

Evaluating the Use of Phase Change Materials in Concrete Pavement to Melt Ice and Snow

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Abstract: This paper investigates the potential use of phase change materials (PCM) in concrete pavements to store heat, which can be used to reduce ice formation and snow accumulation on the surface of the concrete pavement. The thermal properties of the PCMs are evaluated using a low-temperature differential scanning calorimeter (LT-DSC) while a longitudinal guarded comparative calorimeter (LGCC) is used to evaluate the thermal response of cementitious mortar containing the PCM. Paraffin oil (petroleum based) and methyl laurate (vegetable based) were selected as PCMs since they have high enthalpies of fusion (~130–170 J/g) and have desirable freezing temperatures (~2–3°C) during the liquid to solid phase transformation. Two approaches were used to place the PCM in the mortar specimens: (1) placing the PCM in lightweight aggregate (LWA) in mortar and (2) placing the PCM in an embedded tube that is placed in mortar. The durability and stability of the PCMs in the cementitious system were studied by monitoring the change in enthalpy of fusion, mass loss, pulse velocity, and compressive strength. When the PCM was placed in the mortar specimen using LWA, the paraffin oil can release a considerable amount of heat during phase transformation, which can be used to melt ice and snow. However, it was observed that the methyl laurate reacts with the cementitious matrix, causing damage to the mortar. Both paraffin oil and methyl laurate showed promising performance to melt ice and snow when the PCM was placed in an embedded tube in the mortar specimen. **DOI: 10.1061/(ASCE)MT.1943-5533.0001439.** © 2015 American Society of Civil Engineers.

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Introduction

Phase change materials (PCMs) are organic, inorganic, or eutectic materials that can produce a considerable amount of energy during their phase transformation from liquid to solid (Ling and Poon 2013; Regin et al. 2008). The energy that is required to change the phase of a substance is known as latent heat or heat of fusion. PCMs have the potential to be used to store thermal energy from ambient, solar, or applied heat sources (Kalnæs and Jelle 2015;

Ling and Poon 2013). Organic PCMs are classified as paraffin and nonparaffin (Kalnæs and Jelle 2015). Most organic PCMs do not require undercooling to provide nucleation during freezing, and are generally noncorrosive, which are both desirable characteristics for concrete pavement applications (Abhat 1983; Hawes et al. 1992; Regin et al. 2008; Sharma et al. 2009). Inorganic PCMs are classified as salt hydrates and metallic. Metallic PCMs are not within the desired temperature range for building applications (Kalnæs and Jelle 2015). The salt hydrates may corrode metals, need to be supercooled to induce freezing due to poor nucleating properties, experience phase segregation and thermal properties alterations after multiple freeze-thaw cycles due to release/gain of water, and may interact with the cementitious binder (Abhat 1983; Kalnæs and Jelle 2015; Ling and Poon 2013; Shi et al. 2011; Sutter et al. 2008). Eutectics of organic and inorganic compounds are a minimum-melting composition of two or more components that freeze and melt congruently forming mixtures of component crystals (Sharma et al. 2009) that can allow for the design of a particular mixture of organic or inorganic PCMs to create optimum operating temperatures in concrete pavement applications (Kalnæs and Jelle 2015; Karaipekli and San 2008; Lane 1989; Sharma et al. 2009).

PCMs have been conventionally used in solar water-heating systems, green houses, building walls and floors, window shutters, concrete to mitigate thermal cracking, and mass concrete (Abhat 1983; Choi et al. 2014; Fernandes et al. 2014; Hembade et al. 2014; Kuznik and Virgone 2009; Regin et al. 2008; Sharma et al. 2009; Whiffen and Riffat 2012). PCMs have been proposed to be used in the concrete infrastructure to alter anti-icing practices in concrete pavements (Bentz and Turpin 2007; Ling and Poon 2013; Liston et al. 2014; Sakulich and Bentz 2012b, a). Due to the PCM's high heat of fusion, the stored energy can be released during cooling (i.e., during a freezing/icing event), thereby delaying or

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preventing ice formation (Liston et al. 2014; Sakulich and Bentz 2012b, a). This can help to eliminate or decrease the deicing salt demand to melt ice or snow on the surface of concrete pavements. Deicing salts can cause premature deteriorations in concrete pavement and any decrease in salt usage can improve the durability of concrete (Farnam et al. 2014a, b, 2015a, c, d; Qian et al. 2014).

Fig. 1 shows a schematic of using PCM in concrete pavement to melt ice and snow (Memon et al. 2015; Sakulich and Bentz 2012b, a). Using PCMs in the concrete infrastructure requires specific thermal, physical, and chemical properties that are compatible with the concrete. The desirable thermal properties of a PCM for successful use in the concrete pavement to melt ice and snow include a phase transition temperature slightly above 0°C , high latent heat of fusion, high specific heat (heat capacity), and high thermal conductivity (Bentz and Turpin 2007; Ling and Poon 2013; Sakulich and Bentz 2012a, b). The PCM's physical properties should include high density, small volume change between phases, and low vapor pressure. PCM's chemical properties should include stability, compatibility with concrete, nontoxic, and non-flammable (Hawes et al. 1992; Liston et al. 2014). PCMs that are economical, commercially available, and environmentally friendly are also important.

The efficient use of PCMs in concrete pavement application requires an efficient method to place PCMs into the concrete pavement without extensive changes to the construction practice. Different methods have been proposed to use PCM in concrete elements (Hawes and Feldman 1992; Hawes et al. 1992; Sakulich and Bentz 2012b). Fig. 2 illustrates schematics of three methods of incorporating a PCM into concrete. Fig. 2(a) indicates filling tubes with PCM to raise the latent heat of the concrete-tube system, thereby preventing freezing of ice or snow accumulation on the concrete pavement (Pasupathy and Velraj 2006). Fig. 2(b) illustrates incorporating the PCM directly using lightweight aggregate (LWA) or encapsulated PCM in the concrete pavement (Chen et al. 2013; Sakulich and Bentz 2012b; Tyagi et al. 2011). Fig. 2(c) depicts the inclusion of PCM within the concrete pores and voids on the surface of the pavement; this method has also been used for applying sealants to pavement surface (Jones et al. 2013).

