THERMAL ANALYSIS OF CONCRETE INCORPORATED WITH PHASE CHANGING MATERIALS (USING NOVEL EPOXY & GRAPHITE COATED GELATIN ENCAPSULATION)

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This is to certify that the

Final Year Project, titled

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CIVIL ENGINEERING

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"In the name of Almighty Allah, the Most Beneficent, the Most Merciful"

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PCMs	Phase Changing Materials
HVAC	Heating, Ventilation and Air Conditioning
BIM	Building Information Modelling
PMD	Pakistan Meteorological Department
BS	Butyl Stearate
DD	1-Dodecenol
PEG	Polyethylene Glycol
TD	1-Tetradecenol
UV	Ultra-Violet
LWA	Light Weight Aggregate
SSPCM	Shape Stabilized Phase Changing Materials
SCC	Self-Compacting Concrete
DSC	Differential Scanning Calorimetry
OPC	Ordinary Portland Cement
ASTM	American Society for Testing and Materials
SEM	Scanning Electron Microscopy
ASHRAE	American Society for Heating, Refrigerating and Air-conditioning Engineers
ITZ	Interfacial Transition Zone
CSH	Calcium Silica Hydrate
ACI	American Concrete Institute

ABSTRACT

With rising global temperatures, depleting natural resources, and unsustainable energy supply, energy-efficient construction is becoming necessary. Concrete being the most utilized construction material in the world can be modified in several ways to achieve the purpose, like improvement in its thermal properties. One of the methods to mitigate the energy losses is by incorporating PCMs in it. However, the use of PCMs comes with its own disbenefits of deteriorating the structural performance and durability of concrete. Major effects include substantial reduction in the mechanical strength, deficient fire-resistance of concrete, and corrosion of reinforcement. Along with that no practical application of PCMs in buildings causes a lack of information on the performance of PCM-concrete combination.

This research was conducted to study a novel and an affordable technique to incorporate PCM (Paraffin wax) in concrete. We used macro-encapsulation process, using commercially available gelatin capsules coated with graphite and epoxy resin. PCM was added in a variety of percentages by weight of cement and then compared with a control sample to understand the affect in thermal and mechanical properties with varying percentage of PCM. However, loss in compressive strength of concrete was observed to a great extent making the concrete not suitable for structural members but suitable for non-structural envelope members of a building.

Along with strength measurements, thermal properties were also assessed under which the concrete showed interesting results. The thermal conductivity was lowered considerably with respect to control samples and the encapsulation technique ensured no leakage of the paraffin wax in the concrete matrix.

Lastly, a BIM-based analysis was done on Autodesk Revit to simulate the performance of our PCM incorporated concrete under various weather conditions around the calendar. The Revit model showed that the peak cooling loads of the model were reduced, and a much lower cost of utility was speculated.

Future research areas have been identified in the end which can be studied to make PCM incorporated concrete using our encapsulation technique can be an innovative solution to produce an affordable and energy efficient built environment.

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CHAPTER 1

INTRODUCTION

1.1 Background

Rising global temperatures are resulting in constantly growing energy demand. This coupled with depleting natural resources and continuously degrading environment has led to the development of sustainable and energy efficient buildings. The running costs of buildings is increasing endlessly because of growing fuel costs and moreover, the high supply of energy by power generation companies is resulting in a huge carbon footprint. Researchers are making efforts to reduce energy consumption of buildings by increasing thermal insulation and increasing the efficiency of HVAC systems. For this purpose, different areas of study are being focused upon which include, using renewable energy resources like solar energy and wind energy etc. and increasing the thermal energy storage capacity of buildings. Methods of increasing thermal energy storage capacity of buildings can be divided in two categories: ones that store latent thermal energy and the ones that store sensible thermal energy of the buildings.

1.1.1 Background of thermal energy storage systems

Thermal energy storage systems can be classified into two main types: physical processes and chemical processes. Physical processes of storing thermal energy are subdivided, depending on the type of heat stored. Fig. 1 list down different methods of storing thermal energy.

1. Sensible heat
2. Latent heat

Some factors that are to be considered while choosing thermal energy storage material are listed below:

1. Chemical stability
2. Cyclability
3. Mechanical strength
4. Thermal conductivity

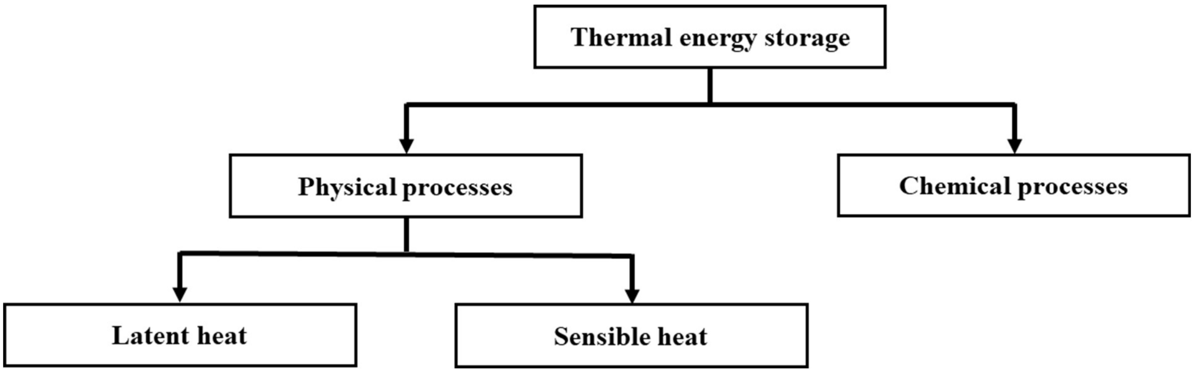

Fig. 1: Types of thermal energy storage systems (from [1])

Sensible heat is the heat stored in a material due to temperature change.[2] Sensible thermal energy storage systems involve changing the temperature of the storage material to absorb or release thermal energy. Some of the sensible thermal energy storage methods in buildings are the water wall and the Trombe wall (Shown in Fig. 2), which are based on the specific heat of materials and temperature variation.[3]

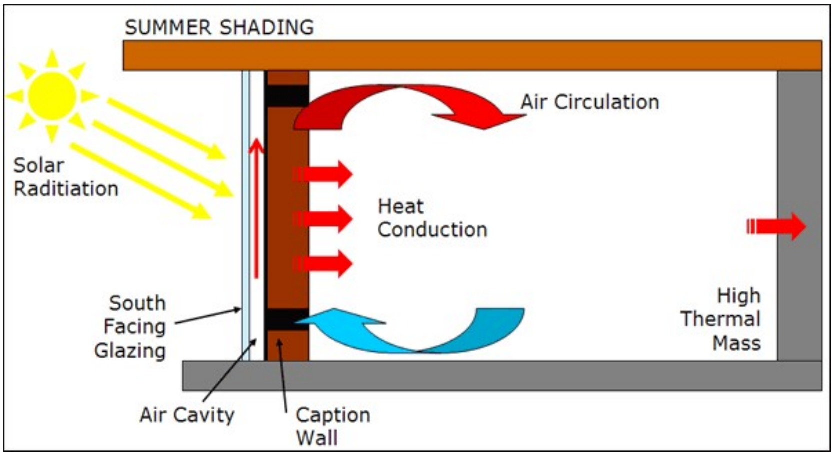

Fig. 2: Working principle of a Trombe wall

In latent thermal energy storage systems, the temperature of the material is changed so that it converts from one state to another i.e., solid to liquid or liquid to gas. While changing from solid state to liquid state, the material absorbs a specific amount of heat energy, which is known as latent heat. Conversely, as the material converts from liquid state to solid state, it releases back the absorbed latent heat. These type of storage systems are more efficient as the amount of thermal energy absorbed per unit volume by a material in solid state is much more than that stored by raising the sensible heat of substance through a small temperature range. There are further two types of latent thermal energy storage systems:

1. Active latent thermal energy storage systems: These systems employ mechanical equipment like HVAC systems to change the phase of the materials
2. Passive latent thermal energy storage systems: These systems change the phase of the materials without the use of mechanical equipment. Thermal energy from the sun can be used for the phase change process.

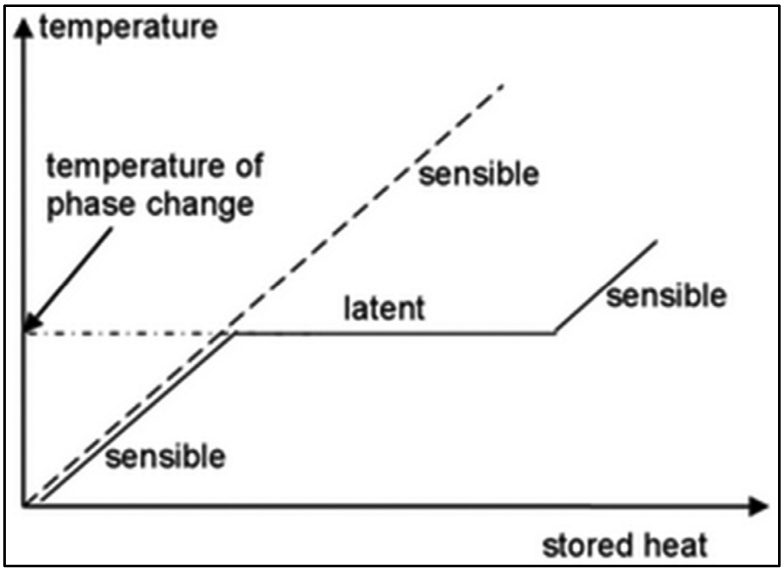

Fig. 3: Heat storage as latent and sensible heat (from [1])

1.1.2 Advantages of Latent thermal energy storage systems

In comparison to sensible thermal energy storage systems, latent thermal energy storage systems have several advantages.

1. The energy storage density is much higher
2. Using appropriate PCMs, thermal storage can be achieved at human comfort temperatures
3. It achieves more efficient energy management
4. It provides much broader design applications

1.2 Phase Changing Materials (PCMs)

Phase Changing Materials (PCMs) are used extensively in passive latent thermal energy storage systems. PCMs provide a large heat capacity over a limited temperature range, and they could act like an almost isothermal reservoir of heat.[4] As the outdoor temperature increase, PCMs undergo an endothermic process, in which they absorb heat, to change their own phase. When the temperature decreases below a specific temperature, an exothermic process is initiated in which the PCM releases back the absorbed thermal energy to change back its phase.

Widespread research is being conducted on the incorporation of PCMs in concrete as it is the most widely utilized building material. The effectiveness and efficacy of the use of PCMs for enhancement of energy performance of a building is affected by many essential parameters like, the type of PCM, the position of PCM in the structure, melting and freezing temperatures of the PCM, response of PCMs on different properties of concrete etc. Fig. 4 shows the response of phase changing materials to thermal energy.

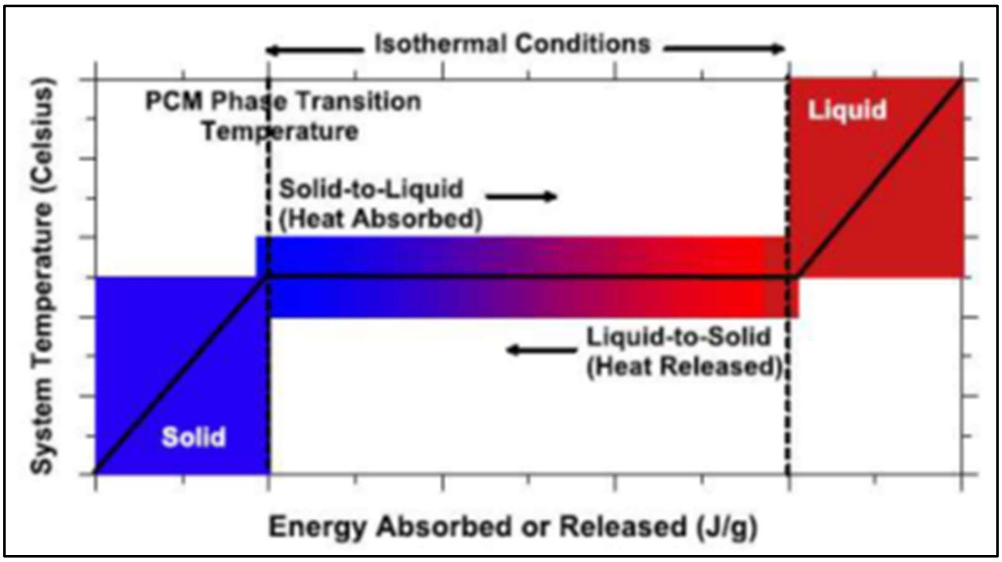

Fig. 4: A schematic illustrating the temperature - energy response of a PCM [5]

1.3 Building Envelope

Building envelope is an integral element in deciding the energy performance of a building and regulating indoor thermal requirements for the comfort of the occupants. All the structural elements that (slabs, walls etc.) that separates the indoor environment from the outdoor environment form the envelope of the building. One of the successful strategies of incorporating PCMs in buildings to make them energy efficient is to include the PCMs in the building envelope. It effectively controls the heating and cooling loads of the building and reduces the indoor temperatures. This leads to remarkable savings in the energy costs.

