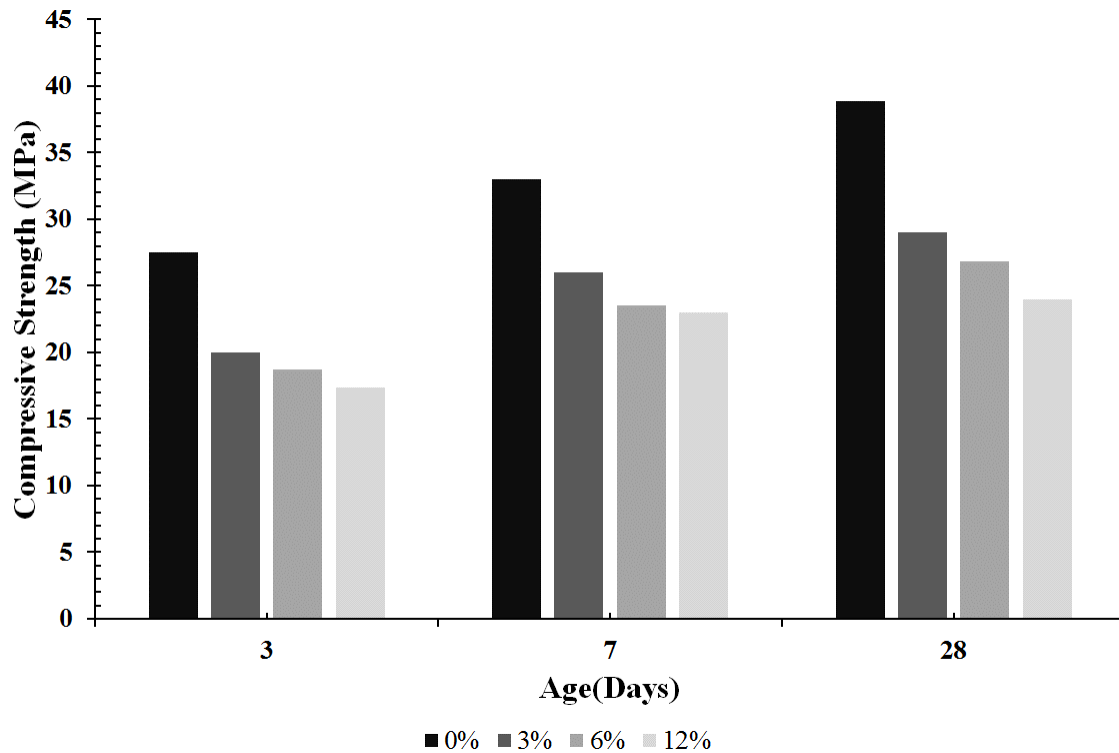


Figure 4.10 Compressive strength for mortar incorporated MPCMs 43&28 under 5 hours hot temperature curing

Figure 4.11 delineate the compressive strength behavior for mortar specimens incorporate with MPCM 43 and MPCM 28 under 10 hours hot temperature curing regime. The compressive strength increased for all specimens over the investigated period. For instance, at the age of 28 days, both 6% and 12% MPCMs mixtures had exhibited a strength less than that of the control with about 40% for both mixtures. On the other hand, 3% MPCM mixture at 28 days has developed better strength compared to 6% and 12% MPCMs mixtures with about 16% and 19% respectively.

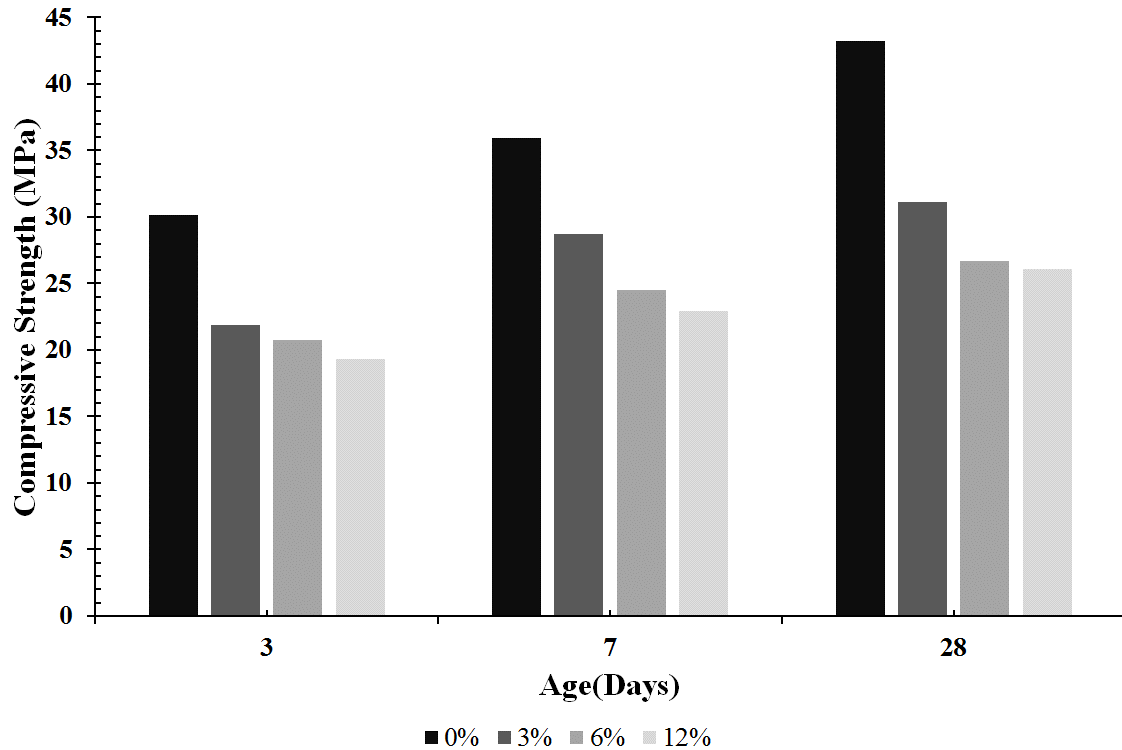


Figure 4.11 Compressive strength for mortar incorporated MPCMs 43&28 under 10 hours hot temperature curing

In the literature review, a study by Kheradmand (2014) reported the use of different kinds of MPCMs in one combination. Which they called it a hybrid microencapsulated PCMs. It was reported to be consistent in the result regarding the effect of the used MPCMs individually. Our findings here confirms the phenomenon of having different MPCMs with different behavior. However, our focus is mainly the compressive strength. Results confirm that each type of MPCMs contribute to the hybrid mortar mechanical strength in a similar way when it was added alone.

4.3.2 Tensile Strength

Figure 4.12 shows the tensile strength development for several mortar mixtures incorporating MPCM 28 cured under 5 hours hot temperature. Tensile strength increased over time for all mixtures. Generally, tensile strength has developed over time for the incorporated mortar. It can be observed that all mixtures with MPCM incorporation, achieved lower strength compared to that of the control mixture. For instance, the tensile strength at the age of 28 days for 6% MPCMs mixture compared to the control is lower with around 22%. Also, 12% MPCMs mixture achieved higher tensile strength with about 4% compared to the 6% MPCMs mixture at age 28 days.

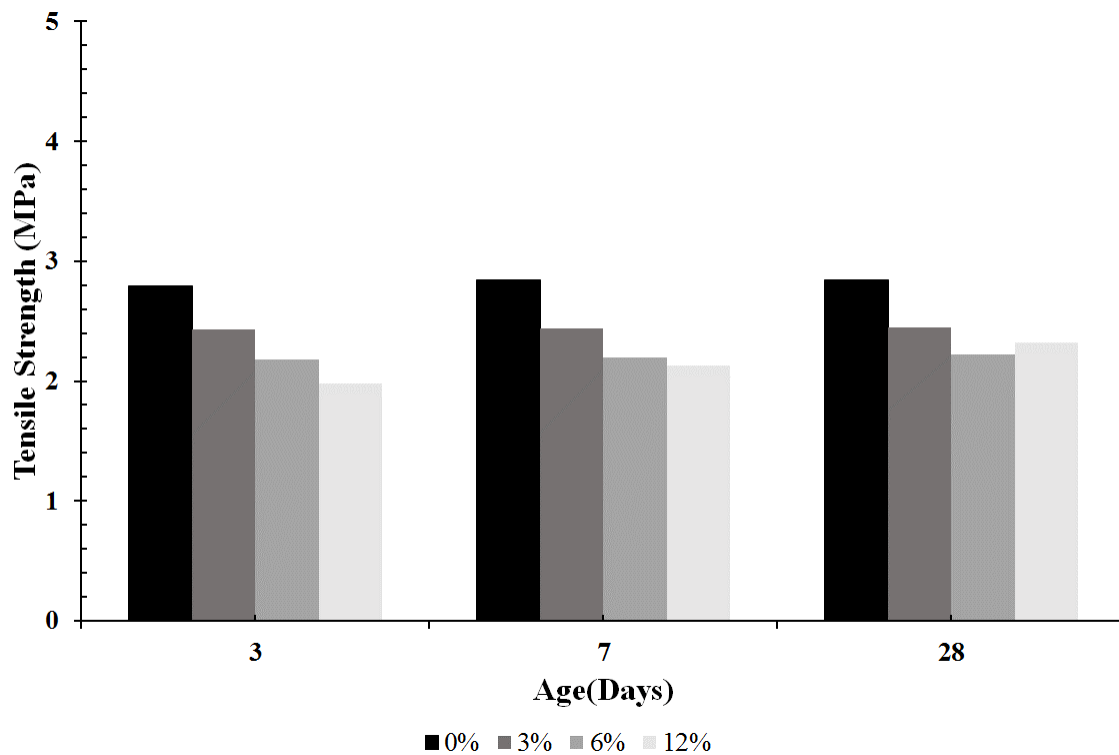


Figure 4.12 Mortar incorporated MPCM 28 tensile strength under 5 hours hot temperature

Figure 4.13 displays the tensile strength development for mortar incorporated MPCM 28 under 10 hours curing regime. Generally, tensile strength development overtime for all mortars. For instance, at the age of 28 days, 6% MPCMs mixture compared to the control has developed less tensile strength with a reduction around 16% compared to that of the control. Moreover, 12% MPCMs mixture at the age of 28 days has achieved lower strength compared to that of the control with about 41%.

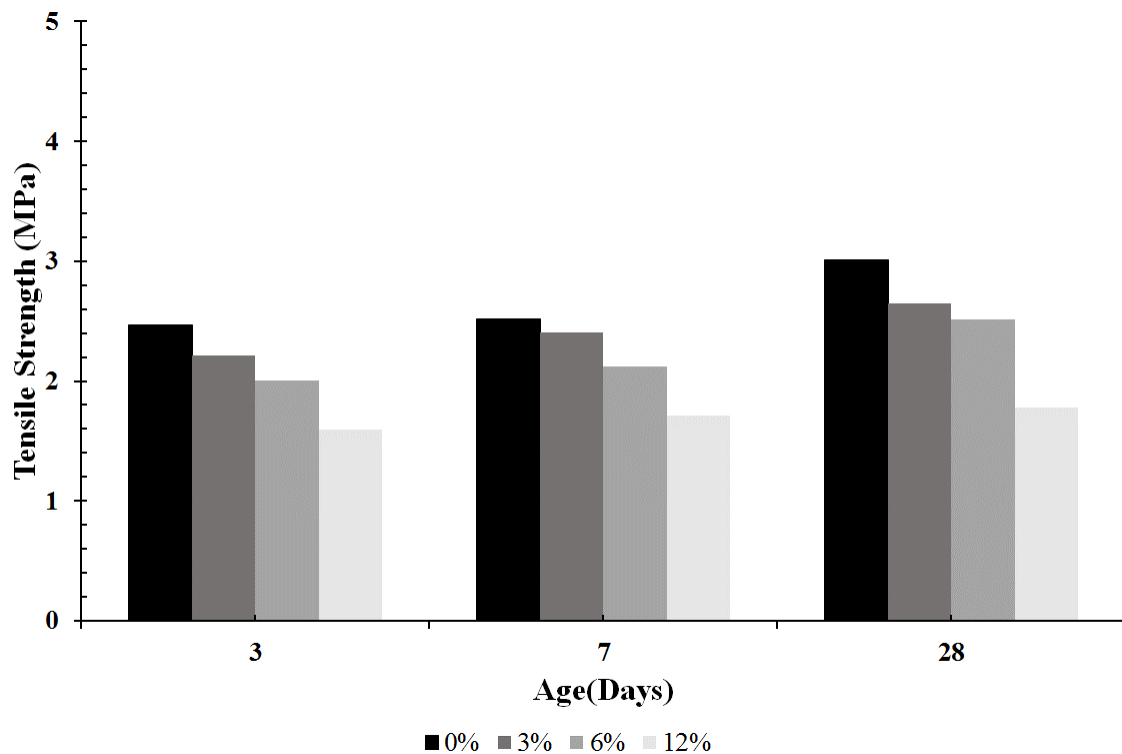


Figure 4.13 Mortar incorporated MPCM 28 tensile strength under 10 hours hot temperature.

This phenomenon can be ascribed to the effect of MPCMs incorporation in cementitious mixtures on the mechanical properties. For this test, particle will suffer a tensile load, which is more dependable on the developed connection inside the matrix. As mentioned before, breakage of the microcapsules with leaked paraffin that plays a part in hindering the interaction between cement

particles and water, which comes from the softness nature of PCM microcapsules. Also, its weakness against load resistance (Eddhahak-Ouni, 2014).

Finally, mechanical strength has developed in the same pattern for cementitious mortar incorporating MPCMs. **Figure 4.14** displays the relation between compressive strength and tensile strength for incorporated mortars. Generally, strength developed over time for all the investigated mortars. As MPCMs increased, mechanical strength were decreased.

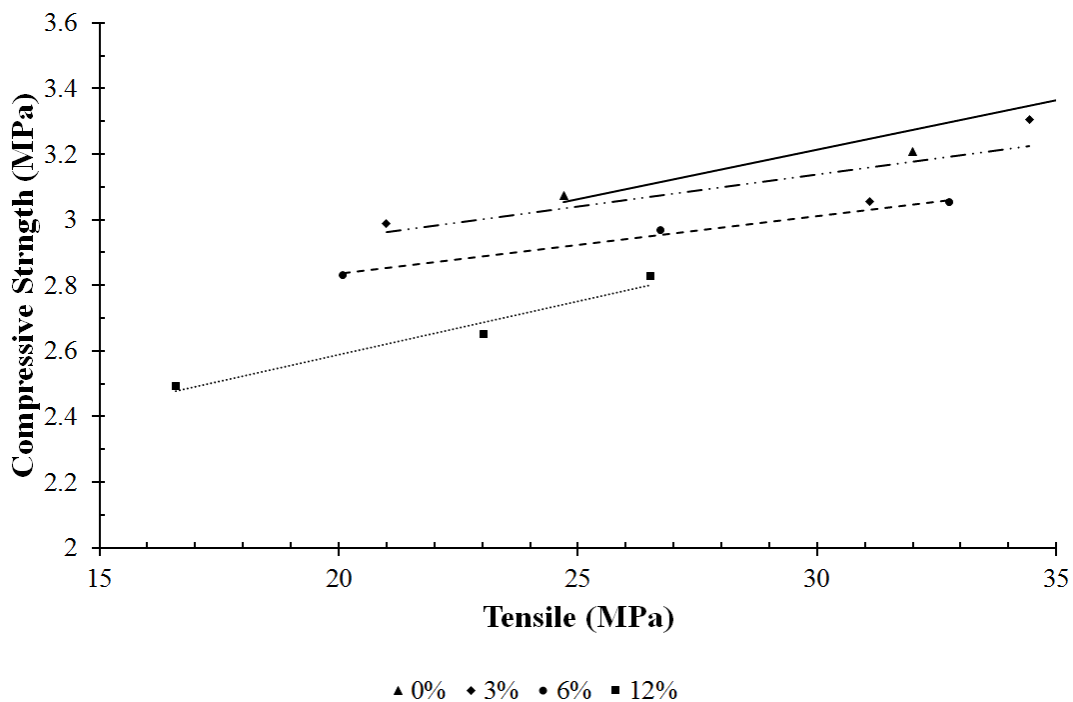


Figure 4.14 Mechanical strength development for mortar incorporating MPCM

Compressive/Tensile

4.3.3 Heat of Hydration

Hydration process starts from the first second of mixing. The hydration is a chemical reaction that happens between the binder and the mixing water. The hydration process is described and followed to elaborate on the effect of MPCMs incorporation on the heat storage and liberation under a hot

temperature curing regime. The samples were prepared at room temperature ($23^{\circ}\text{C} \pm 2^{\circ}\text{C}$) in the calorimeter testing cup. Then, samples were placed in the calorimeter under 23°C for 3 hours. After that, temperature of the calorimeter were raised manually to 60°C . The calorimeter increased the temperature to 60°C in 1.5 hours. Then the temperature was lowered manually again to the 23°C . The calorimeter had taken the same time in the cooling down with about 1.5 hours. Finally the temperature were set on 23°C until the end of the test.

Mortar samples were studied for 22 hours, the test was carried out to capture the peaks of the heat flow produced from the hydration process. After 3 hours, the temperature was raised gradually up to 60°C . Then, after a couple of hours, it was lowered gradually again to 23°C . It can be observed that while raising the temperature, the 3% MPCMs curve had a drop. Which can be ascribed to the effect of the MPCMs at this point, it starts absorbing heat, and temperature exceeds the melting point for phase change temperature. Besides, this stored heat will be released back to the surroundings when the temperature drops down again to hit the phase change temperature in (**Fig. 4.15**).

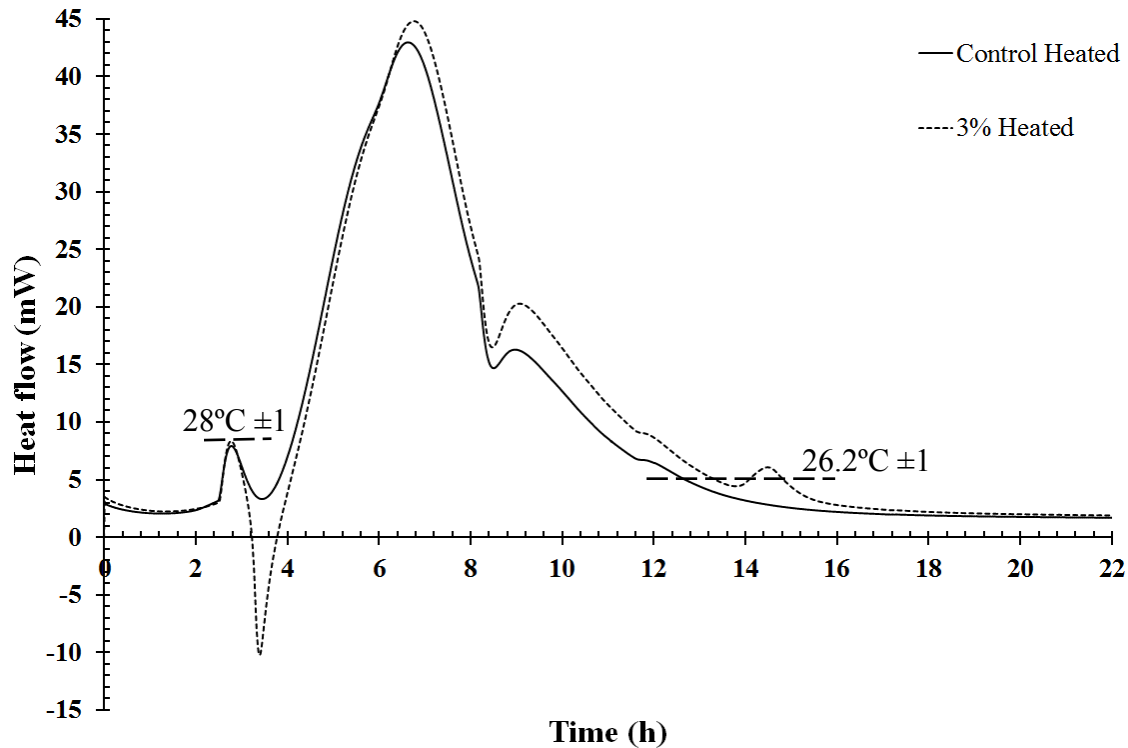


Figure 4.15 Heat flow for control OPC & 3% MPCM 28 OPC, both under hot temperature

To capture the heat flow profile in more details for the MPCM 28 incorporation dosage. **Fig. 4.16** shows the heat liberation profile for mortar incorporated 6% and 12% MPCM 28. Generally, the heat liberation profile followed the same pattern for the 3% MPCM 28 incorporated mortars. Also, this figure offers a more clear display for the heat stored and released, by more MPCM 28 incorporation. Taking into consideration the amount of heat stored in MPCMs, the heat liberated again in the cooling down period is noticeable.

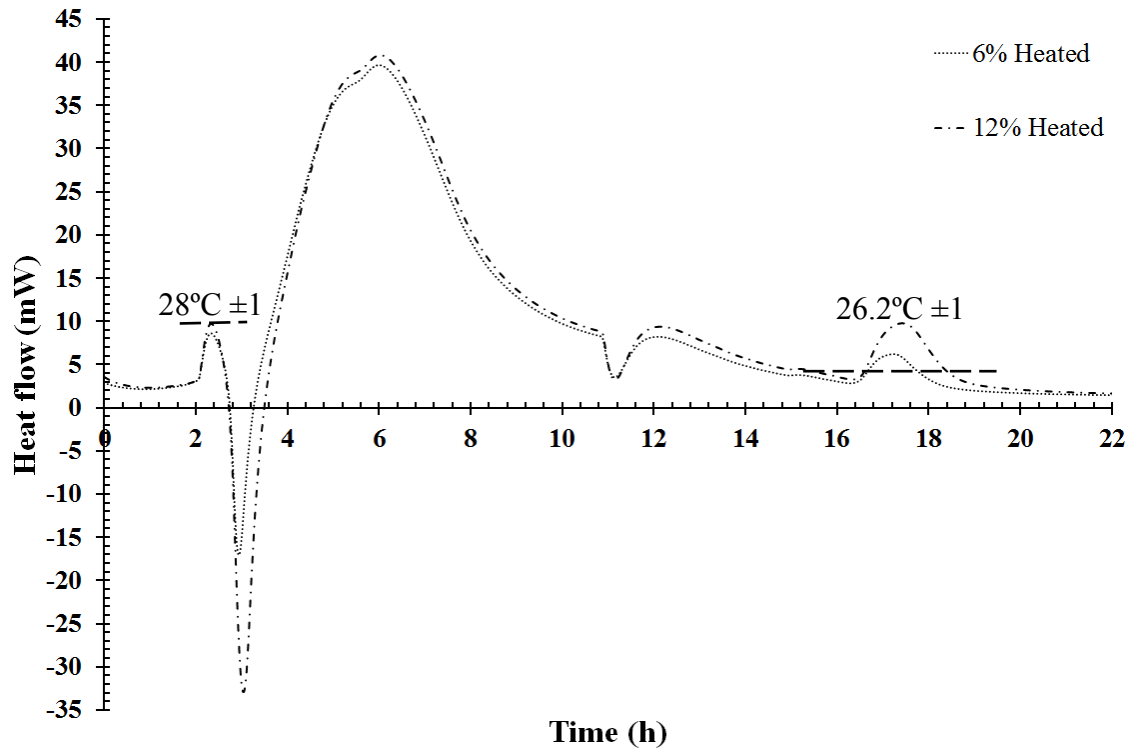


Figure 4.16 Heat flow for Control OPC & 6% & 12% MPCM 28, Both Under Hot Temperature

Figure 4.17 illustrates the heat hydration for mortar incorporating MPCM 43. After raising the temperature, an exothermic peak takes place. Afterward, the heat evolution continues and reach its maximum. On the other hand, the difference in the maximum heat evolution can be noticed between control and 3% MPCM 43 incorporated mortars. The curves show that MPCM 43 incorporation caused a decrease and a delay in the maximum heat of hydration, in contrary to the MPCM 28, which caused an increase and delay at low dosage. The explanation for this phenomenon is mostly dependable on the size of the MPCM particles, which falls in the small group of the cement particles with a size around $10\mu\text{m}$. This small particle work as a physical catalyst for the chemical reaction with their higher surface area (Luo, 2013).

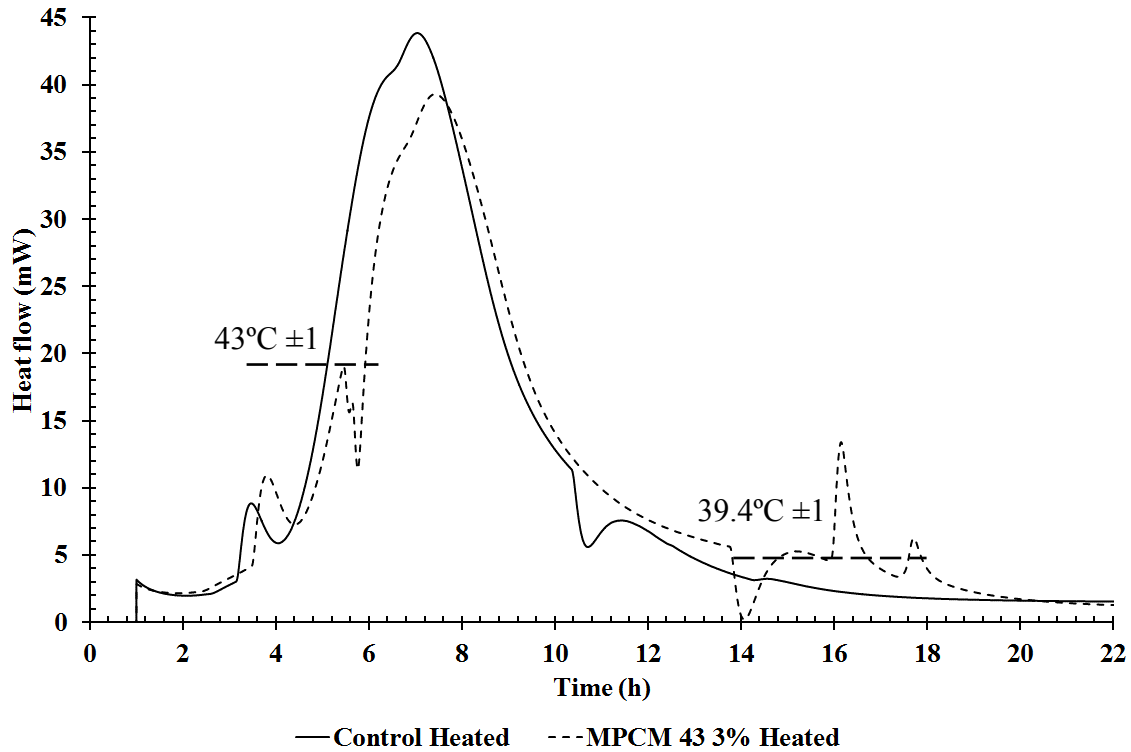


Figure 4.17 Heat flow for control OPC and 3% MPCM 43 OPC, both under hot temperature.

However, it can be observed that while raising the temperature, the second curve has another drop after the dormant period, which can be ascribed to the melting of MPCM 43. It changes the phase from solid to liquid and stores the heat. Furthermore, this stored heat will be released back to the surroundings when the temperature drops down again to hit the phase change temperature, and it can be noticed clearly in **Fig. 4.18**.

