# EXPERIMENTAL INVESTIGATION ON THE INFLUENCE OF PHASE CHANGE MATERIAL (PCM) ON THE PROPERTIES OF CEMENT MORTAR

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**ABSTRACT.** This paper investigates the hydration properties, compressive strength, durability properties and microstructure characteristics of cement mortar integrated with inorganic salt based phase change materials (Sodium Carbonate). Experimental studies were carried out by incorporating PCM into cement mortar with 1%, 2% and 3% by weight of cement. Hydration properties such as initial and final setting times, temperature variations with the passage of time were measured and compressive strength for four curing ages i.e., 3, 7, 28 and 56 days was determined. Durability aspects were studied by means of rapid chloride penetration test (RCPT) at the end of 28 and 56 days of curing ages and also through chemical shrinkage at 7, 14 and 28 days of curing period. SEM studies were also conducted to understand the microstructure of PCM incorporated cement mortar. The outcomes of the present study depicted that presence of PCMs in the cement paste has reduced the hydration temperature with the increase in percentage of PCMs. However, reduced trend of compressive strength in comparison to that of control mortar was observed. The obtained RCPT values inferred that mix inclusive of PCMs was observed to have higher rate of charges passed compared to that of control mortar. However, with respect to the measurement of shrinkage PCM mix showed the less shrinkage as compared to the control mix.

**Keywords:** Phase change material, Cement mortar, Compressive strength, Durability, Shrinkage.

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# INTRODUCTION

The energy efficiency of buildings is considered as today's most important intent for guiding principles of energy at national - international levels (Lombard et al, 2009). The chief portion of worlds energy produced is consumed for buildings and its comforts and so promoting energy efficient sustainable buildings is the need of an hour. This can be accomplished through reducing the level of energy consumption and improving the energy conservation level in buildings (Soares et al, 2013).

The effectual approach of energy conservation can be achieved by thermal storage with the potential incorporation of latent heat storage in building constituents and structures (Kiviste and Lindberg., 2014). At this juncture a significant latent heat storage material known as phase change materials (PCMs) are gaining attention owing to its momentous thermal energy storing ability within a lesser volume (Sharma et al., 2009). PCMs application in the constituents of buildings mostly like walls, ceiling and floors has a capacity to arrest the solar energy and enhances the comfort level of human life by sustaining the desired temperature for a longer period of time (Stovall et al., 1995 and Stritih., 2003), making it as one of the utmost promising technologies in evolving energy efficient buildings.

PCMs are the materials with an ability to absorb and release heat when the material changes from one phase to another i.e. solid-liquid phase (Kanimozhi et al., 2017). Organic and inorganic PCM's are the two forms of PCMs which are mostly preferred for building applications, where most of the PCMs has a capability to retain its latent heat without any changes in physical or chemical properties even after thousands of cycles (Ling et al, 2013). Literature says typically solid and dense materials like concrete possesses good thermal storage capacity (Cao et al., 2017), therefore if concrete material having an excellent thermal insulation and fire resistant properties is choosen as a medium for PCMs it can lead to an increase in overall energy storage capacity.

The integration of PCM in cementitious system functions as both heating and cooling arrangement for a building by its repeated cycles of interchanging sequential process of melting during daytime by absorbing surplus amount of heat and process of solidifying in cooler nights by releasing back the heat into the environment (Zhang et al, 2013). The integration of PCM in cementitious system may have a risk of leakage and thus can change the concrete properties. Hence it is important to select an appropriate PCM and its inclusion methods in cementitious system and the present investigation is framed to study the effect of inorganic phase change materials (PCM) i.e sodium carbonate on different properties such as physical, mechanical, durability and microstructure of cement mortar. For this PCM was incorporated into mortar in 1%, 2% and 3% by total weight of cement and the above properties were studied.

# EXPERIMENTAL STUDY

## Material and its properties

The materials used in the present study are ordinary Portland cement (OPC) from ACC cements of grade 53, the required tests for assessing the properties of cement were carried out according to IS 4031. The locally available river sand conforming to zone II passing through

4.75mm sieve was used. The properties of sand such as fineness modulus and specific gravity were determined as per IS 2386-1963 (Part I). The PCM used in this study is Sodium carbonate

The properties of cement and fine aggregate used in the experimental study are presented in Table 1 and the Figure 1 depicts the particle-size distribution characteristics of fine-aggregates (using sieve analysis). Whereas, the properties of PCMs adopted for the present investigation as per manufactures report is shown in Table 2.