1.4 Problem Statement

In the climates like that of the subcontinent (especially Pakistan), which is predominantly characterized by hot, desert like conditions (source: Koppen climate classification for Pakistan), people consume huge amounts of expensive energy for cooling purposes, which puts extra strain on the country's already diminishing resources. According to PMD, average monthly maximum temperatures in central and southern Pakistan reached over 45°C in peak summer months of the year 2021. Therefore, the employment of latent thermal energy storage buildings' strategy will prove to be very useful to help people achieve thermal comfort in their homes and offices as well as save energy.

For this purpose, we devised an affordable, novel method to incorporate the PCMs in the concrete. Paraffin wax as PCM was encapsulated in commercially available gelatin capsules which were coated with epoxy and graphite powder. We conducted several tests and analyzed its effect on the mechanical, thermal, and morphological properties of the PCM incorporated concrete. Several conclusions are drawn from the obtained results which discuss the efficiency, effectiveness as well as practicality of PCM incorporated concrete using this novel technique. In addition to this, effect of silica fume on the mechanical properties of PCM incorporated concrete was also investigated briefly.

Furthermore, a comparison was drawn between the cooling loads obtained for a study site which used PCM incorporated concrete in its envelope elements and cooling loads obtained using other insulation techniques. This analysis gave insight about the effectiveness of the PCM incorporated concrete in a better way.

1.5 Project Objectives

The objectives of this research are focusing on:

1 To determine the physical properties like, melting and freezing points, enthalpy of phase change processes and melting and freezing onset temperatures of the paraffin wax used
2 To ascertain the effect on the mechanical properties which include compressive strength, stress-strain behavior, and elastic moduli of PCM incorporated concrete using gelatin encapsulation and to compare them with conventional concrete
3 To experimentally evaluate the effect of Paraffin wax as PCM in gelatin encapsulation on the thermal properties comprising of thermal conductivity and thermal resistance of concrete and to compare them with thermal properties of ordinary concrete.
4 To check the affect, if any, of addition of silica fume in the sample containing highest percentage of PCM on its mechanical and thermal properties
5 The study of micro-structure of concrete containing PCM and silica fume, to analyze the ITZ formed between concrete matrix and epoxy plus graphite coated gelatin encapsulation. Moreover, to derive possible reasoning for changes in mechanical properties of PCM incorporated concrete from that analysis.
6 To quantify the thermal properties of PCM incorporated concrete in the form of required cooling loads of a suitable building site in order to explore the practicality and efficacy of our novel PCM incorporated concrete.

CHAPTER 2

LITERATURE REVIEW

2.1 Phase Change Materials (PCMs)

A phase changing material (PCM) is a substance which releases and absorbs sufficient thermal energy to transit from one phase to another, which makes it a useful material for heating and cooling purposes. Phase change can be solid-liquid, liquid-gas, or solid-gas, however PCMs with solid-liquid phase transition are widely used. There are majorly three classifications of PCMs: organic, inorganic and eutectic.

From a concrete design point of view, it is important to identify what kinds of PCM are suitable for use in concrete, because different kinds of PCM have different chemical natures and melting / transition temperatures.[6]

2.1.1 Organic PCMs

Organic PCMs are chemically stable, inert, and safe. They possess high latent heat capacity with self-nucleating properties along with having an ability to melt congruently without posing problems of supercooling. However, they have low thermal conductivities and are flammable which make them slightly unsuitable for commercial usage.

Organic PCMs can be further described as paraffin and non-paraffin types. [7] Paraffins are open chain saturated alkanes that have a chemical structure of C_nH_{2n+2}. It generally comes with a melting point range of 20°C to 70°C depending on the number of carbon atoms it contains. The more carbon atoms present in the chain, the higher the melting point of the paraffin.[6] Extensive use of organic paraffin in concrete has been successfully demonstrated by previous studies as it is inactive in alkaline medium and is inexpensive.[6]

Fatty acids, esters, alcohols, and glycols make up most of the organic non-paraffin PCMs with a chemical structure of ($CH_3(CH_2)_{2n}COOH$). Their melting point is similar to those of paraffin PCM, and they have excellent melting and freezing properties.[6] Butyl Stearate (BS), 1-Dodecenol (DD), Polyethylene glycol (PEG), 1-Tetradecenol (TD) and Dimethyl sulfoxide are some non-paraffin organic PCMs that are studied from concrete's point of view. However, these are two or three times costlier than paraffin based PCMs and possess an undesirable odor which limits its use in building applications.

2.1.2 Inorganic PCMs

Inorganic PCMs comprise of salt hydrates (M_nH_2O) and metals. In comparison with organic PCMs, inorganic PCMs possess very high latent heat storage capacity, are inflammable and have sharper phase transitions. However, they undergo incongruent melting which reduces the reversibility and also suffer from supercooling which are significant problems.

$Na_2SO_4 \cdot 10H_2O$ and $CaSO_4 \cdot 6H_2O$ etc. are some common examples of salt hydrates PCMs. Disadvantages of salt hydrates PCMs outweigh their advantages for use in building applications. Another major issue with them is that with repetitive cycles of thermal loading the hydrated mass keeps on decreasing which eventually degrades its use.

2.1.3 Eutectics

Eutectic PCMs are mixtures of inorganic and / or organic PCMs which have a single melting point. The melting point is lower than the melting point of the constituent PCMs and have a single crystalline structure. Eutectics undergo congruent phase change without segregation and have a sharp phase change temperature. Eutectics can be inorganic - inorganic, organic - organic or organic - inorganic.

2.2 Criteria for selection of PCMs

According to literature review [4] [8] the main criteria for selecting PCMs is listed below.

1. The PCM should have suitable phase change temperature according to its application. Factors like climate, location, type of structure etc. should be considered.
2. Selected PCM should have high specific heat and latent heat of transition per unit volume along with high thermal conductivity so more effect from latent heat storage can be gained with as little volume of PCM as possible.
3. Volume change upon phase transition should be as little as possible and PCM should have low vapor pressure at operating temperature to prevent the risk of rupture of encapsulation.

4. The PCMs should be chemically stable non-reactive, non-corrosive, non-toxic and inflammable so that they do not degrade with time and / or damage the encapsulation, along with being safe to use and having low environmental impact.
5. An attractive PCM should have high crystallization and nucleation rates with little or no supercooling of the liquid phase.
6. To increase the practicality of the technology and to make it economically feasible, the PCM should be cost effective and available in abundance.

L. Braganca et al. [9] studied application of PCMs in buildings. Properties of some of the different PCM substances that were investigated are tabulated in Table 1.

Table 1

Properties of different investigated PCM substances

	Compound	Melting Temperature (°C)	Heat of Fusion (kJ/kg)	Thermal Conductivity (W/m.K)
Inorganic	$Na_2SO_4.10H_2O$	32.4	254	0.544
	$Ba(OH)_2$.8H2O	78	265.7	0.653
Organic	Dimethyl-sulfoxide	16.5	85.7	---
	Paraffin C22-C45	58 – 60	189	0.21
	Naphthalene	80	147.7	0.132
	Polyglycol E400	8	99.6	0.187
Fatty Acids	Propyl palmiate	10	186	---
	Capric acid	32	152.7	0.153
	Lauric acid	42 - 44	178	0.147

Keeping in mind the above-mentioned criteria, we employed Paraffin wax as our PCM in the concrete.

2.3 Paraffin Wax

Greyish black sludge is the last product obtained from the petroleum refining process after the extraction of asphalt. This is called paraffin. It is then converted into paraffin wax (Fig. 5) by treating it with bleaching agents and other chemicals. The phase change temperature of paraffin wax depends on its molecular mass and its chain length; hence it can be combined in suitable ratios to achieve the desirable phase change temperatures. In general, the longer the average length of the hydrocarbon chain, the higher are the melting temperature and heat of fusion. [10] Melting temperatures range from 23°C to 67°C. [10] Paraffin wax shows considerable cyclic stability and remains stable for up to 1500 cycles even. Moreover, it does not suffer from the problem of sub cooling as well as its storage density is found to be a function of the mixture composition. [11] They possess high latent heat storage capacities over a narrow temperature change along with having moderate thermal energy storage density. [10] However, the low thermal conductivity of paraffin as of concern but it can be improved by employing methods like use of finned tubes, metallic fillings etc. Furthermore, paraffin wax is safe, compatible with almost all types of containers and can be easily incorporated in heat storage systems. [10]

Research shows that the high thermal mass of concrete coupled with the ability to incorporate different materials into it makes it a suitable host for PCMs. [12] In order to prevent the risk of PCMs leakage in concrete, the selection of an appropriate PCM and incorporation technique in the concrete product is therefore important.[6] Keeping in mind the above-mentioned knowledge, organic PCMs are preferrable in concrete. Therefore, paraffin wax is selected as the suitable PCM to be used in the concrete.

Concrete is the most utilized material in the world; however, it comes with its own limitations like high energy consumption and a huge carbon footprint. Therefore, innovative solutions are being developed to make concrete more sustainable.

Fig. 5: Commercially available paraffin wax

2.4 Encapsulation of PCMs

There are generally a few probable means of PCM incorporation in concrete. The three predominant methods are:

i. Immersion: simple immersion of the porous concrete in the melted PCM (liquid state)
ii. Impregnation: vacuum impregnation of the PCM in porous aggregates.
iii. Direct mixing: direct mixing of an encapsulated PCM in the concrete mix during the concrete mixing stage. [6]

2.4.1 Immersion Technique

The immersion technique was first introduced by Hawes [6] and it is done by soaking the concrete samples in a PCM which is in liquid state. The liquid PCM seeps into the porous capillaries of the concrete product. Some of the principal factors affecting the immersion of PCM into the concrete sample are concrete structure, viscosity of the PCM and duration of immersion in the liquid PCM.[13] Generally, the immersion technique is a long process, spanning over several hours. The size and the form of capillary pores, voids and fissures in the concrete matrix will affect the volume and flow of the PCM incorporated as well as the effect of surface tension.

The viscosity of the PCM must be low enough at the immersion temperature to achieve effective penetration into the concrete and high enough to prevent excessive drainage from the product when it is removed from the bath.[13] Hawes and Feldman concluded that the immersion time must be adequate to allow the liquid PCM to soak into the voids of the concrete blocks. As a rule of thumb, the higher the temperature of the liquid PCM bath, the faster the speed of immersion process.

2.4.2 Impregnation Technique

From their investigations Zhang et al.[3] state that it is very difficult for even highly porous aggregate to absorb large quantity of liquid PCM by simple immersion. The main reason being that the pore spaces of the aggregate are blocked up with air, which prevent the liquid from entering the pore spaces. Therefore, the impregnation technique involves three steps [14] in which air and moisture are pumped out of the aggregate pores using a vacuum pump, then the aggregate is immersed in liquid PCM under vacuum environment and lastly, the PCM is allowed to solidify gradually before bringing the PCM incorporated aggregates to normal atmospheric pressure. These PCM carrying aggregates are then mixed into the concrete. It can be seen from Zhang et al. experiment results that lower the density of porous aggregate, higher the PCM absorption capacity of the aggregates.

2.4.3 Direct Mixing Technique

In this technique, the PCM is first encapsulated in any form of shell that is physically stable and chemically inert before being mixed in the concrete. The choice of encapsulant is critical since it must not react with either the PCM or the concrete; it must have reasonably good heat transfer properties; it must also withstand the rigors of manufacturing, transport, construction, and use. [15] Also, when the PCM is in liquid state, the capsules prevent the liquid PCM from leaking out. [6] The scale of encapsulation can be anywhere between nanoscale to macroscale depending on the physical size of the encapsulation.

2.4.3.1 Micro-encapsulation

According to Tyagi et al. [16], PCM particles are uniformly coated with a thin layer of any polymeric material, which is known as microencapsulation. This coating saves the PCM from leaking and degradation. Some of the methods of microencapsulation listed by Tyagi et al. are:

1. Pan coating
2. Air-suspension coating
3. Interfacial polymerization
4. Matrix polymerization
5. Spray drying
6. Centrifugal extrusion

Different shapes of PCM micro-encapsulations are depicted in the fig. 6.