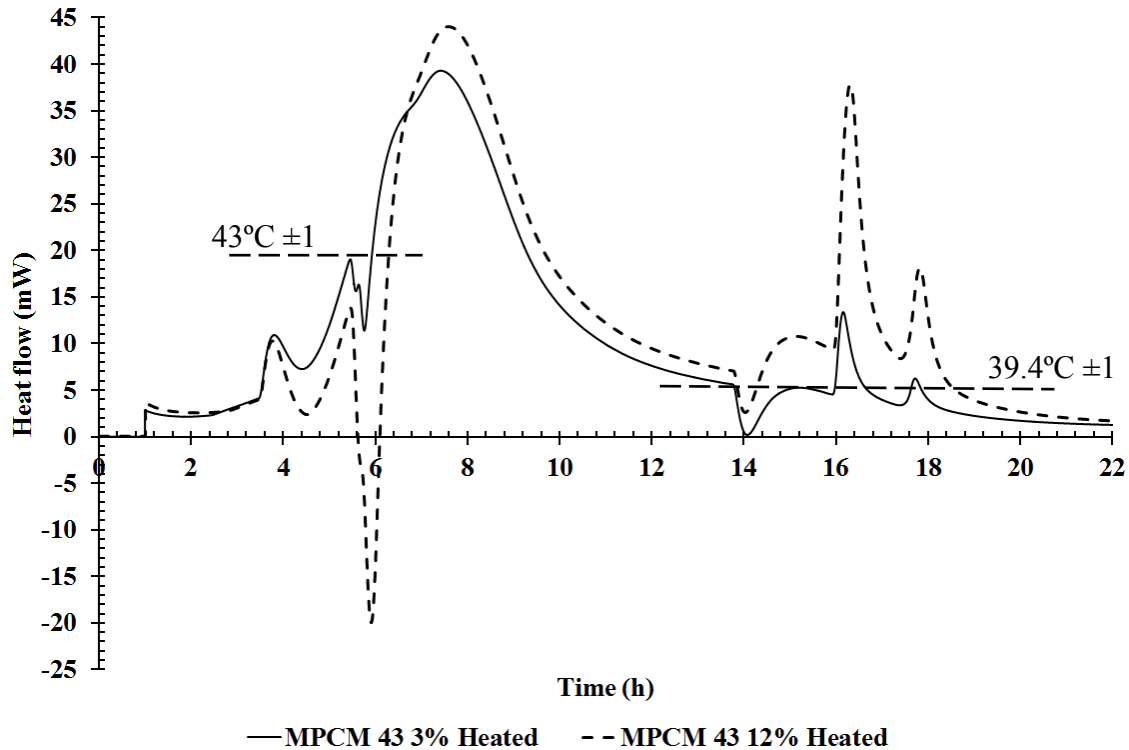


Figure 4.18 Heat flow for 3% MPCM 43 & 12% MPCM 43 OPC, Both Under Hot Temperature

Commonly it can be noticed that MPCM 28 worked better under hot temperature compared to MPCM 43. With higher peaks captured, and more heat liberated and stored. To spot the effect of higher dosages of MPCM 43 incorporation, **Fig. 4.18** displays the heat liberation profile for mixtures incorporating 3% and 12% of MPCM 43. Generally, the heat liberation followed the same expected profile. A noticeable drop in the heat liberation curve, especially at the point that incorporated MPCM 43 starts absorbing heat (phase change temperature). The difference can be ascribed to the difference in the dosage. MPCM 28 did a better performance in heat storage with the same amount compared to that of the MPCM 43. This claim can be observed in **Fig 4.19** which shows a comparison between MPCM 43 and MPCM 28 heat liberation at the same incorporation dosage (i.e. 12%). MPCM 28 stored almost the same heat compared to MPCM 43. Moreover, a

clear delay for the heat absorption is observable, since both MPCMs have different phase change temperature and undergoes the same heating pattern.

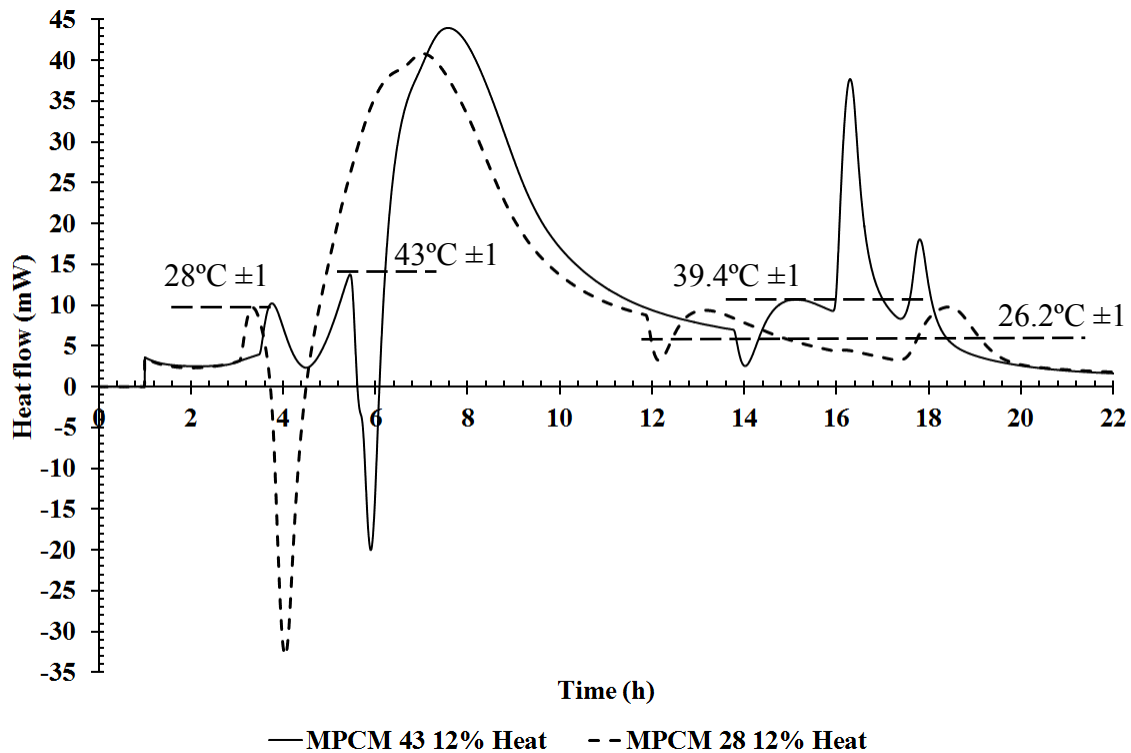


Figure 4.19 Heat flow for OPC 12% MPCM 43 & OPC 12% MPCM, both under hot temperature

4.3.4 Heat Profile

Following the heat profile is one of the most useful ways to proof and detect the effect of PCMs. Phase change materials used in this chapter have different phase-change temperatures, in both (i.e. melting and solidification). The test setup is shown in **Fig. 4.20**.



Figure 4.20 Heat profile test setup

Figure 4.21 displays the full-scale heat profile for two control samples subjected to a heating up to 60 °C, where temperature was plotted as a function of time. Generally, the heat was increased after approximately 4.5 hours from mixing. The temperature for all samples was increased as indicated by the thermocouples inserted inside the chamber and planted in the middle of the cylindrical specimen (5cm X 10cm). After 5 hours, a portion of the samples was moved out of the chamber and placed under laboratory conditions to allow natural cooling. Then the other portion of the samples (labeled 10 hours) were left inside the curing chamber up to 10 hours of hot temperature curing and again let them cool down naturally.

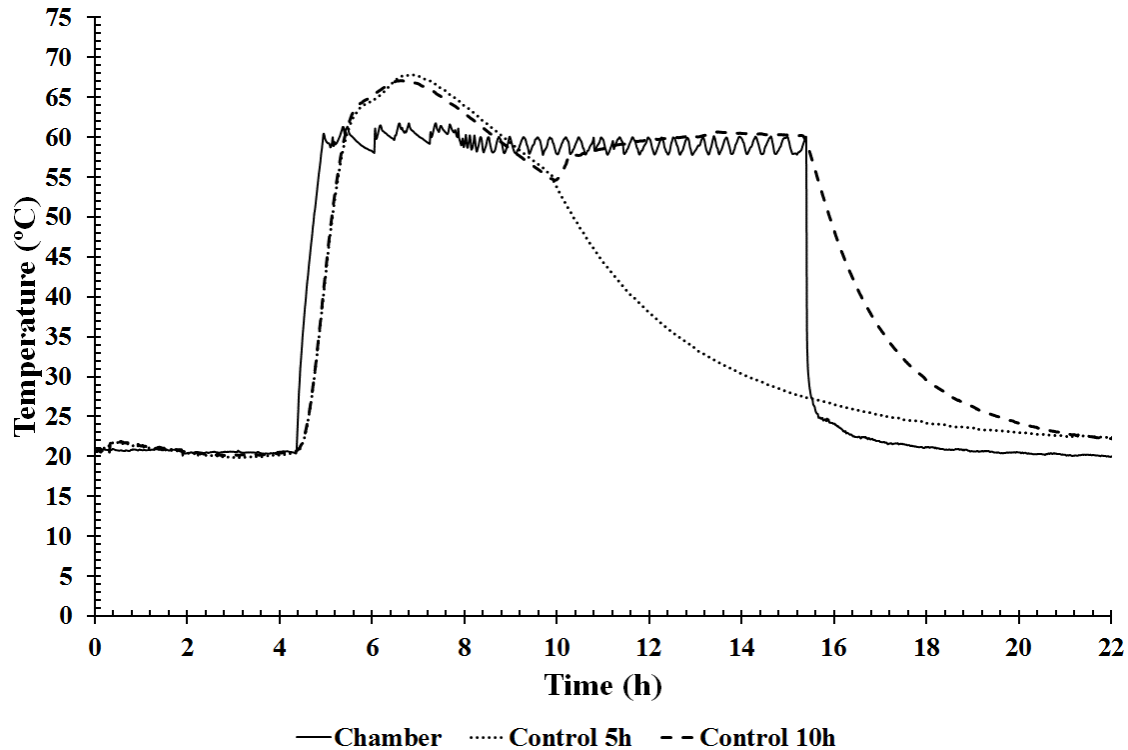


Figure 4.21 Temperature profile for control OPC samples for 5/10 hours 60 °C

Thus, to record the temperature at different points, several thermocouples were used. This temperature variation and changes may be used under the ASTM E1225-09, D5470-12. Moreover, C1045-07 (Sharifi, 2015). The authors also narrated that using such temperature data, and the heat flow can be calculated. ASTM E1225-09 stands for the test method for thermal conductivity of solids using the guarded similar longitudinal heat flow technique. By the principle definition of the rate flow, as the rate of energy passing through a sample due to a temperature gradient. Besides, it should be mentioned that in their experimental work, (Sharifi, 2015), they have adapted quasi-steady-state conditions, which means that cooling and heating rates were somewhat low to reach steady-state conditions. Which is the state proposed for the ASTM equations mentioned earlier? In this experimental work, the method adopted is using the thermocouples inside the samples themselves to capture the temperature change directly. Moreover, the rate of increasing the

temperature and decreasing the temperature was not following the slow rate or the steady-state rate. Since the main purpose of the experiment is to capture the temperature variations. Subsequently, mentioning the energy needed to change the temperature of cementitious samples was reported to be higher for the incorporated samples in the referred of work (Sharifi, 2015), which proves that PCMs increases the thermal mass and decrease temperature deteriorations. One more thing to add, the variations in the slope (how steep or how obvious) is dependable on the sample homogeneity itself. Which comes from the fact that the entire sample will not reach the same temperature at the same time. **Figure 4.22** displays temperature profile as a function of time for OPC samples with and without MPCM 28. As the temperature of the chamber reached the phase change temperature of MPCM 28, the curve for the incorporated mortar had experienced a change in the slope associated with a delay in temperature raising. Afterward, the curve continued in parallel with the control curve. This indicates that incorporated MPCM had reached its capacity of absorbing and storing heat (fully melted). During natural cooling down, as the temperature drops below the phase change temperature, MPCM 28 curve showed a change in the slope, and a higher temperature exerted associated with a delay in cooling down. In other words, the temperature was higher compared to that of the control until the end of the test.

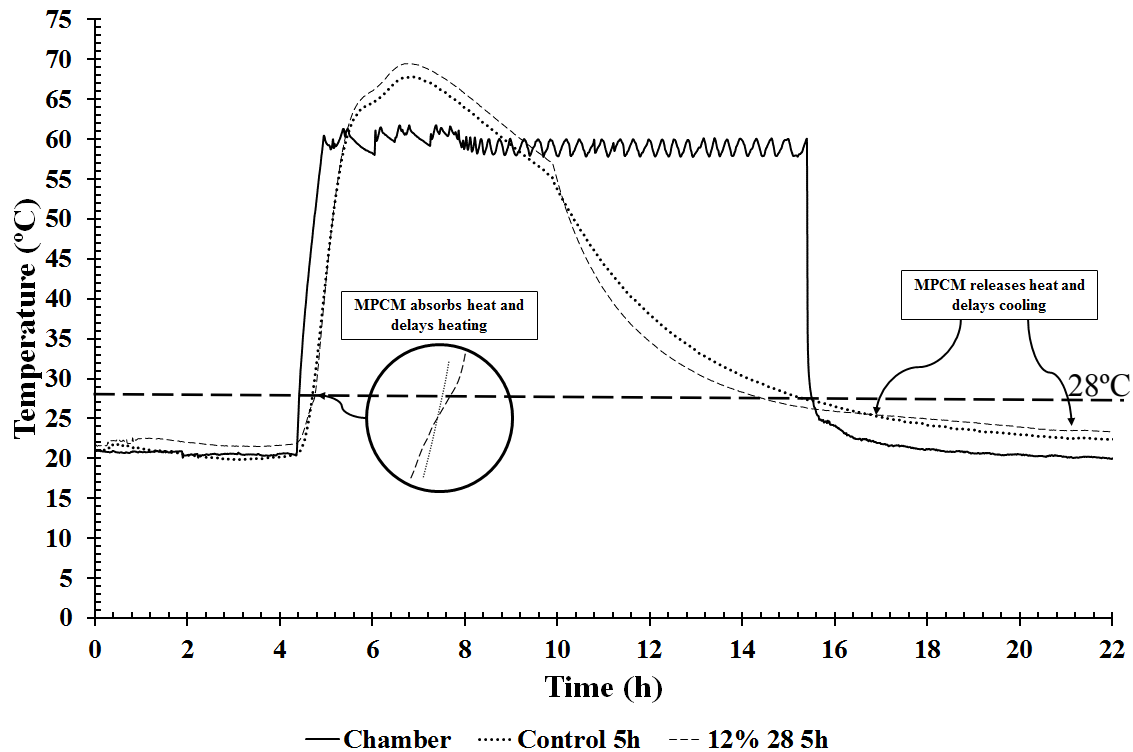


Figure 4.22 Temperature profile for control OPC samples for 5/10 hours 60 °C and 12% MPCM 28 for 5 hours

MPCM 43 showed approximately the same behavior in the charging period. **Figure 4.23** displays temperature profile as a function of time for OPC samples with and without MPCM 43. As the temperature of the chamber reached the phase change temperature of MPCM 43, the curve for the incorporated mortar had experienced a change in the slope associated with a delay in temperature raising. Afterward, the curve continued in parallel with the control curve, indicating the fully mutation for MPCM 43. During natural cooling down, as the temperature drops below the phase change temperature, curve showed a change in the slope, and a higher temperature exerted associated with a delay in cooling down.

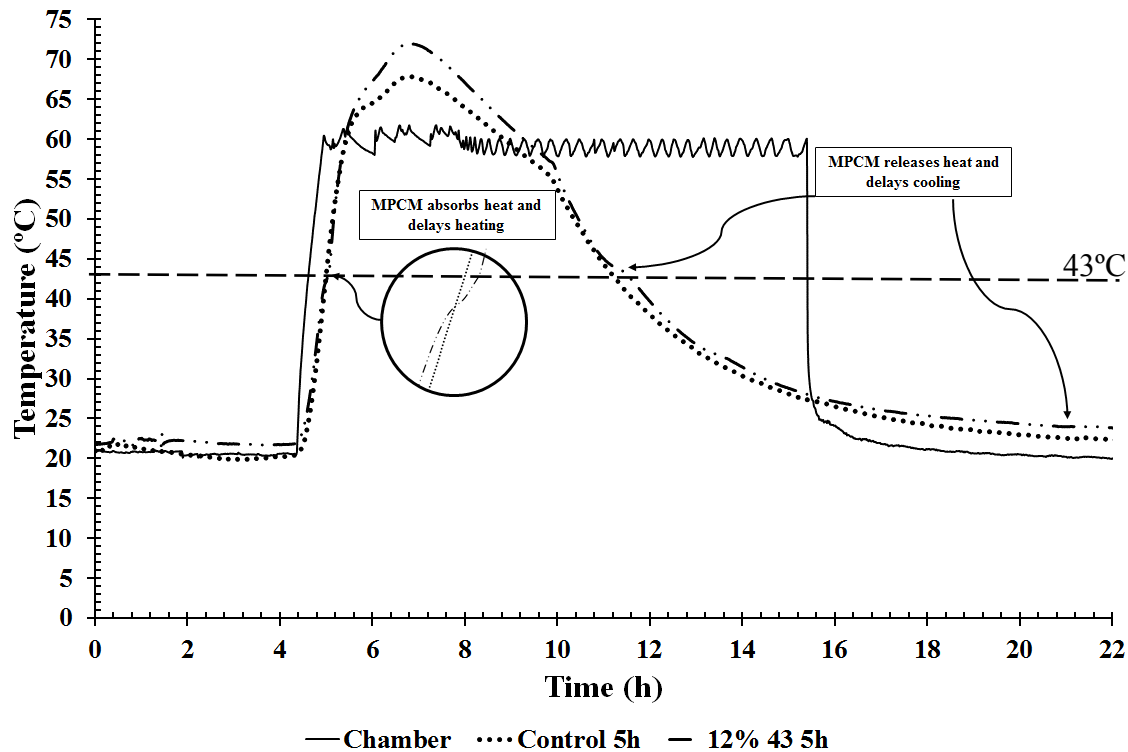


Figure 4.23 Temperature profile for control OPC samples for 5 hours 60 °C and 12% MPCM 43 for 5 hours

Finally, during the discharging period, MPCM 43 acted differently compared to the MPCM 28. **Fig. 4.24** displays temperature profile as a function of time for OPC samples with and without MPCM 43 and MPCM 28 combination. As the temperature of the chamber reached the phase change temperature of MPCM 28, and then that of the 43, heat profile curve experienced a change in the slope associated with a delay in temperature raising at two different places as previously captured for each MPCM individually. Afterward, the curve continued in parallel with the control curve, indicating that incorporated MPCM reached its capacity of absorbing and storing heat (fully melted). Then, during natural cooling down when the temperature drops down to the phase change

temperature. Incorporated sample curve showed a change in the slope and a slower temperature cool down; in other words, a delay.

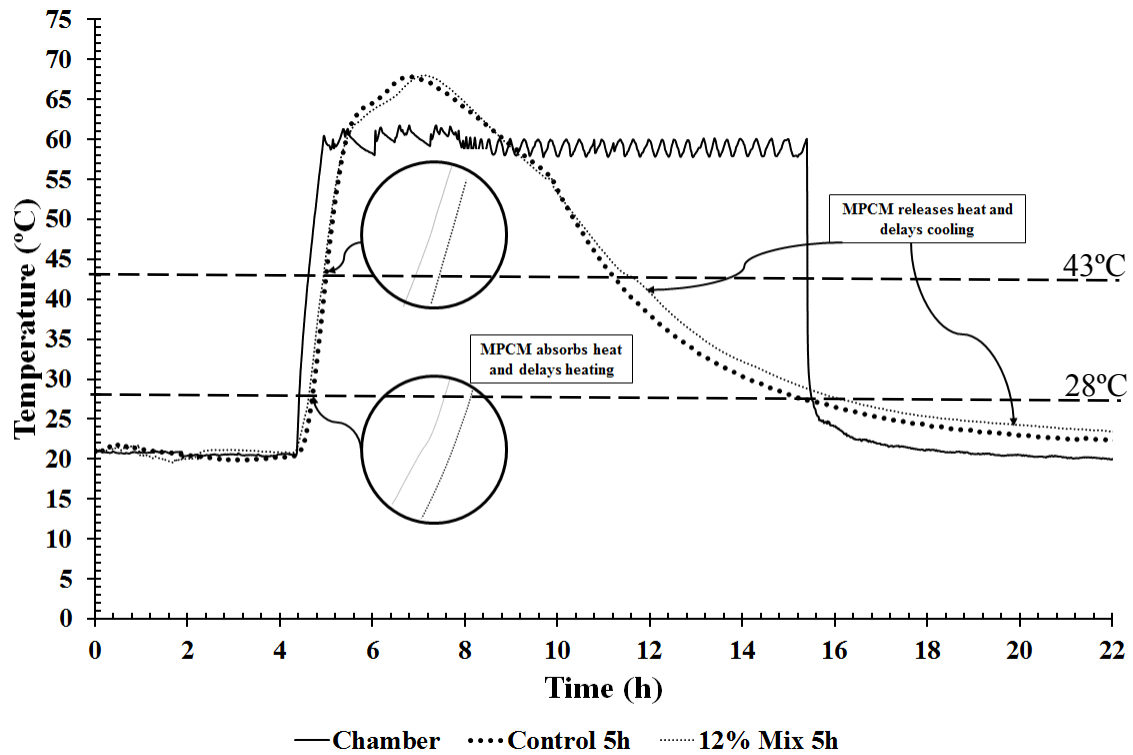


Figure 4.24 Temperature profile for control OPC sample for 5 hours 60 °C and 12% MPCM Mix (MPCM 43 & MPCM 28) for 5 hours

CHAPTER FIVE

PERFORMANCE OF ALKALI-ACTIVATED MORTAR INCORPORATING PHASE CHANGE MATERIALS

5.1 Introduction

A lot of research had been conducted recently to produce an alternative binder for the Ordinary Portland Cement (OPC). This is due to the annual carbon dioxide (CO₂) emission by OPC production (Rehan, and Nehdi, 2005). Alkali-activated material (AAM) is one of the most effective alternatives. It utilizes industrial byproducts and converts them to binding materials like cement. This topic is gaining much attention to research and business worldwide (Thokchom, and Mandal, 2012). All by-products are rich with alumina and silica, such ground granulated blast furnace slag (GGBFS), and fly ash can be used. This replacement method became popular not only for the reduction of the greenhouse gas emissions but also for reusing the byproducts in construction, in other words, recycling wastes (Duxson, 2007; Rangan, 2008). Several attempts have been made to investigate the effect of curing conditions on the properties of alkali-activated materials. A range of curing temperatures were characterized between 40 °C and 85 °C, to complete the geopolymerization reactions. Since the curing at ambient temperature might not serve in the geopolymerization reaction progress. Thus, curing temperature may be applied to develop geopolymerization reactions using a heat source. Which may result in prudent mechanical

properties for AAM. Also, employing elevated temperature contributes to the chemistry of the AA binders products in fresh paste (Doležal, 2006). The research effort by Palomo (1999) narrated that compressive strength of AAM cured for 24 hours at 85 °C was higher than that of AAM cured for 24 hours at 65 °C. In their study they have investigated fly ash with the percentages of 0.25 and 0.30 liquid/solid, with activator concentrations of 8-12 M. Also, they stated that compressive strength was less when curing time exceeded 24 hours.

Moreover, a study for the metakaolin-based AAM under ambient (21-23 °C) and heat (40-65 °C) conditions, with controlled relative humidity for 24 hours by Perera (2007), revealed that curing at 30% relative humidity was better than that of 70% relative humidity. Also, another study by Heah (2011), disclosed that metakaolin-based AAM curing under ambient condition was not feasible compared to that of cured at a higher temperature (40 °C, 60 °C, 80 °C, and 100 °C), where temperature curing effect expedited achieved strength after 1-3 days. Nonetheless, extending curing time and higher temperature-induced failure of samples at a later age, which was reported to be caused by the thermolysis of Si-O-Al-O bond.

On the other hand, Rovnaník (2010) reported that for metakaolin-based AAM cured at temperatures of (40-80 °C) mechanical strength was accelerated, but at the age of 28 days, mechanical strength for AAMs cured at ambient or slightly decreased temperature were better than that cured at high temperature. Furthermore, some other researchers reported that the longer the pre-curing (1 to 7 days) before applying the heat curing (45 °C, 65 °C, and 85 °C for 5-20 h) is more beneficial in gaining more strength (Kani, 2009). Besides, the dissolution of the precursor is very important in producing AAM. Sindhunata (2006) Stated that high-temperature curing influenced increased the total pores volume and surface area, which favor the dissolution of the

precursor in AAMs. Also, at low temperature (below 30 °C), the binder formed contained precipitation of dissolved species instead of poly-condensation of silicate and aluminate.

Different procedures can be employed to apply high-temperature curing. It can be accomplished by dry or steam heat curing techniques. It was reported that steam curing resulted in a higher compressive strength compared to that of the dry heating (Doležal, 2006). A study for both techniques by Kovalchuk (2007) were they compared the 28 days age compressive strength for AAMs cured at 150 °C dry and steam cured at 95°C. They concluded that steam cured specimens achieved better compressive strength compared to that dry curing, with 71 MPa and 31.8 MPa, respectively. Generally, decent curing is required for AAM in order to achieve sound mechanical and durable product (Van Jaarsveld, 2002).

This chapter focus on illustrating effects of adding MPCMs as a volume replacement for sand on the performance of AAM mortar cured at ambient temperature ($T = 23^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and relative humidity (RH) = $50\% \pm 5\%$). High temperatures ($T = 60^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and relative humidity (RH) = $80\% \pm 5\%$).

5.2 Experimental Work

5.2.1 Materials

The same mixture composite and ingredients, as previous chapters, were used except cement. In this chapter, cement was replaced by alkali-activated binder (AAB). The AAB composed of Ground Blast Furnace Slag (GBFS) is an industrial by-product commonly used in AAM mixture design. In conventional concrete it's used mostly to bring down the heat of hydration, resist abrasion and wearing from ground water, and fight other environmental conditions (Kumar, 2010). This by-product is resulting from steel manufacturing. It is the lime of the flux and the aluminates and silicate of ore and coke ash which have been chemically combined to form blast furnace slag.

The used GBFS as alumina silicate source materials, and activated by alkali solution. The alkali solution is a mix of sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₂). The GBFS is off-white in colour. It comprised of calcium silicate and alumina (about 90%) meets the requirement of pozzolanic material. **Table 5.1** shows its chemical composition. The specific gravity is 2.89 g/cm³ and median particle size is 12.4 μm with specific surface area of 4950 cm²/g.

Table 5.1 Chemical composition and physical properties of GBFS

		GBFS
	SiO ₂	32.97
	Al ₂ O ₃	17.97
	CaO	35.08
	Fe ₂ O ₃	0.72
	SO ₃	0.72
	Na ₂ O	--
	MgO	10.31
Density	g/cm ³	2.88
Major crystalline phases		Gehlinitite
Glass content	(%)	93
Loss on ignition		0.58
Specific surface area	m ² /gm	3.15

5.2.2 Testing and Specimen Preparation

MPCM 28 was added as a replacement of sand at a rates 3, 6, and 12% by volume. The water-solid ration for all mixtures was maintained at 0.4. The preparation of the samples included the following steps: (a) preparation of alkaline activator solution (water and NaOH) and let it cool down to room temperature, (b) two minutes mixing the sand with GBFS (dry mixing), (c) adding alkaline activator solution and continue mixing for two more minutes, (d) adding the sodium silicate to the mixer and continue mixing for one minute, (e) adding MPCM to the mixer and continue mixing for one minute. Compressive strength, heat of hydration, water absorption,

thermal transfer, and RCPT, were conducted following the same procedure as the previous chapters. The same curing regime of the previous chapters applied either for ambient or hot temperature.

5.3 Results and Discussions

5.3.1 Followability

Reduction percentages in the average flow table readings for AAM mixtures with MPCM 28 incorporation are shown in **Fig. 5.1**. For instance, mixtures incorporating 3% MPCM induced a reduction of about 4% in the average spread diameter compared to that of the control. Moreover, 6% and 12% MPCM exhibited a reduction of 9% and 14% in the average followability index, respectively, compared to that of the control. This indicated that AAM followed the same trend for OPC, as sand is replaced by MPCMs, resulting in a lower flowability for AAM.