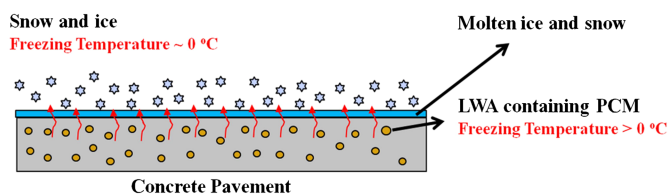


Fig. 1. Schematic of using PCM in concrete pavement to melt ice and snow using lightweight aggregate (LWA)

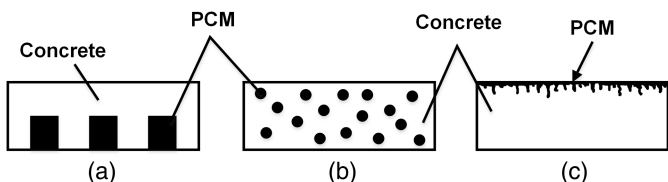


Fig. 2. Schematic illustrating three methods to incorporate PCM into concrete: (a) using pipes of PCM; (b) using particles containing PCM; (c) filling concrete surface voids via PCM absorption

Previous researchers (Bentz and Turpin 2007; Ling and Poon 2013; Liston et al. 2014; Sakulich and Bentz 2012b, a; Stoll et al. 1996) have shown that PCMs have the potential to change deicing and snow removal practice in concrete pavement and that they can be used to decrease the number of freeze-thaw cycles in concrete pavement, thereby increasing concrete freeze-thaw durability. Previous studies have primarily characterized the thermal properties of PCMs themselves for concrete pavement applications using a low-temperature differential scanning calorimeter (Abhat 1983; Bentz and Turpin 2007; Liston et al. 2014) and predicted the PCM concrete performance to prevent ice formation by means of a numerical simulation (Sakulich and Bentz 2012b). In a few cases, some durability and physical stability aspects were addressed (Hawes et al. 1992; Sakulich and Bentz 2012a).

First, this research uses a longitudinal guarded comparative calorimeter (LGCC) to characterize the thermal response of an entire mortar-PCM system to melt ice and snow. Second, this research evaluates the physical and durability performance of mortar containing PCMs. The main objective of this research is to determine whether PCMs are effective enough on ice and snow melting when they are used inside a cementitious material. It is essential that potential PCMs for deicing applications also show promising performance when they are used in concrete in terms of released heat, desired phase transformation temperature, chemical and physical stability, and concrete durability.

Experimental Program

Mortar specimens were prepared containing paraffin oil and methyl laurate, as PCM. Two approaches were selected to incorporate PCM into the mortar specimens: (1) using lightweight aggregate (LWA) and (2) using an embedded tube.

A low-temperature differential scanning calorimeter (LT-DSC) (Farnam et al. 2014a; Villani et al. 2015), TA Q2000 with an operating range of -90 to 550°C , was used to determine the thermal properties of the PCMs. A longitudinal guarded comparative calorimeter (LGCC) (Farnam et al. 2014b) was also used to evaluate the thermal response of mortar specimens containing PCM during cooling and heating. The durability and stability of PCM in mortar specimens were determined by (1) evaluation of the chemical reactions between PCM and mortar constituents using LT-DSC, (2) evaluation of physical properties of the mortar using ultrasonic pulse velocity and compressive strength measurements, and (3) determination of PCM mass loss over time.

Materials

Two types of ordinary portland cement (OPC) were used: Type I and Type V with a Blaine fineness of 375 and $316\text{ m}^2/\text{kg}$, respectively. The chemical composition of the cements, as reported by the manufacturers, is indicated in Table 1. An expanded shale LWA was used in this study. These aggregates were manufactured by Buildex in their Marquette plant with a fineness modulus of 2.94 and a specific density of 1.5 (Castro et al. 2011; Miller et al. 2014). The properties of the LWA are shown in Table 2. The absorption of PCM into LWA was evaluated using two different conditions: ambient and vacuum conditions, which are described in the literature (Liston et al. 2014; Zhang et al. 2004). Two types of PCM were used, including (1) paraffin oil, which is a petroleum-based material with a specific density of 0.77, and (2) methyl laurate, which is a vegetable-based material with a specific density of 0.87. Both PCMs have a vapor pressure less than 0.01 mm Hg.

Table 1. Properties of Ordinary Portland Cements (OPC)

Item	Percent by mass (%)	
	Type I	Type V
Silicon dioxide (SiO ₂)	19.43	21.3
Aluminum oxide (Al ₂ O ₃)	5.39	2.6
Ferric oxide (Fe ₂ O ₃)	3.18	4.2
Calcium oxide (CaO)	63.45	63.2
Magnesium oxide (MgO)	2.97	4.5
Sulfur trioxide (SO ₃)	3.38	2.8
Loss on ignition	0.88	1.20
Sodium oxide	0.35	N/A
Potassium oxide	0.77	N/A
Free lime	N/A	0.38
Insoluble residue	0.25	0.18
Total equivalent alkali as Na ₂ O	0.86	0.21
Tricalcium silicate (C ₃ S)	60	64
Dicalcium silicate (C ₂ S)	10	13
Tricalcium aluminate (C ₃ A)	9	0
Tetracalcium aluminoferrite (C ₄ AF)	10	13

Table 2. Gradation and Absorption for Lightweight Aggregate (LWA)

Specimen	Sieve number	Mass retained (mass %)	Water absorption (mass %)	PCM absorption (mass %)	PCM absorption, 24 h (mass %)
Graded	4	1.8	N/A	23	13
LWA	8	13.3	35	28	16
	16	28.4	33	27	20
	30	22.7	31	27	20
	50	13.6	29	23	19
	Pan	20.2	26	N/A	N/A
Mixed LWA	—	—	32 ± 0.5	N/A	18.8 ± 0.5

Specimen Preparation and Conditioning

Use of Lightweight Aggregate to Incorporate PCM in Mortar

Mortar mixtures were prepared with an aggregate volume fraction of 55% and a water-to-cement ratio (w/c) of 0.42 by mass. To prepare mortar, oven-dried LWAs were soaked in PCMs for 24 h. After saturation with PCM, the PCM on the surface of LWAs was removed using a centrifuge technique (Miller et al. 2014) to reach relatively the saturated-surface-dry (SSD) condition. The aggregate was placed in a centrifuge bowl of known mass; the centrifuge bowl was then placed in the centrifuge, covered with a 4- μ m filter and lid, and tightened into place; the centrifuge was then powered on and the test was run for 3 min at a speed of 2,000 rpm (Miller et al. 2014).