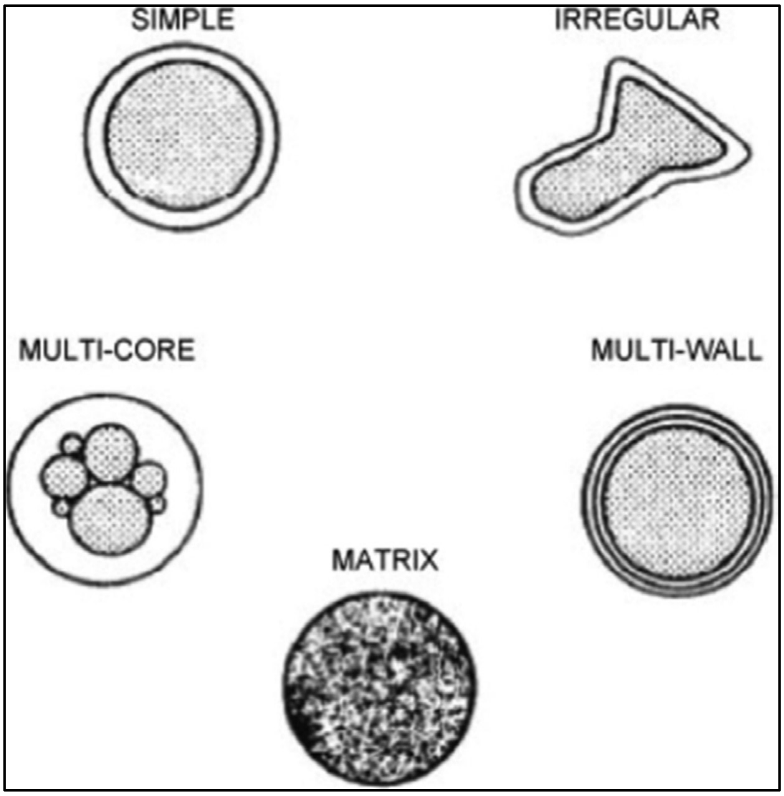

Fig. 6: Morphology of different types of microcapsules (from [1])

2.4.3.2 Macro-encapsulation

Many researchers have also studied and investigated the feasibility and effect of macro-encapsulated PCM in concrete. Macro-encapsulation method is basically storing PCM in any form of containers like tubes, spheres, panels etc. which can then be incorporated in building elements in one way or another.[17] Shi et al. incorporated PCM in concrete walls by encapsulating in steel box and then investigated how different positions of the macro-encapsulated PCM affect the indoor temperatures and the humidity levels.[18] Similarly, Zhijun et al. assessed the development of structurally-functional concrete by using hollow steel balls as the encapsulant to store PCM and investigating the thermal and mechanical properties of that concrete.[17]

On the contrary, there have not been much research on the use of gelatin capsules as the medium to store PCM and then incorporating them in concrete. However, some literature is available regarding inclusion of gelatin and cellulose capsules in development of different kind of concrete products. Like Leyang et al.[19] developed a self-healing concrete by containing Epoxy acrylate based free radical-cationic UV curable adhesive in commercially available gelatin and cellulose medicinal capsules coated with waterproofing mortar and acrylic acid-based polymer coating.

2.4.4 Enhancement of thermal conductivity of PCM encapsulations

Depending on the material of the encapsulant, encapsulation of the PCM leads to decreased thermal conductivity which leads to inadequate heat transfer to the PCM itself, thereby not fulfilling the purpose of latent heat storage by the PCM. To solve this problem, several heat transfer enhancement techniques [4] are employed such as:

i. The dispersion of highly conductive particles in PCM (e.g., Pb, Graphite, Cu, Al etc.)
ii. Impregnation of highly conductive porous material with the PCM.

Memon et al. [20] coated PCM incorporated LWA with epoxy resin and then used graphite powder to improve its thermal conductivity.

2.5 Properties

Addition of PCMs in any form in the concrete mix affects both, the thermal and mechanical properties.

2.5.1 Mechanical Properties

H.-W. Min et al. [21] investigated the effect of shape stabilized PCM (SSPCM) by impregnating the PCM into the structure of xGnP on thermal and mechanical properties of the concrete. They concluded that the maximum stress at the failure decrease as SSPCM content is increased. Similar trend was followed by the tensile strengths and elastic moduli of the samples as well. SSPCM containing concrete mixture had smaller amount of cement and aggregate, which resulted in this loss of strength. M. Hunger et al. [7] mixed micro-encapsulated PCM in self-compacting concrete. Their experimental results showed that increasing the PCM doses led to significant decrease in compressive strengths. This loss of strength can be attributed to the fact that the strength of PCM micro-capsules is very low in comparison to the strength of the concrete mix. Different studies (Ma and Bai [22], Ling et. al. [6], Norvell et. al. [23]) reviewed by Adesina et al. [12] reported a decrease in compressive strength of approximately 30% when 30% by volume of PCM was incorporated in the concrete. Fig. 7 shows the effect of PCMs on the compressive strengths of concrete. Moreover, the flexural load capacity, toughness and stiffness of the concrete was also reported to be decreased. Reduction in the mechanical properties of the concrete upon addition of PCMs can be attributed to the physical nature (i.e., toughness) of the matrix which reduces the toughness of all the whole matrix. In addition, low bonding between the cementitious matrix and the PCM encapsulation might also be responsible for the lower mechanical strength.[12]

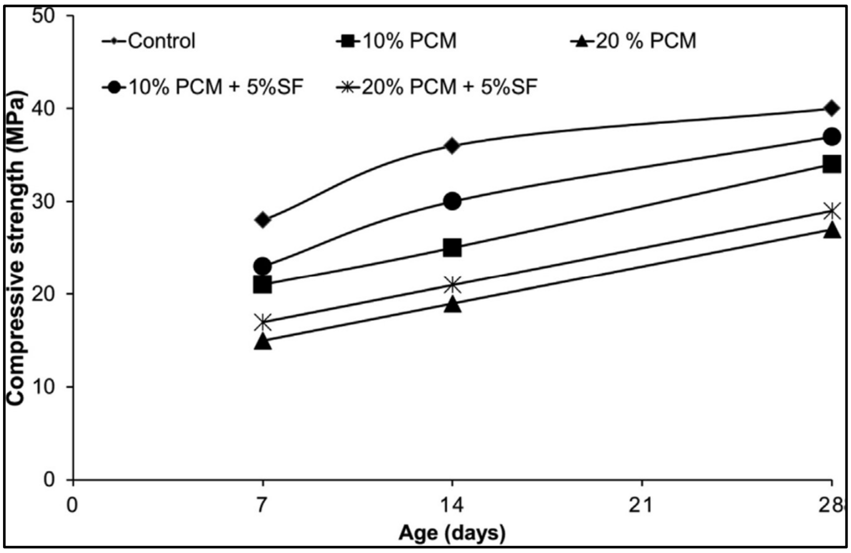

Fig. 7: Effect of PCM on the compressive strength of concrete (data from [5]*)*

2.5.2 Thermal Properties

On the other hand, many studies report that, thermal properties of concrete improve noticeably, when PCM is incorporated in it. However, the type of PCM used and technique of PCM incorporation affects the extent of improvement in thermal properties. Hunger et al. [7] researched the effect of microencapsulated PCM on Self-Compacting Concrete (SCC) and they concluded that addition of PCM particles into the mass of concrete resulted in a reduction of thermal conductivity, which can be attributed to low thermal conductivity of paraffin. Their results are depicted in fig. 8. B. Savija [24] stated that multiple studies report that a reduction in thermal conductivities was observed when PCMs were added in the concrete. Dehdezi et al. [25] conducted thermal, mechanical, and microstructural analysis of concrete containing microencapsulated PCM and they concluded that there is a general reduction in thermal conductivity upon addition of PCM in concrete. They attributed this observation to the decreased inter-particle contact and creation of air pockets because of increased air-void content in the concrete, as a result of entrapped air from PCM addition. Dehdezi et al. and Hunger et al. [7], concluded that the volumetric heat capacity of PCM incorporated concrete increased significantly across the PCM melting temperature range as the volume of PCM increase in the concrete. Similarly, Dong et. al. [17] and Memon et al. [20] used a self-designed thermal performance setup to report that concrete incorporated with macro-encapsulated PCM can improve the thermal performance of building especially in summers, in which the ambient temperature is above the phase changing temperature of the PCM.

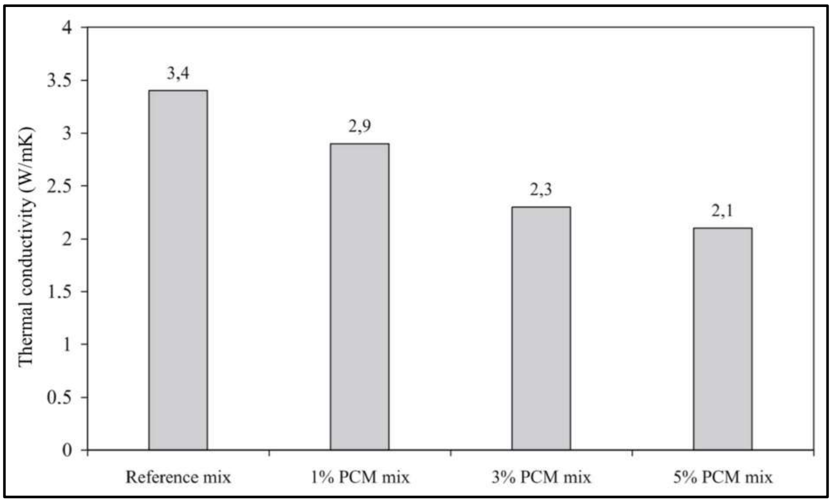

Fig. 8: Thermal conductivity of the self-compacting concrete PCM mixes [7]

CHAPTER 3

METHODOLOGY

3.1 Materials and preparation:

3.1.1 Materials

Paraffin wax procured from a supplier in Lahore, Pakistan was used as the PCM. Table 2 shows the properties of the paraffin wax used, that were obtained by performing Differential Scanning Calorimetry (DSC) analysis. Gelatin capsules that are used for medical purposes were procured from local supplier. Epoxy resin, which was easily available in the market was used to coat the paraffin wax containing gelatin capsules. The mixing ratio of epoxy adhesive and the hardener was kept as 1:1. Graphite powder was added to the liquid epoxy to improve its thermal conductivity. Coarse aggregate with a nominal maximum size of 19 mm was used and fine aggregate, having a fineness modulus of 2.216 was used in the concrete mix design. Aggregate gradation was done according to ASTM C136. Fig. 9 and fig. 10 show the gradation curves of the coarse and fine aggregate, respectively. OPC conforming to ASTM C150, Type I was used as the binder. Table 3 list downs the properties of the OPC. Silica fume was procured from a local supplier.