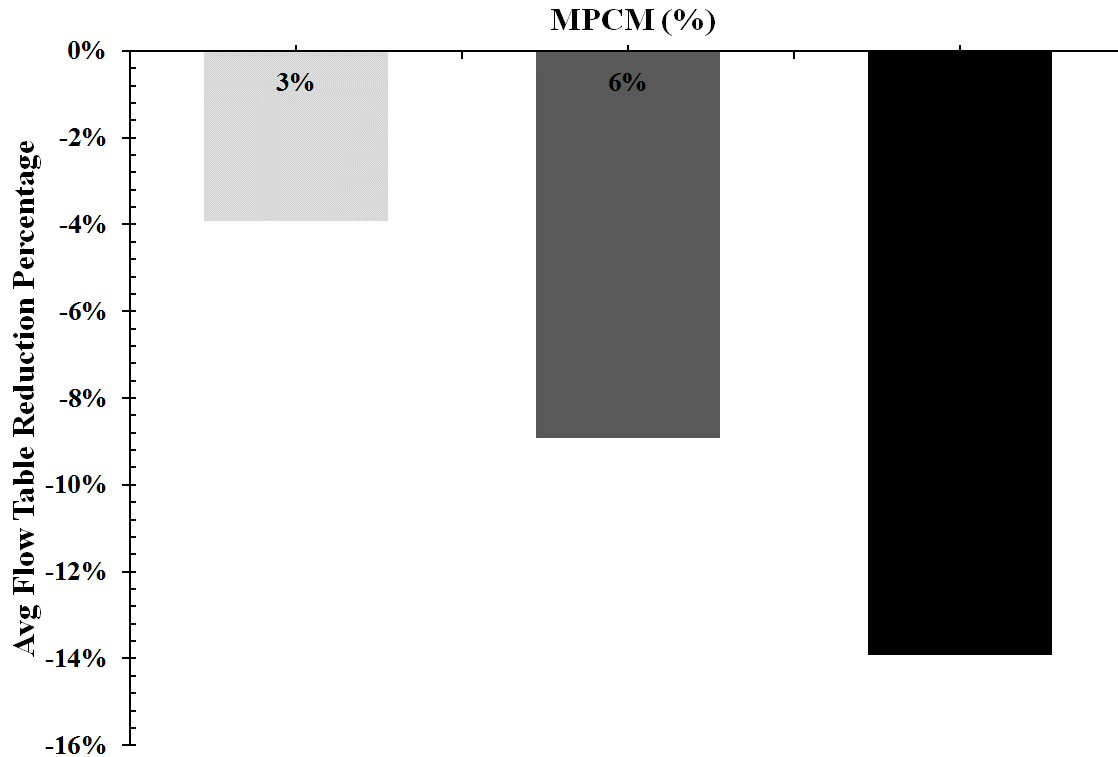


Figure 5.1 Reduction in average flow table readings for incorporated AAM mortars vs control AAM mortar

The reduction in the flow table readings (reduced workability), is confirming with the previous findings of Pilehvar (2018). It can be ascribed to the higher amount of water absorbed as the authors reported. On the other hand, the research effort by Pilehvar (2017) explained the reduction happens by the viscosity of sodium silicate in alkaline solutions, which is the reason behind making AAM mixtures more cohesive. Also, the difference in the particle size has the effect of reducing workability of the mixtures (Park, 2009).

5.3.2 Compressive Strength

Figure 5.2 shows the compressive strength development for AAM incorporating MPCM 28 mortars under the ambient curing condition. Generally, compressive strength increased for all

mixtures over the investigated period. Moreover, the MPCM content increased, the compressive strength decreased. For instance, at age 28 days, mixture incorporating 3% exhibited compressive strength about 5% lower than that of the control. Moreover, at the same age, increasing the percentage of MPCM to 6% resulted in about 10% reduction compared to that of the control.

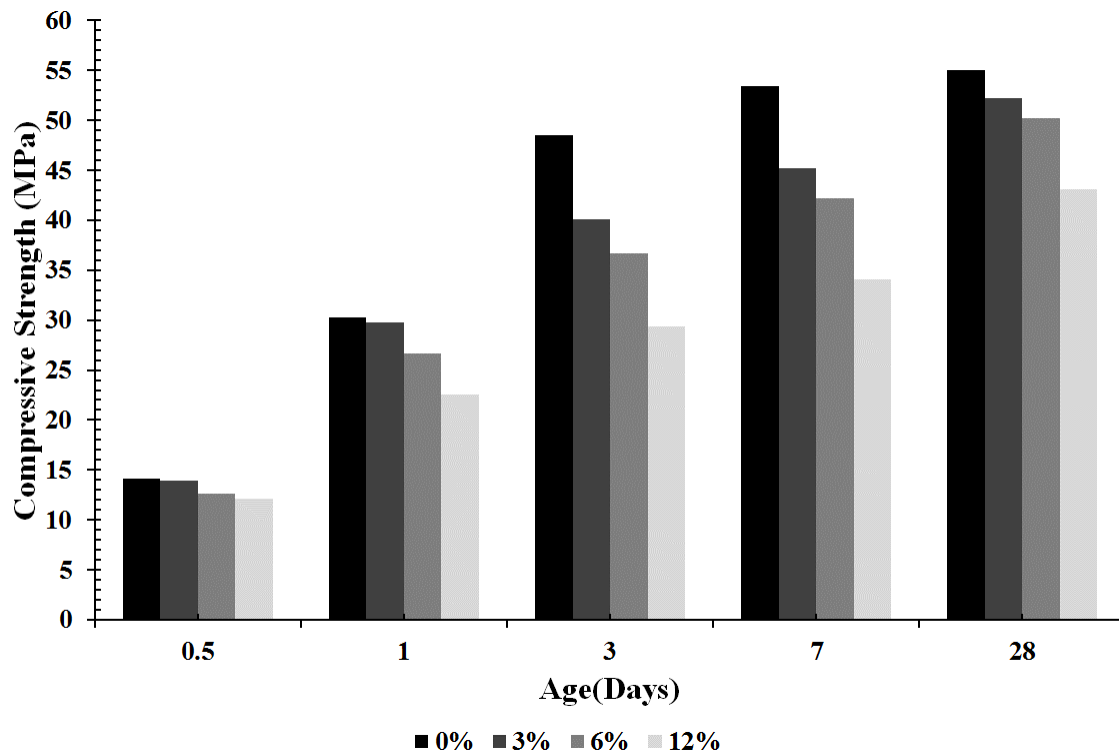


Figure 5.2 Compressive strength development for AAM mortar incorporated MPCM 28 under ambient temperature

The reduction of the compressive strength can be simply associated with the softness nature of MPCMs particles (Shadnia, 2016). This is confirming with previous findings of Meshgin (2012); Yang (2016). Shadnia (2015) SEM pictures showed that increasing MPCMs incorporating in AAM increased the number of broken MPCM particles. Authors referred that to the small stiffness of MPCMs particles against loading, which results in the strength reduction. On the other hand, their

SEM images showed a good bonding between AAM binder and MPCM particles. In our study the SEM images in **Fig. 5.3** shows the connection built between MPCMs and AAM.

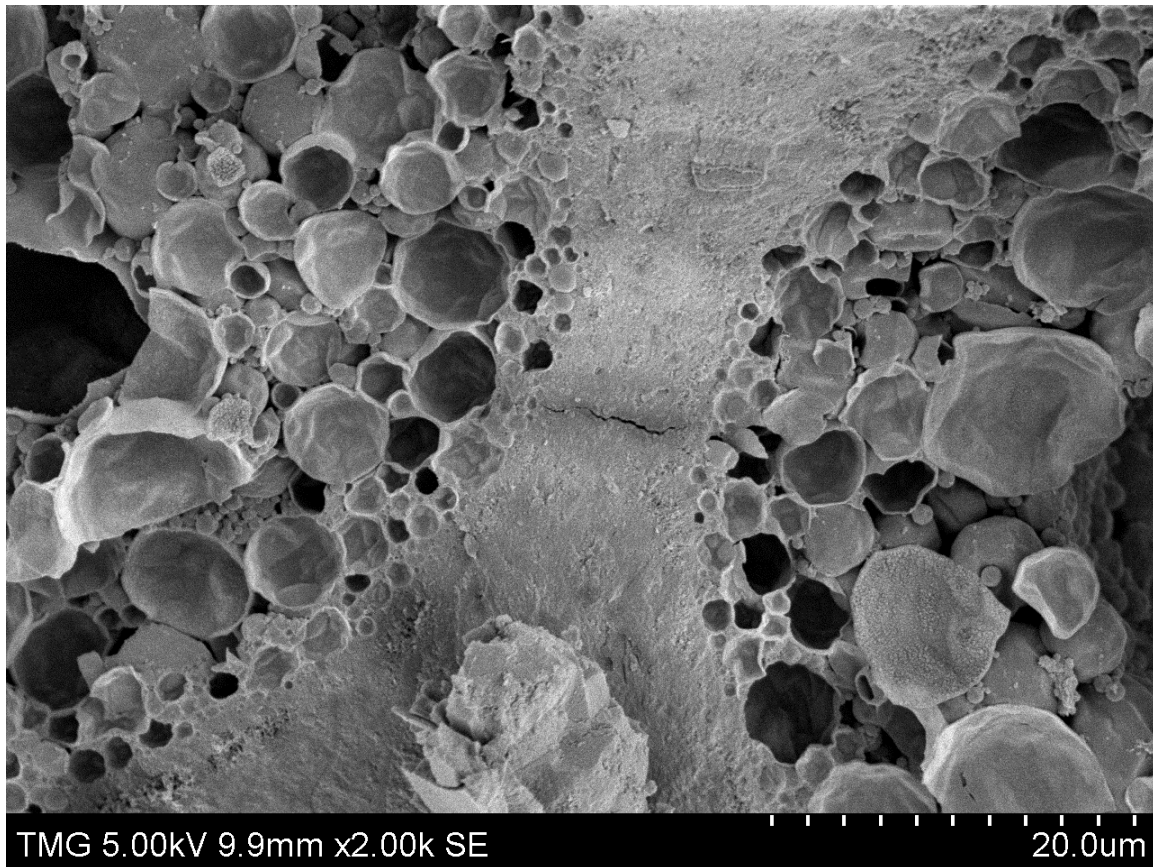


Figure 5.3 AAM bonding with MPCMs

AAM with and without the MPCM was also subjected to hot temperature curing regimes. (i.e., the same two regimes used in the previous chapter). **Figure 5.4** displays the compressive strength developed by control AAM mortar under different curing regimes. Generally, the compressive strength increased for all the mixtures over the investigated period. Also, as the hot curing extended, specimens gained higher compressive strength. For instance, at the age of 28 days, AAM mortar undergoes 5 hours and 10 hours hot curing achieved 28% and 41% increase in strength compared to that of the control. Which confirms with the findings of Heah (2011) and Rovnaník

(2010). As well as the findings by Shadnia (2015), where they reported that compressive strength for AAM is mainly gained in the first seven days, and hot temperature curing accelerated the strength gain. Wallah and Rangan (2008) cured AAM at 60 °C, and stated that the more time under hot temperature is curing the more strength gain. But, they found that compressive strength gain for AAM was rapid until 24 hours, then it was moderate. They recommended using the aid of hot temperature curing for the first 24 hours, which is more convenient and practical.

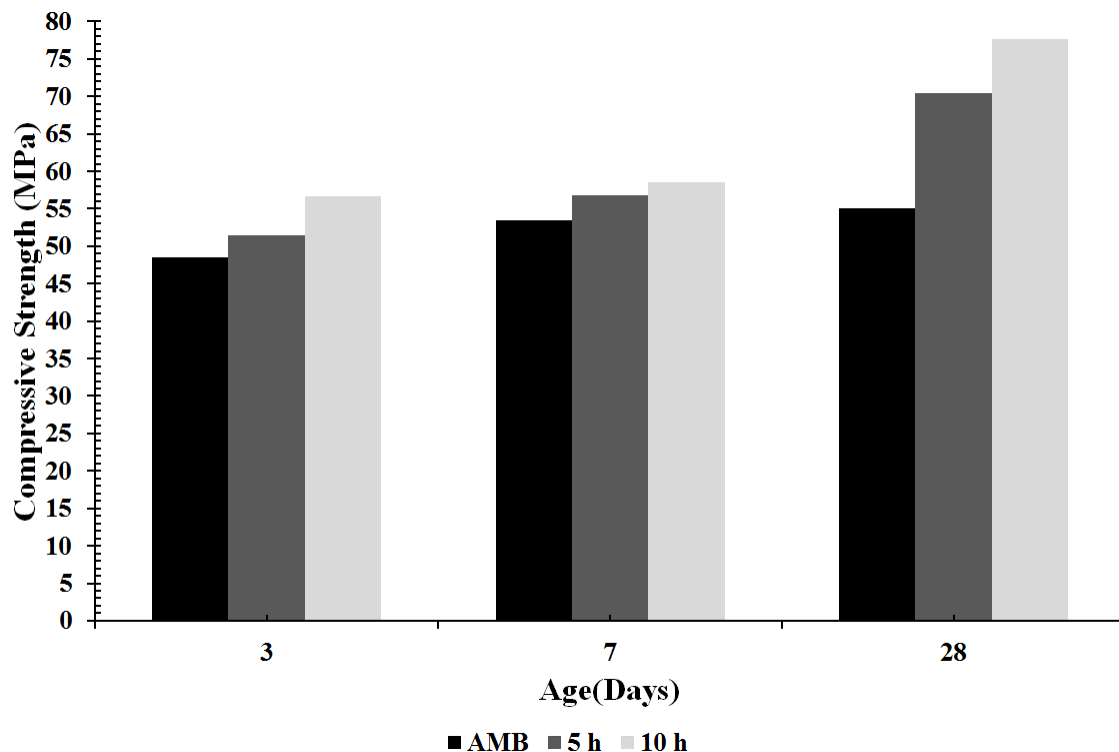


Figure 5.4 Compressive strength development for AAM mortar under different curing conditions

Figure 5.5 displays the compressive strength development for AAM mortar with MPCM 28 incorporation under 5 hour hot temperature treatment. Generally, compressive strength increased with time for all specimens. Increasing MPCM resulted in a lower compressive strength. For instance, 3% MPCM mixture at the age of 28 days, had achieved about 7% lower compressive

strength less than that of the control. Moreover, increasing MPCM percentage to 6% and 12% induced higher strength reduction (i.e. 17% and 28%, respectively)

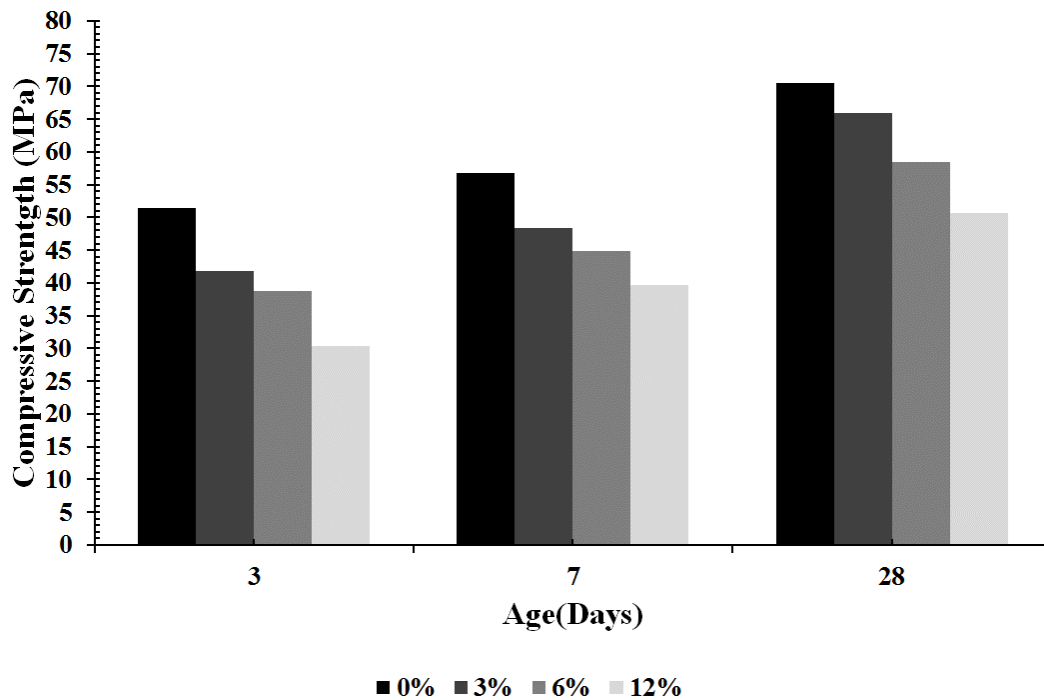


Figure 5.5 Compressive strength development for AAM mortar incorporated MPCM 28 under 5 hours hot curing

Figure 5.6 displays the compressive strength development for mortar with and without the incorporation of MPCM 28 under 10 hours hot curing. The same trend observed in the 5 hours hot curing was followed for the 10 hours hot curing. Increasing the MPCM percentage resulted in compressive strength drop. For instance, at the age of 28 days, compressive strength for 3% MPCM was about 13% less compared to that of the control. Moreover, increasing the MPCM incorporation percentage to 6% and 12% resulted in higher strength reduction (i.e. 23% and 34% respectively).

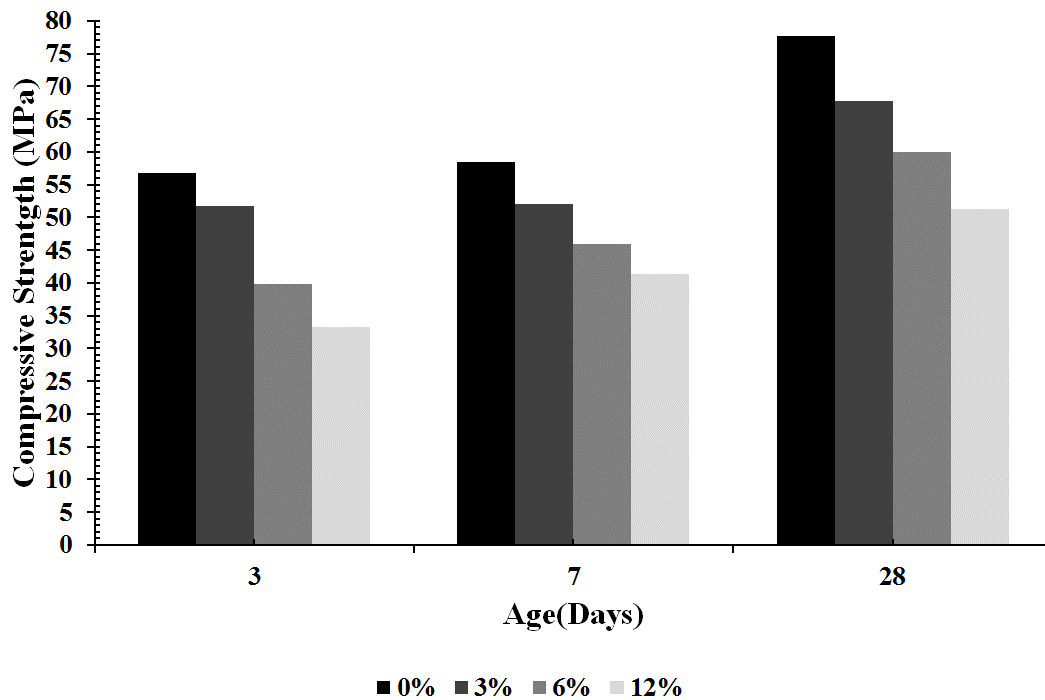


Figure 5.6 Compressive strength development for Geopolymer mortar incorporated MPCM 28 under 10 hours Hot Temperature

The more curing temperature and time increased, the more compressive strength gained. This trend is confirming with the previous findings of Rangan (2008); Zhang (2011); Ryu (2013). Moreover, the temperature effect was reported to accelerate the geopolymerization resulting in the fast gaining of the compressive strength (Khale, 2007; Bingöl, 2013). On the other hand, the curing temperature was reported mainly to have an optimum temperature (beyond this temperature the mechanical properties are adversely affected) dependent on the NaOH concentration, where geopolymerization at low alkalinity is less sensitive to curing temperature. Many researchers studied the effect of curing temperature on AAMs properties (Ahmari, 2012; Ren, 2015; Zhang, 2016). The reduction in the compressive strength resulted from the addition of MPCMs is referred to as several reasons. The low stiffness of the MPCM particles compared to that of the sand, lead to deform and break

the MPCM capsules under compression (Meshgin, 2012; Shadina, 2015). Also, MPCMs might reduce the strength because of the air bubbles and air voids it introduces to the medium (Lecompte, 2015), as well as the poor contact between MPCMs and the matrix (Cui, 2015).

5.3.3 Heat of Hydration

The mechanism during geopolymerization is not fully understood; several proposed models are there in order to describe the alkali activation of alumina-silicate materials. A general mechanism of geopolymerization is divided into three main steps, which are destruction-coagulation, coagulation-condensation, and condensation-crystallization (Duxson, 2007). In sodium hydroxide activated slag, like the one used in this research. The hydration is slow and controlled, with the gradual formation of hydration products during the phases. The hydration reaction is reported to keep progressing along 60 hours, and it was also reported to have induction periods to 24 hours (Gebregziabiher, 2016). Authors reported that at elevated temperature, the bulk reaction of the hydration is mostly complete at 4 hours, compared to 12 hours at ambient. It can be observed from **Figure 5.7**, that the incorporation of MPCMs resulted in a drop in the heat flow for all the incorporated specimens. Moreover, the more MPCM is incorporated, the more severe the drop is. In other words, the amount of the heat stored by MPCM during the heating cycle is increased, as well as the amount if the hear liberated in the cooling period. This is a clear indication that the heat capacity of AAM is increased by MPCM incorporation. It can be seen that the amount of heat stored is increased. As well as the same trend for OPC incorporated specimens. A delay and shift of the peak are noticeable.

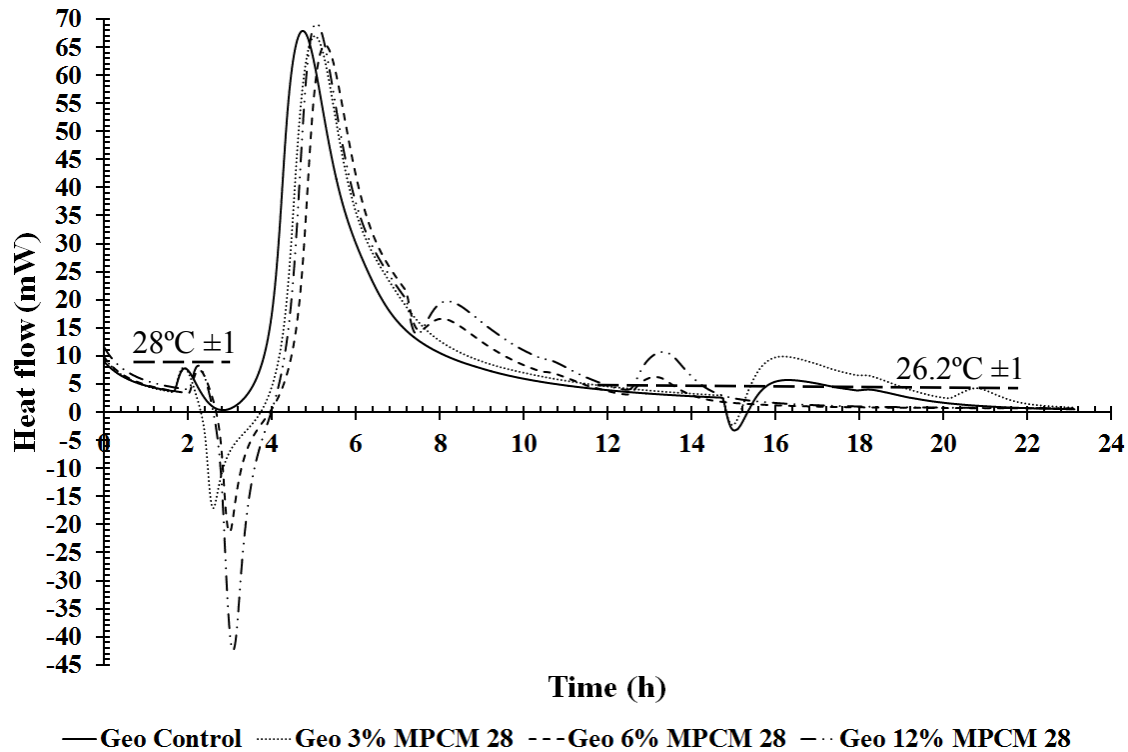


Figure 5.7 Heat flow for control AAM with and without MPCM 28 incorporation under hot temperature

5.3.4 Heat Profile

Figure 5.8 displays the full scale heat profile for control sample subjected to a heating up to 60 °C, where temperature plotted as a function of time. Generally, the heat was increased after approximately 4.5 hours from mixing. The temperature for all samples was gradually increased as captured with the thermocouples inserted inside the chamber and planted in the middle of the cylindrical specimen (5cm X 10cm). 1 hour, the temperature reading for AAM reduced about 26 °C, even before increasing the chamber temperature. After 5 hours, samples were moved out of the chamber and placed under laboratory conditions to cool down.

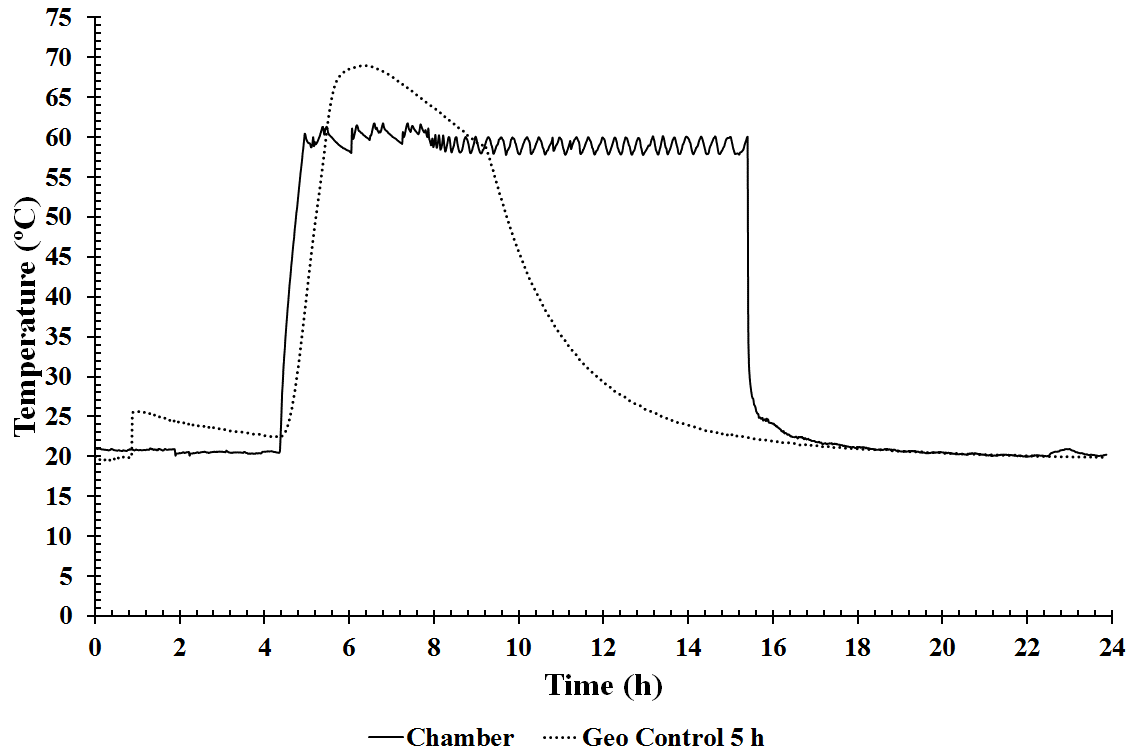


Figure 5.8 Temperature profile for control geopolymer samples for 5 hours 60 °C

This temperature variation and changes may be used under the ASTM E1225-09, D5470-12. And C1045-07 (Sharifi, 2015). The authors also narrated that using such temperature data, the heat flow can be calculated. ASTM E1225-09, stands for the test method for thermal conductivity of solids by means of the guarded comparative longitudinal heat flow technique. By the principle definition of the rate flow, as the rate of energy passing through a sample due to a temperature gradient. In addition, it should be mentioned that in their experimental work, (Sharifi, 2015), they have adapted a quasi-steady state conditions, which means that cooling and heating rates were somewhat low to reach steady state conditions. Which is the state proposed for the ASTM equations mentioned earlier. In this experimental work the method adapted is using the thermocouples inside the samples themselves to capture the temperature change directly.

