INCORPORATION OF PHASE CHANGE MATERIAL IN LIGHTWEIGHT CONCRETE FOR PASSIVE BUILDING APPLICATIONS



FINAL YEAR PROJECT UG-2014

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2018

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DEDICATION

We dedicate this research to our advisor Dr. Rao Arsalan Khushnood

And

To our parents who helped and supported us.

ACKNOWLEDGEMENTS

First of all we are Thankful to ALLAH Almighty who gave us strength and courage to accomplish this project. This project would not have been completed without the efforts of many individuals and instructors. We are really thankful to all of those who contributed in making our project a success.

We would like to express our immense gratitude to our supervisor, Dr. Rao Arsalan Khushnood for his support and guidance that helped us achieving our aims and goals of the project.

We would also like to thank Sherafgan who was a great help throughout the project and to all faculty members for their sincere advice and cooperation.

ABSTRACT

Materials possessing structural as well as thermal properties are one of directions of the sustainable constructions. Phase change materials (PCMs) are the latent thermal storage materials that has the capacity to store large amount of thermal energy during its phase change from one state to another. In the past, the use of PCM in buildings was either in the form of non-structural panels or by incorporation in structural panels in its encapsulated form. The encapsulation is an expensive and a time consuming process so our research was mainly focused on the elimination of the macro-encapsulation layer from PCM impregnated aggregate and to make the non-encapsulated lightweight aggregate concrete structurally useable, thermally efficient and reliable. The PCM used for this research was paraffin wax that has melting point 23.46 °C and latent heat storage capacity of 192.98J/g. PCM was intruded in LWA through vacuum impregnation that increased the absorption from 11% to 23%. Encapsulation layer was made with epoxy (1:1 of resin and hardener) mixed with graphite (10% by weight of epoxy). The compressive strength and elastic modulus, microstructure, thermal properties, thermal stability, chemical compatibility and thermal performance of two concretes i.e. Non-encapsulated Light weight aggregate concrete (PCM-LWAC) and encapsulated Light weight aggregate concrete (ME-LWAC) were investigated by compressive strength machine (CSM), Scanning electron microscopy (SEM), Differential scanning calorimetry (DSC), Thermogravimetric analyzer (TGA), Fourier transformation infrared spectrum analysis (FT-IR) and Thermal performance test (indoor and outdoor).

From the in depth investigation of test results it can be concluded that the compressive strength of PCM-LWAC and ME-LWAC at 28 days was 22.41MPa and 21.51MPa respectively which is greater than the structural strength limit i.e. 17MPa, showing that both the types of concretes can be used in structural members. From FT-IR analysis the chemical compatibility of PCM with epoxy and LWA was ensured. TGA results showed that PCM-LWAC and ME-LWAC were thermally stable up to 150°C. DSC curves showed that the melting point, freezing point and latent heat storage of PCM-LWA and ME-LWAA are 26.13°C, 30.15°C and 20.14J/g and 22.78°C, 29.19°C and 12.5J/g respectively, concluding that both the developed materials are within the human thermal comfort zone (20°C to 32°C). Thermal Performance Test outdoor showed the temperature difference between LWAC and PCM-LWAC is 3.5°C, while for LWAC and ME-LWAC is 4.5°C. Thermal performance indoor results verified the results of outdoor. Since there are no marginal differences between thermal and mechanical properties of ME-LWAC and PCM-LWAC, hence the encapsulation layer can be removed.

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List of Abbreviations

PCM	Phase Change Material
PCM-LWA	Non-encapsulated PCM intruded lightweight aggregate
ME-LWA	Macro-encapsulated Lightweight Aggregate
LWA	Light Weight Aggregate
LWAC	Light Weight Aggregate Concrete
ASTM	American Standard for Testing of Materials
ACI	American Concrete Institute
DSC	Differential Scanning Calorimetery
TGA	Thermo-gravimetric Analysis
SEM	Scanning Electron Microscopy
EDX	Electron Dispersive X-Ray spectroscopy
FTIR	Fourier Transform Infrared Spectroscopy

Chapter 1

INTRODUCTION

1.1 Introduction

Energy demand has been increased due to rapid economic growth which leads to high strain on fossil fuels and renewable energy resources which, upon usage, results in emission of harmful gasses. In order to reduce the demand of purchased energy, energy resources needs to be used efficiently and new techniques of energy storage should be introduced [1]. As a result, researchers are paying more attention towards the introduction and innovation of green building materials having energy saving capabilities for the construction of building industry [2]. Studies showed that Building sector utilize 40% share of the total energy consumption[3][4][5]. For construction of building sector, human being use concrete in such a tremendous quantity that its annual production has been increased to 25 billion metric tons[6]. Massive concrete, i.e. to store thermal energy during day and release it slowly during night can be effectively utilized, therefore reduces the need for artificial heating and cooling [7][8].

To deal with this problem and to save a portion of energy in buildings, enhancing the energy storage capacity of concrete is required through some modification in it, for example incorporation of phase change material (PCM) into concrete mixes [1],[9],[10]. Thermal energy storage materials are of two types 1. Sensible heat storage 2. Latent heat storage materials.

"Phase change are the latent heat storage materials that can store large amount of thermal energy in its phase change from solid to liquid and vice versa".

PCM is classified into three main types. 1. Organic (mostly paraffin) 2. Inorganic 3. Eutectic. Reasonable investigation has been carried out on both the organic and inorganic PCM. Inorganic PCM has promising nature of high latent heat of storage, reasonable thermal conductivity, non-flammable, and economical[1]. However, their usage get restricted due to some of its undesirable characteristics for example corrosive nature, phase decomposition, and super cooling[9]. In contrast, generally organic PCM are chemically stable, free from phase decomposition & super cooling, non-corrosive, non-toxic and have a high latent heat of melting and freezing. Therefore, organic PCM was chosen for the incorporation into concrete for thermal energy storage purposes.