Mortar was prepared in a standard mortar mixer using LWAs containing PCM in SSD condition in accordance with ASTM C305-12 (ASTM 2012). Mortar was also prepared using LWAs saturated with water (in SSD condition) as a control mortar. The mass of cement, mixing water, and LWA (in SSD condition) were 593, 249, and ~940 kg per m³ of total material volume, respectively, providing 145–150 kg/m³ of PCM in the mortar. The mortar was cast in 25.4 × 25.4 × 300 mm (1 × 1 × 11.81 in.) molds and the specimens were demolded after 24 h. All mortar bars were then sealed in double plastic bags and cured for 28 days in these sealed conditions at 23 ± 0.5°C. After 28 days of curing, the mortar bars were cut using a wet saw to 25.4 × 25.4 × 50.8 mm (1 × 1 × 2 in.) specimens. These specimens were then placed in a chamber at

23 ± 1°C and a relative humidity of 50 ± 1% for 7 days to remove moisture prior to the LGCC experiment.

The mortar was also cast in 50.8 × 50.8 × 50.8 mm (2 × 2 × 2 in.) molds to determine its compressive strength. These specimens were demolded after 24 h and were then sealed and cured for 56 days at 23 ± 0.5°C before the compression test.

Use of Embedded Tube to Incorporate PCM in Mortar

A plastic tube with a diameter of 10 mm and a length of 50.8 mm was located longitudinally in the center of the 25.4 × 25.4 × 50.8 mm (1 × 1 × 2 in.) mold. Mortar was then prepared and cast in the prepared mold. The mortar had a fine aggregate (i.e., natural sand) volume fraction of 55% and a w/c ratio of 0.42 by mass. The specimens were demolded after 24 h. The specimens were cured for 28 days in a sealed condition at 23 ± 0.5°C. After 28 days of curing, the specimens were placed in a chamber at 23 ± 1°C and a relative humidity of 50 ± 1% for 7 days to remove water moisture. The embedded tube was then filled using PCMs and the top and bottom of the embedded tube were sealed using plastic tape. Approximately 105–125 kg/m³ of PCM was incorporated into the mortar specimens using the embedded tube. The specimens were then tested in an LGCC experiment. To evaluate the chemical reactivity of the plastic tube with PCM, a piece of plastic tube was immersed in a beaker containing PCM for a week and the mass change in the plastic piece was monitored; no mass change or change in plastic stiffness was observed during the immersion time.

Testing Procedure

Low-Temperature Differential Scanning Calorimetry

To determine the thermal properties of PCMs, low-temperature differential scanning calorimetry (LT-DSC) was used. The heat flow and temperature associated with phase transitions in these materials was obtained. Stainless steel high-volume pans, specifically 100- μ L specimen size TA high-volume pans, with hermetic lids were used to contain the specimens. The initial temperature of the test was set to equilibrate at 40°C for 10 min. After the initial temperature became stable, the LT-DSC cell was cooled to –80°C at a cooling rate of 5°C/min. The temperature of the specimen was then increased to 40°C, again at a rate of 5°C/min. The mass of a specimen was 11 ± 2 mg.

Longitudinal Guarded Comparative Calorimetry

Longitudinal guarded comparative calorimetry (LGCC) was used to determine the thermal response of a mortar specimen containing PCM during cooling and heating (Farnam et al. 2014a, b). Mortar specimens (25.4 × 25.4 × 50.8 mm) containing PCM through either LWA or an embedded tube were tested in the LGCC device. A temperature gradient was generated in the test specimen to produce a one-dimensional heat flow. Two-meter bars with known thermal properties were used on the top and bottom of the mortar specimens, and temperatures at different locations were monitored to calculate the heat flow within the specimens.

The temperature of mortar specimens was varied from 24 to –40°C by using a two-stage cold plate (Cascade CCP-22, TECA, Chicago, Illinois). The cooling and heating rates were –2° and 4°C/h, respectively. At 24 and –40°C, the temperature was kept constant for 1 and 4 h, respectively, to allow the specimen to reach thermal equilibrium. The test was complete after one cycle.

Chemical and Physical Durability Assessment

Durability and stability of PCM in mortar specimens were determined by (1) an evaluation of chemical reactions between PCM and mortar constituents using LT-DSC, (2) an evaluation

of physical properties of mortar using pulse velocity and compressive strength measurements, and (3) a determination of neat PCM mass loss over time.

It was shown that LT-DSC can be used to perform a calorimetry study to assess the chemical reaction between the cementitious matrix and reactive materials at different temperatures (Farnam et al. 2014a; Farnam et al., unpublished data, 2015; Villani et al. 2015). Ground powder specimens were selected for this test since this test is intended to only evaluate the potential reactivity of mortar constituents with PCMs. Mortar constituents consisted of LWA, hydrated cement paste for both Type I and Type V cement, and calcium hydroxide. During the test, the temperature cycles ranged from 25 to -90°C in the cooling path and from -90 to 70°C in the heating ramp at a rate of a $5^{\circ}\text{C}/\text{min}$. The powder (9–11 mg) was mixed with an equal mass of PCM in a high-volume stainless steel pan and tested in LT-DSC: immediately after mixing (0 days), 1 day after mixing, 7 days after mixing, and 28 days after mixing. The change in enthalpy of fusion over time was monitored, which can indicate a chemical reaction between mortar constituents and the PCM.