Moreover, the rate of increasing the temperature and decreasing the temperature were not following the slow rate or the steady state rate. Since the main purpose of the experiment is to capture the temperature variations. Subsequently, mentioning the energy needed to change the temperature of cementitious samples was reported to be higher for the incorporated samples in the referred of work (Sharifi, 2015), which proof that PCMs increases the thermal mass and decrease temperature deteriorations. One more thing to add, the variations in the slope (how steep or how obvious) is dependable on the sample homogeneity itself. This is attributed to the fact that entire sample will not reach the same temperature at the same time.

To record the temperature at different points, several thermocouples were used. **Figure 5.9** displays the temperature profile as a function of time for AAM mortar samples incorporating 0%, 3%, 6%, and 12% of MPCM 28. The temperature profile for several mixtures is shown in the graph separately, especially at the beginning of the test (before increasing the chamber temperature). This gives a clear indication about the temperature increase that happens inside AAM mortar compared to the control. Increasing the MPCM incorporation resulted in more heat at the beginning of the geopolymerization process. As the temperature of the chamber reached the phase change temperature of MPCM 28, the curve for the incorporated mortar had shown a change in the slope associated with a delay in temperature raising. Afterwards, the curve continued in parallel with the control curve. This indicates that the incorporated MPCM, had reached its storing heat capacity (fully melted). During natural cooling down, as the temperature drops below the phase change temperature, MPCM 28 sample curve showed a change in the slope and a higher temperature exerted associated with a delay in cooling down. Also, as the MPCM percentage increased the more exerted heat showed till the end of the test. In other words, the temperature was higher compared to that of the control until the end of the test.

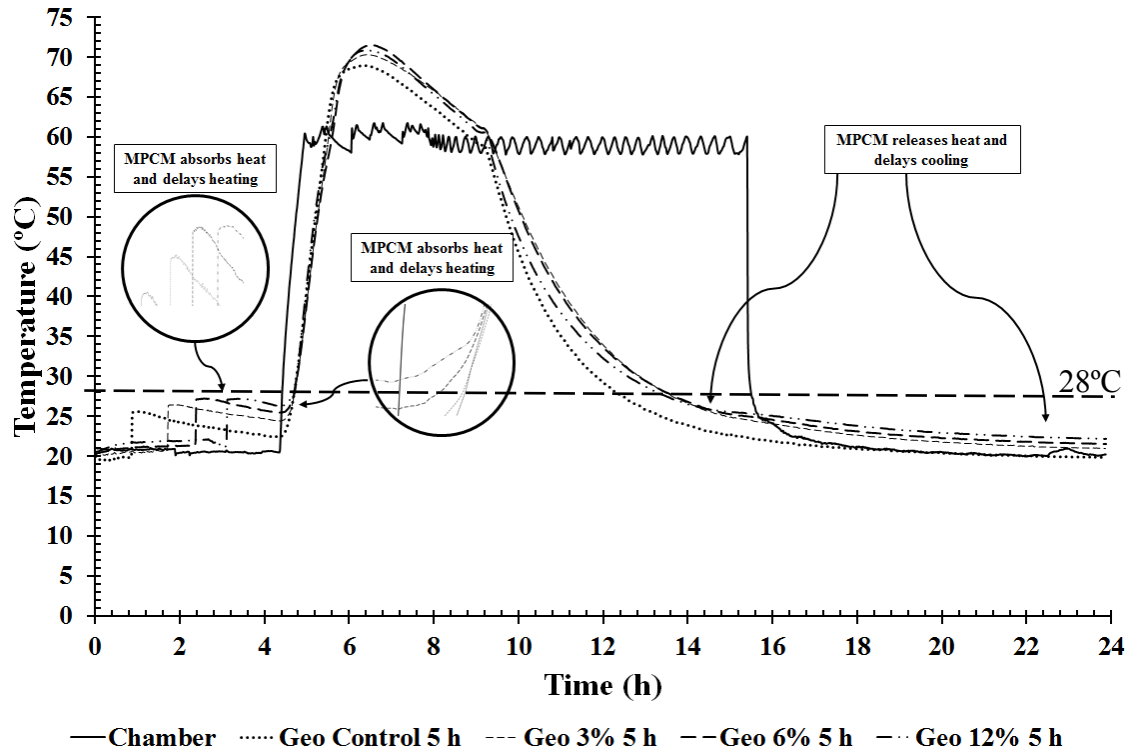


Figure 5.9 Temperature profile for control OPC samples for 5/10 hours 60 °C and 12% MPCM 28 for 5 hours

Finally, MPCM 28 act somewhat in a different way in AAM samples compared to the OPC. And the reason behind this phenomenon can be ascribed to the temperature increased at the beginning of the geopolymerization process compared to the hydration process. Where in the AAM more heat is exerted by the geopolymerization reactions at the beginning of the process which may affect the phase change status of MPCMs impeded.

5.3.5 Thermal Transfer

Thermal conductivity is one of the major interest for research of MPCMs incorporated construction materials. Several research stated that thermal conductivity had increased by PCMs addition as well as the heat storing capacity (Frigione, 2019). On the other hand, the thermal conductivity for

materials with MPCMs incorporation was reported to be low (Khodadadi, 2013). Knowing that PCMs increases the heat transfer due to its large surface areas per unit volume (Riffat, 2015). The effect of microencapsulation shells is considerable, and its effect on reducing the heat transmission of the raw phase change materials (Whiffen, 2012). On the other hand, MPCMs shells itself affects the heat transfer for cementitious mediums differently from the core phase change materials.

Figures 5.10-5.11 show the rate of the heat change through AAM disc specimens with and without MPCM 28 incorporation, versus time using two channels located as follows (Ch1 1.5 cm from the face, Ch3 4.5 cm from the face). Exposed to dry heat gun with at a distance of 5 cm, with temperature of 100 °C from one of its faces. Generally, temperature increased with time for all specimens. It can be observed from **Figs (5.10-5.11)** that channel #1 reached 70 °C in 17 min, 20 min, 29 min, and 30 min for mixtures incorporating 0%, 3%, 6%, and 12% MPCMs. This indicates that the addition of MPCMs had increased the thermal capacity. Hence, some of the heat was consumed to melt the PCMs.

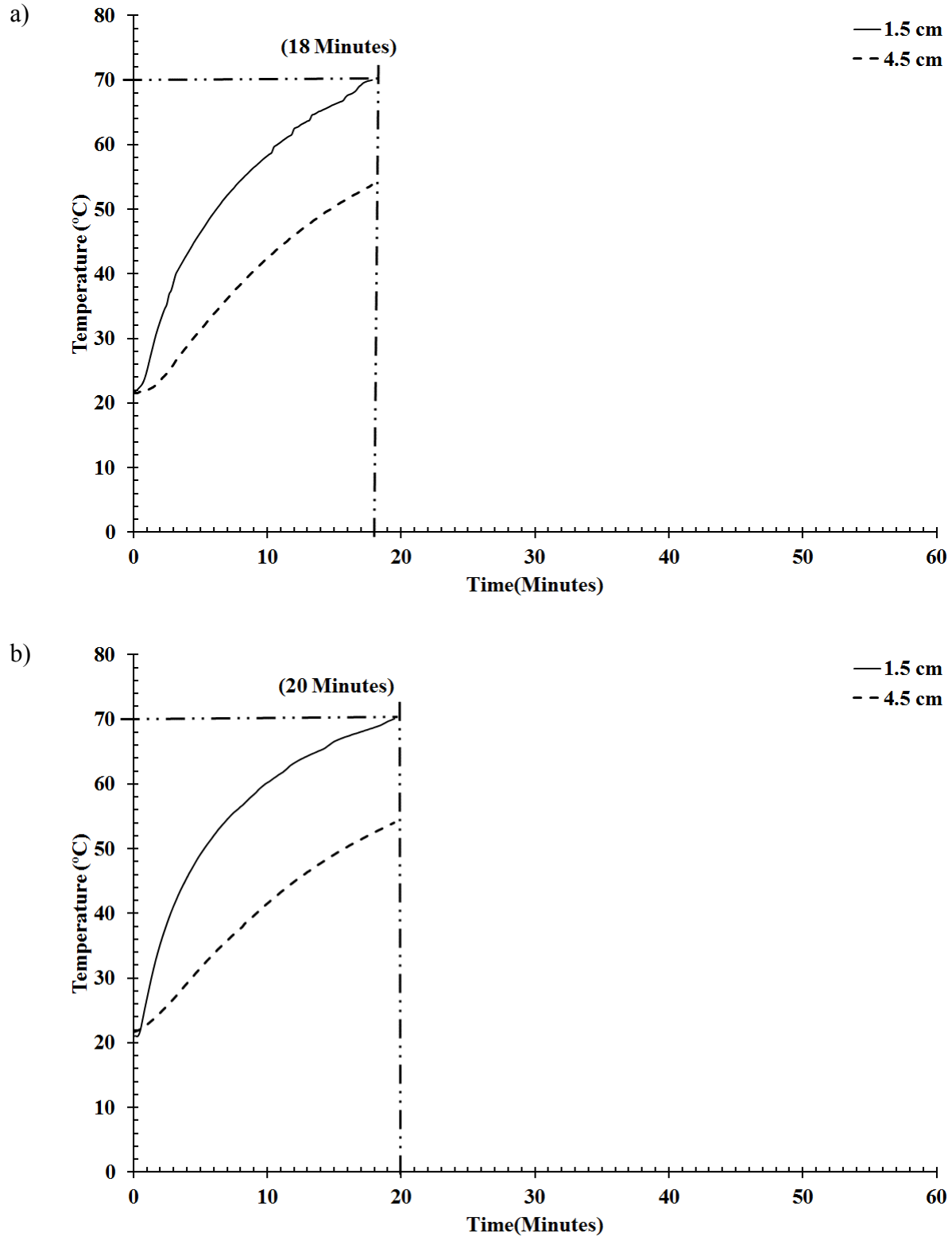


Figure 5.10 Thermal behavior a) control AAM specimen and b) 3% MPCM 28 AAM specimen

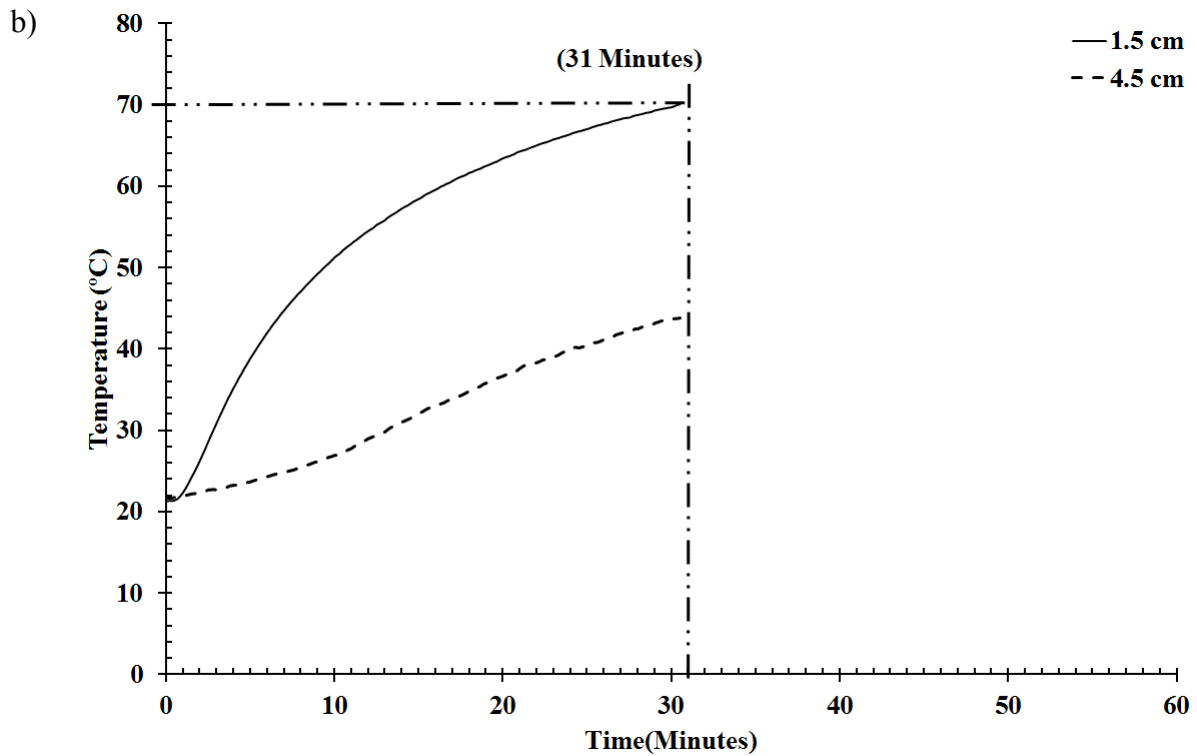
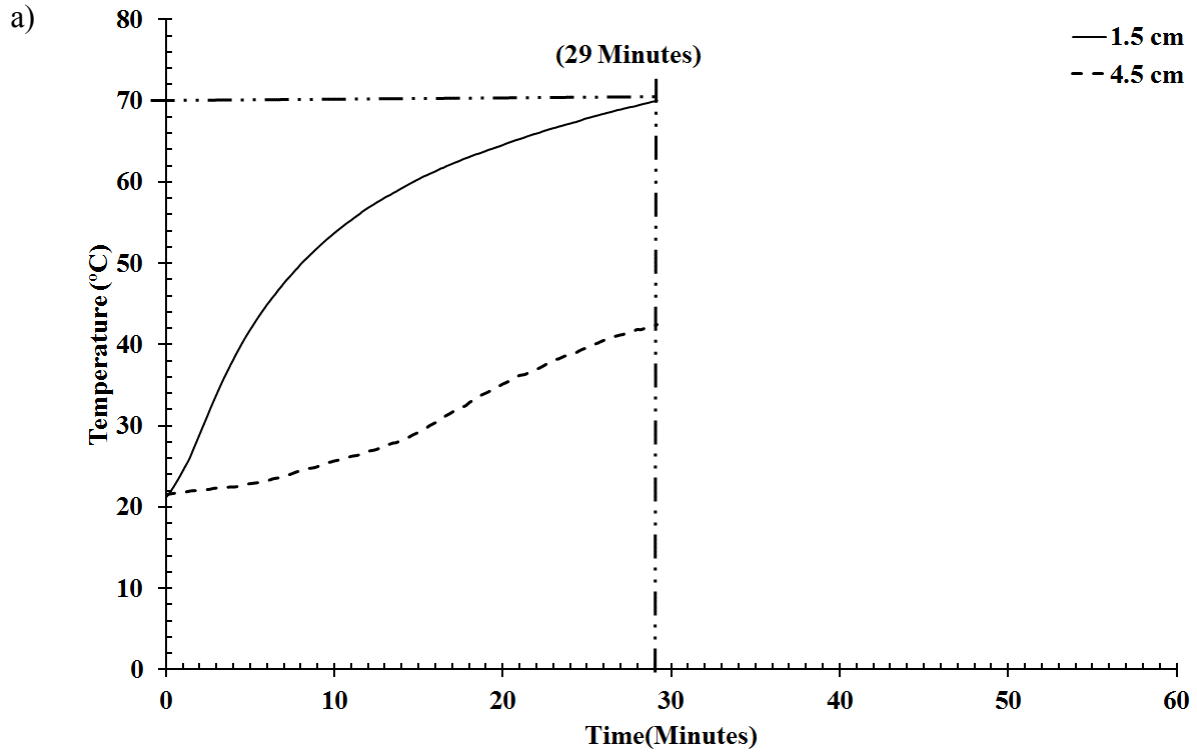


Figure 5.11 Thermal behavior a) 6% MPCM 28 AAM specimen and b) 12% MPCM 28 AAM specimen

Table 5.2 summarizes the time required for all mixtures to reach 70 °C. At channel #1, along with the difference between channel (Ch1) and channel (Ch3) temperatures. At time that channel #1 reached 70 °C, mixture with 3% MPCM reached 70 °C with about three more minutes. This is caused by the MPCM heat storage capacity and resulted in overall delay for the heat transfer (Bahrar, 2018). The temperature variation is approximately the same in the 3% compared to that of the control. Moreover, increasing MPCM dosage up to 6% resulted in slower heat transfer with ten minutes more, along with increasing the temperature variance between both channels of 26 °C. This could be ascribed to the decreased thermal conductivity with increasing MPCM dosages (Jayalath, 2016). Furthermore, mixture with 12% reached 70 °C in 31 minutes with about two minutes more compared to the 6% MPCM mixture, and with a temperature variance of 29 °C. Moreover, MPCMs addition is reported to increase the amount of entrapped air. Which made this materials with a good insulation enhancements (Hunger, 2009). Hence, created entrapped air is responsible of causing a delay in the natural convection of heat. On the other hand, replacing sand with MPCMs, knowing that sand thermal conductivity is higher compared to MPCMs, is expected to result in a less thermal conductivity, thus lower heat transfer rate (Jayalath, 2016). Also, broken microcapsuls will increase voids. This findings confirms with previous findings by (Shadin, 2015). Where adding MPCMs was reported to increase the heat capacity of AAM, and reduce the heat transfer rate inside of AAM mediums. As well as reducing the thermal conductivity while increasing the latent heat capacity (Cao, 2017).

Table 5.2 Temperature variation between Ch1 and Ch3 at 70 °C

<i>Specimen</i>	ΔT (°C)	Δt (Minutes)
<i>Control AAM</i>	16	18
<i>3% MPCM 28 AAM</i>	17	20
<i>6% MPCM 28 AAM</i>	26	29
<i>12% MPCM 28 AAM</i>	29	31

5.3.6 Sorptivity and Absorption

Figures 5.12-5.14 display the initial water absorption for control AAM mortar specimen compared to 3%, 6%, and 12% AAM incorporating MPCM mixtures. Each point in these graphs is an average of two readings. A linear relationship between cumulative absorption and the square root of time is obtained with a correlation coefficient of around 0.95 for all samples. The sorptivity was calculated as the slope of this line. It was observed that initial sorptivity is decreasing by the increasing of the MPCM dosage. This could be referred to the densification action happens by adding MPCMs, and the ability of MPCM particles to obstruct and interrupt the capillary network (Kheradmand, 2018). This results in a reduction of the total permeable pores of the AAM matrix. Therefore, the more you increase the inclusion of MPCMs, the lower the slope of the line and the rate of capillary suction in diminishing

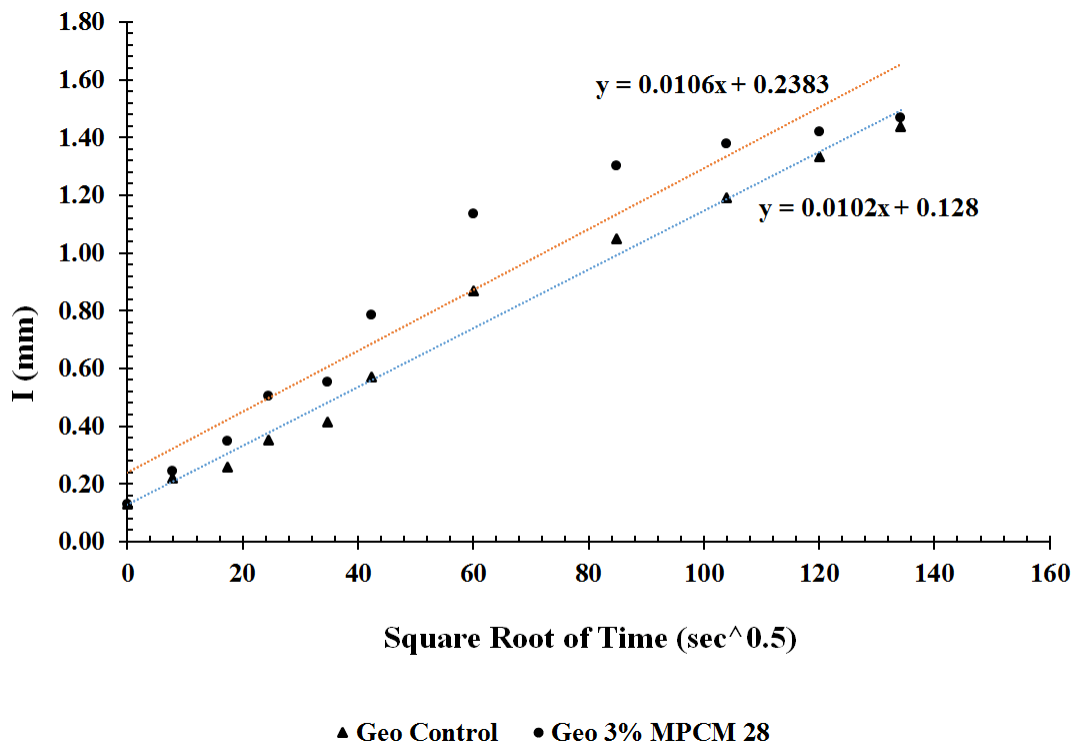
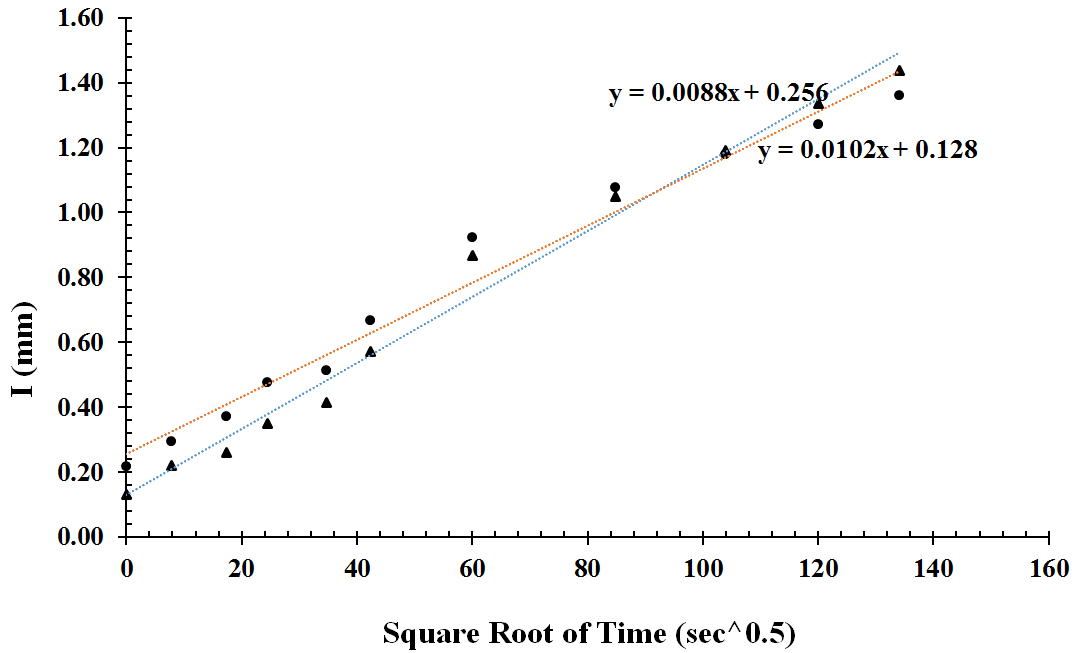
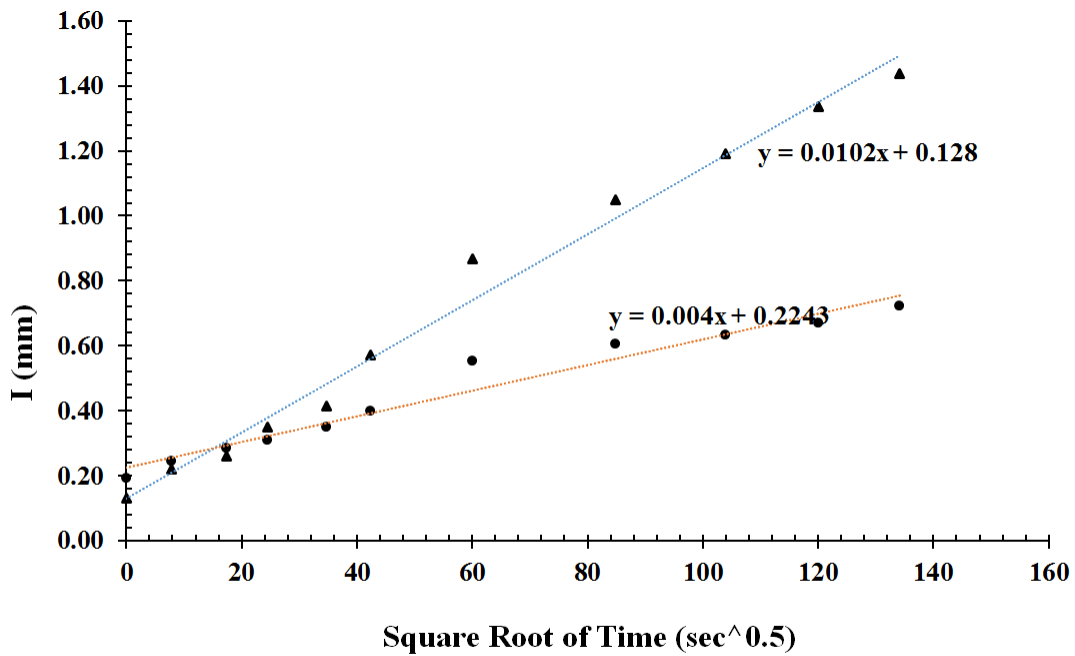


Figure 5.12 Initial Sorptivity Geo/Geo 3% MPCM.



▲ Geo Control • Geo 6% MPCM 28

Figure 5.13 Initial Sorptivity Geo/Geo 6% MPCM.



▲ Geo Control • Geo 12% MPCM

Figure 5.14 Initial Sorptivity Geo/Geo 12% MPCM.

Figures 5.15-5.17 show the secondary sorptivity results for AAM specimens with and without the MPCM incorporation. Generally, all the specimens have reached a level of saturation at a certain point. This is called the saturation point for the specimen. At this point the weight of the specimen is constant and will not increase. For instance, the slope of the line for 6% MPCM incorporation is close to zero. Thus, a different phenomenon is observed for the secondary sorptivity compared to the initial sorptivity. The rate of change for the water sorption decreased faster for the initial sorptivity compared to the secondary, while the rate of change in the secondary sorptivity is relatively slow and mutual. This can be attributed to several factors, like the nature of the MPCMs as a non-sorptive inclusion (Hall, 1993). Therefore, the interface of the MPCMs incorporated redirected the flow around its parameter, and played an intensifying role in the sinuosity of the transport path for the capillary (Wei, 2017).