1.2 Problem Statement

PCM with encapsulation has the capability to make thermally efficient concrete but has reduced compressive strength. The development of encapsulation layer needs special expertise and a great care from environmental destruction. Hence, this research aimed at the elimination of the encapsulation layer to reduce the cost and time along with achieving thermal efficiency and reliability and enhanced mechanical properties.

1.3 Objectives

This research emphasis to compare structural AND thermal properties of concretes prepared by using:

- Macro encapsulated PCM intruded aggregates.
- Non-encapsulated PCM intruded aggregates.

1.4 Research Significance

Researchers successfully attempted to develop thermal energy storage lightweight aggregate concrete with sufficient thermal properties, utilizing lightweight aggregate (LWA) as a supporting materials for PCM [2], [11], [7]–. However their research lack in-depth study of making thermally efficient, thermally reliable and mechanically sound PCM Lightweight concrete without encapsulation layer.

1.5 Research Methodology

To develop PCM lightweight aggregate concrete without encapsulation layer and PCM lightweight aggregate concrete with encapsulation layer, having appreciable range of structural and thermal properties the following step were followed.

- For this study, calculated amount of commercial grade organic *Phase Change Materials* (*PCM*) was imported from Shanghai, China.
- The PCM selected was a paraffin wax having a transition temperature 25.5 °C that best suited to the temperature in Pakistan.
- PCM was first melted in oven and then impregnated into pores of Light Weight Aggregate (LWA) through the set up named as *Vacuum Impregnation Chamber* at vacuum pressure of 0.1MPa developed by the pump.
- Non encapsulated LWA was prepared by intrusion of PCM into LWA and then allowing the extra PCM to drain out from pores in oven at 50°C for almost 12 hours.
- To prepare encapsulation layer for encapsulated LWA, epoxy and graphite were mixed together.

- Impregnated PCM LWA without encapsulation and with encapsulation were used to prepare concrete samples.
- Compressive strength, split tensile and elastic modulus tests were carried out on the samples.
- To assess thermal properties Differential Scanning Calorimetry (DSC) was carried out that provided precise information about the melting point, boiling point and latent energy storage per gram of sample.
- Thermal stability of PCM and PCM intruded aggregates were checked using thermogravimetric analysis (TGA).
- XRF and Fourier Transform Infrared Spectroscopy (FT-IR) was performed to study chemical behavior of the material that includes functional group analysis.
- Thermal performance indoor test was performed on LWAC, PCM-LWAC and ME-LWAC slabs to check the difference of inside and outside temperature.
- Thermal performance outdoor test was performed on LWAC, PCM-LWAC and ME-LWAC slabs to check the difference of inside and outside temperature in natural transition of temperatures and the shifting away of load from peak energy hours.
- The results were compared to past available literature to show improvement and accuracy in results.

1.6 Research Scope

Scope of the research was to achieve thermal and structural properties by using Paraffin as PCM, having melting point 25.52 °C, freezing point 31.99 and latent heat storage capacity 192.98 J/g, into concrete with the elimination of the macro-encapsulation layer around it to have reduced cost, time and increased strength.

1.7 Thesis Organization

This thesis document is organized step by step from Introduction to PCM and methodology of research in chapter 1, literature review in chapter 2, materials used and development methods of Macro-encapsulated and Non-encapsulated PCM intruded concrete in chapter 3 and in chapter 4 results of tests and conclusion are discussed.

Chapter 2

LITERATURE REVIEW

2.1 Types of Phase Change Material

There are three categories of PCM that has different characteristics i.e. different physical and chemical properties. Figure 1 shows the various types of PCM:



2.2 Principle of PCM

PCMs are the materials that store energy by latent heat storage principal[12]. Materials working on Latent heat storage principle stores energy when they change their phase from solid to liquid and releases the stored energy when transition of phase reverses.



Figure 2 working principle of PCM

2.3 PCM in Literature

The idea behind using PCM is not unusual, however it is getting into demand as the energy demand is increasing in every aspect of life. Buildings being the highest energy consumer needs modification in architectural as well as construction perspective. Concrete, as the most abundant materials used by human being, needs modification accordingly to be thermally efficient e.g. thermocrete- a PCM incorporated concrete that has satisfactory structural and thermal properties [7], [9]. Some unique features that make concrete as the best choice to be used as carrier or supporting material for PCM are (a) Its application in tremendous quantity (b) Its flexibility to be molded into variety of shapes and sizes (c) Production of desired quality and quantity (d) Ease with which test procedures can be performed^[7]. Reduction in energy consumption to improve thermal performance of the buildings through thermal energy storage scenarios will not only reduce the dependency on purchased energy such as fossil fuels but would also reduce the risk of total greenhouse gas emission [13]. Storage of thermal energy through incorporation of PCM is a simple and effective option for application to building envelops, to enhance thermal performance for better working efficiency of buildings [14]. Apart from its large latent heat storage, PCM also has a large number of favorable features like (a) At curing stage, it can reduce thermal cracking in mass concreting such as dams [7]; (b) It can also reduce damage through freeze/thaw cycles which may have a very beneficial impact on service life of structures such as decks of bridges [10]; (c) Reduce the overall shrinkage strain in matrix, thus making it more durable.

2.3 Application of PCM into Cementitious System

Until now three different types of procedures are being followed to incorporate PCMs into concrete. Researchers reported both merits and demerits for incorporated PCM into cementitious matrices. Three types of commonly practiced mechanism for incorporation of PCM into concrete are: (a) Through direct incorporation and simple immersion [2], [10] (b) Form stable composites PCM [15], [16][17] (c) Micro/macro-encapsulation. In case of Micro-encapsulation, it has been experimentally investigated that using different percentages of PCM showed significant strength degradation and high investment render it feasibility at commercial level. Hunger et al.[18] used (1%, 3%, and 5%) in his formulation and the resulting cement mortar was found to be thermally efficient but mechanical strength reduced up-to 69% in case of 5% usage. He claimed that the huge loss in compressive strength was due to the significant difference between the strength of micro capsules and the surrounding cement matrix. Moreover, the reduction was attributed to the damage of the PCM micro capsules during mixing stage leads to mixing of PCM with surrounding matrix and interrupt the hydration kinetics. Due to these scenarios macro-encapsulation as a novel form is the best option, and is preferably used.