Acoustic emission testing has shown promising performance for assessing the damage development in cementitious materials (ASTM 2009; Farnam et al. 2014a, 2015b). To evaluate the dynamic elastic modulus of mortar specimens, compressional wave speed (pulse velocity) was measured through the length of the specimens using a pulsed wave generated by two coupled AE sensors in accordance with ASTM C597-09 (ASTM 2009). The dynamic elastic modulus was compared to the one obtained from a control mortar specimen to measure the damage index. Compressive strength measurements were also performed in accordance with ASTM C109/C109M-13 (ASTM 2013) to evaluate the strength of mortar specimens containing PCM. To evaluate the potential mass loss over time, ~ 10 g of PCMs were stored in a beaker and kept in a chamber at $23 \pm 1^{\circ}\text{C}$ and a relative humidity of $50 \pm 1\%$ for 38 days and the specimen mass was monitored during this time.

Results and Discussion

Thermal Properties of PCMs

The heat flow measurement obtained using the LT-DSC for both paraffin oil and methyl laurate is shown in Fig. 3(a). The heating

ramp is shown and the result is normalized by the mass of PCM. In addition, the heat released during the phase transformation is calculated and is shown in Fig. 3(b). Methyl laurate shows one endothermic behavior (i.e., one peak) at a temperature near 1.9°C (which indicates the liquid/solid phase transformation) with an enthalpy of fusion of ~ 160.4 J/g. Paraffin oil shows two endothermic peaks at temperatures near -28.4 and 2.9°C with enthalpies of ~ 15.4 and 129.4 J/g, respectively. For paraffin oil, the phase transformation (at about -28.4°C) may not be useful in snow melting and deicing applications, since ice and snow melt at a temperature near 0°C . For a PCM to have a good performance in snow melting and deicing applications, it is necessary that the PCM has a phase transformation temperature (most importantly the onset temperature) somewhat greater than 0°C and a high enthalpy of fusion (Bentz and Turpin 2007; Ling and Poon 2013; Sakulich and Bentz 2012b, a). Both paraffin oil and methyl laurate show reasonable thermal properties for snow melting and deicing applications since they can produce a sufficient heat (~ 130 – 160 J/g) during their phase transformation at ~ 2 – 3°C .

Thermal Response of Mortar Containing PCMs

Using Lightweight Aggregate to Incorporate PCM in Mortar

The LGCC was used to perform thermal cycling on mortar specimens. The heat flow was calculated (Farnam et al. 2014b) during cooling and heating for mortar specimens containing PCM using the LWA approach and is shown in Fig. 4. For Fig. 4(a), the Type I cement (Table 1 for chemical composition) was used to prepare mortar specimens, while the Type V cement was used to prepare mortar for Fig. 4(b). Mortar specimens containing paraffin oil show heat release (an exothermic peak) during cooling associated with the paraffin oil phase transformation (i.e., freezing or solidification) and heat absorption (endothermic peak) during heating associated with paraffin oil melting. The heat release during paraffin oil solidification (freezing) can be used to melt ice and snow on the surface of the concrete pavement.

Mortar specimens containing methyl laurate did not release/absorb a significant/measurable amount of heat during cooling/heating for specimens prepared using Type I cement in the temperature range tested. This is partially solved when Type V cement is used to prepare the mortar specimen containing methyl laurate,

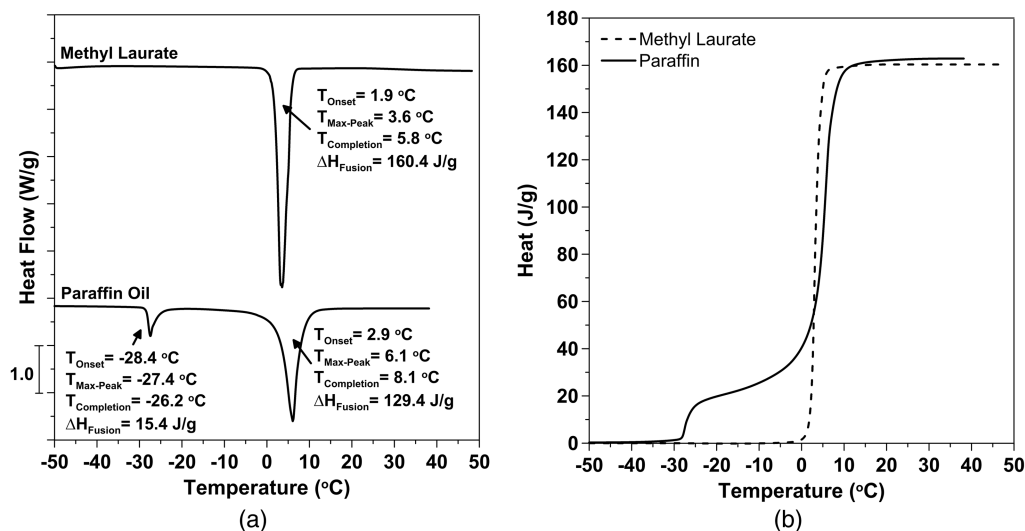


Fig. 3. Thermal responses of paraffin oil and methyl laurate obtained using LT-DSC (normalized per gram of PCM): (a) heat flow versus temperature; (b) heat (enthalpy) versus temperature