Table 1 Properties of the cement and fine aggregate used in the study

| MATERIAL USED | G | FINENESS ($M^2$/KG) | SC (%) | SETTING TIME (MIN) | | BULK DENSITY ($KG/M^3$) | | FM | WATER ABSORPTION (%) |
|---|---|---|---|---|---|---|---|---|---|
| | | | | IST | FST | Loose | Dense | | |
| OPC-53 | 3.15 | 300 | 32 | 110 | 170 | - | - | - | - |
| Fine Aggregate | 2.56 | - | - | - | - | 1524 | 1780 | 3.14 | 1 |

*G- Specific Gravity, SC- Standard Consistency, FM-Fineness Modulus

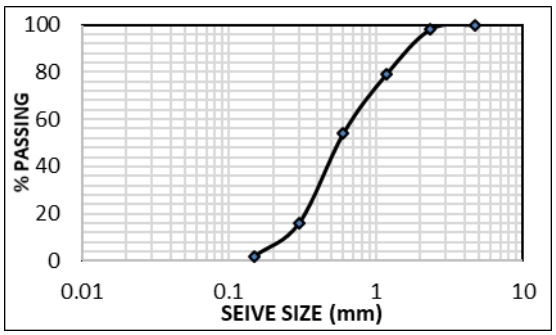



Figure 1 Particle-size distribution characteristics of the fine aggregates used in the study

Table 2 Physical properties of PCMs used in the study

| TYPE OF PCM | APPEARANCE | ODOUR | DENSITY ($G/CM^3$) | SOLUBILITY |
|---|---|---|---|---|
| Sodium carbonate | White powder | - | 2.53 | 220 g/l |

**Mortar mix proportion**

The cement mortar was designed according to IS 4031 (Part 6):1988 specifications. Mortar was produced by incorporating different types of PCMs into it. The details of mortar mix proportions are presented in Table 3.

Table ***Error! No text of specified style in document.*** Mix proportion for different mortar mixes

| PCM REPLACEMENTS | MIX DESIGNATIONS | CEMENT ($KG/M^3$) | PCM (%) | FINE AGGREGATE ($KG/M^3$) | WATER (ML) |
|---|---|---|---|---|---|
| Control (0% PCM) | C | 568.4 | 0 | 1705.1 | 88 |
| 1% Sodium carbonate | S1 | 562.7 | 1 | 1705.1 | 88 |
| 2% Sodium carbonate | S2 | 557.0 | 2 | 1705.1 | 88 |
| 3% Sodium carbonate | S3 | 545.6 | 3 | 1705.1 | 88 |

**Methodology**

Cubes of size 70.6 mm and cylinders of 100mm diameter and 50mm height were cast for compressive strength test and RCPT respectively for mortar mixes. While, for performing setting time and chemical shrinkage tests, cement paste was mixed with certain amount of PCM and water suitably. At the same time thorough hand mixing of materials was adopted for the production of uniform mortar and the desired compaction of the mortar was achieved with the help of a table vibrator. These compacted samples were stored in a humid condition and were de-molded after 24 h of storage, subsequently these samples were stored in a submerged condition (curing tank). At the end of 3, 7, 28 and 56 days of curing period, samples were taken out from the curing tank, after it achieves the saturated surface dry condition, were tested for their compressive strength by means of compression testing machine.

The rapid chloride ion permeability tests were conducted on three identical mortar specimens of size 100mm diameter and 50mm height, corresponding to 28 and 56 days of curing. The cylindrical sample surface was layered with the epoxy to reduce the evaporation loss. A rapid chloride ion permeability test (RCPT) apparatus was employed for conducting these tests, as per ASTM C-1202. The cylindrical samples were subjected to 60 V, for 6 h, and the permeability was determined in terms of the charge passed through it (in Coulomb). The average of the three values obtained from three identical cores has been considered in the study.