2.4 Macro encapsulation of phase change material

Any of the container that can be used as a supporting material for liquid paraffin may be termed as carrier. The best carrier than can be used in concrete is LWA[19]. To safely develop thermal energy storage concrete, encapsulation at micro and macro level is the best option. Micro encapsulation, as mentioned earlier leads to significant reduction in mechanical properties [10]. Therefore, novel form of macro encapsulation is the preferred option left. In this study LWA aggregate was used as a carrier for PCM. Encapsulation was accomplished with the Macro encapsulation layer paste. The developed ME-LWAC was tested for determining, compressive strength, thermal properties, thermal stability, and micro structure was observed to check various properties of the developed concrete.

Chapter 3

EXPERIMENTAL INVESTIGATION

3.1 Procurement of Materials

3.1.1 Paraffin

Paraffin named as 12-Hydroxy stearic acid, was procured from China. It had a melting point 25.52°C, freezing point 31.99°C and latent heat storage capacity of 192.98 J/g.

3.1.2 Light Weight Aggregate

The light weight aggregate (LWA) used was synthetically manufactured by Pakistan Council of Scientific and Industrial Research (PCSIR), Peshawar. The LWA was made of expanded slate and the bulk density given by the manufacturer was 800 kg/m³ and the 24-hour absorption capacity was found out to be 13%.

3.1.3 Sand

The fine aggregate used was procured from Lawrencepur conforming to ASTM standard C778-17 of Fineness Modulus (FM) 3.46, falling into moderately coarse category of sand. Specific gravity of sand was found to be 3.42.

3.1.4 Cement

'Bestway' Ordinary Portland Cement (OPC) of grade 43, type I, confronting to ASTM standard C150-04, having mean particle size 15.41 µm, was used.

3.1.5 Graphite

Technical grade graphite was used to enhance the thermal conductivity of the macro encapsulation layer.

3.1.6 Superplasticizer

Sicament 512PK super plasticizer from the manufacturer SICA was used in a proportion of 0.4% by weight of cement to achieve sufficient workability.

3.2 Methodology

3.2.1 Development of encapsulation layer paste

The encapsulation layer paste was prepared by mixing epoxy (resin and hardener) and graphite powder. The mixing ratio of adhesive to hardener was kept 1:1 as recommended by the manufacturer. The graphite powder, having a high thermal conductivity, was used to improve the thermal conductivity of the encapsulation layer for better thermal performance of the macro-encapsulated light-weight aggregate and hence an increased overall efficiency of the developed thermal energy storage light-weight aggregate concrete. The graphite used was 10% by weight of epoxy which was found to be optimum from past literature review. This encapsulation paste gives approximately 1-1.5 hrs. open time for encapsulation before setting.

3.2.2 Development of macro-encapsulated light-weight aggregate (ME-LWA)

For the preparation of ME-LWA, the first step is the absorption of liquid paraffin into the lightweight aggregate. By simple immersion of the LWA into the water for 1 hour, it absorbed 11% of water by weight as the pores of the LWA were blocked by air. And since the viscosity of liquid paraffin is higher than that of water, its intrusion into the pores by simple immersion would be even lower. Therefore, to intrude a higher quantity of liquid, the removal of air from the porous carrier is required. The effective and enhanced absorption of PCM into porous LWA was made possible through vacuum impregnation setup, and hence was assembled at NUST Institute of Civil Engineering (Structural Engineering Lab). Results showed that absorption capacity of LWA increased to 29.5% through vacuum impregnation. The detailed and labeled schematic sketch of vacuum impregnation setup is shown in Figure 3.



Figure 3 Vacuum impregnation setup

At first, washed and oven dried porous LWA was kept inside vacuum chamber for 30 min to remove air from pores of the LWA. The melted paraffin was then introduced into the chamber from above, and a pressure of -0.1 MPa was maintained for another 60 minutes to ensure complete absorption into the pores. During this whole process, vacuum chamber was kept in water bath maintaining temperature of 60°C-65°C to keep the paraffin in melted form. At the end, LWA impregnated with paraffin was taken out and kept in refrigerator below 4°C to ensure that paraffin get solidified within pores of LWA. The PCM absorption capacity of porous LWA was found to be 23 wt. % by the following formula.

Percentage absorption capacity of LWA for paraffin = (WLWA-Paraffin - WLWA) * 100 / **WLWA**

Where WLWA-Paraffin is the weight of impregnated LWA and WLWA is the weight of LWA in oven dried condition.

The impregnated LWA was then encapsulated using the encapsulation paste and cement was applied to the epoxy coated LWA in order to prevent sticking of the individual particles as shown in the figure.



Figure 4 preparation of ME-LWA

3.2.3 Development of non-encapsulated PCM-LWA

The process is similar to the development of ME-PCM up to the point of vacuum impregnation but after the PCM is intruded into the LWA, instead of cooling the aggregate in a refrigerator, it was placed on a paper and heated in an oven at a temperature of about 50°C and its weight was measured every half an hour unless there was no change in its weight, meaning all the loose PCM has drained out and the PCM that's left inside is retained in its pore and will not seep out upon melting. Using the same absorption capacity relation as in the case of ME-PCM, the aggregate was found out to be retaining up to 19% PCM by weight without any encapsulation.



Aggregate placed at 50°C



Checked for weight loss after every 30 minutes





PCM-LWA ready when no weight loss

3.2.4 Development of thermal energy storage light weight aggregate concrete (LWAC)

Five different mixes were designed for investigation:

- Mix 1: Control LWAC
- Mix 2: 0.5PCM-LWAC
 - o 50% LWA was replaced with PCM-LWA
- Mix 3: 1.0PCM-LWAC
 - 100% LWA was replaced with PCM-LWA
- Mix 4: 0.5ME-LWAC
 - o 50% LWA was replaced with ME-LWA
- Mix 5: 1.0ME-LWAC
 - o 100% LWA was replaced with PCM-LWA

All the mixes were casted with a constant water to cement ratio (w/c) of 0.45 and LWA used were pre-washed and oven dried for 24-hr before casting. The table shows the material required for each mix.