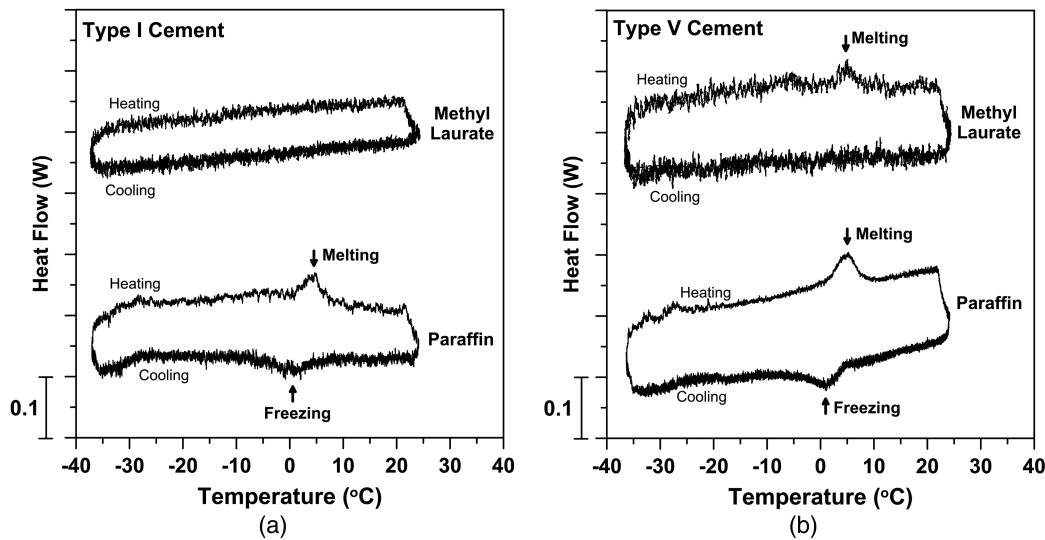


Fig. 4. Heat flow as a function of temperature obtained using LGCC for mortar containing PCM using LWA: (a) mortar prepared using Type I cement; (b) mortar prepared using Type V cement

since a melting peak can be seen during heating. It may be hypothesized that methyl laurate interacts with the cementitious matrix, producing a material that is not beneficial to produce heat in the desired temperature range for snow melting and deicing salt applications. The reaction may be milder for mortar specimens prepared using Type V cement than the ones prepared using Type I cement. The reaction between methyl laurate and the concrete constituents and its influence on concrete properties will be described later in this paper.

Using an Embedded Tube to Incorporate PCM in Mortar

In Fig. 4, it was shown that methyl laurate may interact with the cementitious matrix when the LWA is used to place PCM in the mortar specimens. When PCM is placed in the LWA, there is no barrier between PCM and different mortar constituents (such as calcium hydroxide, aluminate phases, and aggregates). As a result, PCM may interact with different mortar constituents that can produce a material that is not beneficial in melting ice and snow. In the following, it will be also shown that this reaction is destructive and can cause cracking in the mortar specimens. The embedded tube/pipe can be an alternative approach to incorporate PCM in concrete elements. A nonreactive plastic or steel tube/pipe can be used to store PCM in concrete and prevent a direct contact between the PCM and concrete constituents.

Fig. 5 shows heat flow during cooling and heating as a function of temperature for mortar specimens containing PCM using the embedded tube approach obtained from the LGCC experiment. An exothermic phase transformation (freezing peak) and an endothermic phase transformation (melting peak) can be seen for both methyl laurate and paraffin oil. The exothermic phase transformation (i.e., heat release) during PCM solidification (i.e., phase transformation) can be used to melt ice and snow. The released heat can be further restored and recovered in PCM during PCM melting within the (subsequent) heating process.

Performance of Mortar Containing PCM to Melt Ice

The area under the heat flow versus time curve can be used to quantify the amount of heat that is released or absorbed (stored) during a phase transformation (freezing or melting). The total amount of heat release during PCM solidification was calculated for the

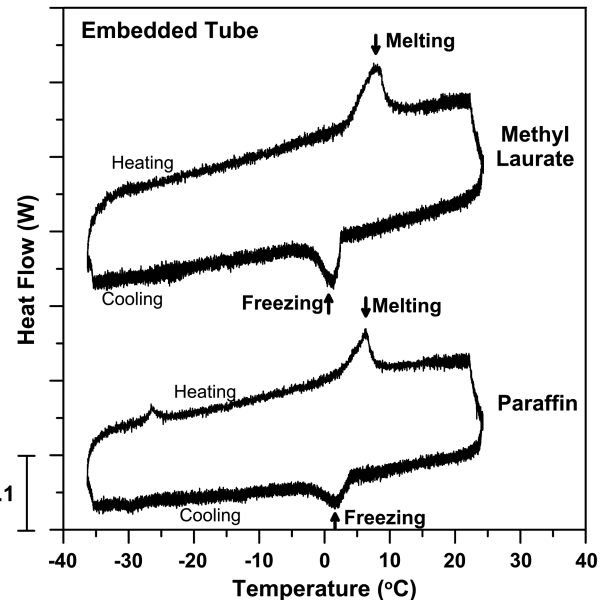


Fig. 5. Heat flow as a function of temperature obtained using LGCC for mortar specimens containing PCM through embedded tube

mortar specimens tested with the LGCC experiment and is shown in Table 3. A desirable amount of heat is released during PCM phase transformation (i.e., freezing) that can be further used to melt ice or snow. It was reported that the heat release measured using the LGCC experiment is approximately 40% less than the actual heat release during phase transformation in the mortar specimen tested in the LGCC experiment (Esmaeeli et al., unpublished data, 2015). As a result, the actual heat release may be much higher than the values reported in Table 3.

The temperatures at which the phase transformation begins or ends were obtained during the LGCC experiment and are reported in Table 4. It should be mentioned that the onset temperature at the bottom surface of the specimen in the LGCC experiment

Table 3. PCM Potential to Release Heat and Melt Ice during Phase Transformation in Mortar Specimen Obtained Using LGCC Experiment (the Results Are Reported ± 1 Standard Deviation for At Least Two Replicates of LGCC Experiment)

PCM incorporation method	PCM type	PCM quantity (kg/m ³) ^a	Heat release (J/g _{PCM})	Heat release (kJ/m ³) ^a	Calculated molten ice (kg/m ³) ^a	Ice-to-concrete depth ratio (%)
LWA ^b	Methyl laurate	150	—	—	—	—
	Paraffin oil	145	78.4 (± 1.3)	11,345 (± 188)	34.0 (± 0.5)	3.7 (± 0.1)
Embedded tube	Methyl laurate	122.0 (± 3.8)	98.0 (± 6.8)	12,111 ($\pm 1,070$)	36.3 (± 3.2)	4.0 (± 0.3)
	paraffin oil	111.7 (± 4.3)	66.9 (± 9.9)	7,430 (± 827)	22.3 (± 2.5)	2.4 (± 0.3)

^aPer cubic meter of mortar specimen.

