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Citation	Composite structures, 215, 214-225 https://doi.org/10.1016/j.compstruct.2019.02.057
Issue Date	2019-05-01
Doc URL	https://hdl.handle.net/2115/81122
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Type	journal article
File Information	Revised Manuscript.pdf



Influence of Continuous and Cyclic Temperature Durations on the Performance of Polymer Cement Mortar and its Composite with Concrete

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ABSTRACT

Polymer cement mortar (PCM) is a widely used cementitious repairing material due to its considerable adhesive property with concrete. However, the polymers are sensitive to elevated temperatures. The behaviours of polymers and PCM at elevated temperatures (e.g., 60 °C) for short, moderate duration and cyclic conditions remain unknown and need to be explored. This work was aimed at studying the mechanical performance of PCM and PCM-concrete composites under the aforementioned exposure conditions. The bond strength in tension was evaluated by interfacial split tensile and flexural strength tests. A reduction in the mechanical strength of PCM was observed when exposed and tested at 60 °C, and the strength recovery was also observed after cooling the specimen. The cyclic temperature condition has the most detrimental influence on the mechanical behaviour of PCM and PCM-concrete interface compared to other exposure conditions. To reveal the damage mechanism, the polymers were extracted from the PCM, and the glass transition (T_g) and melting point temperatures were obtained by differential scanning calorimetry (DSC) analysis. Corresponding to the mechanical reduction of the PCM and interface, the reduction in the T_g value was also observed after elevated temperature and cyclic temperature exposure except the case exposed to moist condition. The maximum strength recovery was observed when the testing temperature was less than T_g . Besides, the molecular weight of the extracted polymers was analysed by gel permeation chromatography (GPC). The ratio of the area regarding the amount of oligomers to the area regarding the molecular weight of the GPC curve increased with the temperature duration, which was consistent with the tensile strength reduction of PCM.

Keywords: Environmental exposure conditions; Polymer cement mortar; Bond strength; Polymer; Glass transition temperature; Molecular weight.

1. INTRODUCTION

Polymers are widely used in the construction industry to prepare cementitious and non-cementitious repairing/strengthening materials. Specifically, by using different amounts of polymers and techniques, polymer concrete, polymer modified concrete or polymer impregnated concrete can be produced [1]. Polymer incorporated concretes have superior properties over ordinary concrete due to the formation of polymer films surrounding the hydrated products between the old concrete substrate and the newly casted polymer modified mortar. The polymer film cannot only reduce the porosity and permeability in the interface but

also provide an additional adhesive strength along with chemical and mechanical bonding [2-4]. Although polymer modified mortar is a strong and durable material, due to the temperature sensitivity of the polymers it is necessary to evaluate its mechanical performance under different environmental conditions, especially when the environmental temperature exceeds 50 °C. Regarding the environmental influence on the interface between concrete and fibre reinforced polymers (FRP), the ACI committee [5] recommends that the environmental load reduction factor ranges from 0.85 to 0.95 for carbon/epoxy systems. However, no guidelines are available for polymer cement mortar behaviour under different environmental loads.

Polymer cement mortar (PCM) can be prepared in a laboratory by adding the desired amount of polymers to Portland cement mortar. It performs well when cured under a dry condition, which is beneficial for making polymer film [6]. After proper curing, the PCM is considered as more compatible with concrete than other repairing materials [7]. Although it has a strong bond property, additional stresses are generated at the interface between the PCM and concrete due to drying and shrinkage. When exposed to different moisture and temperature levels, deterioration of the interface can be induced [8]. The interfacial bond performance can be assessed by different bond strength tests, such as the interfacial split tensile, bi-surface shear, slang shear and flexural tests [9, 10]. However, for specimens under different environmental conditions, the bond performance assessment requires further experimental research.

The variation in the physical and chemical properties of polymer may alter the microstructure of the PCM and ultimately the behaviour of the PCM. The two main physical properties of the polymers are the glass transition temperature and the melting point temperature, which can be measured by differential scanning calorimetry (DSC). A significant change in the mechanical properties of the PCM are observed before and after the glass transition temperature [2, 11]. The polymer can also be decomposed in number of ways: (1) Chain scission (random-chain scission, end-chain scission and chain-stripping), (2) Cross linking, in which bonds are created between polymer chains, (3) Side chain elimination, and (4) Side chain cyclization. The molecular weight (M_n) is another important physical property of the polymers. A reduction in M_n is observed only in chain scission decomposition and may be referred to as de-polymerization or unzipping. Due to unzipping, the amount of oligomers and monomers increases, which can be assessed experimentally by gel permeation chromatography (GPC). Since the degree of polymerization is analogous with the M_n , the higher M_n is, the higher the degrees of polymerization and mechanical strength are [12]. Different environmental conditions, e.g., alkali silica reaction, freeze thaw cycles, carbonation, chloride ion penetration, etc., may degrade the polymers and ultimately result in a reduction of M_n or degree of polymerization.