Initial setting time and final setting time were measured as per IS: 4031-Part-5 1988. Initial setting time is measured as interval between the phases when water is added to cement and phase at which 1 mm needle fails to penetrate the cement paste placed in vicat's mould by 5 mm to 7 mm from the bottom. Whereas, final setting time was measured as interval between the phases when water is added to cement and at which 5mm needle does not make any indentation on cement paste placed in vicat's mould. Along with this temperature was also measured with the aid of mercury thermometer from the mixing stage of cement with PCM till final setting time to understand the temperature variation during hydration of cement paste after incorporating PCM.

Chemical shrinkage was determined by means of dilatometry method as specified by ASTM C 1608 standards. The erlenmeyer flask with cork and pipette arrangement was set up and cement paste was placed in the flask to about 7mm-10mm thickness then the rest of the flask is filled with water till the zero pipette level, at last paraffin oil was placed in the top of the

pipette to minimize water evaporation from the tube during the testing period. The drop in water level in a hydrating cement paste was periodically recorded after 7, 14 and 28 days and the chemical shrinkage of cement paste was considered as the change of water level in the pipette. Figure 2 represents the experimental set up for measuring chemical shrinkage in accordance to ASTM C 1608 standards

SEM analysis in the present investigation was studied using scanning electron microscope from Jeol (JSM-638OLA) with a magnification of 20000X and spatial resolution of 50 to 100 nm. The crushed small chunk samples were collected (from its inner core) from compressive strength tested mortar cubes and subjected to drying via hot air oven at 110$^{o}$C so as to expel the moisture content. Then these samples were placed in sample holders and gold sputtering was done to provide an even coating was on the specimen surface.

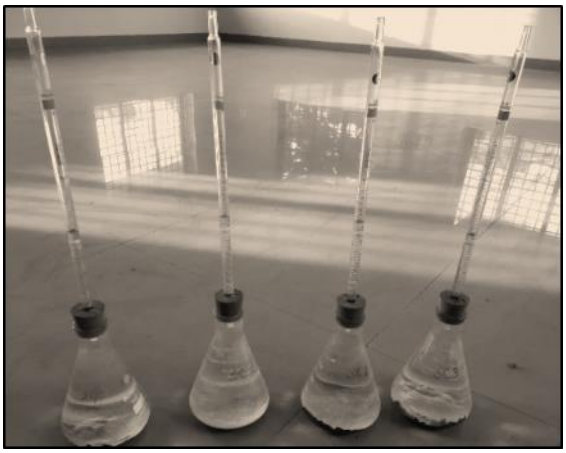


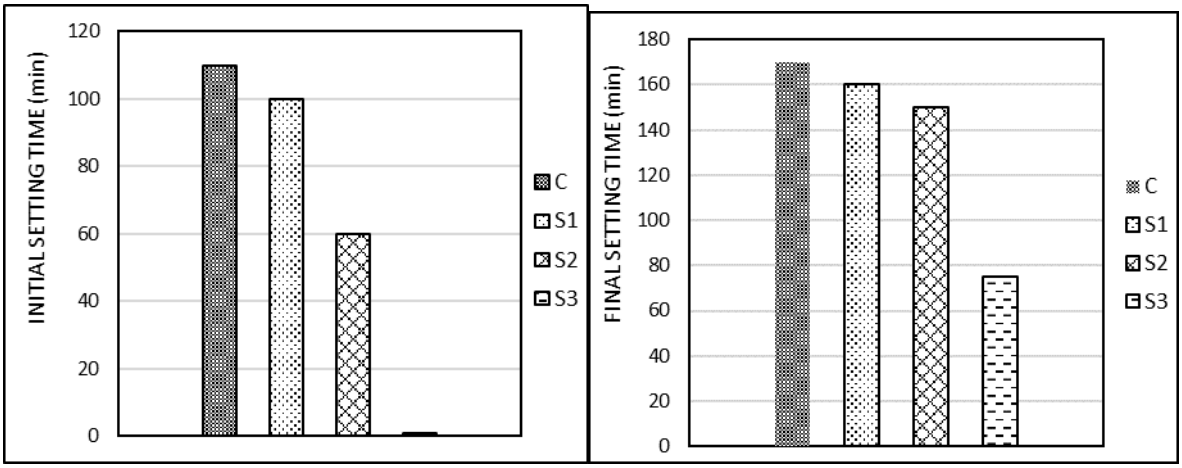
Figure 2 Experimental set up for measuring chemical shrinkage (as per ASTM C 1608 standards)