^bThe average value for samples prepared using both Type I and Type V are reported.

Table 4. Temperatures at the Top and the Bottom of Mortar Specimens Associated with the Onset and the Completion of PCM Phase Transformation during LGCC Experiment (the Results Are Reported ± 1 Deviation for At Least Two Replicates of the LGCC Experiment)

PCM incorporation method	PCM type	Temperature of specimen (°C)			
		PCM freezing onset		PCM freezing completion	
		Bottom	Top	Bottom	Top
LWA ^a	Methyl laurate	—	—	—	—
	Paraffin oil	3.0 (± 0.3)	6.0 (± 0.2)	-13.8 (± 0.7)	-9.8 (± 0.7)
Embedded tube	Methyl laurate	1.2 (± 0.1)	3.5 (± 0.1)	-12.7 (± 1.5)	-9.6 (± 1.5)
	Paraffin oil	3.0 (± 0.1)	5.4 (± 0.4)	-8.2 (± 3.1)	-5.2 (± 3.4)

^aThe average value for samples prepared using both Type I and Type V are reported.

may be a better indication of the temperature that is required on the top surface of the concrete pavement to melt ice and snow. In concrete pavement, the heat flow is toward the top surface of pavement during cooling, while in the LGCC experiment the heat flow is toward the bottom surface of the mortar specimen during cooling since the cold plate is located at the bottom. The onset temperature of the phase transformation at the bottom surface of the specimen was near 1.2 to 3°C, which is in the range of desired temperatures for deicing applications for concrete (Bentz and Turpin 2007; Ling and Poon 2013; Sakulich and Bentz 2012b, a).

The amount of concrete that can be melted using PCM in the mortar specimen was calculated using Eq. (1). The amount of ice can be simply converted to the equivalent amount of snow using the snow density and the snow packing (Schmucki et al. 2014). Eq. (1) describes a procedure that can be used to determine the thickness of the ice on the surface of a concrete element that can be melted using the energy released by the PCM phase transformation

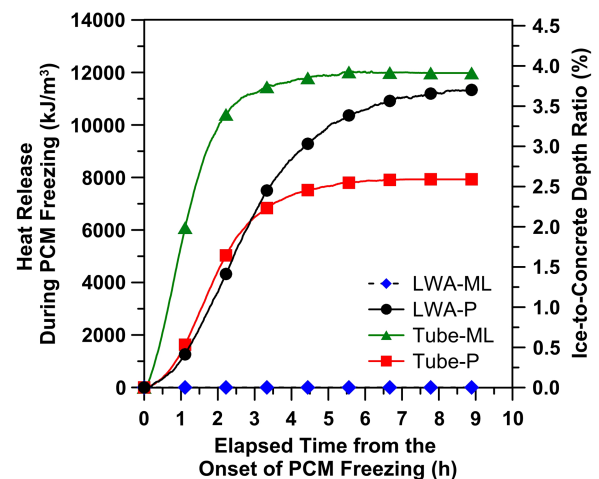
$$\text{Ice to concrete depth ratio} = \frac{d}{D} = \frac{Q}{V_{\text{concrete}} \cdot \rho_{\text{ice}} \cdot \Delta H_{\text{water/ice}}} \quad (1)$$

where d = thickness of ice on the surface of concrete element that can be melted using PCM incorporated in the concrete element; D = thickness of the concrete element; Q (kJ) = energy released during the PCM phase transition in the concrete element, which is obtained here using LGCC experiment for mortar specimen; V_{concrete} (m³) = volume of concrete element, which is here the volume of mortar specimen; $\Delta H_{\text{water/ice}}$ = heat of fusion for ice during melting, which is equal to 334 kJ/kg; and ρ_{ice} = density of ice, which is equal to 916.7 kg/m³.

The ice to concrete depth ratio (%) was calculated for mortar specimens tested in the LGCC experiment using Eq. (1) and is shown in Fig. 6 as a function of elapsed time from the onset moment of PCM phase transformation. In addition, the amount of heat released during the PCM phase transformation per cubic meter of concrete was calculated from the LGCC experiment and is shown in Fig. 6. As expected, mortar specimens containing methyl laurate

using the LWA approach show no heat release since a chemical reaction appears to alter the methyl laurate in LWA. Using the embedded tube approach, the methyl laurate specimen shows a considerable amount of heat release during its PCM phase transformation. The amount of ice-to-concrete depth ratio after a complete methyl laurate phase transformation within the embedded tube is near 4%, and this occurs in a relatively quick manner since the slope of the curve for methyl laurate is steeper than the ones for paraffin oil. This analysis assumes that heat conduction through the concrete is not a limiting factor in transferring energy from within the concrete to its top surface where the ice/snow is located.

Mortar specimens containing paraffin oil using both LWA and the embedded tube indicate significant heat release during the paraffin oil phase transformation. The amount of heat release is higher

**Fig. 6.** Released heat during PCM freezing in mortar specimen as a function of elapsed time from the onset of PCM freezing (notice that the quantity of PCM per volume of mortar sample varies for each case which is reported in Table 3)