Generally, MPCMs act as nucleation sites and accelerate the geopolymerization leading to a denser microstructure. Besides acting as filler materials (especially for small particles in the range from $< 3\mu\text{m}$ and up to $10\ \mu\text{m}$). At the same time, broken MPCMs induce higher voids. The role of this voids will depend on two factors: location and connectivity. Isolated voids away from the surface will mainly have effect the mechanical performance. However, surface connected voids will affect the capillary void structure and water absorption.

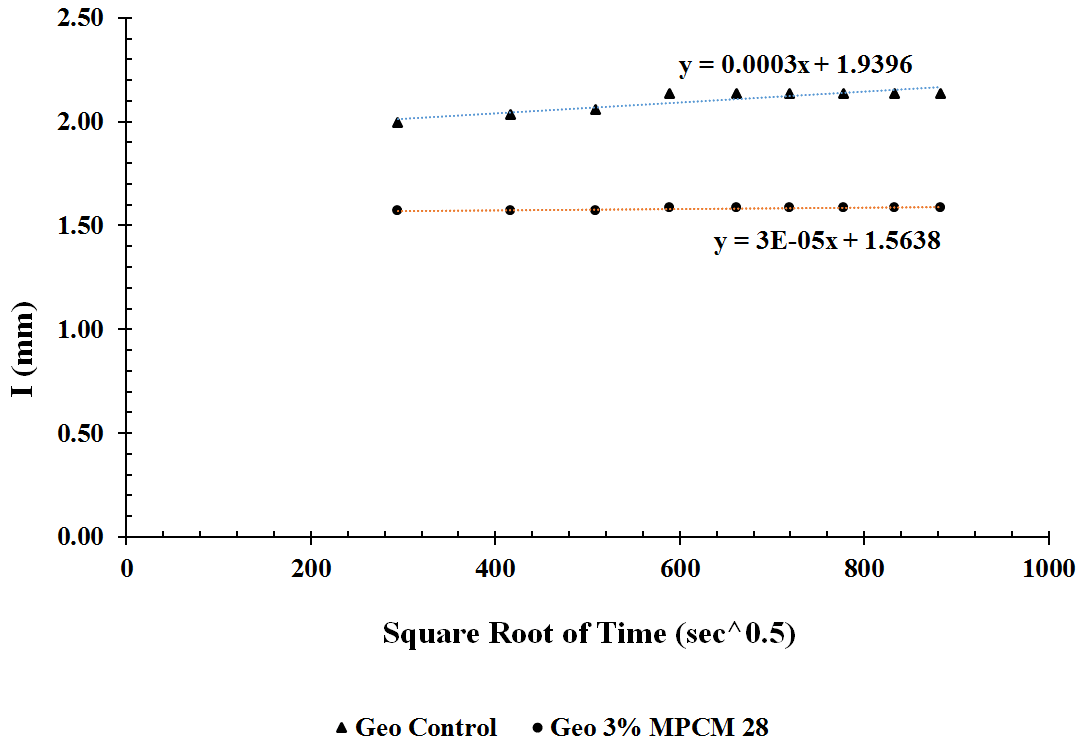


Figure 5.15 Secondary Sorptivity Geo/Geo 3% MPCM.

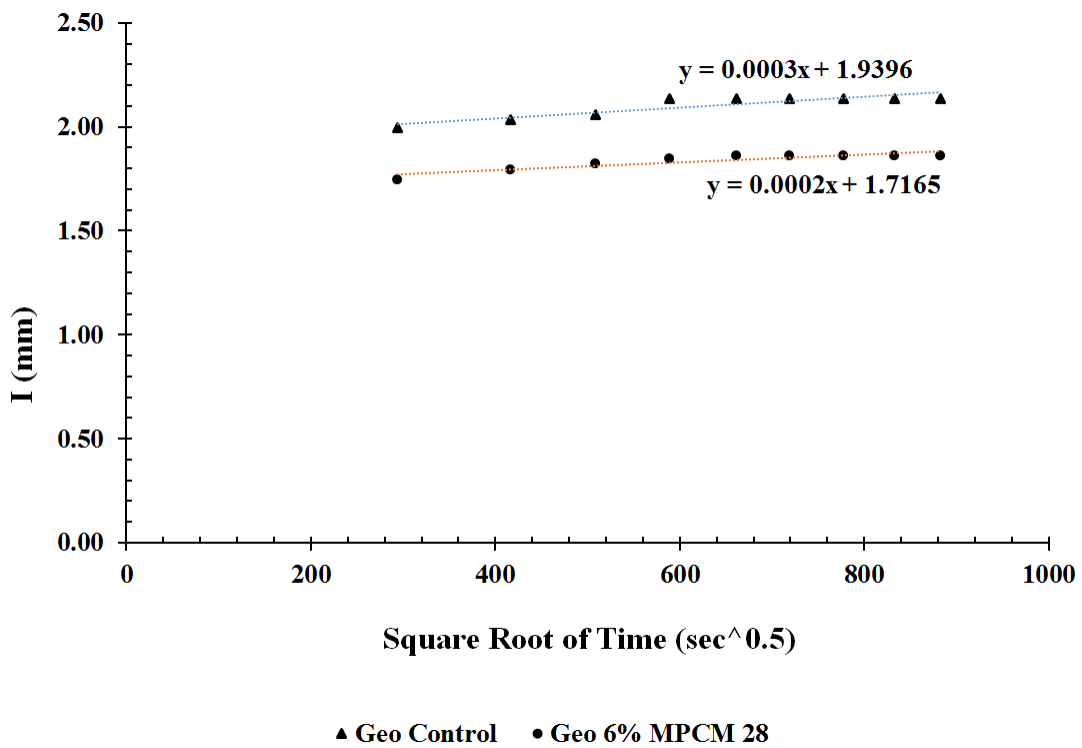


Figure 5.16 Secondary Sorptivity Geo/Geo 6% MPCM.

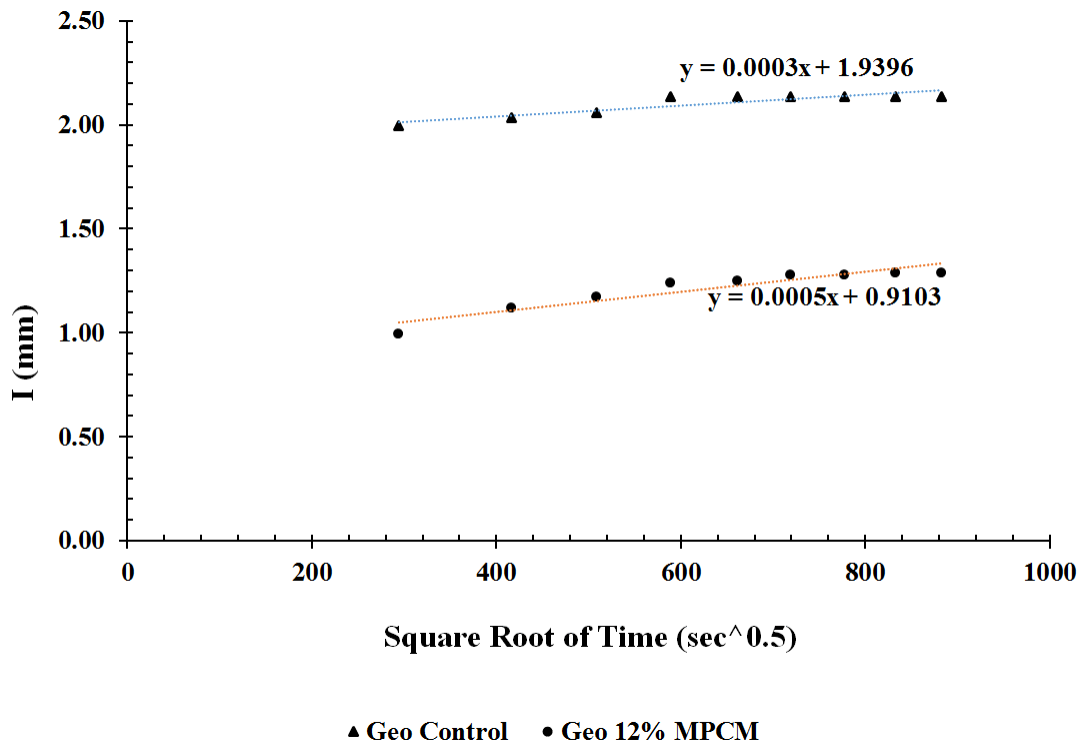


Figure 5.17 Secondary Sorptivity Geo/Geo 12% MPCM.

5.3.7 RCPT

Figure 5.18 shows different total charge passed through AAM specimens with and without the incorporation of MPCMs at the age of 28 days. Generally, increasing MPCMs contents resulted in a lower chloride ion penetration. According to ASTM C 1202 Limits shown in Table 3.5. Accordingly, the specimen can be classified as follows: the first two columns are the AAM without MPCM incorporation and have decreased the sand from control AAM mixture to 3% NO PCM mixture, both can be classified as highly permeable. Moreover, the 3%, 6% and 12% mixtures, can be classified as moderately permeable, keeping in mind that the permeability is decreasing as the

MPCMs incorporation is increasing. This can be ascribed to the nature of the AAMs as a ceramic materials at hardened ages.

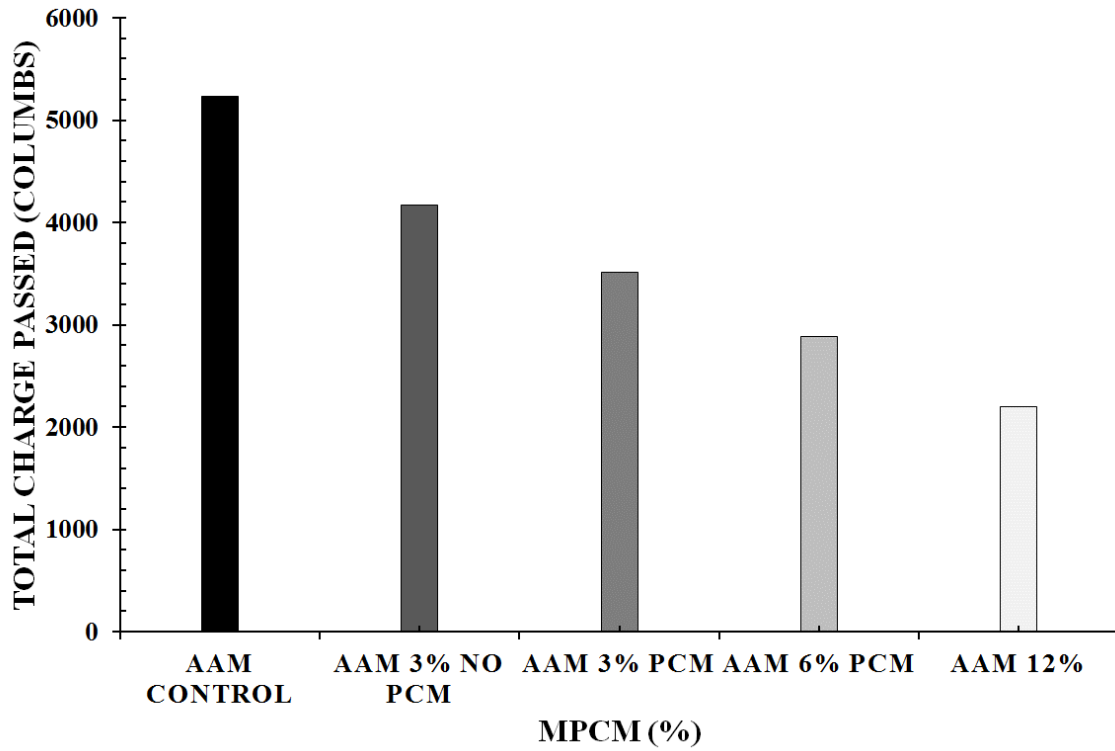


Figure 5.18 Total charge passed for AAM specimens with and without the incorporation of MPCMs

CHAPTER SIX

PERFORMANCE OF CEMENT AND ALKALI-ACTIVATED MORTAR INCORPORATING PHASE CHANGE MATERIALS IN SUBFREEZING CONDITION

6.1 Introduction

During cold weather, several construction materials and cementitious materials are being used. Part of them are being cast in-situ, and some others are prepared in the factory and moved to the field. Construction projects are ongoing during relatively cold (freezing, below 0 °C) in some areas in North America. In such cold climates, the subfreezing temperature may reign for long times, slowing down or even suspending construction projects. ACI Committee 3106R-10 has defined the cold weather as a period of three or more successive days with an average daily temperature below 4 °C and does not exceed 10 °C for more than one-half of any 24 hours (ACI 306 Committee, 2010). In such cold situation and winter construction, for cast-in-situ concrete, heating large areas for extended periods is considered, along with the use of accelerating admixtures to ensure adequate development of compressive strength. On the other hand, such methods are combined with extra cost and low-quality control management. Precast option commonly considered for its rigorous advantages in performance and quality control under adverse/cold weather, as precast elements are being cast and cured in a quality-controlled plant (Provost-Smith, 2017). Most

research efforts studied the effect of subfreezing conditions on the compressive strength. Repeatedly, most of the research outcomes demonstrated that compressive strength gain was adversely affected by the exposure to subfreezing conditions, especially at early ages. The effect of curing concrete made using cement type 1 and cement type 3 at $-4\text{ }^{\circ}\text{C}$ on the compressive strength was studied by Klieger (1958). A considerable reduction in the 28 days compressive strength with 13% and 70% for both types was reported, respectively. Another research efforts by Gardner (1990) disclosed that curing concrete at $0\text{ }^{\circ}\text{C}$ had negative effects on the compressive strength of concrete. On the other side, some research work reported no adverse effect on the compressive strength for concrete cured at $0\text{ }^{\circ}\text{C}$ and $2\text{ }^{\circ}\text{C}$ (Gardner, 1976 & 1988). Moreover, high-strength concrete incorporating silica fume and fly ash, cured in ocean water at $-5\text{ }^{\circ}\text{C}$, resulted in a reduction of the 7 days compressive strength of 37% and 81%, respectively Husem, (2005); Marzouk (1990). MPCM incorporation in construction materials has been studied extensively by many researchers. In order to employ such materials in thermally active and temperature affected construction materials. The main benefit of utilization MPCM in construction materials, depends on its ability to store and release heat. This chapter focuses on illustrating the effects of adding MPCMs as a volume replacement for sand on the performance of mortar cured at low temperatures (i.e. temperature (T) $=-15^{\circ}\text{C} \pm 2^{\circ}\text{C}$) meeting the temperature of the phase change for MPCM -10 .

6.2 Materials

Materials and testing procedures were the same as previous chapters (Chapter#3, Chapter#4, and Chapter#5). Differences are the applied curing regime and the melting temperature for, MPCMs.

Table 6.1 summarizes the properties for the used MPCMs according to the manufactured.

Table 6.1 MPCMs Properties

MPCM 6	Manufacturing Properties
Melting Point (°C)	6
Latent Heat Capacity (J/g)	165-175
Moisture (%)	< 3%
Appearance	White to Slightly White Powder
MPCM-10	
Melting Point (°C)	-10
Latent Heat Capacity (J/g)	170-180
Moisture (%)	< 3%
Appearance	White to Slightly White Powder

6.3 Testing and Specimen Preparation

All mortar cubes were compacted using the vibration table and the tamping rod. All samples were demolded after 24h, then stored at temperature $=-15^{\circ}\text{C} \pm 2^{\circ}\text{C}$. In addition, to determine the heat evolution profile inside of the mixtures, cylindrical samples 50 mm X 100 mm were prepared, and thermocouples type (T) were used to monitor the temperature change inside of the samples to follow temperature evolution in the samples from casting time.

The curing regimes were followed for this chapter consists of two curing regimes, one under the ambient condition for the first 24 hours, and then moved to the cold condition. The other curing regime followed a heat curing during the first 24 hours which is followed by moving to cold condition. **Figure 6.1** displays the second curing regime followed in this chapter,

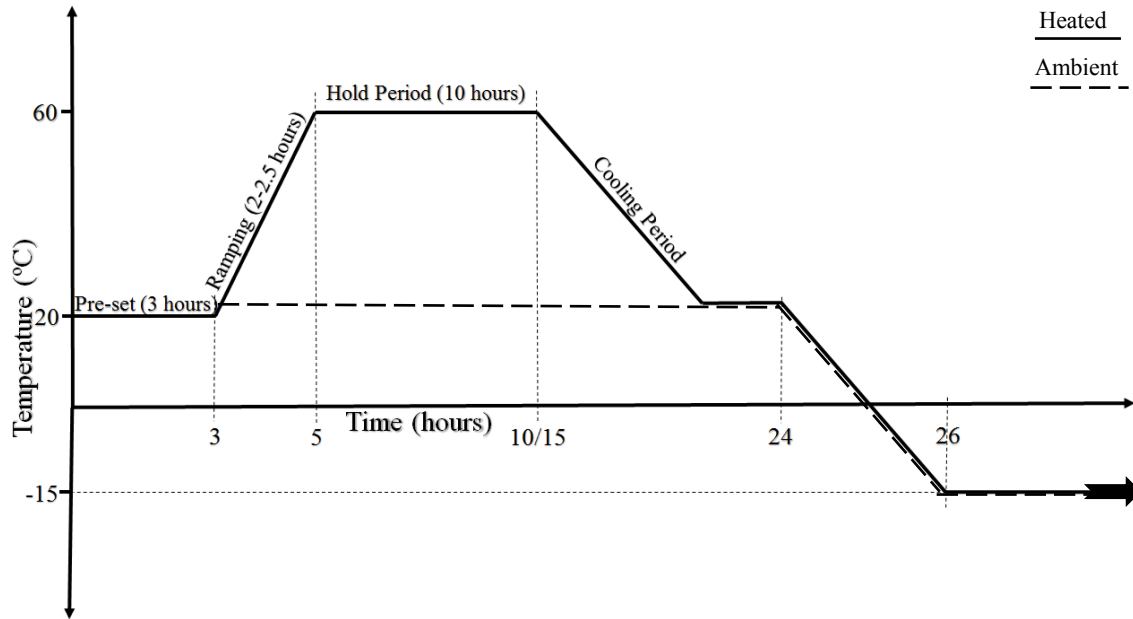


Figure 6.1 Curing Regime

6.4 Results and Discussions

6.4.1 Compressive Strength

Figure 6.2 shows the compressive strength development for OPC mortar specimens with and without MPCM 28, under the first curing regime of ambient for 24 hours. MPCM 28 was utilized here to study the effect of microcapsules inside mixtures undergoes subfreezing conditions. Generally, the compressive strength increased for all the samples over the investigated period. Also, specimens incorporating MPCM showed the usual trend of achieving lower strength compared to that of the control. For instance, at age 28 days, 3% achieved compressive strength less than the control with about 13%. Moreover, 6% and 12% at age 28 days achieved a compressive strength less than the control with about 20% and 30%, respectively.

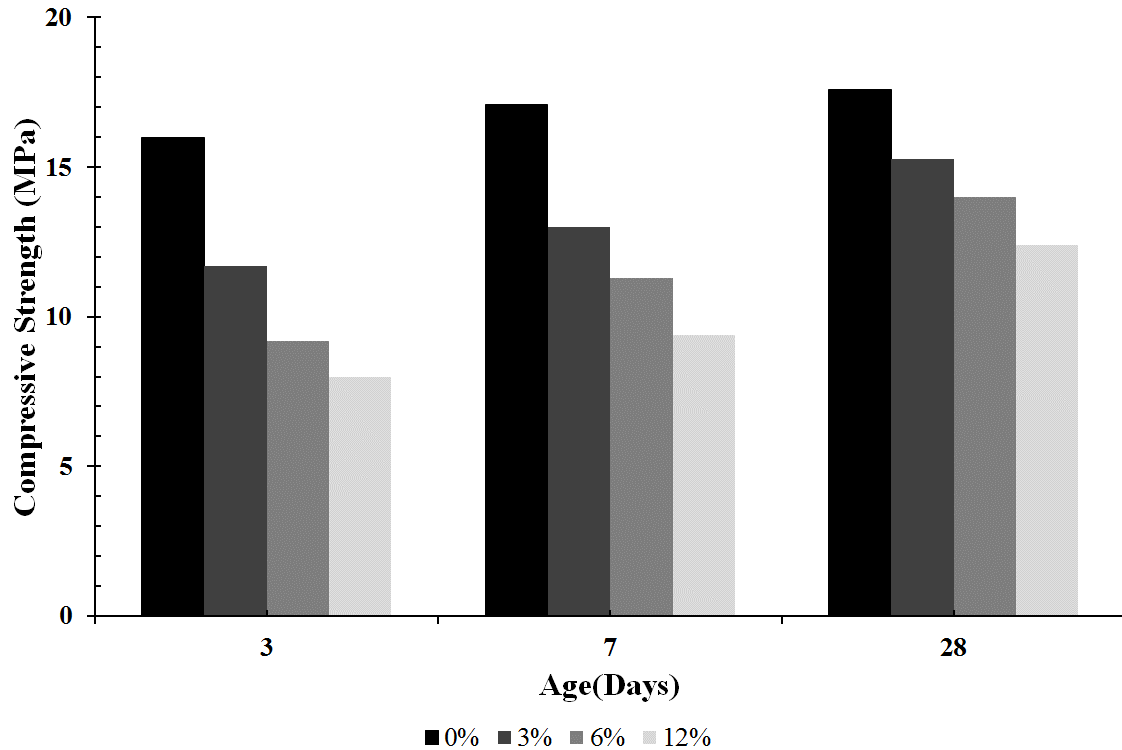


Figure 6.2 Compressive strength development for OPC mortar incorporated MPCM 28 under AMB then freezing condition

Figure 6.3 displays the compressive strength development for OPC mortar specimens with and without the incorporation of MPCM 6, under the first curing regime of ambient for 24 hours. Generally, the compressive strength increased for all the samples over the investigated period. Also, specimens incorporating MPCM 6 showed the usual trend of achieving lower strength compared to the control. For instance, 6% MPCM mixture showed better compressive strength at all ages after moving specimens to cold condition. For instance, at age 28 days, 3% attained compressive strength less than the control with about 15%. Moreover, 6% at age 28 days gained compressive strength lower than that of the control with about 11% and 4% more strength compared to 3% MPCM mixture. In addition, the 12% MPCM mixture achieved lower compressive strength at age of 28 days compared to the control with about 20%.

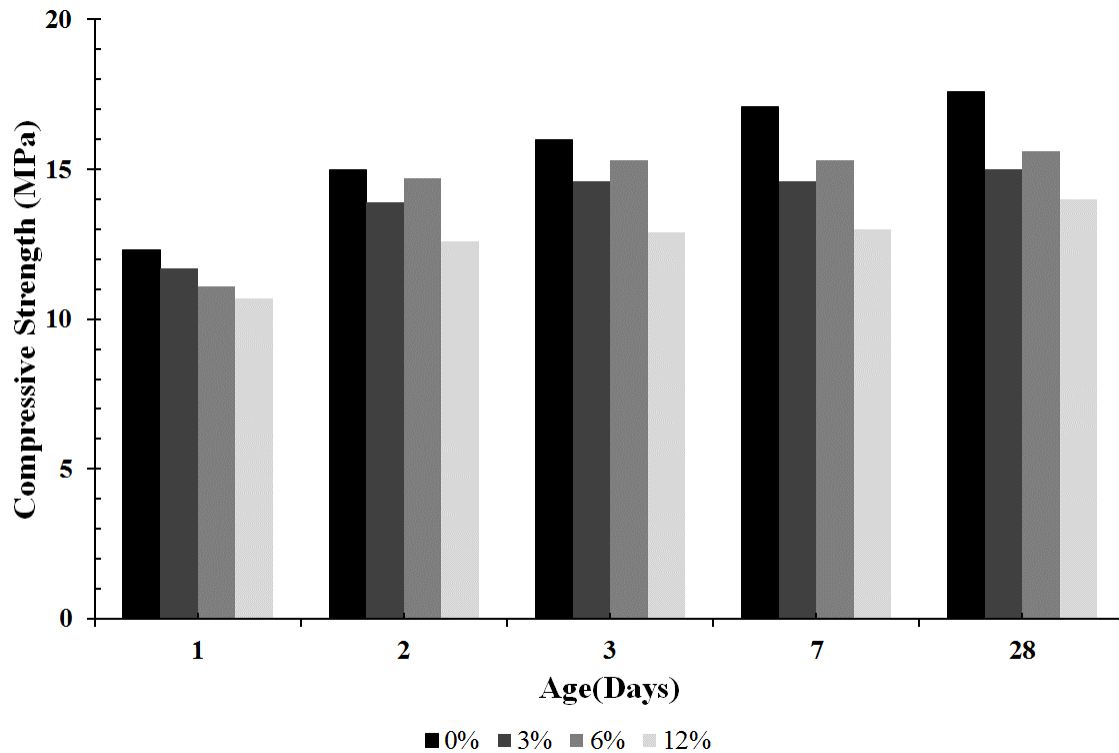


Figure 6.3 Compressive strength development for OPC mortar incorporated MPCM 6 under AMB then freezing condition

Figure 6.4 displays the compressive strength development for OPC mortar specimens with and without MPCM 6, under the second curing regime of heat curing for 10 hours. Generally, compressive strength increased for all the samples over the investigated period. Also, specimens incorporating MPCM 6 showed the usual trend of achieving lower strength compared to the control. For instance, at age 28 days, 3% MPCM mixture achieved compressive strength lower than that of the control with about 23%. Moreover, 6% and 12% at age 28 days achieved a compressive strength lower than the control with 30% and 34%, respectively.

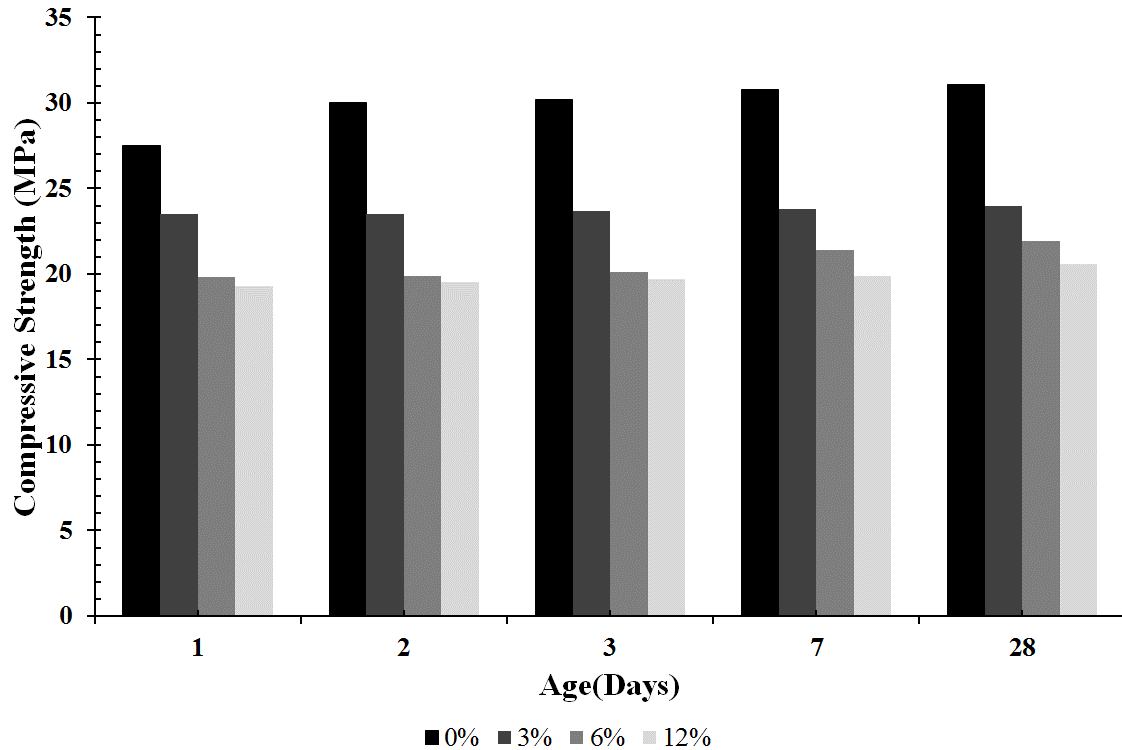


Figure 6.4 Compressive strength development for OPC mortar incorporated MPCM 6 under hot temperature 10 hours then freezing condition

Figure 6.5 displays the compressive strength development for OPC mortar specimens with and without MPCM -10, under the first curing regime, ambient for 24 hours. Generally, the compressive strength increased for all the samples over the investigated period. In addition, specimens incorporating MPCM showed the usual trend of achieving lower strength compared to the control. For instance, at age 28 days, 3% MPCM mixture achieved compressive strength lower than that of the control with about 16%. Moreover, 6% and 12% MPCM mixtures at age 28 days achieved a compressive strength lower than that of the control with about 27% and 30%, respectively.