C No	Nomenalations	Cement	Water	Sand	LWA	PCM-LWA
5. NO	Nomenciature	(Kg/m ³)				
1	LWAC	445.1	200.25	445.1	668	0
2	0.5PCM-LWAC	445.1	200.25	445.1	334	387
3	1.0PCM-LWAC	445.1	200.25	445.1	0	761.56
4	0.5ME-LWAC	445.1	200.25	445.1	334	467.6
5	1.0ME-LWAC	445.1	200.25	445.1	0	935.2

Table 1 Materials required in Kg/m3 of concrete volume.

3.3 TESTING

3.3.1 Micro-Structure Analysis

The microscopic morphology of LWA, LWAC, 1.0PCM-LWAC and 1.0ME-PCM LWAC was examined with third generation TESCAN VEGA3 scanning electron microscope. The specimen was .first cut (10 mm x10 mm x4 mm) by using rotary saw cutter then surfaces were leveled by



Figure 6 samples of SEM

#400 and polished by #2000 fineness paper. The prepared specimen was then washed by Ethanol and then dried up in oven for 1 day to remove all the impurities from pores. Finally, the gold sputtering process was performed for all the specimens.

3.3.2 Mechanical tests

To find out the mechanical properties of the all the types of concretes following tests were performed:

3.3.2.1 Compression Test

To determine the mechanical properties such as compressive strength of LWAC, compression test was performed. For the compression test, cubes of 100 mm3 were casted and tested at 7 and 28 days in accordance with ASTM C39. The loading rate on which tests were performed was set at 0.6 MPa/s. The model of compression machine used was MCC-08, having minimum and maximum test load 0.1 kN and 5000 kN respectively.



Figure 8 Cube being tested for Compression in Compression

Testing Machine



Figure 7 sample after testing

3.3.2.2 Elastic Modulus Test

To determine the elasticity of LWAC elastic modulus test was performed. For the test of young's modulus, cylinders of 100mmx200mm were casted and were tested at 28 days in accordance to ASTM C128. Loading rate was set on 0.25 MPa/s in compression machine and dial gauge was



Figure 9 cylinder with assembly in compression machine

attached with the assembly to check the deflection of cylinders. The model of compression machine used was MCC-08, having minimum and maximum test load 0.1 kN and 5000 kN respectively. Stress v/s strain graph was plotted and Tangent Elastic Modulus was calculated from the graph.

3.3.2.3 Split Tensile Strength Test

Cylinders of size 100mm x 200mm were casted for Mix 1, Mix 3, and Mix 5; cured for a period of 28 days and tested in UTM for their split tensile strength using the split tensile assembly. The test was performed according to ASTM standard C496. The failure load was determined for the cylinder and strength calculated with the following formula:

$$T = \frac{2P}{\Pi DL}$$

Where P = Applied load



Figure 10 cylinder after split test

D = Diameter of the cylinder

L = Length of cylinder

3.3.3 Thermal Analysis

Thermal properties including melting point, freezing point, thermal conductivity, thermal stability and latent heat storage capacity were determined using following tests.

3.3.3.1 Differential Scanning Calorimeter (DSC)

Melting point, freezing point and latent heat storage capacity were determined by using TA instrument, Model No. DSC6000.

The test procedure is as follows:

- Set TA at 0.0°C.
- Regulate isothermal condition for 5 min.
- For endothermic phase (melting) an increasing ramp of 2.00 °C/min from 0°C to 60.00 °C.
- At 60.00°C regulate isothermal condition for 5.00 min.
- For exothermic phase (freezing) a decreasing ramp of 2°C/m
- Figure 8 Typical DSC 6000in from 60.00°C back to 0.00°C.

Analysis of the data obtained from above steps was done on TA software to find out the physical properties (melting point, freezing point and latent heat storage capacity) of PCM. The maximum deviation in phase change temperature and latent heat values was observed to be ± 0.15 -0.20 °C and ± 0.30 -0.50 J/g. As a purging environment, nitrogen gas was used, and 20ml/min of flow rate was maintained for the whole experiment.





Figure 11 Chiller

Figure 12 DSC 6000

3.3.3.2 Thermo-Gravimetric Analysis (TGA)

Thermal stability of ME-PCMs was analyzed using SHIMADZU Differential Thermal Gravimetric Analyzer (DTG-60H). The following steps were followed for performing the analysis.

- Set the TGA at room temperature (25.00 $^{\circ}$ C).
- Regulate isothermal condition for 5.00 min.
- Step-up with increasing ramp of 20.00°C/min from room temperature (~25.00 °C) to 600.00°C.
- Observe the DTG curve while experiment is in progress.



Figure 13 SHIMADZU Differential Thermal Gravimetric Analyzer (DTG-60H)

• The TGA curves were plotted in Origin-Pro 8.5, after extracting it from the TA 60WS software in txt format. As a purging environment, nitrogen gas was used, and 20ml/min of flow rate was maintained for the whole experiment

3.3.4 Fourier Transform Infrared Radiations (FTIR)

FTIR was done on LWA, mixture of epoxy and graphite, PCM and ME-LWA. Chemical compatibility was checked by comparing the comparing the peaks of three graphs with the ME-LWAC.



Figure 14 FT-IR apparatus

3.3.5 Thermal Performance Test (Indoor)

Indoor thermal performance test was carried out to determine temperature difference in the cooling compartment for the three concretes (LWAC, ME-LWAC and non-encapsulated PCM-LWAC) when the concrete slab is heated from above. Two wooden compartments with dimensions 250mmx250mmx250mm were prepared with a 40mm thick slab in between. One was heating compartment (upper compartment) where source of heat, a 300W electric heating plate in our case, was attached and aluminum foil was used to concentrate the heat to the center of the slab. A tiny hole was provided in the wall of heating/cooling compartment for the inoculation of sensors. Second was cooling compartment (lower compartment) in which the temperatures were measured for each type of concrete slab. One of the two digital thermocouples was inoculated at the center of cooling compartment to read the temperature inside the compartment and other thermocouple was placed outside the setup to note down the outside temperature. Outside temperature was kept constant at 20°C. The heating plate was turned on for 25min and temperature readings noted every 5 minutes for 3hrs.