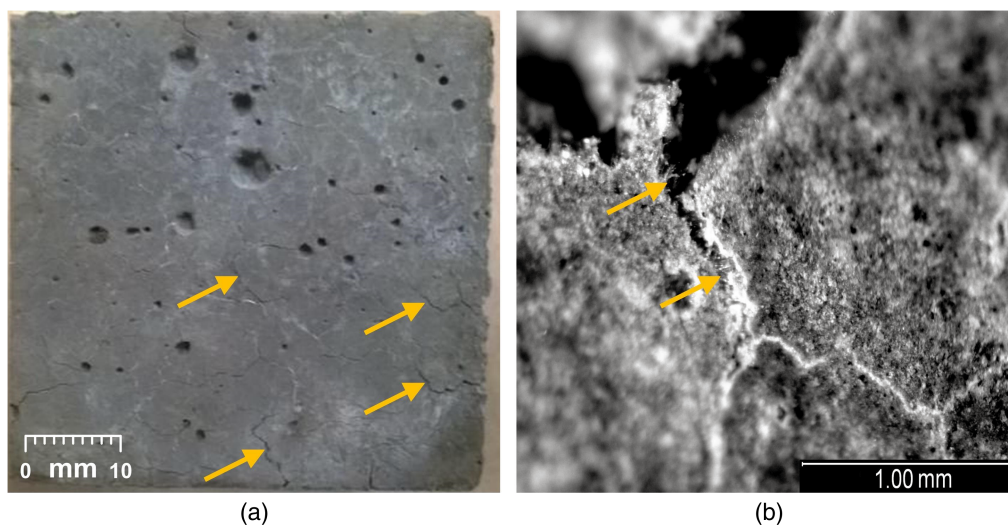


Fig. 7. Images of mortar specimen containing methyl laurate using LWA approach: (a) cracked surface (arrows show cracking); (b) microscopy image (arrows show white color fiber shape material)

for methyl laurate than it is for paraffin oil since methyl laurate has a higher enthalpy of fusion than paraffin oil during solid/liquid phase transformations, as indicated in Fig. 3. As a result, methyl laurate can produce a greater amount of heat during freezing than paraffin oil. The amount of ice-to-concrete depth ratio for paraffin oil is near 2.5–3.5%. For paraffin oil, the heat due to phase transformation is released more gradually in comparison to methyl laurate. Assuming a 355.6 mm (14 in.) thickness for a concrete pavement, paraffin oil can melt 8.9 mm (0.35 in.) to 12.4 mm (0.49 in.) thickness of ice on the surface of the pavement after the complete phase transformation.

Chemical and Physical Durability Assessment

It is necessary that a potential PCM for deicing and snow melting applications remains nonreactive and stable over its lifetime in a concrete element. Any reaction between PCM and concrete constituents or loss of PCM over time may result in a reduction in concrete durability or a reduction in PCM performance to melt ice and snow, respectively.

Evaluation of Chemical Reaction between PCM and Concrete Constituents

During the curing time of mortar specimens containing methyl laurate using the LWA approach, some cracking was observed on the surface of mortar specimens, as shown by arrows in Fig. 7(a). The cracking was developed and propagated over time and a white color fiber shape material grew within the cracking [shown by arrows in Fig. 7(b)]. In addition, no phase change was observed when these specimens were tested in the LGCC experiment (Fig. 4). The cracking was not as severe when Type V cement was used instead of Type I cement to prepare the mortar specimen. In addition, the cracking was not observed when paraffin oil was used in the mortar specimen.

Methyl laurate appears to interact with the constituents of the mortar, resulting in cracking and damage. An evaluation of this chemical reaction was performed for methyl laurate to understand the source of reaction and damage. A powder of cement paste (prepared using both Type I cement and Type V cement), calcium hydroxide, or LWA was blended and tested in LT-DSC to assess the chemical interaction. Evaluation of Type I and Type V cements can help to understand whether aluminate phases in concrete create the

reaction, since Type V cement has relatively no tricalcium aluminate while Type I cement has 9% tricalcium aluminate (Table 1).

Fig. 8 indicates the reduction in enthalpy of fusion (or heat of fusion) as a function of time for powders blended with methyl laurate obtained using LT-DSC. Paste made using Type I cement showed a substantial decrease in enthalpy, indicating a relatively fast reaction between aluminate phases and methyl laurate. A reduction in enthalpy can also be seen for calcium hydroxide blended with methyl laurate, and it seems this reaction is more gradual than the reaction created by aluminate phases. The reduction in enthalpy is small for LWA and paste made using Type V cement. It can be concluded that aluminate phases and calcium hydroxide in cementitious materials are the main sources of the reaction between methyl laurate and the cementitious matrix. Use of Type V cement may reduce this reaction since there is relatively no C_3A in Type V cement. However, calcium hydroxide is still present in a concrete made using Type V cement that may participate in reactions and decrease the methyl laurate performance to melt ice and snow as observed in Fig. 4. Other approaches, such as using an embedded tube or encapsulation, can be used to incorporate methyl laurate in

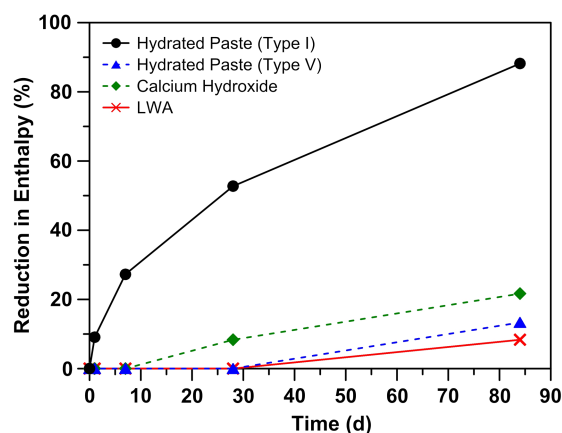


Fig. 8. Reduction in heat of fusion (enthalpy) obtained using LT-DSC for methyl laurate during melting/freezing over time exposed to different constituents of mortar