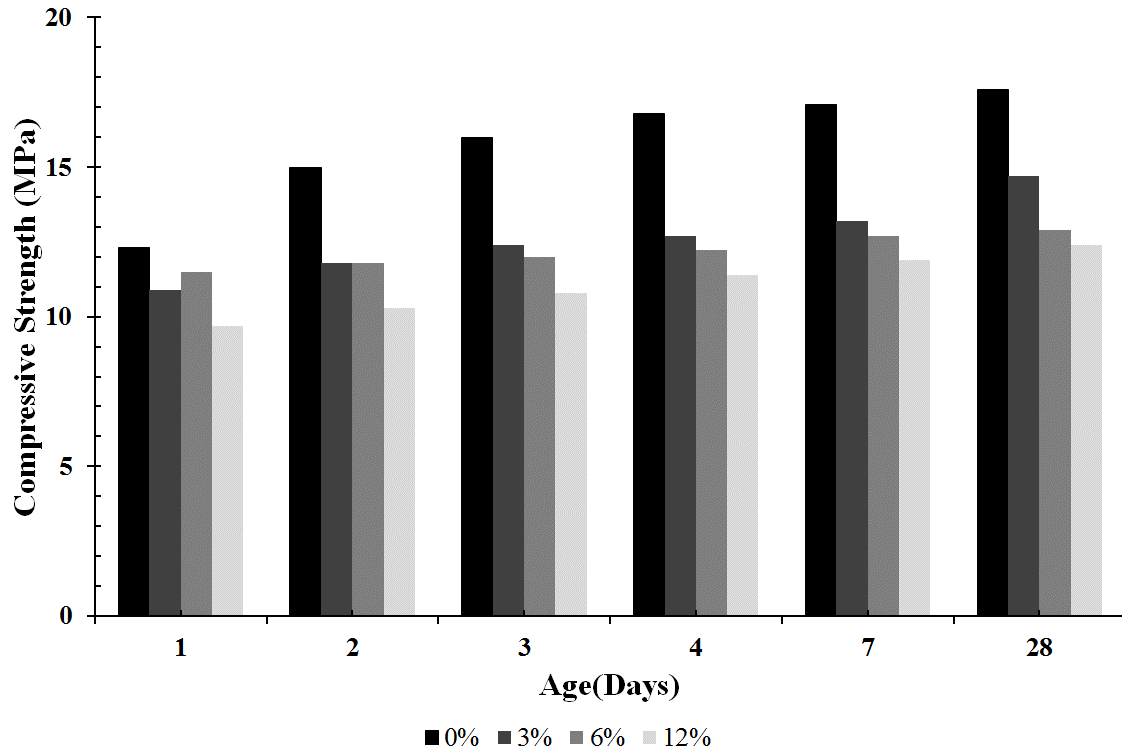


Figure 6.5 Compressive strength development for OPC mortar incorporated MPCM -10 under AMB then freezing condition

Figure 6.6 displays the compressive strength development for OPC mortar specimens with and without MPCM -10, under the second curing regime of heat curing for 10 hours. Generally, the compressive strength increased for all the samples over the investigated period. Also, specimens incorporating MPCM showed the usual trend of achieving lower strength compared to the control. It can be noticed that 6% MPCM mixture showed better compressive strength at all ages. For instance, at age 28 days, 3% MPM mixture attained compressive strength lower than that of the control with about 20%. Moreover, 6% MPCM mixture at age 28 days achieved a compressive strength lower than that of the control with about 15% and 5% more strength compared to 3% MPCM mixture. Also, 12% MPCM mixture gained lower compressive strength at age 28 days compared to that of the control with about 29%.

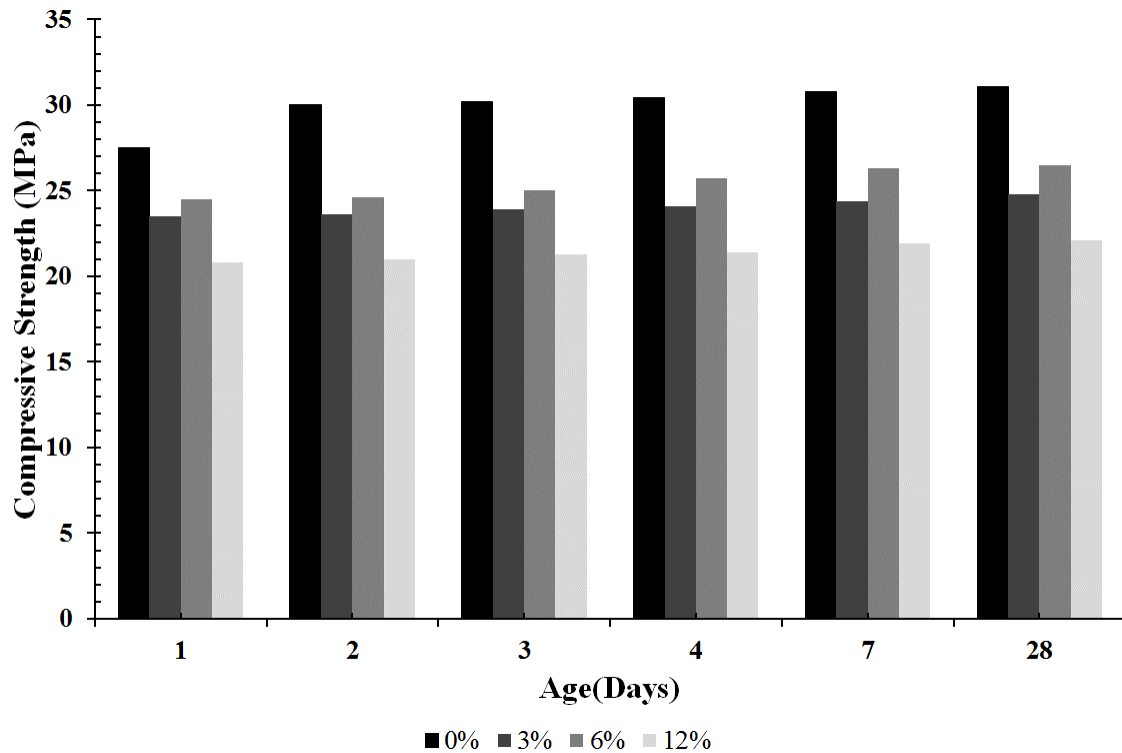


Figure 6.6 Compressive strength development for OPC mortar incorporated MPCM -10 under hot temperature 10 hours then freezing condition

Figure 6.7 displays the compressive strength development for AAM mortar specimens with and without MPCM -10, under the first curing regime of ambient for 24 hours. Generally, compressive strength increased for all the samples over the investigated period. In addition, specimens incorporating MPCM showed the usual trend of achieving lower strength compared to that of the control. For instance, at age 28 days, 3% MPCM mixture attained compressive strength lower than that of the control with about 25%. Moreover, 6% and 12% MPCM mixtures, at age 28 days achieved a compressive strength lower than that of the control with about 37% and 36%, respectively.

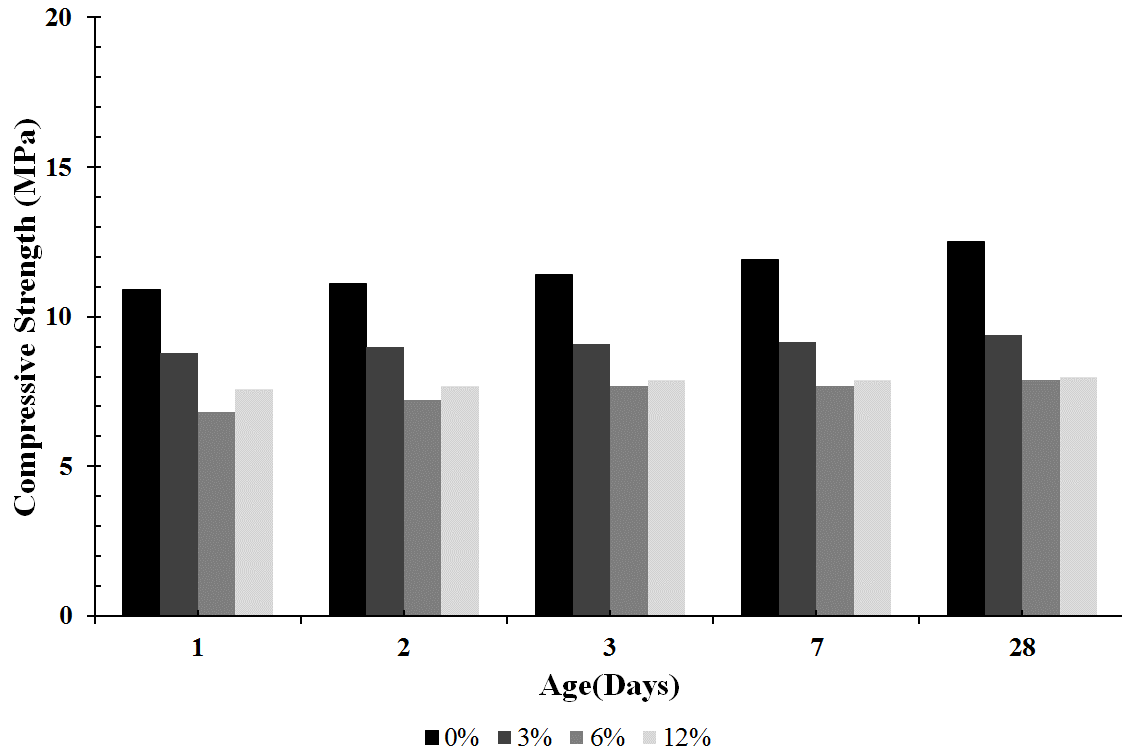


Figure 6.7 Compressive strength development for AAM mortar incorporated MPCM -10 under AMB then freezing condition

Figure 6.8 displays the compressive strength development for AAM mortar specimens with and without MPCM -10, under the second curing regime of heat curing for 10 hours. Generally, the compressive strength increased for all the samples over the investigated period. In addition, specimens incorporating MPCM showed the usual trend of achieving lower strength compared to the control. For instance, at age 28 days, 3% MPCM mixture achieved compressive strength lower than that of the control with about 3%. Moreover, 6% at age 28 days achieved a compressive strength lower than that of the control with about 16%. Also, the 12% achieved lower compressive strength at age 28 days compared to that of the control with about 12%, but more than 6% MPCM mixture with about 4%.

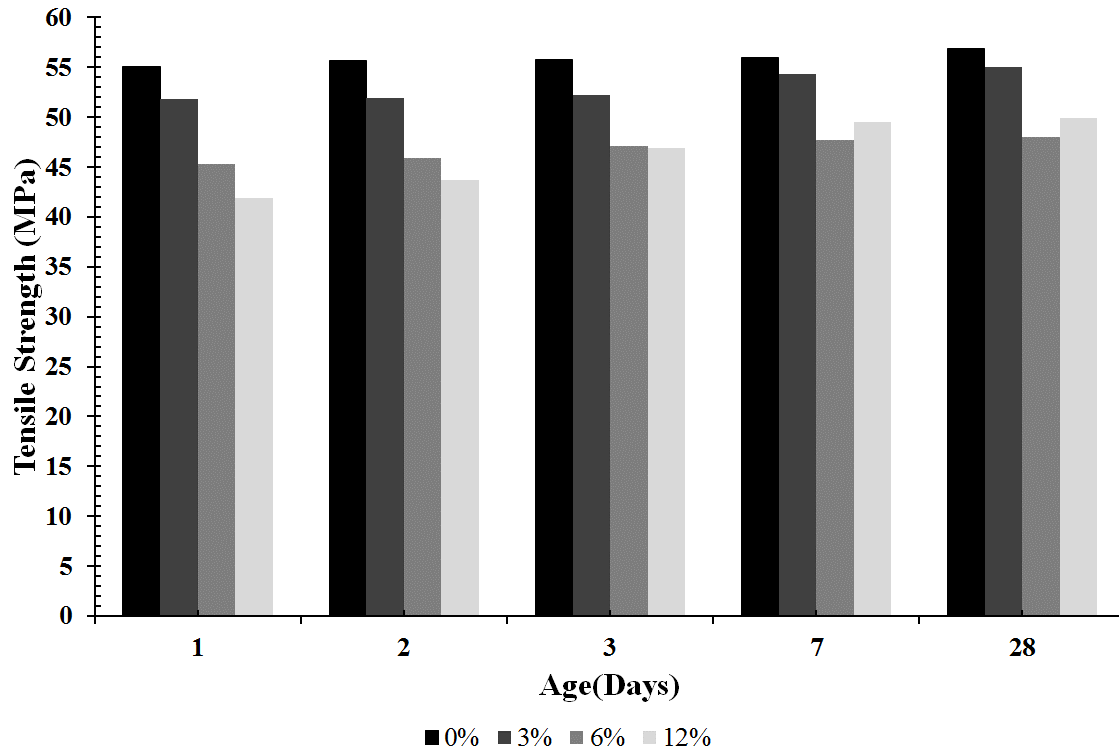


Figure 6.8 Compressive strength development for AAM mortar incorporated MPCM -10 under hot temperature 10 hours then freezing condition

All specimens were cured either in the ambient condition or the heat curing condition for the first 24 hours. Since, initial curing is very important for the progression of hydration/geopolymerization, which followed the findings and recommendations by Gardner (1976): Aitcin (1985): Provost-Smith (2017). Conforming to the previous findings of the previous chapters, adding MPCMs resulted in a reduction in the compressive strength for the incorporated mortars both in the OPC and AAM. Moreover, cementitious materials continued to gain mechanical strength after the exposure to cold temperature. In agreement with the previous findings pertaining curing at cold temperature Gardner (1990): Marzouk (1990): Provost-Smith (2017). Some research efforts debated that heat of hydration will keep the internal temperature of specimens above the subfreezing conditions like Nassif (2013). While it was not detected in heat profile testing coming

in the next section, that comes in agreement with the findings of Provost-Smith (2017). The authors reported that slowed compressive strength development can be mainly attributed to the total reduction of pore size distribution in cementitious materials under subfreezing conditions which may halt or stop the pozzilanic reactions. Some incorporation percentages (i.e, 6% MPCM) had exerted good compressive strength compared to the other percentages. Confirming with the previous chapters, especially for the hot temperature cured specimens. This could be ascribed to the development of microstructure and the hydration progression that may occur due to the nucleation sites effect as well as the latent heat storage discussed earlier.

6.4.2 Heat Profile

Following the heat profile is one of the most useful ways to proof and detect the effect of PCMs. Phase change materials used in this chapter have different phase-change temperatures, in both (i.e., melting and solidification). **Figure 6.9** displays temperature profile as a function of time for OPC specimens with 3%, 6%, and 12% of MPCM -10 exposed to hot temperature cured for 10 hours, and then demolded and placed in the fridge after 24 hours from casting. There are two different peaks captured by the thermocouples inserted in the chamber and planted in the middle of the cylindrical specimen (5cm X 10cm): first one occurs when the chamber temperature reached the freezing temperature of water (-4 °C). The second occurs as the temperature of the chamber reached the phase change temperature of MPCM -10. Moreover, the curves had experienced a change in the slope associated with a delay in temperature dropping, which is caused by the solidification of MPCM -10, and the heat released to the medium. Afterward, curves continued dropping. The higher the MPCMs incorporation percentage, the higher the delay.

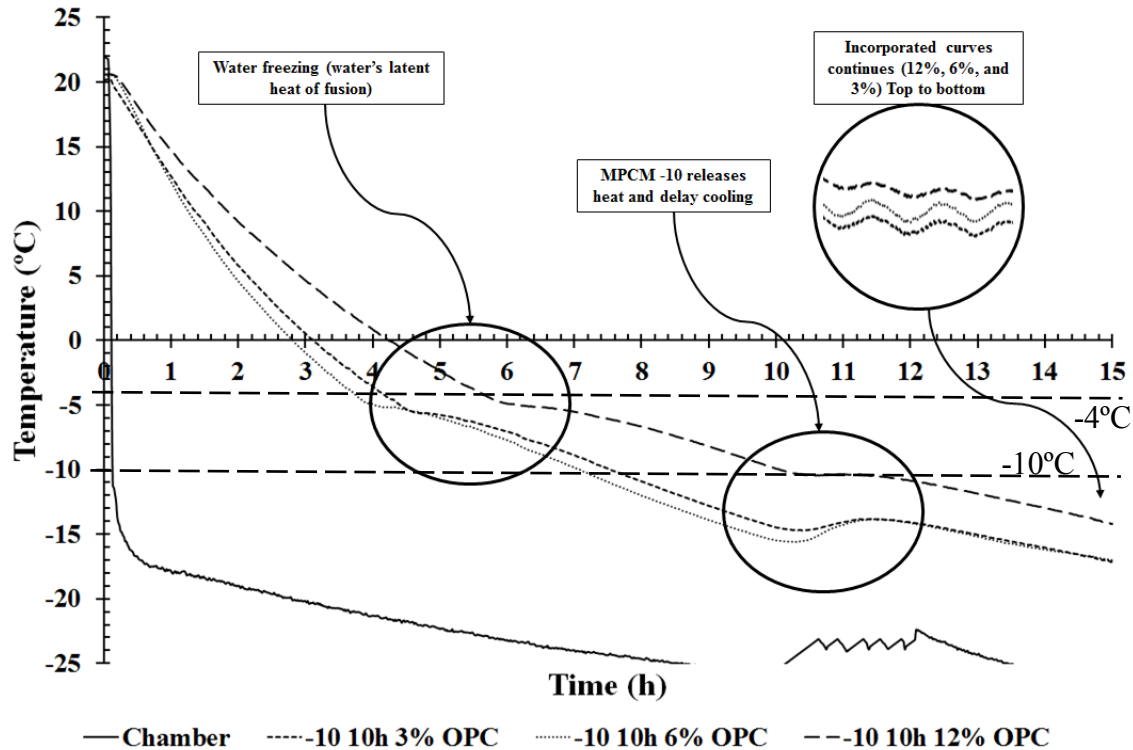


Figure 6.9 Temperature profile for OPC specimens incorporating MPCM -10 exposed to hot cure regime

MPCM -10 showed different behavior while incorporated in AAM under the same conditions as OPC in the previous figure. **Fig. 6.10** displays temperature profile as a function of time for AAM specimens with 3%, 6%, and 12% of MPCM -10 hot temperature cured for 10 hours, and then demolded and placed in the fridge after 24 hours from casting. As the temperature of the chamber reached the phase change temperature of MPCM -10, the curves had experienced a change in the slope associated with a delay in temperature dropping, which is caused by the solidification of MPCM -10, and the heat released to the medium. Afterward, curves continued dropping differently from OPC incorporated specimens curves. Where 12% of incorporation percentage showed more delay. Here it showed less temperature drop. In other words, the less the MPCM incorporation, the more delay in temperature dropping inside of the specimen, which is the same case in **Fig. 6.11**.

Another thing that can be observed here compared to the OPC is that no water freezing peaks appeared. This behavior can be ascribed to the heat treatment of AAM, where the free water that acts as a lubricant and not part of AAM structure will evaporate, and the water released from aluminosilicate matrix will let the geopolymerization to progress effectively (Ng, 2018), and it is consumed and evaporated during heating instead of getting captured by the microstructure of AAM, which is also proved in **Fig. 6.11**, where AAM specimens undergoes ambient curing before the fridge. In other words, the water is still inside the mixture. Moreover, increasing MPCMs incorporation showed denser microstructure for AAM. Hence, the temperature may drop faster inside specimens with increasing MPCMs incorporation.

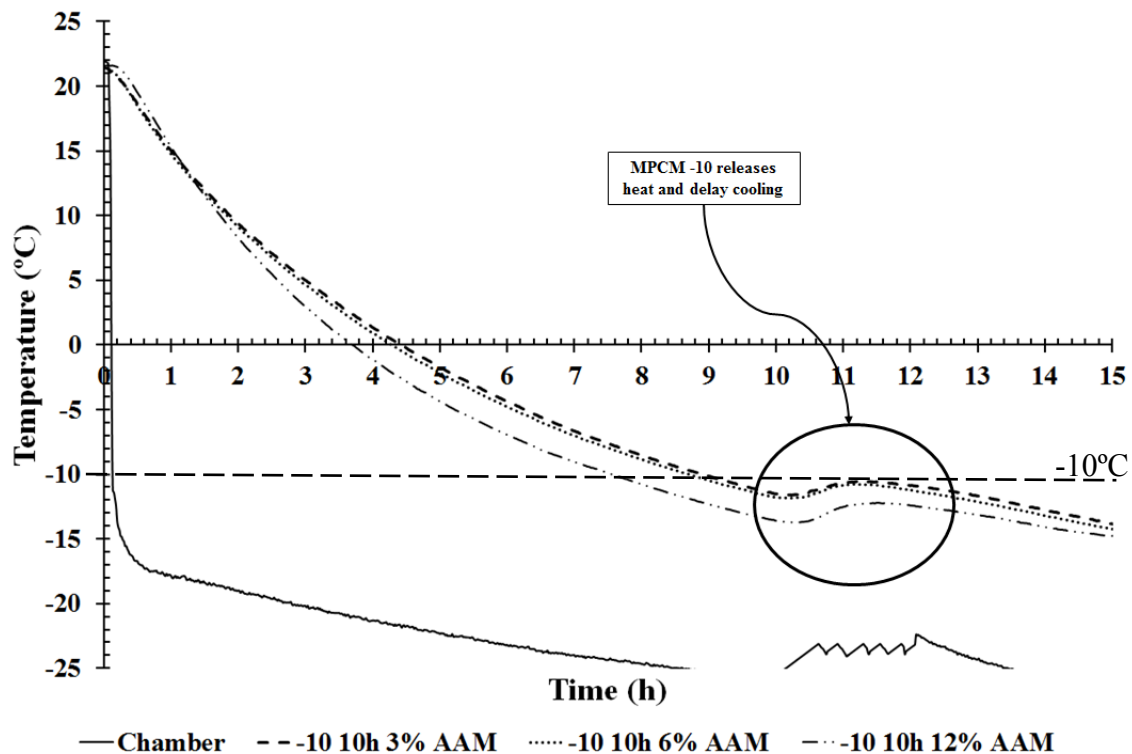


Figure 6.10 Temperature profile for AAM specimens incorporating MPCM -10 exposed to hot cure regime

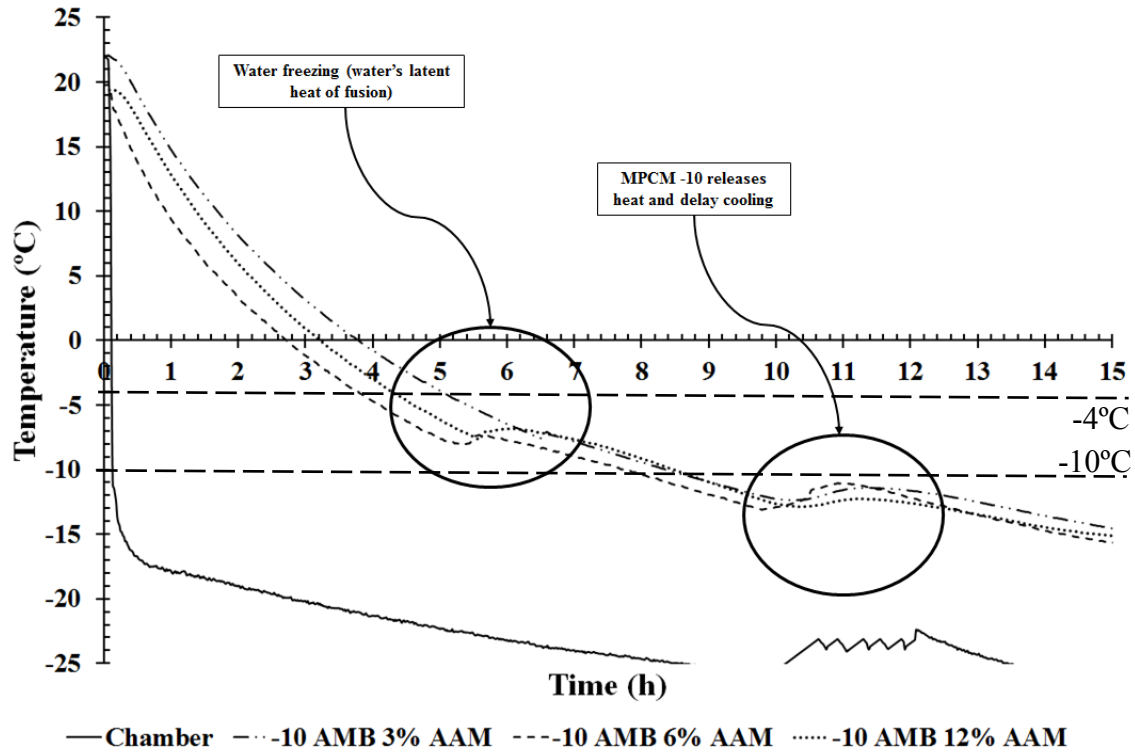


Figure 6.11 Temperature profile for AAM specimens incorporating MPCM -10 exposed to AMB curing regime

Figure 6.12 Displays temperature profile as a function of time for OPC samples with MPCM 6. As the temperature of the chamber reached the phase change temperature of MPCM 6, and then that of the freezing of water, the heat profile curve experienced a change in the slope associated with a delay.