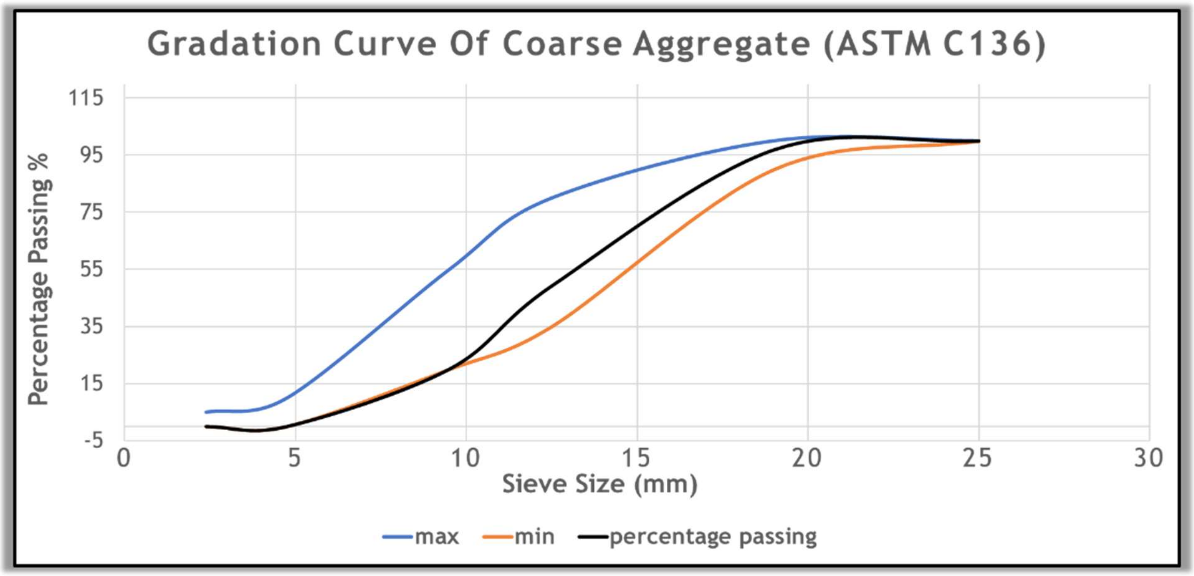

Fig. 9: Gradation curve of coarse aggregate

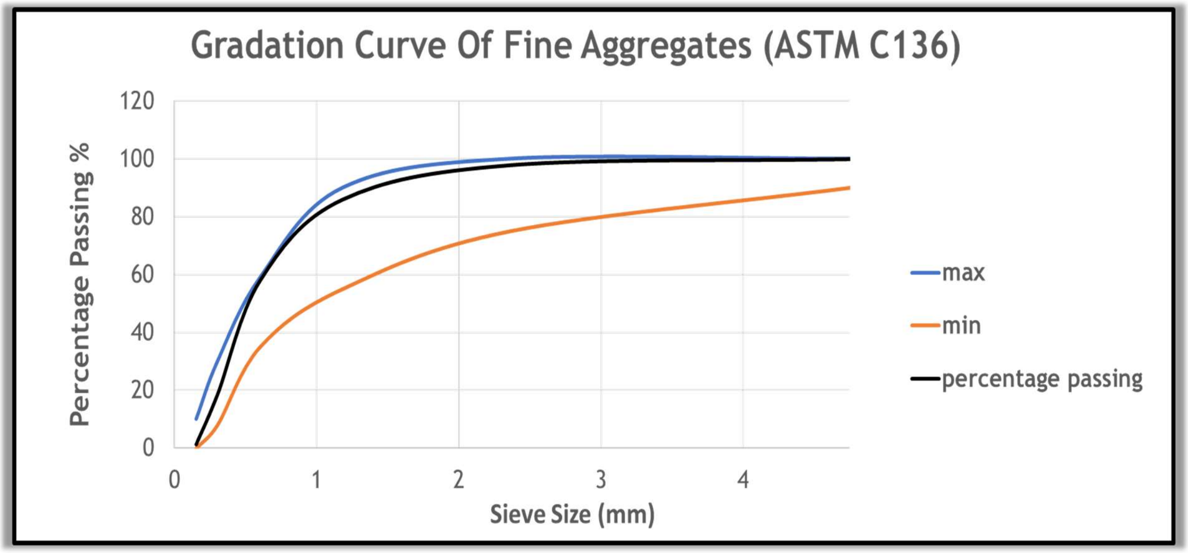

Fig. 10: Gradation curve of fine aggregate

Table 2

Physical properties of Paraffin wax

Melting point	52 °C
Melting onset	39 °C
Enthalpy on melting	53.5 J / g
Freezing point	46 °C
Freezing onset	55 °C
Density	0.9 g / cm^3
Oil content	0.5 %

Table 3

Physical properties of Ordinary Portland Cement

Blaine Fineness	3425 cm^2 / g
Initial setting time	115 min
Final setting time	136 min
Specific gravity	3.16
Normal consistency	26%

3.1.2 Preparation of PCM containing gelatin capsules

Liquid paraffin wax was filled in the gelatin capsules using a syringe with its needle removed. The capsules were then closed and left so that the paraffin wax solidifies. Gelatin capsules are water soluble and are affected by acid and bases. Therefore, to protect the paraffin containing gelatin capsules as well as to make them stronger they were coated with epoxy resin. As the thermal conductivity of epoxy resin is low, it hinders the transfer of thermal energy to the PCM thereby, lowering its performance. So based on the literature, 10% graphite powder by weight of epoxy plus resin was added in the liquid epoxy to enhance the working efficiency of our PCM incorporated concrete. After uniformly coating the capsules, they were shifted to trays containing dry OPC so that they could remain separated and not stick to each other. After 24 hours, the epoxy coating had dried completely, and capsules were ready to be added to the concrete mixes. The three figures below show the process of making the epoxy and graphite coated gelatin capsules filled with paraffin wax.

Fig. 11: Preparation of PCM containing capsules

3.2 Mix designs:

Five different samples of concrete were prepared in which the amount of PCM was varied as 0, 10, 15, and 20 percent by the weight of cement. In 20PCM8SF sample 20% PCM by the weight of cement was added and 8% silica fume by the weight of cement was added as a replacement of cement. “Control” sample represents ordinary concrete without any sort of additions. Details of the mix design are shown in table 4. All these mixes were designed for a constant water cement ratio of 0.5.

Table 4

Mix Designs

Sample	Cement (kg / m^3)	Sand (kg / m^3)	Aggregate (kg / m^3)	PCM (kg / m^3)	Silica Fume (kg / m^3)
Control	420	780	1056	0	0
10PCM	420	780	1056	42	0
15PCM	420	780	1056	63	0
20PCM	420	780	1056	84	0
20PCM8SF	386.4	780	1056	84	33.6

3.2.1 Mixing of Concrete:

For all the mix designs, coarse and fine aggregate were taken in surface dry condition. All the solid particles were mixed in dry state for about 60 sec. After which water was added slowly and the mixing was continued for a further 120 sec. For the samples having PCM, the PCM containing capsules were added into the freshly prepared concrete and mixed by handheld tool. Paraffin containing capsules were added at the last so that the capsules are not damaged during the mixing process.

Concrete mixes were placed in stainless steel cylinders of diameter 100 mm and height 200 mm and oil was applied on their inside surface to prevent the dried concrete from sticking to the molds. Using a scoop, concrete was added in three layers and using a tamping rod, each layer was tamped 25 times. Molds were removed after 24 hours, and samples were then placed in curing tanks for 28 days. Figures below show the process of concrete placement.

Fig. 12: Process of casting PCM incorporated concrete cylinders

Samples for thermal conductivity tests were prepared in the same way as mentioned above, the only difference was of the mold, which was a disc of diameter 50 mm and height 25 mm, as shows in fig. 13.

Fig. 13: Discs of concrete for thermal conductivity test

3.3 Test methods

3.3.1 Compressive strength test:

Compressive strength of concrete cylinders was measured at the age of 28 days in accordance with ASTM C39. Compression machine, with loading rate set as 0.25 MPa per second was used to measure the strength. Compressive strength test was conducted at 25 °C and 80 °C to study the effect of melted PCM on the compressive strength of the samples. At 25 °C the PCM was in solid state inside the capsules and at 80 °C the PCM was in liquid state in the capsules. The samples were loaded until failure and an average was taken of strengths of three cylinders for each mix design. To achieve the temperature of 80 °C, samples were heated in the oven for one hour.

3.3.2 Elastic Modulus:

To determine the elastic modulus, a load-controlled compression machine was used, with a loading rate of 0.25 MPa per second. The age of samples at the time of testing was 28 days and the test was performed at room temperature. Compressometer assembly was attached to the cylinders and the digital deformation dial was calibrated. Two loading cycles were performed; in the first cycle loading was stopped as the loading reached 40% of the compressive strength of the sample, then in the second loading cycle, the samples were loaded until failure. As the load was increased on the samples, the axial deformation in millimeters was recorded at every 10 kN interval during both the cycles. Strain at each load was obtained by dividing the deformation with original length of the cylinders. Stress – Strain curves were plotted, and tangent elastic modulus was determined by calculating the slope of the stress – strain curve till the elastic limit.

3.3.3 Thermal conductivity and Thermal resistance:

Thermal conductivity test was performed in accordance with ASTM E1530 – 06, which uses guarded heat flow meter technique to evaluate the resistance offered by materials to thermal transmission. We conducted the test at 45 °C and at 65 °C, so that the PCM is able to melt properly, and the age of samples was 28 days.

3.3.4 Differential Scanning Calorimetry:

Differential Scanning Calorimetry (DSC) is a thermal analysis technique in which the heat flow into or out of a sample is measured as a function of temperature or time, while the sample is exposed to a controlled temperature program. It is a very powerful technique to evaluate material properties such as melting / freezing points, latent heat storage, purity, and thermal stability. We subjected a 1g sample of paraffin wax to DSC to obtain its melting and freezing points, melting and freezing onsets and enthalpy changes.

3.3.5 Scanning Electron Microscopy:

The addition of microencapsulated PCM to our concrete would surely affect the mechanical properties along with the thermal properties. To study the change at microstructural level we conducted the SEM test, using back scattered electron technique, for a variety of samples obtained from broken concrete cylinders. The main purpose of this test was to visualize the modification observed in the ITZ and the bond formed between the capsule and cement matrix. The SEM analysis was also used to support our findings regarding mechanical properties. Magnified images of both the gelatin capsule and cement matrix were observed at a magnification of 5000X.

3.4 Revit Energy Analysis:

A single-story, 10 Marla house, located in Sukkur, Pakistan was taken as our study site. The study site consisted of two bedrooms, a TV lounge, a kitchen, and a drawing room. The site was exposed to solar radiation from all the four sides and the roof. Sukkur was chosen specifically, because it experiences very high maximum temperatures in summers, and it is a reasonable representation of urban cities of Pakistan from economic point of view. Different versions of the site were modelled, which are listed below:

1. All the envelope elements were made of concrete having 20% PCM
2. All the envelope elements were made of our control concrete
3. All the envelope elements were made of brick masonry (clay brick shown in fig. 14)
4. All the envelope elements were made of concrete blocks having air gaps for insulation (shown in fig. 15)

Fig. 14: Clay brick

Fig. 15: Concrete block

Energy models of all the versions of the site were created using Autodesk Revit and subsequently, HVAC loads were obtained using the built-in Revit tools. The outdoor dry air bulb was considered to be as 45 °C and the cooling loads were calculated so that the indoor temperature is maintained at 23 °C. HVAC loads were calculated in accordance with ASHRAE standards. Fig. 16 shows the visuals of the modelled study site whereas fig. 17 shows the energy model of the study site.