## RESULTS AND DISCUSSIONS

### Setting time and temperature study

The results of the initial and final setting time on control cement paste and PCM incorporated cement paste is presented in Figure 3a and 3b. It can be noted from the presented graphical representation that on increasing the PCM content, reduced the setting time around 55 and 45% respectively compared to normal cement paste. The accelerating setting time effect of cement paste was observed more significant with 2 and 3% addition of PCM content.

The results of surface temperature variation during setting time of control cement paste and PCM incorporated cement paste is presented in the Figure 4. It can be noted from the presented plot that control cement paste reached a peak temperature of 34.5$^{\circ}$C at 130min. The cement paste mix incorporated with 1% PCM reduces the peak temperature by 1 $^{\circ}$C in comparison to control cement paste and mainted contant temperature. While, 2% PCM showed 50min early peak temperature and then reduces the temperature. The cement paste mix with 3% PCM reduces the temperature by 2 $^{\circ}$C and then mainted the contant temperature.




(a) (b)

Figure 3 Initial (a) and final setting time (b) of cement pastes with different dosages of different PCMs

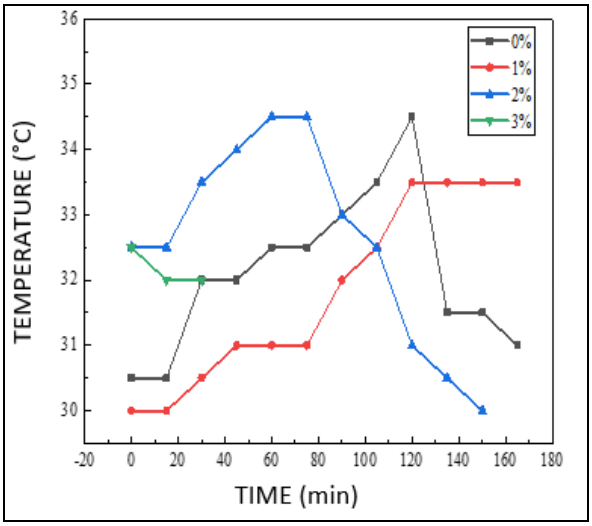



Figure 4 Temperature variations of different cement pastes measured upto final setting time after the start of mixing with water as per ASTM C 187

## Compressive strength

Compressive strength of cement mortar specimens were determined corresponding to different curing ages and results are presented in Figure 5. It can be noted from the graphical representation that salt based PCM incorporated cement mortar showed lower compressive strengths compared to control mortar. This may be attributed to the presence of PCM content inside the pores of cement mortar is weakened the mechanical strength. According to Sharifi and Sakulich (2015), if PCM is added directly to the concrete, it will interfere with hydration reaction and therefore the compressive strength of concrete will be reduced.