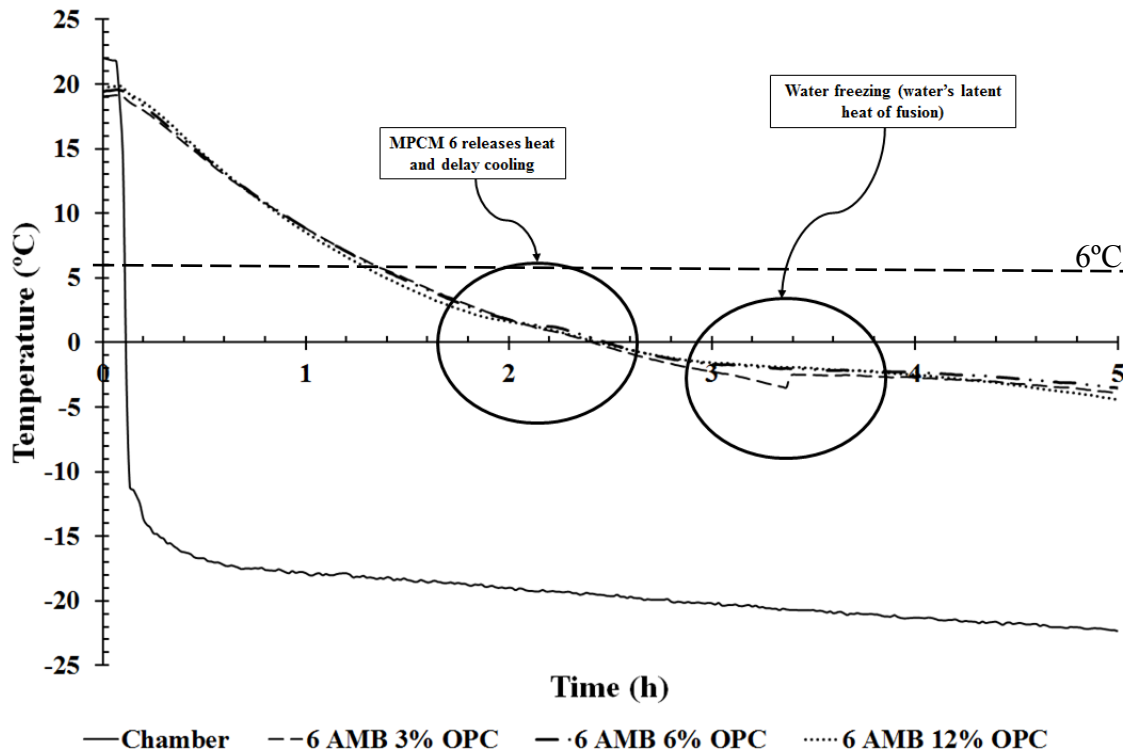


Figure 6.12 Temperature profile for OPC specimens incorporating MPCM 6 specimens exposed to AMB regime

Figure 6.13 shows temperature profile as a function of time for OPC and AAM samples with MPCM -10. As the temperature of the chamber reached the phase change temperature of MPCM -10, peaks of the phase transition of the incorporated MPCMs occurred. It can be observed that temperature dropping profile inside OPC samples was slower than that of AAM, and the delay caused by MPCM is more pronounced in AAM. This can be attributed to the presence of water with OPC sample compared to the AAM sample, where the latent heat of fusion appeared in the OPC curve.

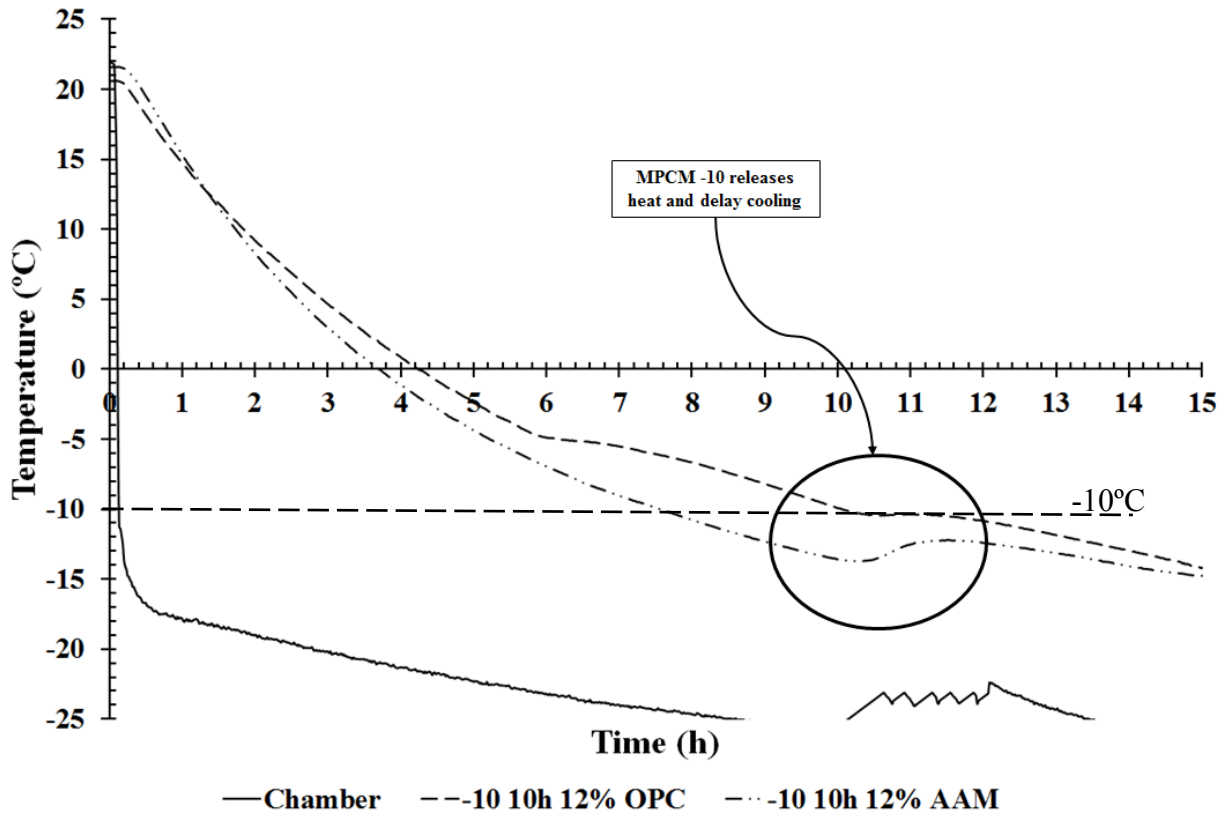


Figure 6.13 Temperature profile for OPC and AAM specimens incorporating MPCM -10 exposed to hot cure regime

CHAPTER SEVEN

Conclusions and Future Work

7.1 Conclusions

In this thesis, the effects of implementing MPCMs with different melting points on fresh, mechanical, thermal, and durability performance for cement-based and alkali-activated materials were investigated. The findings of this study are considered a keystone for ongoing research to better understanding and enhancing the knowledge about construction materials incorporating MPCMs. Moreover, MPCMs performance under different exposure conditions (i.e., ambient, hot and cold temperatures) was investigated. The following is a brief conclusion for the reported results.

- Adding MPCM reduces workability for OPC and AAMs mixtures. There is a need to use workability aids (i.e. superplasticizer) to maintain the desired fresh properties.
- The density had reduced by the addition of MPCMs, which is ascribed to the low specific gravity of MPCMs compared to sand. This can pave the way to produce lightweight construction materials.
- Adding MPCM reduces strength for OPC and AAMs mixtures, which is mainly attributed to the brittle nature of microcapsules and voids induced after its fracture. However, the reduction in AAMs mixtures strength was lower than that of OPC mixture. This indicates

that producing a denser microstructure can partially compensate for this reduction in strength.

- Adding MPCMs had induced an increase in the hydration peak at low dosages, which can be attributed to the nucleation effect of MPCMs and stimulates the hydration. On the other hand, a delay in the hydration peaks at medium to high dosages for both OPC and AAM as well. This can be ascribed by storing and releasing heat by MPCMs.
- The thermal capacity for OPC and AAMs mixtures incorporating MPCMs were affected. A delay in the heat transfer was observed, and it was ascribed to MPCMs changing phase and storing the heat.
- RCPT showed completely different results for OPC and AAMs mixtures incorporating MPCMs. In the OPC, adding MPCMs has increased the total charged passed, while on AAM the total charge passed was decreased by the addition of MPCMs.
- The absorption rate had been affected by MPCMs addition for OPC and AAMs mixtures. It was decreased for both OPC and AAM, unlikely it was more pronounced in the AAM. It could be ascribed to the development of the denser microstructure for AAM.
- Heat curing had enhanced the mechanical strength for control OPC and AAM. On the other hand, adding MPCMs have included a reduction in the compressive strength at all ages and percentages under both ambient and hot temperature curing. Nonetheless, 6% of MPCMs incorporation percentage has exerted good compressive strength compared to the other incorporation percentages.
- Exposure to cold temperature for both OPC and AAM, either it was cured at ambient or hot temperature during the first 24 hours resulted in a lower compressive strength compared to that exposed to ambient temperature after first 24 hours.

- Pozzolan reactions (hydration/geopolymerization) appeared to be halted and stopped under subfreezing conditions. While all specimens have gained mechanical strength especially during the first 3 days. While the development was slowed afterwards.
- MPCM incorporated specimens exposed to subfreezing conditions were affected by the incorporation as expected. But at some incorporation percentages (i.e. 6%), the specimens had attained better compressive strength and the rate of strength development was higher compared to the other incorporation percentages.

7.2 Future Work

For future work, it is worthy of investigating the following points:

- Developing much more stable and stiff microcapsules could increase the reliability of MPCMs inclusion in construction materials, as strength reduction is the main drawback reported worldwide. Improved microcapsules with less tendency to agglomeration while taking into consideration the compatibility with cementitious materials mediums.
- Despite that, there are many studies recommended adding MPCMs as sand replacement in cementitious mixtures, rather than addition on top of the mix. Developing a standard mix design for MPCMs incorporation is with a crucial benefit.
- A long term study for the properties of MPCMs incorporated OPC and AAM would give a clearer vision of the choice of MPCMs and its long term effects.
- Economic feasibility can be optimized for MPCMs incorporation in green buildings and different construction materials under several environmental conditions. This should help in assessing and addressing these materials into the construction.

References

- AASHTO T132, “Standard Method of Test for Tensile Strength of Hydraulic Cement Mortars Standard”. American Association of State Highway and Transportation Officials. 2013.
- ACI 306 Committee. (2010). Guide to cold weather concreting. ACI 306R-10, American Concrete Institute, Farmington Hills, Michigan, USA, 26.
- Adamson, A. W., & Gast, A. P. (1967). *Physical chemistry of surfaces* (Vol. 15). New York: Interscience.
- Aguayo, M., Das, S., Castro, C., Kabay, N., Sant, G., & Neithalath, N. (2017). Porous inclusions as hosts for phase change materials in cementitious composites: Characterization, thermal performance, and analytical models. *Construction and Building Materials*, 134, 574-584.
- Aguayo, M., Das, S., Maroli, A., Kabay, N., Mertens, J. C., Rajan, S. D., ... & Neithalath, N. (2016). The influence of microencapsulated phase change material (PCM) characteristics on the microstructure and strength of cementitious composites: Experiments and finite element simulations. *Cement and Concrete Composites*, 73, 29-41.
- Ahmad, M., Bontemps, A., Sallée, H., & Quenard, D. (2006). Experimental investigation and computer simulation of thermal behaviour of wallboards containing a phase change material. *Energy and buildings*, 38(4), 357-366.
- Ahmad, M., Bontemps, A., Sallée, H., & Quenard, D. (2006). Thermal testing and numerical simulation of a prototype cell using light wallboards coupling vacuum isolation panels and phase change material. *Energy and buildings*, 38(6), 673-681.
- Ahmari, S., Zhang, L., & Zhang, J. (2012). Effects of activator type/concentration and curing temperature on alkali-activated binder based on copper mine tailings. *Journal of Materials Science*, 47(16), 5933-5945.
- Aitcin, P. C., Cheung, M. S., & Shah, V. K. (1985). Strength development of concrete cured under arctic sea conditions. In *Temperature Effects on Concrete*. ASTM International.
- Akeiber, H., Nejat, P., Majid, M. Z. A., Wahid, M. A., Jomehzadeh, F., Famileh, I. Z., ... & Zaki, S. A. (2016). A review on phase change material (PCM) for sustainable passive cooling in building envelopes. *Renewable and Sustainable Energy Reviews*, 60, 1470-1497.

- Akeiber, H., Nejat, P., Majid, M. Z. A., Wahid, M. A., Jomehzadeh, F., Famileh, I. Z., ... & Zaki, S. A. (2016). A review on phase change material (PCM) for sustainable passive cooling in building envelopes. *Renewable and Sustainable Energy Reviews*, 60, 1470-1497.
- Alawadhi, E. M. (2008). Thermal analysis of a building brick containing phase change material. *Energy and Buildings*, 40(3), 351-357.
- Alqallaf, H. J., & Alawadhi, E. M. (2013). Concrete roof with cylindrical holes containing PCM to reduce the heat gain. *Energy and Buildings*, 61, 73-80.
- Altan, E., & Erdoğan, S. T. (2012). Alkali activation of a slag at ambient and elevated temperatures. *Cement and Concrete Composites*, 34(2), 131-139.
- Amaral, C., Vicente, R., Marques, P. A. A. P., & Barros-Timmons, A. (2017). Phase change materials and carbon nanostructures for thermal energy storage: A literature review. *Renewable and Sustainable Energy Reviews*, 79, 1212-1228.
- Ansuini, R., Larghetti, R., Giretti, A., & Lemma, M. (2011). Radiant floors integrated with PCM for indoor temperature control. *Energy and Buildings*, 43(11), 3019-3026.
- Athienitis, A. K., Liu, C., Hawes, D., Banu, D., & Feldman, D. (1997). Investigation of the thermal performance of a passive solar test-room with wall latent heat storage. *Building and environment*, 32(5), 405-410.
- Baetens, R., Jelle, B. P., & Gustavsen, A. (2010). Phase change materials for building applications: a state-of-the-art review. *Energy and buildings*, 42(9), 1361-1368.
- Bahrar, M., Djamai, Z. I., Mankibi, M. E., Larbi, A. S., & Salvia, M. (2018). Numerical and experimental study on the use of microencapsulated phase change materials (PCMs) in textile reinforced concrete panels for energy storage. *Sustainable cities and society*, 41, 455-468.
- Bahrar, M., Djamai, Z. I., Mankibi, M. E., Larbi, A. S., & Salvia, M. (2018). Numerical and experimental study on the use of microencapsulated phase change materials (PCMs) in textile reinforced concrete panels for energy storage. *Sustainable cities and society*, 41, 455-468.
- Banu, D., Feldman, D., & Hawes, D. (1998). Evaluation of thermal storage as latent heat in phase change material wallboard by differential scanning calorimetry and large scale thermal testing. *Thermochimica acta*, 317(1), 39-45.

- Barzin, R., Chen, J. J., Young, B. R., & Farid, M. M. (2015). Application of PCM underfloor heating in combination with PCM wallboards for space heating using price based control system. *Applied energy*, 148, 39-48.
- Basheer, L., Kropp, J., & Cleland, D. J. (2001). Assessment of the durability of concrete from its permeation properties: a review. *Construction and building materials*, 15(2-3), 93-103.
- Bastani, A., Haghighat, F., & Kozinski, J. (2014). Designing building envelope with PCM wallboards: design tool development. *Renewable and Sustainable Energy Reviews*, 31, 554-562.
- Bentz, D. P., & Turpin, R. (2007). Potential applications of phase change materials in concrete technology. *Cement and Concrete Composites*, 29(7), 527-532.
- Bingöl, A. F., & Tohumcu, İ. (2013). Effects of different curing regimes on the compressive strength properties of self compacting concrete incorporating fly ash and silica fume. *Materials & Design*, 51, 12-18.
- Biswas, K., Lu, J., Soroushian, P., & Shrestha, S. (2014). Combined experimental and numerical evaluation of a prototype nano-PCM enhanced wallboard. *Applied energy*, 131, 517-529.
- Borreguero, A. M., Carmona, M., Sanchez, M. L., Valverde, J. L., & Rodriguez, J. F. (2010). Improvement of the thermal behaviour of gypsum blocks by the incorporation of microcapsules containing PCMS obtained by suspension polymerization with an optimal core/coating mass ratio. *Applied Thermal Engineering*, 30(10), 1164-1169.
- Braun, J. E., Montgomery, K. W., & Chaturvedi, N. (2001). Evaluating the performance of building thermal mass control strategies. *HVAC&R Research*, 7(4), 403-428.
- Bullard, J. W., Jennings, H. M., Livingston, R. A., Nonat, A., Scherer, G. W., Schweitzer, J. S., ... & Thomas, J. J. (2011). Mechanisms of cement hydration. *Cement and concrete research*, 41(12), 1208-1223.
- Cabeza, L. F., Castellon, C., Nogues, M., Medrano, M., Leppers, R., & Zubillaga, O. (2007). Use of microencapsulated PCM in concrete walls for energy savings. *Energy and Buildings*, 39(2), 113-119.
- Cai, Y., Sun, G., Liu, M., Zhang, J., Wang, Q., & Wei, Q. (2015). Fabrication and characterization of capric-lauric-palmitic acid/electrospun SiO₂ nanofibers composite as form-stable phase change material for thermal energy storage/retrieval. *Solar Energy*, 118, 87-95.

- Cao, V. D., Pilehvar, S., Salas-Bringas, C., Szczotok, A. M., Rodriguez, J. F., Carmona, M., ... & Kjøniksen, A. L. (2017). Microencapsulated phase change materials for enhancing the thermal performance of Portland cement concrete and geopolymer concrete for passive building applications. *Energy conversion and management*, 133, 56-66.
- Carbonari, A., De Grassi, M., Di Perna, C., & Principi, P. (2006). Numerical and experimental analyses of PCM containing sandwich panels for prefabricated walls. *Energy and Buildings*, 38(5), 472-483.
- Castell, A., & Farid, M. M. (2014). Experimental validation of a methodology to assess PCM effectiveness in cooling building envelopes passively. *Energy and Buildings*, 81, 59-71.
- Castell, A., Martorell, I., Medrano, M., Pérez, G., & Cabeza, L. F. (2010). Experimental study of using PCM in brick constructive solutions for passive cooling. *Energy and Buildings*, 42(4), 534-540.
- Castellón, C., Castell, A., Medrano, M., Martorell, I., & Cabeza, L. F. (2009). Experimental study of PCM inclusion in different building envelopes. *Journal of Solar Energy Engineering*, 131(4), 041006.
- Castellón, C., Medrano, M., Roca, J., Cabeza, L. F., Navarro, M. E., Fernández, A. I., ... & Zalba, B. (2010). Effect of microencapsulated phase change material in sandwich panels. *Renewable Energy*, 35(10), 2370-2374.
- Cohen, B. (2015). Urbanization, City growth, and the New United Nations development agenda. *Cornerstone*, 3(2), 4-7.
- Coppola, L., Coffetti, D., & Lorenzi, S. (2016). Cement-based renders manufactured with phase-change materials: Applications and feasibility. *Advances in Materials Science and Engineering*, 2016.
- Costoya Fernández, M. M. (2008). Effect of particle size on the hydration kinetics and microstructural development of tricalcium silicate (No. THESIS). EPFL.
- Cui, H., Liao, W., Memon, S., Dong, B., & Tang, W. (2014). Thermophysical and mechanical properties of hardened cement paste with microencapsulated phase change materials for energy storage. *Materials*, 7(12), 8070-8087.
- Cui, H., Liao, W., Mi, X., Lo, T. Y., & Chen, D. (2015). Study on functional and mechanical properties of cement mortar with graphite-modified microencapsulated phase-change materials. *Energy and Buildings*, 105, 273-284.

- Cui, H., Memon, S. A., & Liu, R. (2015). Development, mechanical properties and numerical simulation of macro encapsulated thermal energy storage concrete. *Energy and buildings*, 96, 162-174.
- Cui, H., Tang, W., Qin, Q., Xing, F., Liao, W., & Wen, H. (2017). Development of structural-functional integrated energy storage concrete with innovative macro-encapsulated PCM by hollow steel ball. *Applied energy*, 185, 107-118.
- Cui, Y., Xie, J., Liu, J., Wang, J., & Chen, S. (2017). A review on phase change material application in building. *Advances in Mechanical Engineering*, 9(6), 1687814017700828.
- Cunha, S., Aguiar, J. B., Kheradmand, M., Bragança, L., & Ferreira, V. M. (2013). Thermal mortars with incorporation of PCM microcapsules. *Restoration of Buildings and Monuments*, 19(2-3), 171-178.
- Cunha, S., Aguiar, J., & Ferreira, V. (2017). Mortars with incorporation of phase change materials for thermal rehabilitation. *International Journal of Architectural Heritage*, 11(3), 339-348.
- Cunha, S., Aguiar, J., & Pacheco-Torgal, F. (2015). Effect of temperature on mortars with incorporation of phase change materials. *Construction and Building Materials*, 98, 89-101.
- Cunha, S., Lima, M., & Aguiar, J. B. (2016). Influence of adding phase change materials on the physical and mechanical properties of cement mortars. *Construction and Building Materials*, 127, 1-10.
- Cunha, Sandra, José Aguiar, Victor Ferreira, and António Tadeu. "Mortars based in different binders with incorporation of phase-change materials: Physical and mechanical properties." *European Journal of Environmental and Civil Engineering* 19, no. 10 (2015): 1216-1233.
- Doležal, J., Škvára, F., Kopecký, L., Pavlasová, S., Lucuk, M., Dvořáček, K., & Šulc, R. (2006). Concrete based on fly ash geopolymers. *Proceedings of 16th Intern. Baustofftagung IBAUSIL*.
- Dove, P. M., & Han, N. (2007, June). Kinetics of mineral dissolution and growth as reciprocal microscopic surface processes across chemical driving force. In *AIP Conference Proceedings* (Vol. 916, No. 1, pp. 215-234). AIP.
- Dove, P. M., Han, N., & De Yoreo, J. J. (2005). Mechanisms of classical crystal growth theory explain quartz and silicate dissolution behavior. *Proceedings of the National Academy of Sciences*, 102(43), 15357-15362.

- Doyle, M. W., & Havlick, D. G. (2009). Infrastructure and the environment. *Annual Review of Environment and Resources*, 34, 349-373.
- Drissi, S., Ling, T. C., Mo, K. H., & Eddhahak, A. (2019). A review of microencapsulated and composite phase change materials: Alteration of strength and thermal properties of cement-based materials. *Renewable and Sustainable Energy Reviews*, 110, 467-484.
- Duxson, P., Fernández-Jiménez, A., Provis, J. L., Lukey, G. C., Palomo, A., & van Deventer, J. S. (2007). Geopolymer technology: the current state of the art. *Journal of materials science*, 42(9), 2917-2933.
- Duxson, P., Provis, J. L., Lukey, G. C., & Van Deventer, J. S. (2007). The role of inorganic polymer technology in the development of 'green concrete'. *Cement and Concrete Research*, 37(12), 1590-1597.
- Eddhahak, A., Drissi, S., Colin, J., Caré, S., & Neji, J. (2014). Effect of phase change materials on the hydration reaction and kinetic of PCM-mortars. *Journal of thermal analysis and calorimetry*, 117(2), 537-545.
- Eddhahak-Ouni, A., Drissi, S., Colin, J., Neji, J., & Care, S. (2014). Experimental and multi-scale analysis of the thermal properties of Portland cement concretes embedded with microencapsulated Phase Change Materials (PCMs). *Applied Thermal Engineering*, 64(1-2), 32-39.
- Evola, G., L. Marletta, and F. Sicurella. "A methodology for investigating the effectiveness of PCM wallboards for summer thermal comfort in buildings." *Building and Environment* 59 (2013): 517-527.
- Fallahi, A., Guldentops, G., Tao, M., Granados-Focil, S., & Van Dessel, S. (2017). Review on solid-solid phase change materials for thermal energy storage: Molecular structure and thermal properties. *Applied Thermal Engineering*, 127, 1427-1441.
- Falzone, G., Falla, G. P., Wei, Z., Zhao, M., Kumar, A., Bauchy, M., ... & Sant, G. (2016). The influences of soft and stiff inclusions on the mechanical properties of cementitious composites. *Cement and Concrete Composites*, 71, 153-165.
- Fang, X., & Zhang, Z. (2006). A novel montmorillonite-based composite phase change material and its applications in thermal storage building materials. *Energy and Buildings*, 38(4), 377-380.

- Farnam, Y., Esmaeeli, H. S., Zavattieri, P. D., Haddock, J., & Weiss, J. (2017). Incorporating phase change materials in concrete pavement to melt snow and ice. *Cement and concrete composites*, 84, 134-145.
- Feldman, D., Banu, D., & Hawes, D. W. (1995). Development and application of organic phase change mixtures in thermal storage gypsum wallboard. *Solar Energy Materials and Solar Cells*, 36(2), 147-157.
- Fernandes, F., Manari, S., Aguayo, M., Santos, K., Oey, T., Wei, Z., ... & Sant, G. (2014). On the feasibility of using phase change materials (PCMs) to mitigate thermal cracking in cementitious materials. *Cement and Concrete Composites*, 51, 14-26.
- Figueiredo, A., Lapa, J., Vicente, R., & Cardoso, C. (2016). Mechanical and thermal characterization of concrete with incorporation of microencapsulated PCM for applications in thermally activated slabs. *Construction and Building Materials*, 112, 639-647.
- Franquet, E., Gibout, S., Tittlein, P., Zalewski, L., & Dumas, J. P. (2014). Experimental and theoretical analysis of a cement mortar containing microencapsulated PCM. *Applied Thermal Engineering*, 73(1), 32-40.
- Frigione, M., Lettieri, M., & Sarcinella, A. (2019). Phase Change Materials for Energy Efficiency in Buildings and Their Use in Mortars. *Materials*, 12(8), 1260.
- Frigione, M., Lettieri, M., Sarcinella, A., & de Aguiar, J. B. (2018, April). Mortars with Phase Change Materials (PCM) and Stone Waste to Improve Energy Efficiency in Buildings. In *International Congress on Polymers in Concrete* (pp. 195-201). Springer, Cham.
- Gallucci, E., Mathur, P., & Scrivener, K. (2010). Microstructural development of early age hydration shells around cement grains. *Cement and Concrete Research*, 40(1), 4-13.
- Gardner, N. J. (1990). Effect of Temperature on the Early-age Properties of Type I, Type II, and Type III/fly ash concretes with temperature. *Materials Journal*, 87(1), 68-78.
- Gardner, N. J., & Poon, S. M. (1976, July). Time and temperature effects on tensile, bond, and compressive strengths. In *Journal Proceedings* (Vol. 73, No. 7, pp. 405-409).
- Gebregziabih, B. S., Thomas, R. J., & Peethamparan, S. (2016). Temperature and activator effect on early-age reaction kinetics of alkali-activated slag binders. *Construction and Building Materials*, 113, 783-793.
- Gschwander, S., Schossig, P., & Henning, H. M. (2005). Micro-encapsulated paraffin in phase-change slurries. *Solar Energy Materials and Solar Cells*, 89(2-3), 307-315.

- Hadjieva, M., Stoykov, R., & Filipova, T. Z. (2000). Composite salt-hydrate concrete system for building energy storage. *Renewable Energy*, 19(1-2), 111-115.
- Hall, C. (1989). Water sorptivity of mortars and concretes: a review. *Magazine of concrete research*, 41(147), 51-61.
- Hall, C., Hoff, W. D., & Wilson, M. A. (1993). Effect of non-sorptive inclusions on capillary absorption by a porous material. *Journal of Physics D: Applied Physics*, 26(1), 31.
- Haurie, L., Serrano, S., Bosch, M., Fernandez, A. I., & Cabeza, L. F. (2016). Single layer mortars with microencapsulated PCM: Study of physical and thermal properties, and fire behaviour. *Energy and Buildings*, 111, 393-400.
- Hawladar, M. N. A., Uddin, M. S., & Khin, M. M. (2003). Microencapsulated PCM thermal-energy storage system. *Applied energy*, 74(1-2), 195-202.
- Heah, C. Y., Kamarudin, H., Al Bakri, A. M., Binhussain, M., Luqman, M., Nizar, I. K., ... & Liew, Y. M. (2011). Effect of curing profile on kaolin-based geopolymers. *Physics Procedia*, 22, 305-311.
- Huang, M. J., Eames, P. C., & Hewitt, N. J. (2006). The application of a validated numerical model to predict the energy conservation potential of using phase change materials in the fabric of a building. *Solar Energy Materials and Solar Cells*, 90(13), 1951-1960.
- Hunger, M., Entrop, A. G., Mandilaras, I., Brouwers, H. J. H., & Founti, M. (2009). The behavior of self-compacting concrete containing micro-encapsulated phase change materials. *Cement and Concrete Composites*, 31(10), 731-743.
- Husem, M., & Gozutok, S. (2005). The effects of low temperature curing on the compressive strength of ordinary and high performance concrete. *Construction and Building Materials*, 19(1), 49-53.
- Jamekhorshid, A., Sadrameli, S. M., & Farid, M. (2014). A review of microencapsulation methods of phase change materials (PCMs) as a thermal energy storage (TES) medium. *Renewable and Sustainable Energy Reviews*, 31, 531-542.
- Jamekhorshid, A., Sadrameli, S. M., Barzin, R., & Farid, M. M. (2017). Composite of wood-plastic and micro-encapsulated phase change material (MEPCM) used for thermal energy storage. *Applied Thermal Engineering*, 112, 82-88.