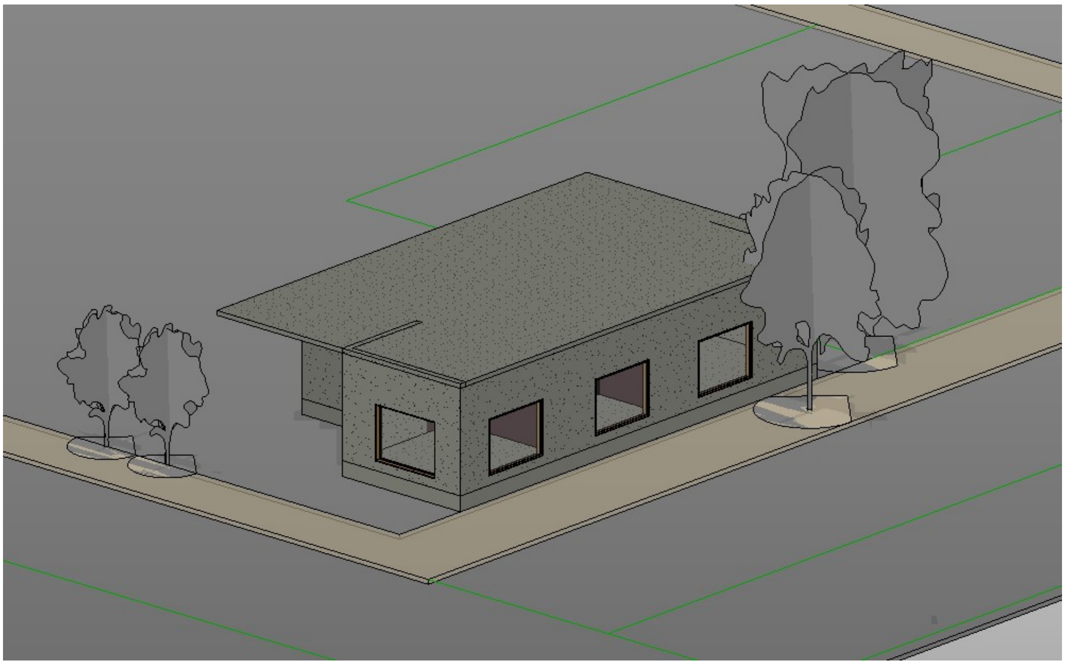

Fig. 16: Model of the study site

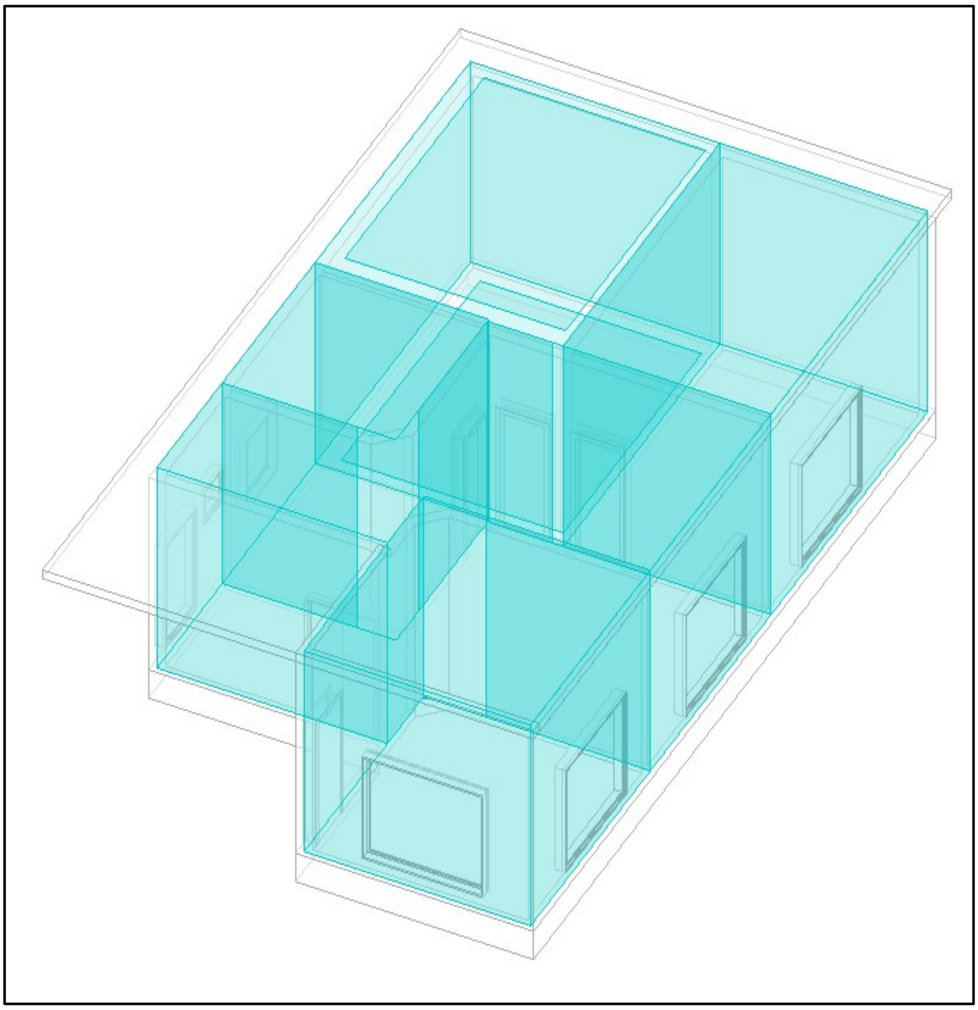

Fig. 17: Energy model of the study site

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Mechanical properties:

4.1.1 Compressive strengths

The compressive strengths of five different mixes i.e., control mix, mix having 10, 15, 20% PCM and mix having 20% PCM along with 8% silica fume were conducted at 25 °C and 80 °C. Following observations can be made from the results, which are depicted in the fig. 18:

- The compressive strength of the samples decreased as the percentage of PCM in them increased. At 25 °C, the percentage decrease of 10%, 15%, 20% and 20% PCM with 8% silica fume with respect to control sample showed a percentage decrease of 29.5%, 44.8%, 57.1% and 50.7% respectively.
- For all the samples, there was only a slight difference between their compressive strength at 25 °C and at 80 °C.
- At both the temperatures, the sample containing 8% silica fume and 20% PCM showed a bit higher compressive strength than the sample containing only 20% PCM.

The successive decrease in compressive strength can be attributed to the major difference between the intrinsic strengths of PCM capsules and the concrete matrix. This disparity in the strengths leads to low overall strength of the matrix. The weak bonding between the PCM capsules and concrete sample is also responsible for the decrease in compressive strength, as the capsules and matrix do not form a dense Interfacial Transition Zone (ITZ), which leads to creation weak zones in the samples. The higher the amount of PCM containing capsules in the concrete, higher would be the number of weak and disturbed ITZs in the matrix. The sample containing silica fume showed small increase in the strength because presence of silica fume in the matrix results in dense ITZs and pozzolanic reaction leads to the creation of secondary C-S-H gel.

4.1.2 Elastic Moduli

Stress – strain curves of all five samples were prepared and are shown in the fig. 19. It can be clearly observed that the stiffness of samples decreases as the amount of PCM incorporated in them is increased.

The elastic moduli of all the samples determined from the stress – strain curves, showed a similar trend as we found in the compressive strengths. Elastic moduli decreased significantly as the percentage of PCM increases. Fig. 20 shows the elastic moduli of all five mix designs.

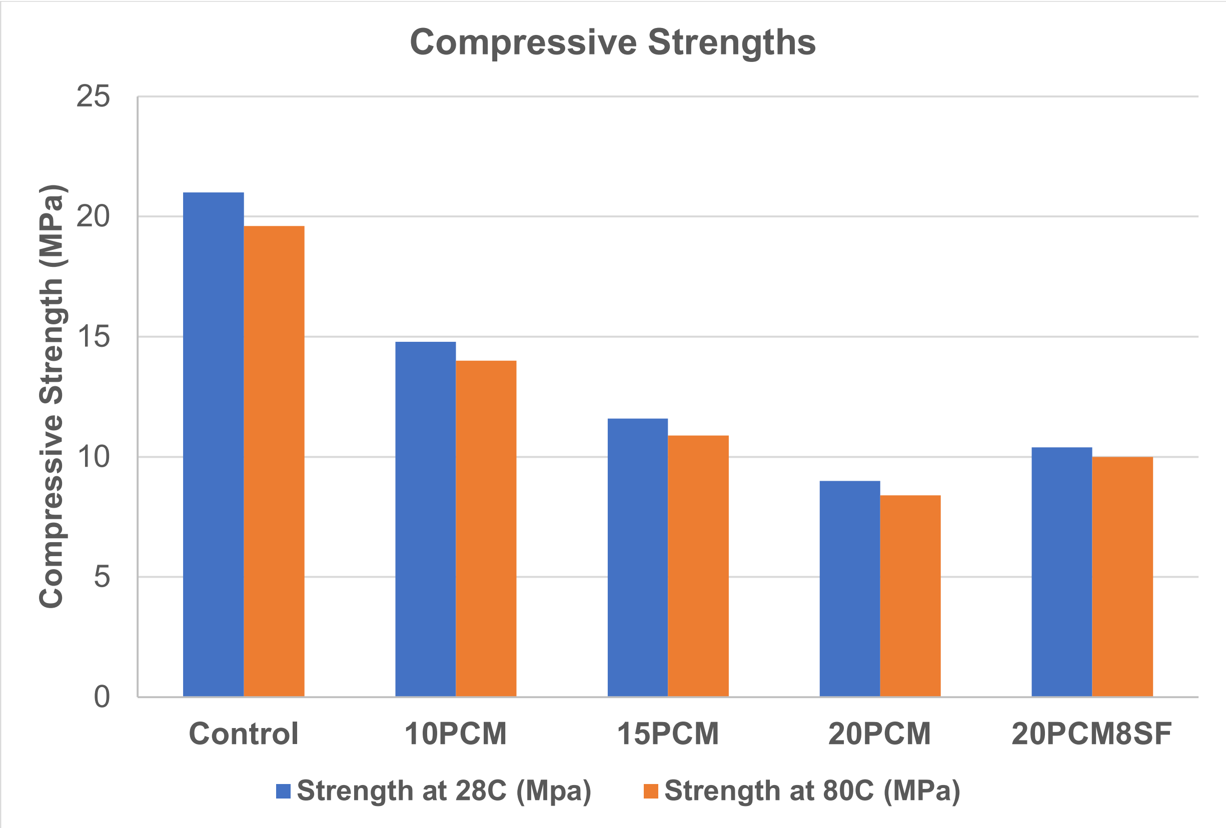

Fig. 18: Compressive strengths of PCM incorporated Concrete

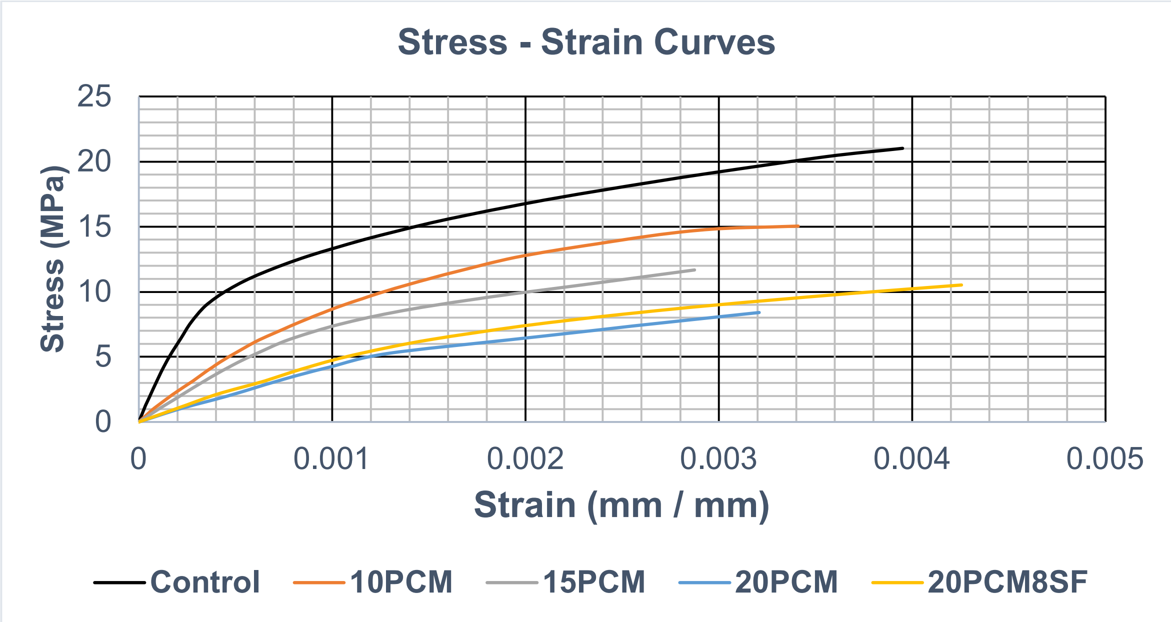

Fig. 19: Stress - strain curves of all the mix designs

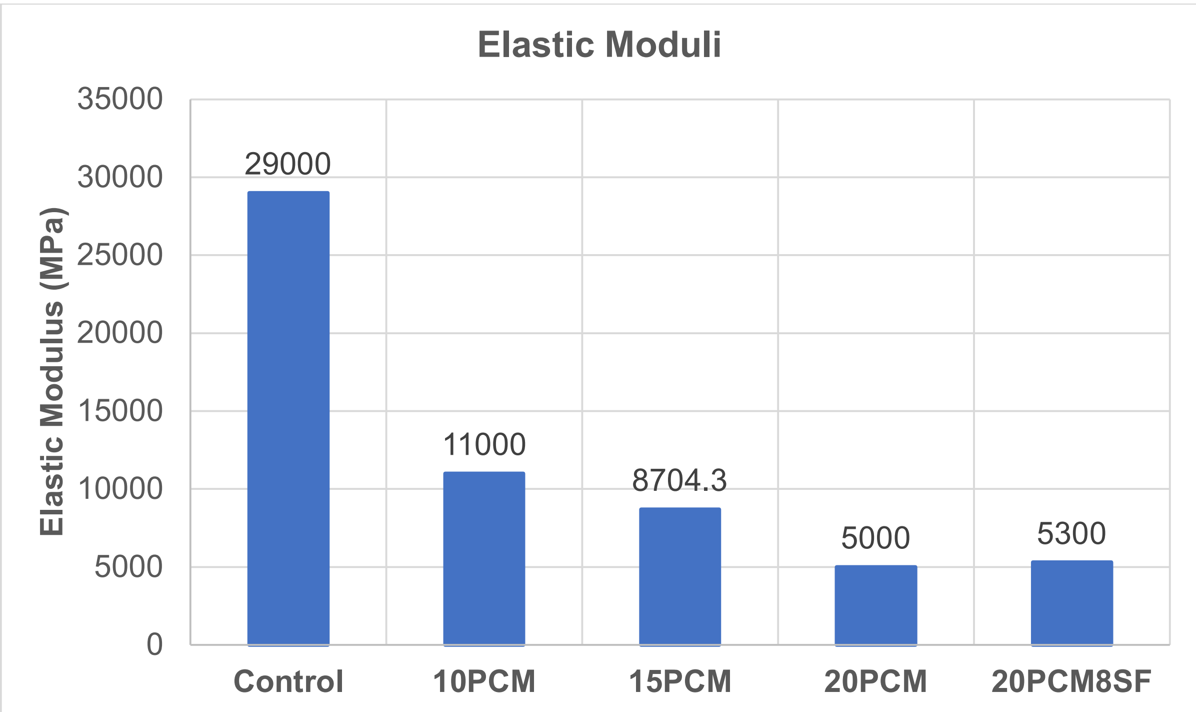

Fig. 20: Elastic moduli of PCM incorporated concrete

4.2 Thermal properties:

4.2.1 Differential Scanning Calorimetry

The DSC results of paraffin wax our mentioned in the section 3.1.1, which shows us that however the melting point of the paraffin wax used is very high i.e., 52 °C, however, the melting process starts at 39 °C which helps us achieve our purpose. Fig. 21 shows the results of the DSC test.