Figure 15 schematic diagram of apparatus of thermal performance test (left) front view (right) top view.

3.3.6 Thermal Performance Test (Outdoor)

To determine the temperature peak shift and the temperature difference among the three concrete types, the thermal performance setup (excluding heating compartment) was placed outside for the natural heating and cooling of compartments for 24 hours.



Figure 16 Thermal Performance apparatus (left) at day (right) at night

3.3.7 Retention Test

Retention test is to find out the quantity of PCM that can be retained in pores. Weigh PCM intruded aggregate (W1). Spread PCM intruded aggregate in a tray having white paper in it. Put tray in oven, having temperature 50°C (more than melting temperature) and let it in oven for 10 min. Then get the tray out and change the papers underneath and again put tray in oven. Repeat this process until paper does not get any wetter that is, PCM does not leach out of aggregate anymore. Weigh the aggregate again (W2).



(a)

(b)

(c)

Figure 17 (a) intruded PCM -LWA (b) loss of PCM at 50°C for 30 min. (c) loss of mass of PCM has stopped

Chapter 4

RESULTS AND DISCUSSION

4.1 Compressive Strength Test

Compressive strength of all the mixes i.e. Mix-1, light weight aggregate concrete (LWAC), Mix-2, LWAC with 50% LWA replaced with non-encapsulated PCM-LWA (0.5PCM-LWAC). Mix-3, LWAC with 100% LWA replaced with non-encapsulated PCM-LWA (1.0PCM-LWAC). Mix-4, LWAC with 50% LWA replaced with ME-PCM (0.5ME-PCM LWAC). Mix-5, LWAC with 100% LWA replaced with ME-PCM (1.0ME-PCM LWAC) was evaluated at the age of 7 and 28 days.

Following are the results that were found out after compression test:

- All mixes' compressive strength results showed that Compressive strength increases with the increase in age of the specimen.
- All mixes achieved about 75% of their compressive strength at 7days.
- In case of Mix-2 (0.5 PCM-LWAC), 15.2% reduction in 28 days strength as compared to control LWAC was observed. While in case of Mix-3 (1.0PCM-LWAC), 19.2% reduction in 28 days strength was observed as compared to control LWAC.
- In case of Mix-4 (0.5ME-PCM LWAC), 17.75% reduction in compressive strength was observed as compared to LWAC. While in case of Mix-5 (1.0ME-PCM LWAC), reduction of 29% reduction in compressive strength as compared to LWAC was observed.
- PCM-LWAC showed 11.6% more compressive strength than ME-PCM LWAC.



Figure 18 compressive strength of ME-LWAC



Figure 19 Compressive strength of PCM-LWAC

4.2 Elastic Modulus

The tests results showed that Elastic modulus of LWAC, PCM-LWAC and ME-LWAC were 19.44, 16.97 and 14.6 GPa respectively. Strain at compressive strength was 0.0023, 0.0024, and 0.0025 for LWAC, PCM-LWAC and ME-LWAC which is well within the range according to past experimental values. It also shows that there is no additional adverse effect of the presence of paraffin or the encapsulation layer is the reduction in elastic modulus is proportional to that of the compressive strength for the three mixes.



Figure 20 elastic modulus results (left) Secant moduli (right) stress-strain curve

4.3 Split tensile strength

The tests results showed that split tensile strength of LWAC, PCM-LWAC and ME-LWAC were 2.6, 2.2, 2.04 MPa respectively for LWAC, PCM-LWAC and ME-LWAC which is well within the acceptable range1/8 to 1/12 of the compressive strength of cubes. It also shows that there is no additional adverse effect of the presence of paraffin or the encapsulation layer as the reduction in split tensile strength is proportional to that of the compressive strength for the three mixes.



4.4 Micro-structural analysis

To study the morphology of the LWA and the two different concretes i.e. 1.0ME-LWAC and 1.0PCM-LWAC SEM was performed on the samples. The result for the LWA shows a high porosity of the aggregate with sufficient interconnectivity of the pores which can result in a higher amount of PCM being retained in the aggregate. From the images of the ME-LWAC, it can be

clearly seen that there is a thick, smooth layer of encapsulation between the aggregate surface and the surrounding mortar which results in a weak bond between the two. On the other hand, the images of PCM-LWAC shows a relatively better interaction between the LWA and the surrounding matrix as the matrix can be seen penetrating into the surface pores of the LWA. This improved ITZ can be associated to the higher strength of the PCM-LWAC as compared to the ME-LWAC in the compressive strength tests.



(a)

(b)

Figure 22 (a) SEM results of LWA (b) SEM results of ME-LWAC showing ITZ



Figure 23 SEM showing ITZ of PCM-LWAC (a) and (b)

4.4 Differential Scanning Calorimeter (DSC)

DSC was performed on three samples: Simple paraffin, PCM-LWA and ME-LWA.



Figure 25 ME-LWA endothermic curve (left) exothermic curve (right)



Figure 26 PCM-LWA endothermic curve (left) exothermic curve (right)

Following deductions were made from the DSC curves:

- Paraffin has melting temperature 24.52°C, freezing temperature 31.99°C and latent heat storage capacity of 192.98 J/g in its pure form.
- When Paraffin was directly intruded in LWA without encapsulation, melting point is 26.13°C, Freezing point 30.55°C and latent heat storage capacity of PCM-LWA is 20.14J/g.
- With the application of Macro-encapsulation on PCM intruded LWA, melting point reduced to 22.78°C, freezing point reduced to 29.19°C and latent heat storage capacity of the composite is 12.5J/g.

As literature describes that the shifting of temperature depends upon the interactive forces developed between material and the carrier for that material. The increase in the melting temperature shows the strong attractive force between the PCM and surface of the aggregate and some of this increase in melting temperature can also be attributed to the pore confinement upon the PCM. Though, this minute phase change temperature difference of PCM in its different forms shows that the PCM is thermally reliable in terms of its phase change behavior.