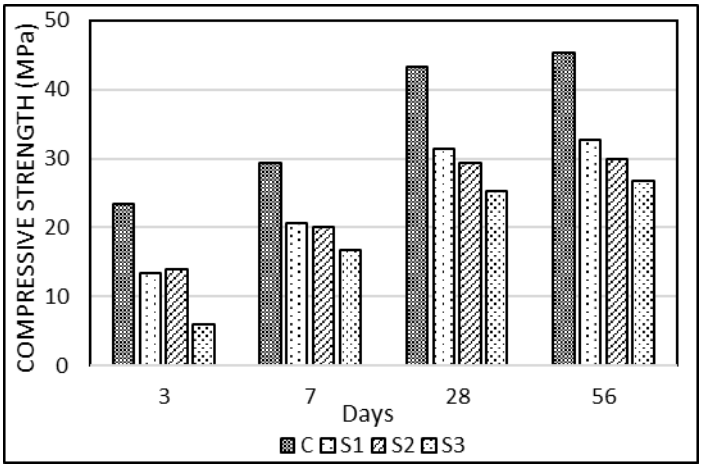
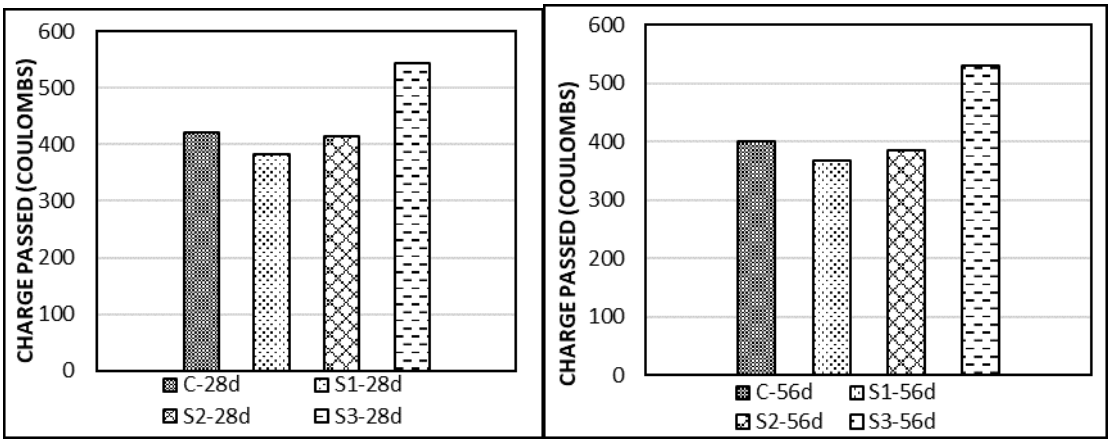



Figure 5 Compressive behaviour of different mortars incorporated by various PCMs

**Rapid chloride-ion permeability test (RCPT)**

The resistance to chloride ion penetration test results of 28 and 56 curing ages are depicted in Figure 6. It can be noted that, as the curing ages increased the chloride ion penetrability decreased for both control and PCM based cement mortar. It was also observed that, as the dosage of PCM in cement mortar increased the higher rate of charges passed compared to that of control mortar. This may be attributed to the reduction of cement content in the PCM mortar mixes and increased porosity. However, it was also observed that, 1% and 2% of sodium carbonate PCM based cement mortar showed lesser permeability compared to control cement mortar the conductivity, which entails that this mix is may be more durable than the control mortar. The chloride ion permeability of all the mixes of PCM are within the range of 100 to 1000 coulombs. As per ASTM C 1202 the value of chloride ion permeability of specimens within 100 to 1000 coulombs has lower permeability and hence they are very durable.




(a) (b)

Figure 6 Rapid Chloride Ion Permeability Test results a) 28day cured samples b) 56day cured sample.

## Chemical shrinkage test

Figure 7 depicts the results of chemical shrinkage for control and PCM added cement pastes at the end of 7, 14 and 28days. It was observed that chemical shrinkage behavior of cement pastes tested was well reflected with its cement hydration process. At the age of 7 days the chemical shrinkage values of both control and PCM based cement pastes were found to be low. This can be attribute to its limited cement hydration and it was observed to be increased with accelerated hydration process. It was observed that presence of PCM in cement paste lowered the chemical shrinkage values compared to that of control cement paste which may be attributed to the lowered rate of cement hydration. The large difference between chemilcal shrinkage values at early age and later age was observed which may be attributed to its slow rate of hydration during early ages. Thus, simply using chemical shrinkage values measured at very early age for internal curing concrete may lead in underestimation of the amount of internal curing materials needed for shrinkage reduction (Yodsudjai and Wang, 2013).

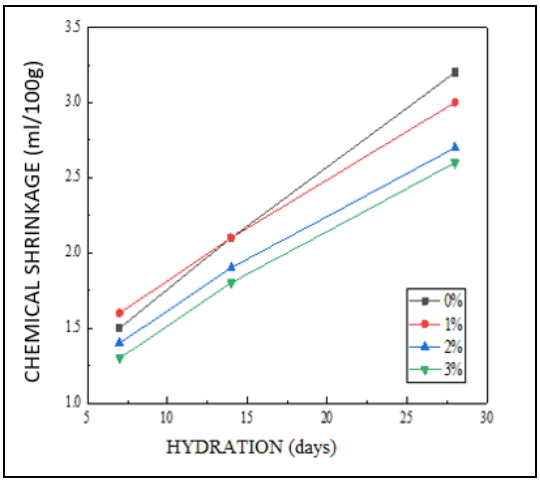



Figure 7 Chemical shrinkage results for various mixes

## Scanning electron microscopic (SEM) –microstructural study

The microstructure study was conducted on all the samples by means of SEM imaging and the images are shown in Figures 8-9.