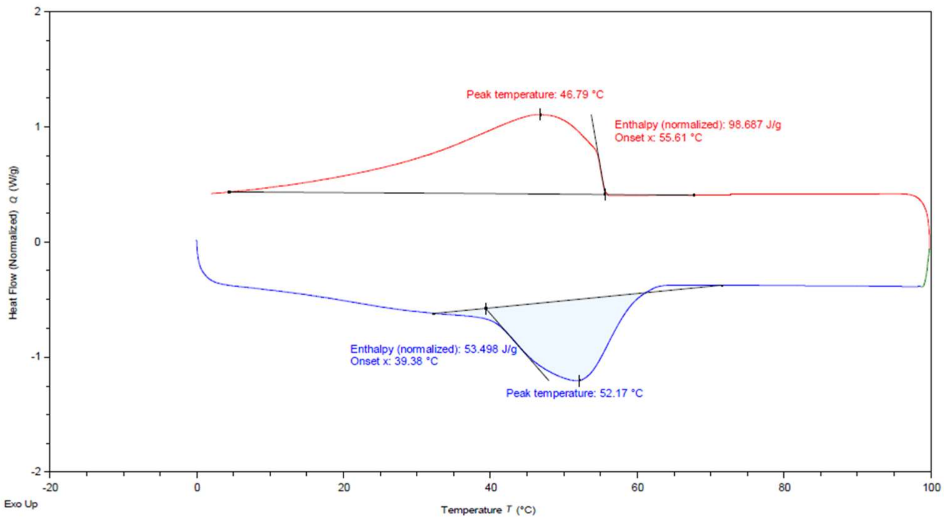

Fig. 21: DSC results of paraffin wax

4.2.2 Thermal conductivity and Thermal resistance

Observation of effect on the thermal properties of PCM incorporation in concrete using our novel encapsulation technique was an eminent segment of this research. DTC-300 was conducted at 45 °C and at 65 °C to obtain the thermal conductivity and thermal resistance of five different samples. The thermal conductivities of all the samples at both the temperatures are shown in fig. 22

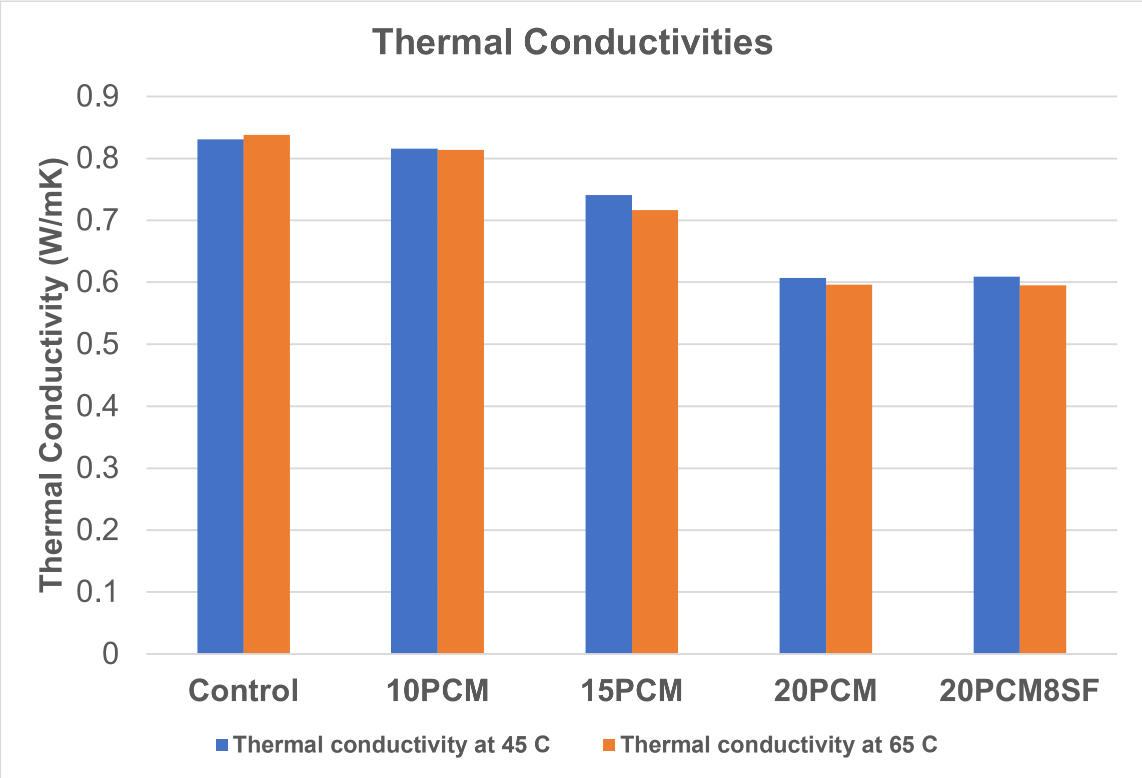

Fig. 22: Thermal conductivities of PCM incorporated concrete

From the results, it can be observed that, as hypothesized, the thermal conductivities of the samples decrease as the percentage of PCM is increased in the mix. A decrease of almost 30% was observed in the thermal conductivity of 20PCM sample with respect to control sample. This trend is observed because PCMs, paraffin wax in this case, have very low thermal conductivities in comparison with that of ordinary concrete. We can deduct from the figure that 20% PCM sample and the sample containing 20% PCM and 8% silica fume have similar thermal conductivities which means that addition of silica fume in the concrete have no major effect on its thermal conductivity.

Similar trend was also observed in the thermal resistances of all the mixes, which are depicted in fig. 23. As the mass of PCM in the mix increased, the thermal resistance offered by the sample also increased. This is because PCMs have high latent heat storage capacity which absorbs and uses thermal energy to change phase rather than let the thermal energy pass through them.

One observation that was made from all the thermal tests was that the improvement in thermal properties was only noticeable when the percentage of PCM in the mix was increased beyond 15%, with respect to control mix, only little improvement was seen in thermal properties of the mix containing 10% PCM.

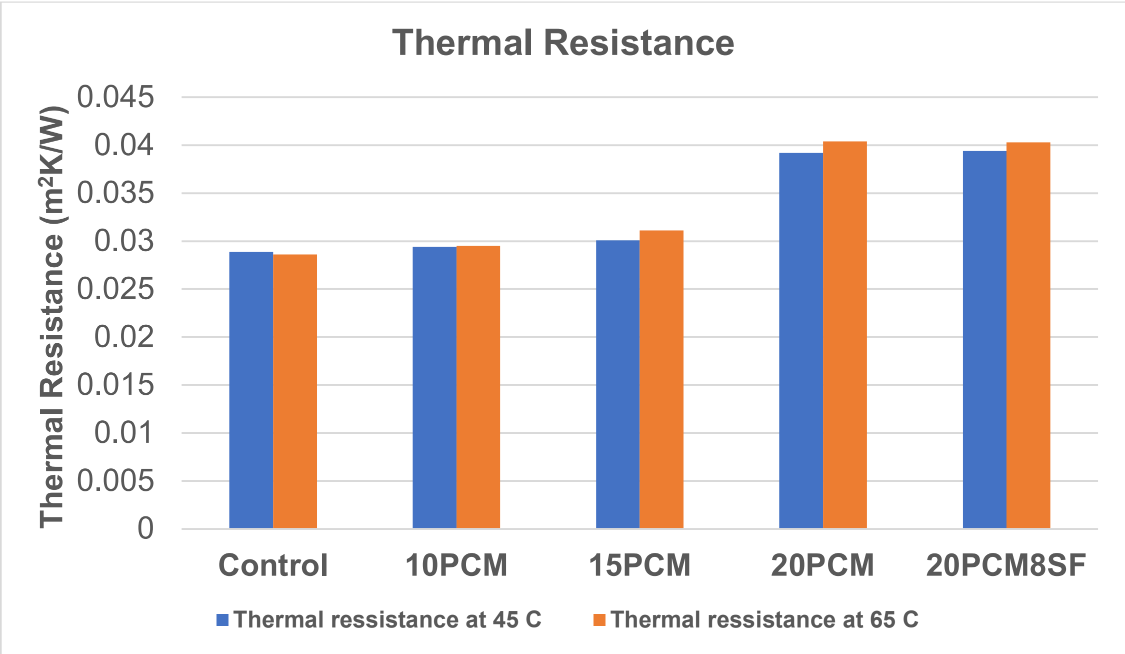

Fig. 23: Thermal resistances of PCM incorporated concrete

4.3 Microstructural study:

The SEM results obtained at 5000X magnification yielded important information. Fig. 24 shows the cement matrix-PCM capsule boundary. We can observe a disconnected ITZ explaining the loss of compressive strength up to great extents and the weak bond formed between the capsule and cement matrix. Due to presence of a weak ITZ the capsule and matrix boundary forms the weakest link and results in early failure of samples under compressive loads. Fig. 25 shows a SEM analysis of epoxy and graphite coating. The capsule shell can be observed to be cracked at various locations due to the action of loading. The weak nature of our coating can be seen clearly which causes a reduction in overall strength of our PCM incorporated concrete as compared to the control samples.