Moreover, the overall phase change temperatures of the PCM in its different forms is well within the human thermal comfort zone so it can be used in the concrete for the development of thermally efficient lightweight concrete to reduce the indoor temperature fluctuation.

4.4 Thermogravimetric Analysis (TGA)

To assess thermal stability of ME-PCMs TGA analysis was carried out. The investigation was carried out to determine the range of temperature up to which developed ME-PCMs and PCM-LWA possess stable nature. Standard TGA technique allows continuous weighing of a specific sample with respect to a reference sample as a function of temperature at desired temperature. The TG curves of the Paraffin, LWA and ME-PCMs are plotted in Figure. TGA curve of LWA showed negligible weight loss, and hence concluded to be the most stable. For paraffin, ME-PCMs and PCM-LWAs, the weight loss initiated at approximately in a rage of 140°C-150°C and ended at approximately 230°C and 250°C with only one step, which correspond to the



Figure 27 TGA curves

Evaporation or volatile nature of paraffin above 100°C. Moreover, the weight loss of PCM-LWA was found to be 26%, which depicts a high retention value of PCM in the LWA and the weight loss for ME-PCM was about 17%. Although the amount of PCM in the encapsulated composite is higher than the non-encapsulated one, the overall increase in mass due to the encapsulation layer results in a decreased amount of PCM per unit weight of the aggregate composite.

Since the weight loss in developed ME-PCMs and PCM-LWAs below 140°C is almost negligible, therefore, the investigated aggregate composites provide an opportunity to be used as thermal energy storage media in buildings.

4.5 Fourier Transform-Infrared Spectroscopy (FT-IR)

To investigate the possible physical and chemical interaction between individual components of the composite Fourier transform infrared spectroscopy (FT-IR) was used. The FT-IR spectra for paraffin, LWA, epoxy and ME-PCMs (composite) was determined and are presented in *Figure 28-31* while their significant peaks and possible assignments of different bands are given in *Table 2-5*.



Figure 28 FT-IR results of Paraffin

Figure 29 FT-IR results of epoxy



Figure 30 FT-IR results of ME-LWAC

The FT-IR spectrum of paraffin showed peaks at 2916 cm-1, 2850 cm-1, 1736 cm-1, 1449 cm-1, 1369 cm-1, 1169 cm-1, 722 cm-1. The two characteristic peaks at 2916 cm-1 and 2850 cm-1 are linked to C-H stretching vibration of the methylene group [20],[21]. The band at 1738 cm-1 represent the stretching vibration of carbonyl group [20]. Moreover, at 1449 cm-1 the paraffin sample also shows strong characteristic peak representing the bending vibration of methyl group, and a week characteristic peak at 1369 cm-1 which represent the bending vibration of hydroxyl group [22], [23]. The bands at 1169 cm-1 [22], [24] and 722 cm-1 [24] are associated with carbonyl stretching and rocking methylene.

The FT-IR spectrum of LWA showed significant peaks at 1029 cm-1,792 cm-1 and 556 cm-1. The band at 1029 cm-1 are associated with the stretching vibration of silica [24], [25] and 792 cm-1 with Si-O bending vibration [22], [26], moreover, iron oxide showed stretching at 556 cm-1 [22]. Epoxy FT-IR analysis, give rise to three significant peaks at wavelength 2920 cm-1, 1455 cm-1 and 1160 cm-1. The first two characteristic peaks are associated to methylene stretching while the last one is related to carbonyl stretching [20], [27], [28].

From the analysis it can be concluded that there is no or negligible shift in characteristic peaks and no new peaks have been raised, depicting that, there is no chemical interaction between the individual components of the developed ME-PCMs. From FT-IR analysis, it can be concluded that the interactions are physical in nature between the PCM, aggregate and the epoxy.

Band (<i>cm</i> ⁻¹)	Bands assignment	Reference
2916	Methylene C-H stretching	[22], [23]
2850	Methylene C-H stretching	[22], [23]
1736	Carbonyl C=O stretching	[22]
1449	Methylene/methyl bending	[25], [14]
1369	Hydroxyl O-H bonding	[24],[17]
1169	Carbonyl C-O stretching	[24], [25]
722	Rocking methylene =C-H	[25]

Table 2 Functional groups against peaks of Paraffin

Table 3 Functional groups against peaks of LWA

Band (<i>cm</i> ⁻¹)	Bands assignment	Reference
1029	Silica SiO ₂ stretching	[25], [26], [17]
794	Si-O bending	[24], [28]
556	Iron oxide Fe_2O_3 stretching	[24], [25], [29]

Table 4 Functional groups against peaks of Epoxy

Band (<i>cm</i> ⁻¹)	Bands assignment	Reference
2920	Methylene C-H stretching	[26]
1455	Methylene/methyl bending	[25], [14]
1160	Carbonyl C-O stretching	[26], [28]

Table 5 Functiona	l groups	against	peaks	of	LWA	composite
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Band (<i>cm</i> ⁻¹)	Bands assignment	Reference
2922	Methylene C-H stretching	[26]
2855	Methylene C-H stretching	[26]
1742	Carbonyl C=O stretching	[14], [13]
1449	Methylene/methyl bending	[25], [14]
1369	Hydroxyl O-H bonding	[28], [14], [30]
1169	Carbonyl C-O stretching	[26], [28]
1029	Silica SiO ₂ stretching	[25],[29], [26]

794	Si-O bending	[27], [28]
556	Iron oxide Fe_2O_3 stretching	[25],[29],[26]

4.6 Thermal performance tests

4.6.1 Indoor

In order to analyze the thermal performance of the concretes i.e. 1.0PCM-LWAC and 1.0ME-LWAC, the different slabs were placed between the heating and cooling compartments of the performance setup and the 300W heating plate was turned on for 25 minutes. The rise and fall of temperature was measured in the cooling compartment for the different concretes at an interval of 5 minutes as shown in the graph.