For hydrated cement mortar, capillary pores are the darkest, calcium hydroxide (CH) is light grey, calcium silicate hydrate (CSH) and other (aluminate) hydration products are dark grey (Bentz and Stutzman, 2006).

Figure 8 depicts the SEM microstructure images of control cement mortar for the curing age of 28 day. It can be noted from the figure that at the end of 28days the formation of CSH and CH by the hydration process of $C_3S$ and $C_2S$.

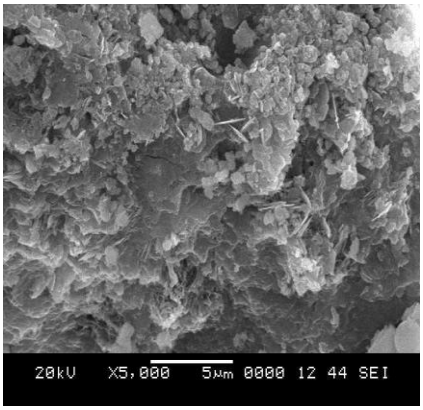
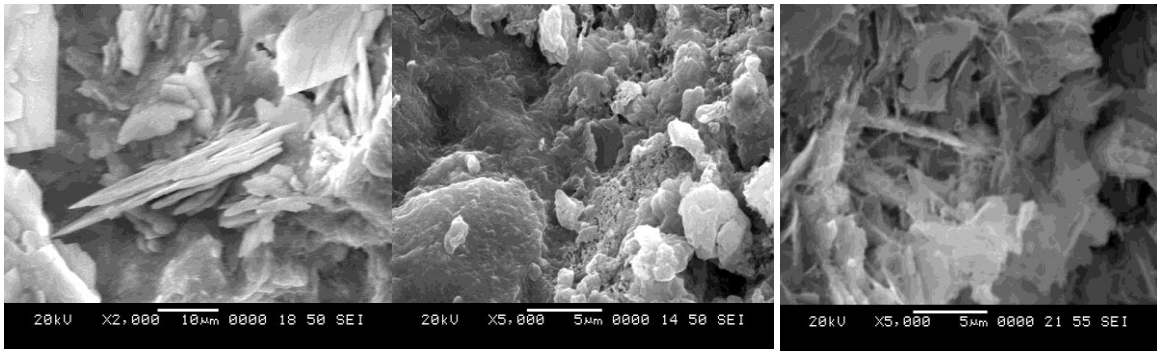



Figure 8 Microstructure images of control cement mortar at the age of 28 day




S1 S2 S3

Figure 9 Morphology of PCM based mortar at the age of 28 day

Figure 9 demonstrates the morphology of PCM based cement mortars with 1%, 2% and 3% by weight of cement at the curing age of 28 days. It can be observed the higher concentration of plate like CH crystals with lesser amount of CSH formations in all the mixes of sodium carbonate. It can also be noted that S3 mix indicates the dispersed needle like structures in the bulk of CH crystals.

## CONCLUSIONS

It can be concluded from the study that setting time was observed to be decreasing in case of S1, S2 and S3 with increase in dosage compared to control mortar acting as an accelerator. Surface temperature evolution was reduced in case of 1% PCM in cement paste. Compressive strength of mortar reduced drastically with the addition of sodium carbonate PCM. RCPT values of both control PCM specimens are within 100 - 1000 coulombs, this showed very low permeability. Decreased rate of chloride ion penetrability was seen with the increased curing ages. PCM added cement paste showed lower chemical shrinkage. Average chemical shrinkage values of all the cement pastes at 7 days were only 30-55% of those at 28 days. The

PCM added specimens showed the more plate like calcium hydroxide structure microstructure compared to that of control.

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