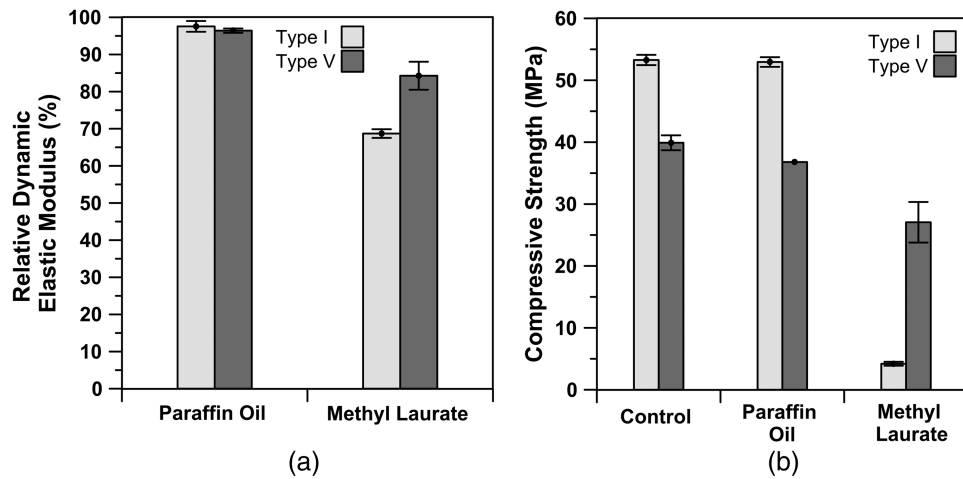


Fig. 9. (a) Relative dynamic elastic modulus with respect to control specimen; (b) compressive strength for mortar specimen made using LWA and different types of cement (Type I and Type V); the error bars indicate ± 1 standard deviation for at least two replicates of testing

concrete to eliminate the reaction between methyl laurate and the cementitious matrix.

Evaluation of Physical Performance

The relative dynamic elastic modulus ($E_{PCM}/E_{Control}$) was calculated using Eq. (2) for mortar specimens containing PCM using the LWA approach and is shown in Fig. 9(a). Compressive strength was also measured and is reported in Fig. 9(b)

$$\frac{E_{PCM}}{E_{Control}} = 1 - \left[\frac{V_{PCM}}{V_{Control}} \right]^2 \quad (2)$$

where E_{PCM} and $E_{Control}$ are the dynamic elastic modulus of the PCM specimen and the control specimen, respectively; and V_{PCM} and $V_{Control}$ are the average wave velocity through the length of the PCM specimen and the control specimen, respectively.

As indicated in Fig. 9, mortar specimens containing methyl laurate using the LWA approach show reductions in both relative dynamic elastic modulus and compressive strength, indicating damage development and cracking in the mortar specimen due to the chemical reaction between methyl laurate and the cementitious matrix. The dynamic elastic modulus for mortar specimens containing paraffin oil using the LWA approach remains relatively unchanged (95% of the control specimen) and the compressive strength is in a desirable range for concrete pavement applications.

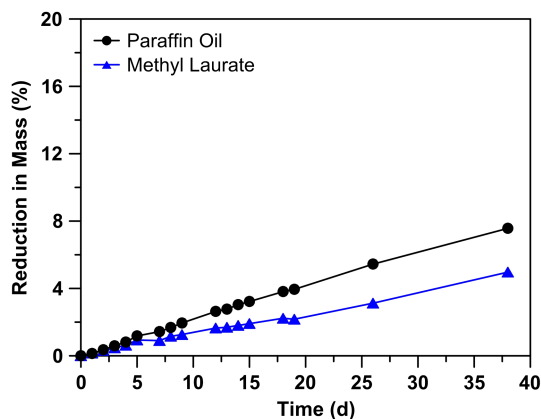


Fig. 10. The stability of PCMs (i.e., change in mass) over time exposed to 50% relative humidity at 23°C

Fig. 10 indicates the mass loss as a function of time for methyl laurate and paraffin oil as they are exposed to a relative humidity of 50% and room temperature (23°C). A relatively small amount of mass loss (~5–8%) was observed after 38 days of exposure. The mass loss is mainly due to PCM evaporation. This amount will be reduced for PCM stored inside a concrete element instead of experiencing direct exposure to 50% relative humidity at 23°C since the PCM will be surrounded by a dense concrete matrix, embedded tube, or a shell provided by encapsulation.

Summary and Conclusion

This paper reports the results of tests to evaluate the thermal response of mortars containing phase change materials (PCM) subjected to a thermal temperature cycle from 24 to -40°C for use in melting snow and ice in concrete pavement. Two approaches were used to place the PCM in the mortar: (1) using lightweight aggregates (LWA) containing PCM and (2) using an embedded tube filled with PCM. Chemical and physical durability aspects of incorporating PCM in concrete were also assessed by monitoring the change in heat of fusion, compressive strength, relative dynamic elastic modulus, and mass loss over time. Methyl laurate and paraffin oil were selected as the PCMs used in this study. These PCMs have desired thermal properties (high heat of fusion during the phase transformation, $\sim 130\text{--}160$ J/g, and appropriate phase transformation temperature, $\sim 2\text{--}3^{\circ}\text{C}$) for snow and ice melting.

When the PCM was stored in LWA and placed in the mortar specimens with PCM, the mortar specimen containing paraffin oil showed a heat release of $\sim 11,000$ kJ per cubic meter of mortar at $\sim 3.0^{\circ}\text{C}$ during freezing (i.e., phase transformation of the PCM). However, methyl laurate showed no heat release due to two chemical reactions that occurred between the methyl laurate and the cementitious matrix. The two reactions appear to occur between the PCM and the aluminate phases or between the PCM and the calcium hydroxide in the cementitious matrix. As such, the use of Type V cement (containing lower tricalcium aluminate) may reduce the rate of the former reaction, but it cannot eliminate the latter reaction since cementitious material made using Type V cement still produces calcium hydroxide. Both chemical reactions appear to create cracking and damage in concrete, thereby reducing the dynamic elastic modulus and compressive strength. The use of paraffin oil as the PCM did not exhibit such reactions.

A second approach was used to place the PCM into concrete using an embedded tube/pipe filled with the PCM. The mortar with embedded tube containing paraffin oil and methyl laurate showed a heat release of ~7,500 kJ (at ~3°C) and ~12,000 (at ~1.2°C) per cubic meter of mortar during the phase transformation, respectively. These heat releases can be further used to melt 22–36 kg of pure ice per cubic meter of mortar on the surface of the concrete pavement. The PCM inside the embedded tube did not exhibit any reaction.

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