Figure 32 results of thermal performance indoor test

In comparison to the control room model, the rooms prepared with ME-PCM and PCM-LWA show lower indoor temperature, right shifted and have a flatter profile. The peak temperature reduction in case of PCM-LWA is 5.6°C whereas the reduction in case of ME-LWAC is 6.6°C. Therefore, the developed thermally efficient LWACs can reduce the indoor temperature during the charging process of Paraffin. When the cooling curves for the room models are compared, it can be seen that the PCM-LWAC and the ME-LWAC panels do not let the temperature to fall as quickly as in the case of the control concrete panel. This shows that the developed thermally efficient concrete room models are less vulnerable to indoor temperature fluctuations.

4.6.3 Outdoor thermal performance test

Three room models, each with a different type of concrete slab i.e. Control LWAC, 1.0PCM-LWAC, 1.0ME-LWAC, were placed outside in open air and the atmospheric temperature along with the inside temperature of each room was noted throughout the day as shown in the figure. It can be seen that Mix 3 has reduced the temperature of the compartment by 4.6°C with a peak temperature shift of 1.5 hours and there is a minute difference in case of mix 5 as it further decreases the temperature by 0.9°C. the result depicts that not only does the thermocrete help us in reducing the amount of energy required for space heating/cooling but it also helps in reducing the cost of that energy by shifting the peak temperatures away from peak load billing hours.



Figure 33thermal performance outdoor test

Chapter 5

CONCLUSIONS AND RECOMMENDATIONS

This research was focused on the elimination of the encapsulation layer from ME-LWA to reduce the cost, time and to enhance the structural properties along with the achievement of thermal properties. Comparison was made for thermal and mechanical properties of PCM-LWAC and ME-LWAC.

5.1 Research Findings

- PCM intrusion in LWA was increased to 2.2 times through Vacuum impregnation compared to simple immersion.
- PCM-LWAC and ME-LWAC has compressive strength of 22.41 MPa and 20.09 MPa.
 PCM-LWAC has more compressive strength than ME-LWAC and hence, PCM-LWAC has a higher strength than ME-LWAC because of the strong ITZ. Since its strength is greater than 17 MPa it can be used structurally.
- DSC results showed that the melting point, freezing point and latent heat storage capacity of PCM-LWA and ME-LWA is 26.13°C, 30.55°C and 20.14 J/g and 22.78°C, 29.19°C and 12.5 J/g respectively. These results showed that PCM-LWAC has temperature within the human thermal comfort zone (25 °C to 32 °C).
- TGA results showed that percentage mass loss of PCM-LWA (29%) greater than ME-LWA (17%) this depicts that ME-LWA has lower amount of PCM per unit weight.
- Thermal performance indoor showed the temperature difference of inside and outside with respect to LWAC was 5.6 °C for PCM-LWAC and 6.6 °C for ME-LWAC.
- Thermal performance test showed the temperature difference of 4.6 °C for PCM-LWAC and 5.5 °C for ME-LWAC with respect to LWAC and an energy shifting away from peak billing hours for 1.5 hrs. For PCM-LWAC and 1.8 hrs. for ME-LWAC with respect to LWAC.
- FT-IR results showed that no new peaks were generated when ME-LWA was prepared using the PCM, epoxy and LWA showing the compatibility of PCM with other components of concrete.
- Thermal cycling test showed that the same trend was observed even after multiple freezemelt cycles, showing thermal reliability of PCM-LWAC and ME-LWAC.

5.2 Conclusions

Keeping in view the findings of the research we can have following conclusions:

- Deterioration in strength was successfully reduced from 33% for ME-LWAC to 19.2% for PCM-LWAC by eliminating encapsulation layer and hence improving ITZ.
- At 28 days, Strength of PCM-LWAC is greater than 17 MPa, hence can be used as structural concrete.
- The peak temperature difference between the ME-LWAC and PCM-LWAC is about 1°C which shows that even after removing the encapsulation layer, there is a limited compromise in the thermal performance of the developed concrete.
- The Developed thermocretes i.e. PCM-LWAC and ME-LWAC are found to be chemically compatible (by FTIR) and thermally stable up to a temperature of 150°C (by Thermogravimetric analysis).
- The encapsulation layer can be eliminated as the slight temperature reduction (1°C) of ME-LWAC compared to PCM-LWAC comes at the expense of high cost, strength and increased need of time and technical expertise for its preparation.
- From the results it can be concluded that the developed concrete can be utilized in structural application and can be used to reduce the demand of purchased energy or shift the energy loads during peak hours.

5.3 Recommendations

- Work should be carried out to improve the fire resistance of the thermally efficient concrete.
- Investigation should be done on the effects of freeze-thaw cycles on the thermo Crete.
- Dynamic simulations should be carried out to estimate the energy requirements reduction in a full sized thermally efficient concrete structure.