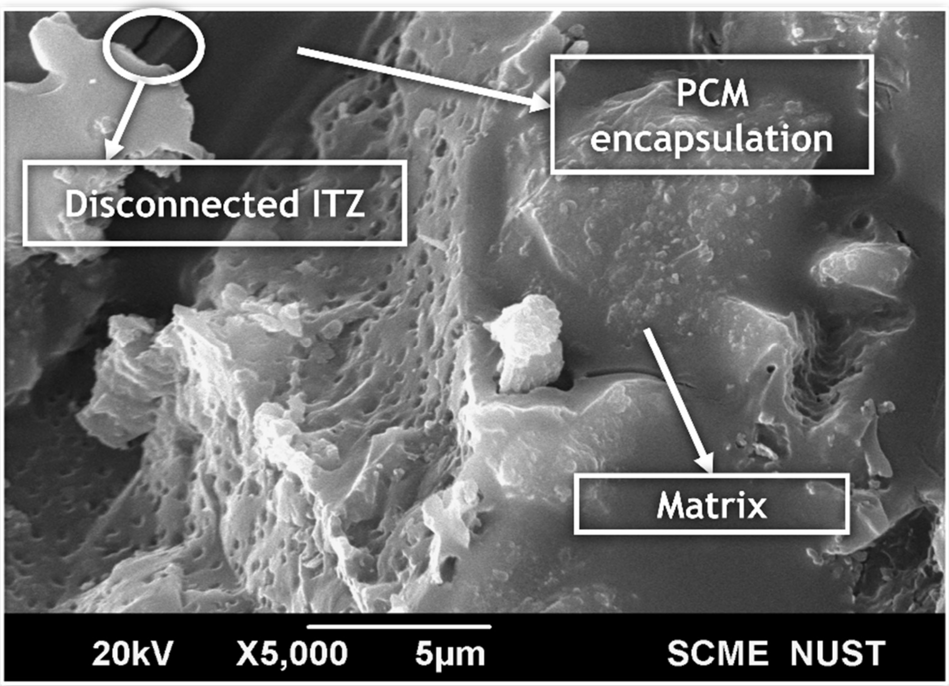

Fig. 24: SEM image showing concrete matrix, PCM encapsulation and its ITZ

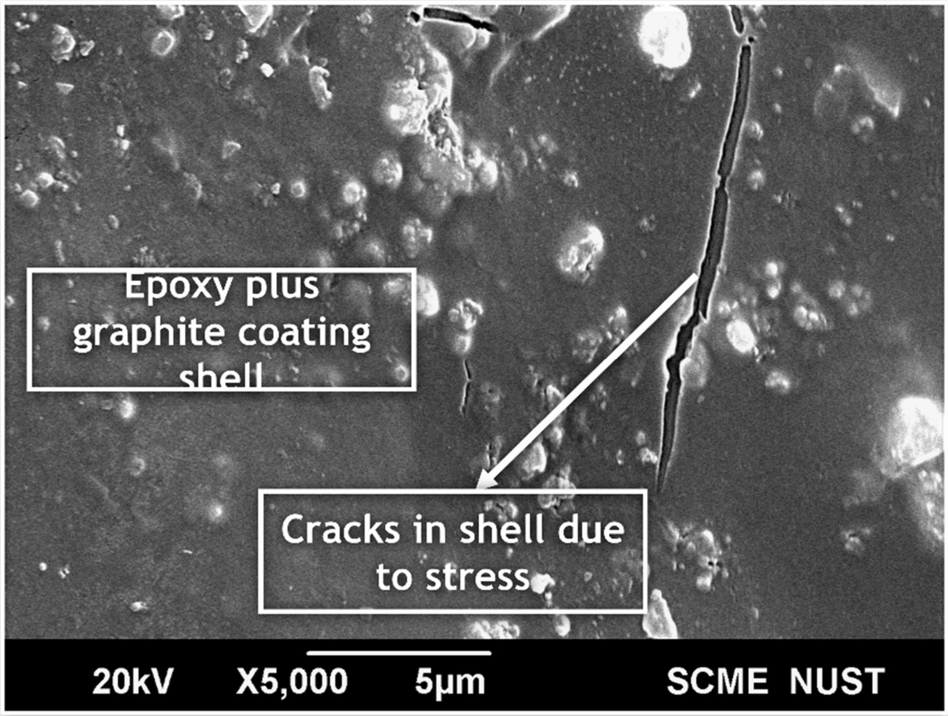

Fig. 25: SEM image showing close-up of epoxy and graphite coating

4.4 BIM Energy Analysis:

From the application point of view of our PCM incorporated concrete, we modelled and calculated cooling loads of our study site having concrete with 20% PCM in its envelope elements. Those cooling loads were then compared with cooling loads of the same study site with modified insulation techniques or no insulation technique at all. The obtained cooling loads are depicted in the form of bar graph in fig. 26.

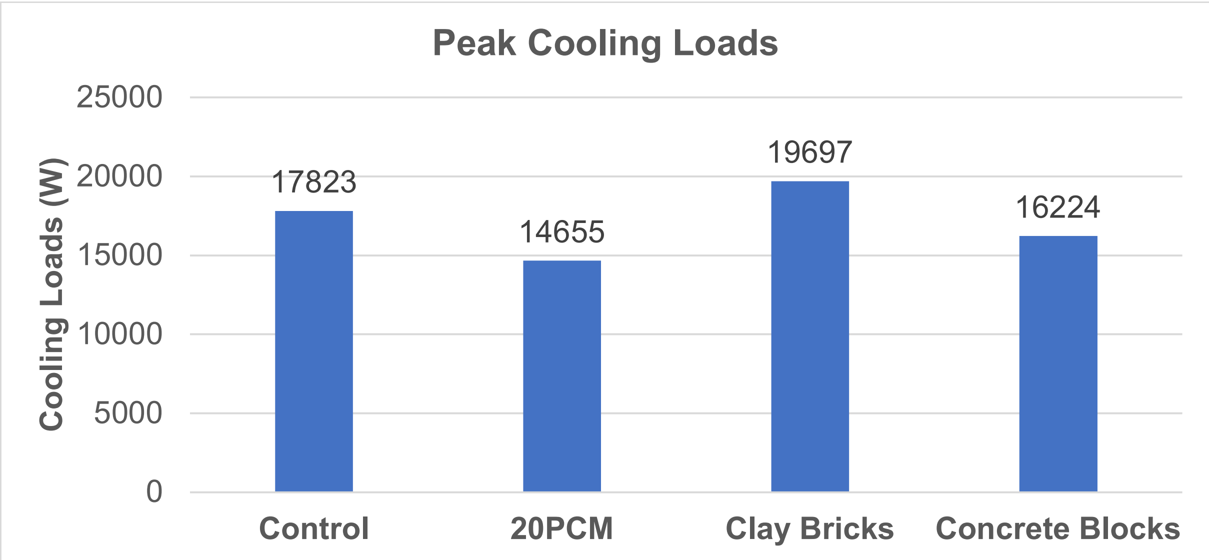

Fig. 26: Cooling loads of different building envelopes

It can be clearly observed that the energy required to cool the indoor temperature of the site having 20% PCM incorporated concrete is much less than the energy required for the same purpose in the site made with control concrete. The difference is found to be almost 18%.

CHAPTER 5

CONCLUSIONS

5.1 Conclusions Derived

We developed a novel technique of incorporating PCM in concrete using gelatin encapsulation coated with epoxy resin and graphite powder. Therefore, the focus of the research was to analyze the thermal and mechanical properties of this newly developed concrete as well as to study its microstructure. The application of this new PCM incorporated concrete was also investigated. Based on the test results, following conclusions can be drawn:

- The compressive strengths of PCM incorporated concrete decrease as the percentage of PCM is increased. PCM incorporated concrete using novel, epoxy and graphite coated encapsulation cannot be used for structural applications, as its strength is less than the minimum strength of structural concrete described by ACI 318, which is 17 MPa. However, this newly developed concrete can still be used for non – structural elements in buildings in the form of blocks etc.
- From the thermal conductivity and thermal resistivity tests, it can be concluded that incorporation of PCM using epoxy and graphite coated gelatin encapsulation improves the thermal properties and make it suitable for latent thermal energy storage purposes in the building
- Since there is no major difference between the compressive strengths of samples measured at 25 °C and at 80 °C, this means that the PCM inside the epoxy and graphite coated gelatin capsules is not leaking when converted to liquid state, proving that this novel encapsulation technique is effective and useful.
- Silica fume can be added to improve the strength of PCM incorporated concrete by densifying the concrete matrix.
- From SEM images, it was found that epoxy and graphite coated gelatin encapsulation do not form a well-integrated ITZ due to difference in intrinsic properties of the encapsulation and the matrix. The images confirm the reason behind the decrease in strength of this concrete.

Although, decrease in strength is observed but notable improvements in thermal properties and simulations show PCM incorporated concrete with epoxy coated gelatine encapsulation is breakthrough research in the field of affordable thermally efficient concrete

CHAPTER 6

APPLICATION & SIGNIFICANCE

6.1 Advantages and Significance

Data from Pakistan Meteorological Department helps us establish the fact that our PCM incorporated concrete will be beneficial in areas of Pakistan that experience very high temperatures in summers. Fig. 27 (a) to 27 (f) shows the distribution of mean daily maximum temperatures in Pakistan from the month of April to September in the year 2021. From the figures we can derive that from April to September, almost half of Pakistan experiences temperatures greater than 39 °C with maximum mean temperatures reaching even over 45 °C, which is more than the melting onset temperature of paraffin wax that is used as the PCM. The above conclusions from the Revit models are evidence that our novel technique of PCM incorporation in concrete increases the latent thermal storage capacity of the buildings and lead to low indoor temperatures and decreased temperature fluctuations. This leads to lower energy consumption for HVAC purposes and the peak loads are also shifted. As a rough estimate, over 70 million people can become beneficiary of this type of concrete. The practicality of our PCM incorporated concrete is also increased due to the fact that all the material used, from paraffin wax to gelatin capsules and epoxy resin, are reasonably priced in the local market and within the reach of a common man. Our technique of PCM incorporation in concrete only slightly increase its production cost. However, if this slight increase is weighted against the benefits that it is providing, it will be safe to say that PCM incorporation using gelatin capsules coated with epoxy and graphite powder will be successful development, of course after some deeper research and investigations.

Issue may arise in regions where daily maximum temperatures in summers are not very high. This is because the paraffin wax that we used was melting at very high temperatures. However, to make it suitable for regions having mild summers the PCM incorporated concrete can be made in the exact same way as mentioned above. Just the gelatin capsules should be filled with paraffin wax having lower melting points that is suitable for such regions.

Our innovative technique of PCM incorporation in concrete using epoxy and graphite coated gelatin capsules is also very simple which makes it easy to be used by labor. No special education or skills would be required by labor to cast-in-situ and place this PCM incorporated concrete. Thereby, increasing its usefulness and practicality.