- Jayalath, A., San Nicolas, R., Sofi, M., Shanks, R., Ngo, T., Aye, L., & Mendis, P. (2016). Properties of cementitious mortar and concrete containing micro-encapsulated phase change materials. *Construction and Building Materials*, 120, 408-417.
- Jeong, S. G., Jeon, J., Cha, J., Kim, J., & Kim, S. (2013). Preparation and evaluation of thermal enhanced silica fume by incorporating organic PCM, for application to concrete. *Energy and Buildings*, 62, 190-195.
- Johra, H., & Heiselberg, P. (2017). Influence of internal thermal mass on the indoor thermal dynamics and integration of phase change materials in furniture for building energy storage: A review. *Renewable and Sustainable Energy Reviews*, 69, 19-32.
- Joulin, A., Zalewski, L., Lassue, S., & Naji, H. (2014). Experimental investigation of thermal characteristics of a mortar with or without a micro-encapsulated phase change material. *Applied Thermal Engineering*, 66(1-2), 171-180.
- Kalnæs, S. E., & Jelle, B. P. (2015). Phase change materials and products for building applications: A state-of-the-art review and future research opportunities. *Energy and Buildings*, 94, 150-176.
- Kani, E. N., & Allahverdi, A. (2009). Effects of curing time and temperature on strength development of inorganic polymeric binder based on natural pozzolan. *Journal of materials science*, 44(12), 3088-3097.
- Karaipekli, A., & Sarı, A. (2008). Capric–myristic acid/expanded perlite composite as form-stable phase change material for latent heat thermal energy storage. *Renewable Energy*, 33(12), 2599-2605.
- Kasaeian, A., Pourfayaz, F., Khodabandeh, E., & Yan, W. M. (2017). Experimental studies on the applications of PCMs and nano-PCMs in buildings: A critical review. *Energy and Buildings*, 154, 96-112.
- Kashchiev, D., & Van Rosmalen, G. M. (2003). Nucleation in solutions revisited. *Crystal Research and Technology: Journal of Experimental and Industrial Crystallography*, 38(7-8), 555-574.
- Khadiran, T., Hussein, M. Z., Zainal, Z., & Rusli, R. (2015). Encapsulation techniques for organic phase change materials as thermal energy storage medium: A review. *Solar Energy Materials and Solar Cells*, 143, 78-98.

- Khale, D., & Chaudhary, R. (2007). Mechanism of geopolymerization and factors influencing its development: a review. *Journal of materials science*, 42(3), 729-746.
- Kheradmand, M., Abdollahnejad, Z., & Pacheco-Torgal, F. (2018). Alkali-activated cement-based binder mortars containing phase change materials (PCMs): mechanical properties and cost analysis. *European Journal of Environmental and Civil Engineering*, 1-23.
- Kheradmand, M., Azenha, M., de Aguiar, J. L., & Krakowiak, K. J. (2014). Thermal behavior of cement based plastering mortar containing hybrid microencapsulated phase change materials. *Energy and buildings*, 84, 526-536.
- Khudhair, A. M., & Farid, M. M. (2004). A review on energy conservation in building applications with thermal storage by latent heat using phase change materials. *Energy conversion and management*, 45(2), 263-275.
- Khudhair, A. M., & Farid, M. M. (2004). A review on energy conservation in building applications with thermal storage by latent heat using phase change materials. *Energy conversion and management*, 45(2), 263-275.
- Kim, H. B., Mae, M., & Choi, Y. (2017). Application of shape-stabilized phase-change material sheets as thermal energy storage to reduce heating load in Japanese climate. *Building and Environment*, 125, 1-14.
- Kim, S., Chang, S. J., Chung, O., Jeong, S. G., & Kim, S. (2014). Thermal characteristics of mortar containing hexadecane/xGnP SSPCM and energy storage behaviors of envelopes integrated with enhanced heat storage composites for energy efficient buildings. *Energy and Buildings*, 70, 472-479.
- Kim, S., Paek, S., Jeong, S. G., Lee, J. H., & Kim, S. (2014). Thermal performance enhancement of mortar mixed with octadecane/xGnP SSPCM to save building energy consumption. *Solar Energy Materials and Solar Cells*, 122, 257-263.
- Klieger, P. (1958, June). Effect of mixing and curing temperature on concrete strength. In *Journal Proceedings*(Vol. 54, No. 6, pp. 1063-1081).
- Konuklu, Y., & Paksoy, H. Ö. (2009). Phase change material sandwich panels for managing solar gain in buildings. *Journal of Solar Energy Engineering*, 131(4), 041012.
- Koschenz, M., & Lehmann, B. (2004). Development of a thermally activated ceiling panel with PCM for application in lightweight and retrofitted buildings. *Energy and buildings*, 36(6), 567-578.

- Kovalchuk, G., Fernández-Jiménez, A., & Palomo, A. (2007). Alkali-activated fly ash: effect of thermal curing conditions on mechanical and microstructural development—Part II. *Fuel*, 86(3), 315-322.
- Kumar, S., Kumar, R., & Mehrotra, S. P. (2010). Influence of granulated blast furnace slag on the reaction, structure and properties of fly ash based geopolymer. *Journal of materials science*, 45(3), 607-615.
- Kuznik, F., & Virgone, J. (2009). Experimental investigation of wallboard containing phase change material: Data for validation of numerical modeling. *Energy and Buildings*, 41(5), 561-570.
- Kuznik, F., David, D., Johannes, K., & Roux, J. J. (2011). A review on phase change materials integrated in building walls. *Renewable and Sustainable Energy Reviews*, 15(1), 379-391.
- Lachheb, M., Younsi, Z., Naji, H., Karkri, M., & Nasrallah, S. B. (2017). Thermal behavior of a hybrid PCM/plaster: A numerical and experimental investigation. *Applied Thermal Engineering*, 111, 49-59.
- Lai, C. M., Chen, R. H., & Lin, C. Y. (2010). Heat transfer and thermal storage behaviour of gypsum boards incorporating micro-encapsulated PCM. *Energy and Buildings*, 42(8), 1259-1266.
- Lecompte, T., Le Bideau, P., Glouannec, P., Nortershauser, D., & Le Masson, S. (2015). Mechanical and thermo-physical behaviour of concretes and mortars containing phase change material. *Energy and buildings*, 94, 52-60.
- Lecompte, T., Le Bideau, P., Glouannec, P., Nortershauser, D., & Le Masson, S. (2015). Mechanical and thermo-physical behaviour of concretes and mortars containing phase change material. *Energy and buildings*, 94, 52-60.
- Lee, T., Hawes, D. W., Banu, D., & Feldman, D. (2000). Control aspects of latent heat storage and recovery in concrete. *Solar energy materials and solar cells*, 62(3), 217-237.
- Li, H., Liu, X., & Fang, G. (2010). Preparation and characteristics of n-nonadecane/cement composites as thermal energy storage materials in buildings. *Energy and Buildings*, 42(10), 1661-1665.
- Ling, T. C., & Poon, C. S. (2013). Use of phase change materials for thermal energy storage in concrete: an overview. *Construction and Building Materials*, 46, 55-62.

- Lu, S., Li, Y., Kong, X., Pang, B., Chen, Y., Zheng, S., & Sun, L. (2017). A Review of PCM Energy Storage Technology Used in Buildings for the Global Warming Solution. In *Energy Solutions to Combat Global Warming* (pp. 611-644). Springer, Cham.
- Lucas, S. S., Ferreira, V. M., & De Aguiar, J. B. (2013). Latent heat storage in PCM containing mortars—Study of microstructural modifications. *Energy and Buildings*, 66, 724-731.
- Luo, F. J., He, L., Pan, Z., Duan, W. H., Zhao, X. L., & Collins, F. (2013). Effect of very fine particles on workability and strength of concrete made with dune sand. *Construction and Building Materials*, 47, 131-137.
- Maltais, Y., & Marchand, J. (1997). Influence of curing temperature on cement hydration and mechanical strength development of fly ash mortars. *Cement and concrete research*, 27(7), 1009-1020.
- Marzouk, H. M., & Hussein, A. (1990). Properties of high-strength concrete at low temperatures. *ACI Materials Journal*, 87(2), 167-171.
- Memon, S. A. (2014). Phase change materials integrated in building walls: A state of the art review. *Renewable and sustainable energy reviews*, 31, 870-906.
- Memon, S. A., Cui, H. Z., Zhang, H., & Xing, F. (2015). Utilization of macro encapsulated phase change materials for the development of thermal energy storage and structural lightweight aggregate concrete. *Applied Energy*, 139, 43-55.
- Memon, S. A., Cui, H., Lo, T. Y., & Li, Q. (2015). Development of structural–functional integrated concrete with macro-encapsulated PCM for thermal energy storage. *Applied energy*, 150, 245-257.
- Meshgin, P., & Xi, Y. (2012). Effect of Phase-Change Materials on Properties of Concrete. *ACI Materials Journal*, 109(1).
- Mills, R., & Lobo, V. M. M. (1989). Self-diffusion in electrolyte solutions, vol. 36 of physical sciences data.
- Min, H. W., Kim, S., & Kim, H. S. (2017). Investigation on thermal and mechanical characteristics of concrete mixed with shape stabilized phase change material for mix design. *Construction and Building Materials*, 149, 749-762.
- Morel, F. M. (1983). *Principles of aquatic chemistry*. John Wiley and Sons, New York NY. 1983. 446.

- Navarro, L., de Gracia, A., Castell, A., Álvarez, S., & Cabeza, L. F. (2015). PCM incorporation in a concrete core slab as a thermal storage and supply system: proof of concept. *Energy and Buildings*, 103, 70-82.
- Nepomuceno, M. C., & Silva, P. D. (2014). Experimental evaluation of cement mortars with phase change material incorporated via lightweight expanded clay aggregate. *Construction and Building Materials*, 63, 89-96.
- Neville, A. M. (1995). *Properties of concrete* (Vol. 4). London: longman.
- Ng, C., Alengaram, U. J., Wong, L. S., Mo, K. H., Jumaat, M. Z., & Ramesh, S. (2018). A review on microstructural study and compressive strength of geopolymer mortar, paste and concrete. *Construction and Building Materials*, 186, 550-576.
- Nkwetta, D. N., & Haghghat, F. (2014). Thermal energy storage with phase change material—A state-of-the art review. *Sustainable Cities and Society*, 10, 87-100.
- Outlook, A. E. (2010). Energy information administration. Department of Energy, 92010(9), 1-15.
- Pagliolico, S. L., Sassi, G., Cascone, Y., & Bongiovanni, R. M. (2015). A reference device for evaluating the thermal behavior of installed multilayered wall containing a phase change material. *Energy Conversion and Management*, 106, 1409-1417.
- Palomo, A., Grutzeck, M. W., & Blanco, M. T. (1999). Alkali-activated fly ashes: a cement for the future. *Cement and concrete research*, 29(8), 1323-1329.
- Park, S. K., Kim, J. H. J., Nam, J. W., Phan, H. D., & Kim, J. K. (2009). Development of anti-fungal mortar and concrete using Zeolite and Zeocarbon microcapsules. *Cement and Concrete Composites*, 31(7), 447-453.
- Parrott, L. J. (1992). Water absorption in cover concrete. *Materials and structures*, 25(5), 284.
- Pasupathy, A., Athanasius, L., Velraj, R., & Seeniraj, R. V. (2008). Experimental investigation and numerical simulation analysis on the thermal performance of a building roof incorporating phase change material (PCM) for thermal management. *Applied Thermal Engineering*, 28(5-6), 556-565.
- Pavlík, Z., Trník, A., Keppert, M., Pavlíková, M., Žumár, J., & Černý, R. (2014). Experimental investigation of the properties of lime-based plaster-containing PCM for enhancing the heat-storage capacity of building envelopes. *International Journal of Thermophysics*, 35(3-4), 767-782.

- Pavlik, Z., Trník, A., Ondruška, J., Keppert, M., Pavlíková, M., Volfová, P., ... & Černý, R. (2013). Apparent thermal properties of phase-change materials: an analysis using differential scanning calorimetry and impulse method. *International Journal of Thermophysics*, 34(5), 851-864.
- Perera, D. S., Uchida, O., Vance, E. R., & Finnie, K. S. (2007). Influence of curing schedule on the integrity of geopolymers. *Journal of materials science*, 42(9), 3099-3106.
- Pilehvar, S., Cao, V. D., Szczotok, A. M., Carmona, M., Valentini, L., Lanzón, M., ... & Kjøniksen, A. L. (2018). Physical and mechanical properties of fly ash and slag geopolymer concrete containing different types of micro-encapsulated phase change materials. *Construction and Building Materials*, 173, 28-39.
- Pilehvar, S., Cao, V. D., Szczotok, A. M., Valentini, L., Salvioni, D., Magistri, M., ... & Kjøniksen, A. L. (2017). Mechanical properties and microscale changes of geopolymer concrete and Portland cement concrete containing micro-encapsulated phase change materials. *Cement and Concrete Research*, 100, 341-349.
- Pilehvar, S., Szczotok, A. M., Rodríguez, J. F., Valentini, L., Lanzón, M., Pamies, R., & Kjøniksen, A. L. (2019). Effect of freeze-thaw cycles on the mechanical behavior of geopolymer concrete and Portland cement concrete containing micro-encapsulated phase change materials. *Construction and Building Materials*, 200, 94-103.
- Pomianowski, M., Heiselberg, P., & Zhang, Y. (2013). Review of thermal energy storage technologies based on PCM application in buildings. *Energy and Buildings*, 67, 56-69.
- Pomianowski, M., Heiselberg, P., Jensen, R. L., Cheng, R., & Zhang, Y. (2014). A new experimental method to determine specific heat capacity of inhomogeneous concrete material with incorporated microencapsulated-PCM. *Cement and Concrete Research*, 55, 22-34.
- Provost-Smith, D. J., Elsayed, M., & Nehdi, M. L. (2017). Effect of early-age subfreezing temperature on grouted dowel precast concrete wall connections. *Construction and Building Materials*, 140, 385-394.
- Rangan, B. V. (2008). Fly ash-based geopolymer concrete.
- Rangan, B. V. (2008). Low-calcium, fly-ash-based geopolymer concrete. In *Concrete construction engineering handbook* (pp. 26-1). CRC press.

- Rao, V. V., Parameshwaran, R., & Ram, V. V. (2018). PCM-mortar based construction materials for energy efficient buildings: A review on research trends. *Energy and Buildings*, 158, 95-122.
- Rehan, R., & Nehdi, M. (2005). Carbon dioxide emissions and climate change: policy implications for the cement industry. *Environmental Science & Policy*, 8(2), 105-114.
- Ren, X., Zhang, L., Ramey, D., Waterman, B., & Ormsby, S. (2015). Utilization of aluminum sludge (AS) to enhance mine tailings-based geopolymer. *Journal of materials science*, 50(3), 1370-1381.
- Richardson, A., Heniegal, A., & Tindall, J. (2017). Optimal performance characteristics of mortar incorporating phase change materials and silica fume. *Journal of Green Building*, 12(2), 59-78.
- Richardson, I. G. (2004). Tobermorite/jennite-and tobermorite/calcium hydroxide-based models for the structure of CSH: applicability to hardened pastes of tricalcium silicate, β -dicalcium silicate, Portland cement, and blends of Portland cement with blast-furnace slag, metakaolin, or silica fume. *Cement and Concrete Research*, 34(9), 1733-1777.
- Ricklefs, A., Thiele, A. M., Falzone, G., Sant, G., & Pilon, L. (2017). Thermal conductivity of cementitious composites containing microencapsulated phase change materials. *International journal of heat and mass transfer*, 104, 71-82.
- Riffat, S., Mempo, B., & Fang, W. (2015). Phase change material developments: a review. *International Journal of Ambient Energy*, 36(3), 102-115.
- Roskos, C., Cross, D., Berry, M., & Stephens, J. (2011, May). Identification and verification of self-cementing fly ash binders for 'Green' concrete. In proceedings of the 2011 world of coal ash (WOCA) conference—May 9–12, 2011 in Denver CO, USA.
- Rovnaník, P. (2010). Effect of curing temperature on the development of hard structure of metakaolin-based geopolymer. *Construction and building materials*, 24(7), 1176-1183.
- Royon, L., Karim, L., & Bontemps, A. (2014). Optimization of PCM embedded in a floor panel developed for thermal management of the lightweight envelope of buildings. *Energy and Buildings*, 82, 385-390.
- Ruud, M. D., Mitchell, J. W., & Klein, S. A. (1990). Use of building thermal mass to offset cooling loads. *ASHRAE Transactions (American Society of Heating, Refrigerating and Air-Conditioning Engineers);(United States)*, 96(CONF-9006117--).

- Ryu, G. S., Lee, Y. B., Koh, K. T., & Chung, Y. S. (2013). The mechanical properties of fly ash-based geopolymer concrete with alkaline activators. *Construction and Building Materials*, 47, 409-418.
- Sakulich, A. R., & Bentz, D. P. (2011). Increasing the service life of bridge decks by incorporating phase-change materials to reduce freeze-thaw cycles. *Journal of Materials in Civil Engineering*, 24(8), 1034-1042.
- Sakulich, A. R., & Bentz, D. P. (2012). Incorporation of phase change materials in cementitious systems via fine lightweight aggregate. *Construction and Building Materials*, 35, 483-490.
- Sari, A. (2014). Composites of polyethylene glycol (PEG600) with gypsum and natural clay as new kinds of building PCMs for low temperature-thermal energy storage. *Energy and Buildings*, 69, 184-192.
- Sari, A., Karaipekli, A., & Kaygusuz, K. (2008). Capric acid and stearic acid mixture impregnated with gypsum wallboard for low-temperature latent heat thermal energy storage. *International Journal of Energy Research*, 32(2), 154-160.
- Scalat, S., Banu, D., Hawes, D., Parish, J., Haghghata, F., & Feldman, D. (1996). Full scale thermal testing of latent heat storage in wallboard. *Solar Energy Materials and Solar Cells*, 44(1), 49-61.
- Schossig, P., Henning, H. M., Gschwander, S., & Hausmann, T. (2005). Micro-encapsulated phase-change materials integrated into construction materials. *Solar Energy Materials and Solar Cells*, 89(2-3), 297-306.
- Schossig, P., Henning, H. M., Gschwander, S., & Hausmann, T. (2005). Micro-encapsulated phase-change materials integrated into construction materials. *Solar Energy Materials and Solar Cells*, 89(2-3), 297-306.
- Scrivener, K. L. (1984). The development of microstructure during the hydration of Portland cement.
- Senff*, L., Hotza, D., Repette, W. L., Ferreira, V. M., & Labrincha, J. A. (2010). Rheological characterisation of cement pastes with nanosilica, silica fume and superplasticiser additions. *Advances in Applied Ceramics*, 109(4), 213-218.
- Shadnia, R., & Zhang, L. (2016). EXPERIMENTAL STUDY OF GEOPOLYMER CONCRETE WITH INCORPORATED PCM CURED AT AMBIENT TEMPERATURE. GREEN

GEOPOLYMER WITH INCORPORATED PCM FOR ENERGY SAVING IN BUILDINGS, 73.

- Shadnia, R., Zhang, L., & Li, P. (2015). Experimental study of geopolymer mortar with incorporated PCM. *Construction and building materials*, 84, 95-102.
- Shadnia, R., Zhang, L., & Li, P. (2015). Experimental study of geopolymer mortar with incorporated PCM. *Construction and building materials*, 84, 95-102.
- Sharifi, N. P., & Mahboub, K. C. (2018). Application of a PCM-rich concrete overlay to control thermal induced curling stresses in concrete pavements. *Construction and Building Materials*, 183, 502-512.
- Sharma, A., Tyagi, V. V., Chen, C. R., & Buddhi, D. (2009). Review on thermal energy storage with phase change materials and applications. *Renewable and Sustainable energy reviews*, 13(2), 318-345.
- Shi, X., Memon, S. A., Tang, W., Cui, H., & Xing, F. (2014). Experimental assessment of position of macro encapsulated phase change material in concrete walls on indoor temperatures and humidity levels. *Energy and Buildings*, 71, 80-87.
- Shilei, L. V., Guohui, F., Neng, Z., & Li, D. (2007). Experimental study and evaluation of latent heat storage in phase change materials wallboards. *Energy and buildings*, 39(10), 1088-1091.
- Sieminski, A. (2014). *International energy outlook*. Energy Information Administration (EIA), 18.
- Silva, T., Vicente, R., & Rodrigues, F. (2016). Literature review on the use of phase change materials in glazing and shading solutions. *Renewable and Sustainable Energy Reviews*, 53, 515-535.
- Snoeck, D., Priem, B., Dubruel, P., & De Belie, N. (2016). Encapsulated Phase-Change Materials as additives in cementitious materials to promote thermal comfort in concrete constructions. *Materials and Structures*, 49(1-2), 225-239.
- Soares, N., Costa, J. J., Gaspar, A. R., & Santos, P. (2013). Review of passive PCM latent heat thermal energy storage systems towards buildings' energy efficiency. *Energy and buildings*, 59, 82-103.
- Somorjai, G. A., & Li, Y. (2010). *Introduction to surface chemistry and catalysis*. John Wiley & Sons. Adamson, A. W., & Gast, A. P.

- Song, M., Niu, F., Mao, N., Hu, Y., & Deng, S. (2018). Review on building energy performance improvement using phase change materials. *Energy and Buildings*, 158, 776-793.
- Tam, C. M., Tam, V. W., & Ng, K. M. (2012). Assessing drying shrinkage and water permeability of reactive powder concrete produced in Hong Kong. *Construction and Building Materials*, 26(1), 79-89.
- Telkes, M. (1978, March). Trombe wall with phase change storage material. In *Proceedings of the 2nd National Passive Solar Conference*, Philadelphia, PA, USA.
- Thokchom, S., Mandal, K. K., & Ghosh, S. (2012). Effect of Si/Al ratio on performance of fly ash geopolymers at elevated temperature. *Arabian Journal for Science and Engineering*, 37(4), 977-989.
- Toppi, T., & Mazzarella, L. (2013). Gypsum based composite materials with micro-encapsulated PCM: Experimental correlations for thermal properties estimation on the basis of the composition. *Energy and Buildings*, 57, 227-236.
- Tuncel, E. Y., & Pekmezci, B. Y. (2018). A sustainable cold bonded lightweight PCM aggregate production: Its effects on concrete properties. *Construction and Building Materials*, 181, 199-216.
- Tyagi, V. V., Kaushik, S. C., Tyagi, S. K., & Akiyama, T. (2011). Development of phase change materials based microencapsulated technology for buildings: a review. *Renewable and sustainable energy reviews*, 15(2), 1373-1391.
- Tyagi, V. V., Kaushik, S. C., Tyagi, S. K., & Akiyama, T. (2011). Development of phase change materials based microencapsulated technology for buildings: a review. *Renewable and sustainable energy reviews*, 15(2), 1373-1391.
- Vicente, R., & Silva, T. (2014). Brick masonry walls with PCM macrocapsules: an experimental approach. *Applied Thermal Engineering*, 67(1-2), 24-34.
- Vieira, J., Senff, L., Gonçalves, H., Silva, L., Ferreira, V. M., & Labrincha, J. A. (2014). Functionalization of mortars for controlling the indoor ambient of buildings. *Energy and Buildings*, 70, 224-236.
- Voelker, C., Kornadt, O., & Ostry, M. (2008). Temperature reduction due to the application of phase change materials. *Energy and Buildings*, 40(5), 937-944.
- Wang, S. D., Scrivener, K. L., & Pratt, P. L. (1994). Factors affecting the strength of alkali-activated slag. *Cement and Concrete Research*, 24(6), 1033-1043.

- Wei, Z., Falzone, G., Das, S., Saklani, N., Le Pape, Y., Pilon, L., ... & Sant, G. (2017). Restrained shrinkage cracking of cementitious composites containing soft PCM inclusions: A paste (matrix) controlled response. *Materials & Design*, 132, 367-374.
- Wei, Z., Falzone, G., Wang, B., Thiele, A., Puerta-Falla, G., Pilon, L., ... & Sant, G. (2017). The durability of cementitious composites containing microencapsulated phase change materials. *Cement and Concrete Composites*, 81, 66-76.
- Weinläder, H., Beck, A., & Fricke, J. (2005). PCM-facade-panel for daylighting and room heating. *Solar Energy*, 78(2), 177-186.
- Whiffen, T. R., & Riffat, S. B. (2012). A review of PCM technology for thermal energy storage in the built environment: Part II. *International Journal of Low-Carbon Technologies*, 8(3), 159-164.
- Xiao, M., Feng, B., & Gong, K. (2002). Preparation and performance of shape stabilized phase change thermal storage materials with high thermal conductivity. *Energy conversion and management*, 43(1), 103-108.
- Yang, H. B., Liu, T. C., Chern, J. C., & Lee, M. H. (2016). Mechanical properties of concrete containing phase-change material. *Journal of the Chinese institute of engineers*, 39(5), 521-530.
- Yang, H. S., & Che, Y. J. (2015). Influence of particle size distribution of fine and micro-aggregate on the microstructure of cement mortar and paste. *Materials Research Innovations*, 19(sup1), S1-130.
- Yang, Q., Yang, Q., & Zhu, P. (2003). Scaling and corrosion resistance of steam-cured concrete. *Cement and concrete research*, 33(7), 1057-1061.
- Young, B. A., Wei, Z., Rubalcava-Cruz, J., Falzone, G., Kumar, A., Neithalath, N., ... & Pilon, L. (2017). A general method for retrieving thermal deformation properties of microencapsulated phase change materials or other particulate inclusions in cementitious composites. *Materials & Design*, 126, 259-267.
- Zalba, B., Marin, J. M., Cabeza, L. F., & Mehling, H. (2003). Review on thermal energy storage with phase change: materials, heat transfer analysis and applications. *Applied thermal engineering*, 23(3), 251-283.

- Zhai, X. Q., Wang, X. L., Wang, T., & Wang, R. Z. (2013). A review on phase change cold storage in air-conditioning system: Materials and applications. *Renewable and Sustainable Energy Reviews*, 22, 108-120.
- Zhang, H. Y., Kodur, V., Wu, B., Cao, L., & Wang, F. (2016). Thermal behavior and mechanical properties of geopolymer mortar after exposure to elevated temperatures. *Construction and Building Materials*, 109, 17-24.
- Zhang, L., Ahmari, S., & Zhang, J. (2011). Synthesis and characterization of fly ash modified mine tailings-based geopolymers. *Construction and Building Materials*, 25(9), 3773-3781.
- Zhang, X. X., Fan, Y. F., Tao, X. M., & Yick, K. L. (2005). Crystallization and prevention of supercooling of microencapsulated n-alkanes. *Journal of colloid and interface science*, 281(2), 299-306.
- Zhang, X. X., Tao, X. M., Yick, K. L., & Wang, X. C. (2004). Structure and thermal stability of microencapsulated phase-change materials. *Colloid and Polymer Science*, 282(4), 330-336.
- Zhimin, H., Junzhe, L., & Kangwu, Z. (2012). Influence of mineral admixtures on the short and long-term performance of steam-cured concrete. *Energy procedia*, 16, 836-841.
- Zhou, D., Zhao, C. Y., & Tian, Y. (2012). Review on thermal energy storage with phase change materials (PCMs) in building applications. *Applied energy*, 92, 593-605.
- Zhou, G., Zhang, Y., Wang, X., Lin, K., & Xiao, W. (2007). An assessment of mixed type PCM-gypsum and shape-stabilized PCM plates in a building for passive solar heating. *Solar energy*, 81(11), 1351-1360.