REFERENCES

- [1] S. A. Memon, T. Y. Lo, H. Cui, and S. Barbhuiya, "Preparation, characterization and thermal properties of dodecanol/cement as novel form-stable composite phase change material," *Energy Build.*, vol. 66, pp. 697–705, 2013.
- [2] H. Cui, S. A. Memon, and R. Liu, "Development, mechanical properties and numerical simulation of macro encapsulated thermal energy storage concrete," *Energy Build.*, vol. 96, pp. 162–174, 2015.
- [3] X. Shi, S. A. Memon, W. Tang, H. Cui, and F. Xing, "Experimental assessment of position of macro encapsulated phase change material in concrete walls on indoor temperatures and humidity levels," *Energy Build.*, vol. 71, pp. 80–87, 2014.
- [4] B. Sharma, N. Neithalath, S. Rajan, and B. Mobasher, "Incorporation of Phase Change Materials into Cementitious Systems," no. September, 2013.
- [5] L. Pérez-Lombard, J. Ortiz, and C. Pout, "A review on buildings energy consumption information," *Energy Build.*, vol. 40, no. 3, pp. 394–398, 2008.
- [6] H. Klee, "Briefing: The Cement Sustainability Initiative," *Proc. Inst. Civ. Eng. Eng. Sustain.*, vol. 157, no. 1, pp. 9–11, 2004.
- [7] S. A. Memon, H. Z. Cui, H. Zhang, and F. Xing, "Utilization of macro encapsulated phase change materials for the development of thermal energy storage and structural lightweight aggregate concrete," *Appl. Energy*, vol. 139, pp. 43–55, 2015.
- [8] A. Palacios, A. De Gracia, L. Haurie, L. F. Cabeza, A. I. Fernández, and C. Barreneche, "Study of the thermal properties and the fire performance of flame retardant-organic PCM in bulk form," *Materials (Basel).*, vol. 11, no. 1, 2018.
- [9] R. Baetens, B. P. Jelle, and A. Gustavsen, "Phase change materials for building applications: A state-of-the-art review," *Energy Build.*, vol. 42, no. 9, pp. 1361–1368, 2010.
- [10] A. R. Sakulich and D. P. Bentz, "Incorporation of phase change materials in cementitious systems via fine lightweight aggregate," *Constr. Build. Mater.*, vol. 35, pp. 483–490, 2012.
- [11] S. A. Memon, "Phase change materials integrated in building walls: A state of the art review," *Renew. Sustain. Energy Rev.*, vol. 31, pp. 870–906, 2014.
- [12] A. Jayalath, P. Mendis, R. Gammampila, and L. Aye, "Applications of Phase Change Materials in Concrete for Sustainable Built Environment : A Review."
- [13] S. Ali Memon, T. Yiu Lo, X. Shi, S. Barbhuiya, and H. Cui, "Preparation, characterization and thermal properties of Lauryl alcohol/Kaolin as novel form-stable composite phase change material for thermal energy storage in buildings," *Appl. Therm. Eng.*, vol. 59, no. 1–2, pp. 336–347, 2013.
- [14] A. Karaipekli and A. Sari, "Preparation and characterization of fatty acid ester/building material composites for thermal energy storage in buildings," *Energy Build.*, vol. 43, no. 8, pp. 1952–1959, 2011.
- [15] B. Xu, H. Ma, Z. Lu, and Z. Li, "Paraffin/expanded vermiculite composite phase change material as aggregate for developing lightweight thermal energy storage

cement-based composites," Appl. Energy, vol. 160, pp. 358-367, 2015.

- [16] Z. Zhang, G. Shi, S. Wang, X. Fang, and X. Liu, "Thermal energy storage cement mortar containing n-octadecane/expanded graphite composite phase change material," *Renew. Energy*, vol. 50, pp. 670–675, 2013.
- [17] Y. Wang, T. D. Xia, H. Zheng, and H. X. Feng, "Stearic acid/silica fume composite as form-stable phase change material for thermal energy storage," *Energy Build.*, vol. 43, no. 9, pp. 2365–2370, 2011.
- [18] M. Hunger, A. G. Entrop, I. Mandilaras, H. J. H. Brouwers, and M. Founti, "The behavior of self-compacting concrete containing micro-encapsulated Phase Change Materials," *Cem. Concr. Compos.*, vol. 31, no. 10, pp. 731–743, 2009.
- [19] T. Y. Lo and H. Z. Cui, "Effect of porous lightweight aggregate on strength of concrete," *Mater. Lett.*, vol. 58, no. 6, pp. 916–919, 2004.
- [20] B. Gokulakumar and R. Narayanaswamy, "Fourier Transform Infrared Spectra (FT-IR) Analysis of Root Rot Disease in Sesame (Sesamum Indicum)," *Rom. J. Biophys.*, vol. 18, no. 3, pp. 217–223, 2008.
- [21] G. Sankari, T. S. Aishwarya, and S. Gunasekaran, "F Ourier T Ransform I Nfrared S Pectroscopy and F Luorescence E Mission S Pectroscopic I Nvestigations on R At T Issue," *Sci. Technol.*, vol. 2, no. 11, pp. 20–31, 2010.
- [22] T. S. Renuga Devi and S. Gayathri, "FTIR And FT-Raman spectral analysis of Paclitaxel drugs," *Int. J. Pharm. Sci. Rev. Res.*, vol. 2, no. 2, pp. 106–110, 2010.
- [23] Y. Su, J. Wang, and H. Liu, "FTIR Spectroscopic Study on Effects of Temperature and Polymer Composition on the Structural Properties of PEO–PPO–PEO Block Copolymer Micelles," *Langmuir*, vol. 18, no. 14, pp. 5370–5374, 2002.
- [24] J. Madejová, "FTIR techniques in clay mineral studies," Vib. Spectrosc., vol. 31, no. 1, pp. 1–10, 2003.
- [25] S. A. Memon, H. Cui, T. Y. Lo, and Q. Li, "Development of structural-functional integrated concrete with macro-encapsulated PCM for thermal energy storage," *Appl. Energy*, vol. 150, pp. 245–257, 2015.
- [26] B. Shokri, M. A. Firouzjah, and S. I. Hosseini, "FTIR analysis of silicon dioxide thin film deposited by metal organic-based PECVD," *Proc. 19th Int. Plasma Chem. Soc.*, pp. 1–4, 2009.
- [27] D. H. W. Li, L. Yang, and J. C. Lam, "Zero energy buildings and sustainable development implications - A review," *Energy*, vol. 54, pp. 1–10, 2013.
- [28] H. Akeiber *et al.*, "A review on phase change material (PCM) for sustainable passive cooling in building envelopes," *Renew. Sustain. Energy Rev.*, vol. 60, pp. 1470–1497, 2016.
- [29] H. A. Patel, R. S. Somani, H. C. Bajaj, and R. V. Jasra, "Nanoclays for polymer nanocomposites, paints, inks, greases and cosmetics formulations, drug delivery vehicle and waste water treatment," *Bull. Mater. Sci.*, vol. 29, no. 2, pp. 133–145, 2006.
- [30] Z. Ma and S. Wang, "Enhancing the performance of large primary-secondary chilled

water systems by using bypass check valve," *Energy*, vol. 36, no. 1, pp. 268–276, 2011.