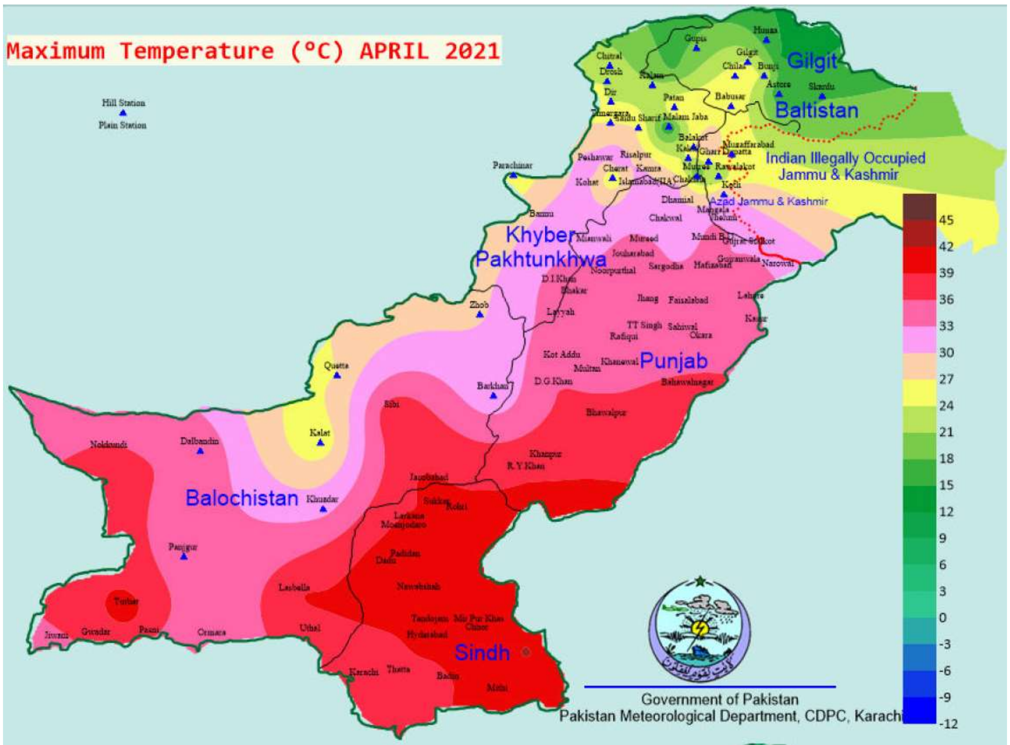

Fig. 27 (a): Mean maximum monthly temperature distribution in Pakistan

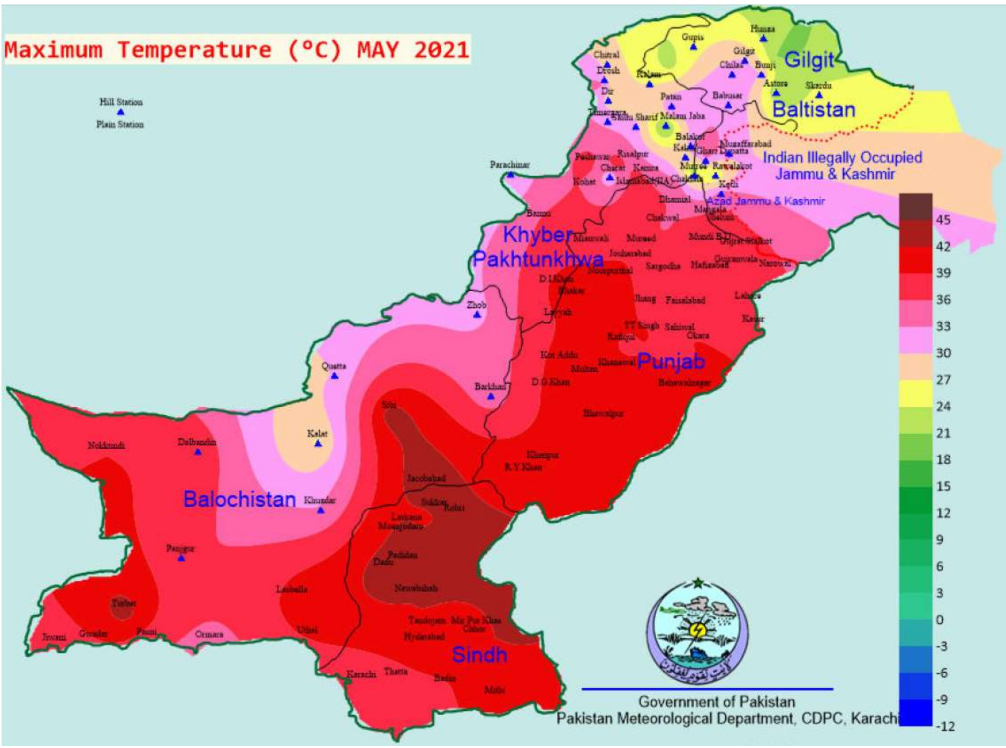

Fig. 27 (b): Mean maximum monthly temperature distribution in Pakistan

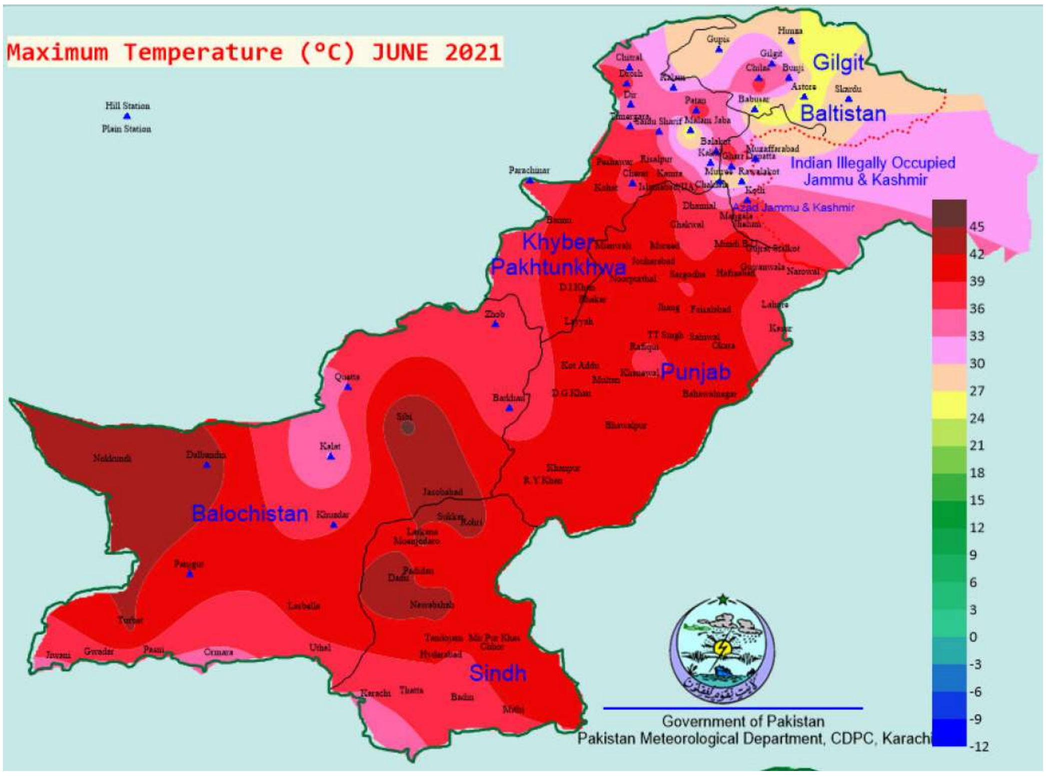

Fig. 27 (c): Mean maximum monthly temperature distribution in Pakistan

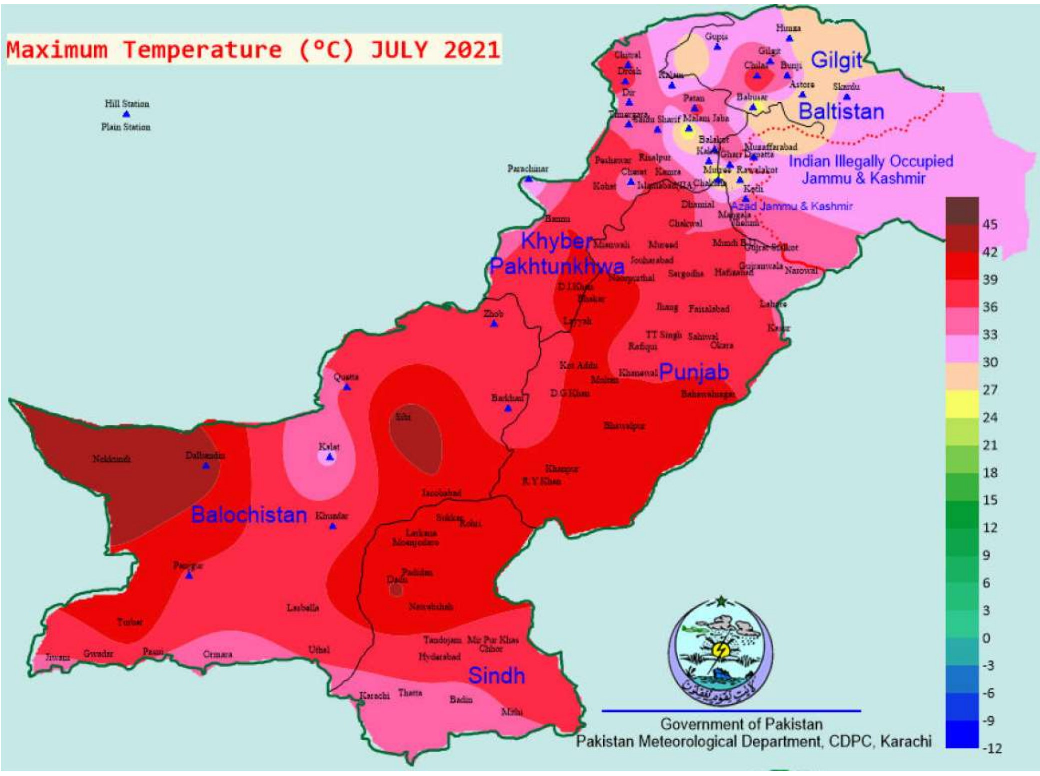

Fig. 27 (d): Mean maximum monthly temperature distribution in Pakistan

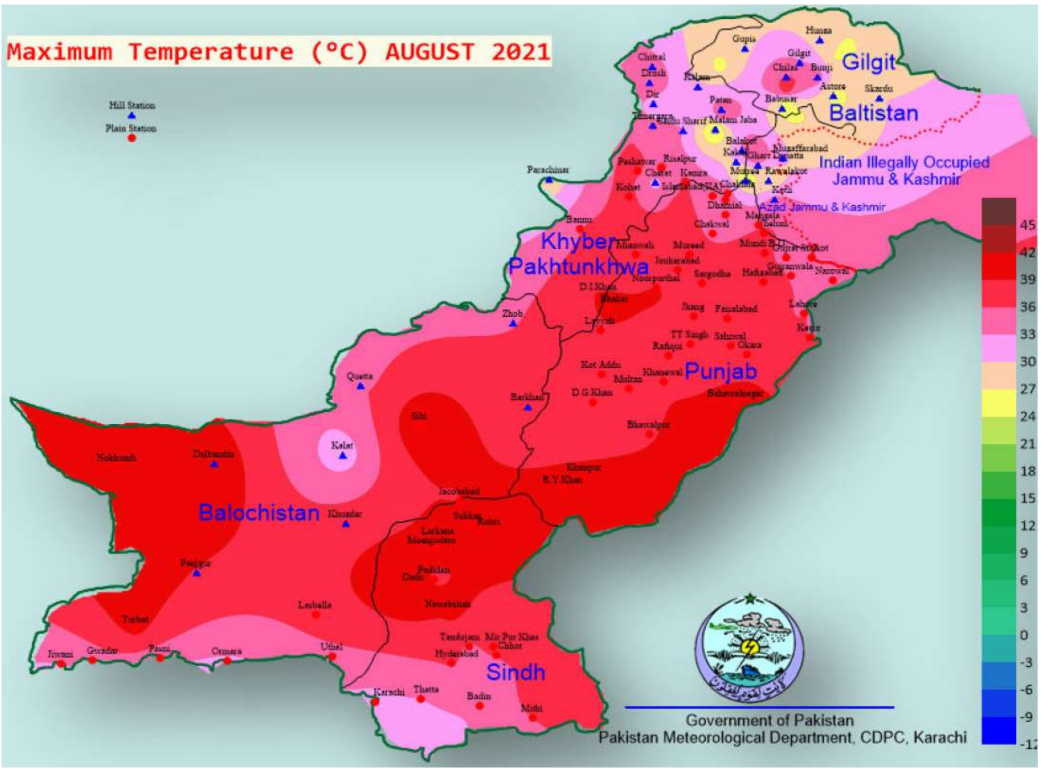

Fig. 27 (e): Mean maximum monthly temperature distribution in Pakistan

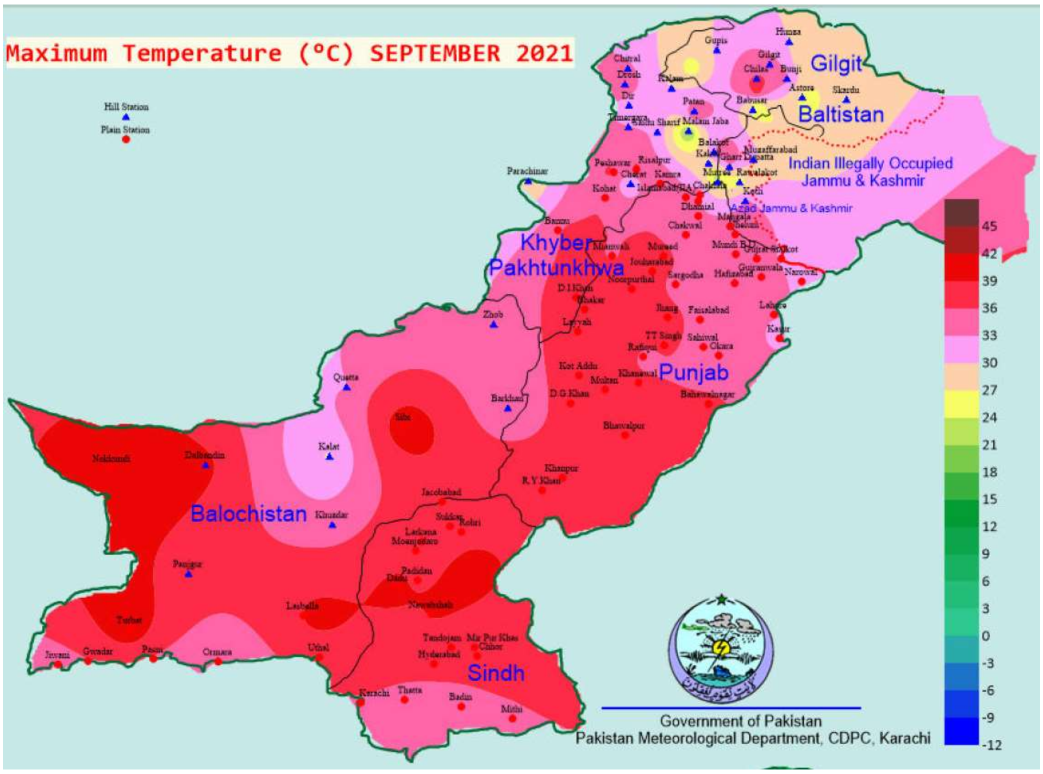

Fig. 27 (f): Mean maximum monthly temperature distribution in Pakistan

CHAPTER 7

FURTHER PROSPECTS

7.1 Future Research Areas

Research and investigations can be performed in following areas in order to establish the efficacy of PCM incorporated using epoxy and graphite coated gelatin encapsulation:

- Techniques to improve the mechanical properties of this concrete
- Enhancement of fire resistance properties, as paraffin wax is an organic material which catches fire easily
- Researchers should explore the reliability of this encapsulation technique under cyclic thermal loads

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