The performance of PCM and PCM-concrete under several environmental conditions, most specifically short duration temperature exposure due to the temperature sensitivity of polymers, was explored experimentally and analytically in our previous studies [13-15]. PCM and its composite specimens at temperature levels of 20, 40 and 60 °C were examined, and a significant tensile strength reduction was observed with an increase in temperature [13-15]. With different wetting/drying cycles and continuous immersion in water for several days, a marginal influence on the tensile strength was also observed [13, 16, 17]. The shear and flexural bond behaviour was investigated at elevated temperatures, and the bond strength reduction for

both bulk and composite specimens were noticed [18, 19]. The study was further extended to the flexural behaviours of a beam overlaid with PCM and exposed to short duration temperature levels at 20, 40 and 60 °C. The de-bonding failure mode was observed at an elevated temperature. More importantly, the flexural strength was reduced with an increase in the temperature level, even when the failure mode was flexural failure [18, 20]. Additionally, the flexural crack spacing and crack width increased with temperature [21]. A detrimental influence was observed for all exposure conditions, which were all short temperature duration exposures. However, the influence of temperature for a moderate duration as well as cyclic temperature conditions remains unclear, requiring further investigation.

The environmental temperature of some regions exceeds 50 °C in the summer (e.g., the Gulf State, Pakistan, some parts of North America). Although the repairing works of concrete structures were properly performed, when exposed to such high temperature conditions the durability of concrete and PCM-concrete interface should be taken into consideration. For all intents and purposes, the mechanical behaviour of PCM-concrete structures under harsh elevated temperature environments remains unknown. Examples of harsh elevated temperatures include exposure to the hottest day of the year, suffering from a peak summer season, significant temperature variation between day and night, and seasonal environmental variations. For a long-term durability design, it is necessary to investigate the aforementioned issues.

Based on the previous studies, a short duration, moderate duration and cyclic temperature conditions were designed to simulate real harsh environmental conditions. The mechanical behaviour of the PCM, the PCM-concrete interface and the properties of polymers were investigated under such exposure conditions. The mechanical strength of the PCM was investigated by conducting compressive, split tensile and three-point bending tests, while the bond performance of PCM-concrete specimens was evaluated by conducting an interfacial split tensile and three-point bending tests. The testing temperature condition was also set as a parameter in this study, from which the behaviour was noted at an elevated temperature as well as after cooling down. Polymers were extracted from the PCM after performing a mechanical test under the designed conditions, and their glass transition and melting point temperatures were measured by a DSC. Additionally, to discuss the degradation mechanism of polymers, the M_n of polymers was also measured through GPC analysis.

2. EXPERIMENTAL DESCRIPTION

2.1 Materials and Specimen Preparation

Concrete was casted in the laboratory using ordinary Portland cement of ASTM Type-I as a binding material with a specific gravity of 3.16. Locally available river sand and crush were used as aggregates, having specific gravities of 2.71 and 2.72, respectively. Tap water was used to mix the constituents to achieve a target compressive strength of 40 MPa. The relatively higher compressive strength of concrete substrate was chosen to achieve a brittle and abrupt failure mode, which is the most critical condition for PCM-concrete interface. Since the bond strength of PCM-concrete interface is highly depending on the constitutive materials' mechanical properties, with higher compressive strength of concrete, the bond behaviour could be poorer and the reduction tendency could be more obvious. The polymer in the PCM can

hardly penetrate into the high strength concrete to form an adhesive layer since it is less porous. Without an efficient adhesive layer between PCM and concrete, the adhesive strength would be lower. The explanation for the mechanism can be found in Ref. [9]. In this case, for real structural strengthening, the most critical degradation can be understood and taken into account at the design stage. The mixture proportions for concrete are provided in Table 1. The PCM was used as repairing material and is commercially available in the form of a 25 kg pack provided by Denka Company Limited, Japan. It is in the form of grey colour PCM powder, and the amount of water required for 1 pack (25 kg) is only 3.5 kg.

Table 1. Mixture proportion for 1 cubic metre of concrete.

Description	Value
Cement (kg/m ³)	453
Water (Litre)	165
Water to cement ratio (w/c)	0.36
Sand (kg/m ³)	843
Crush (kg/m ³)	1035
Target Compressive Strength (MPa)	40

Next, 100 x 100 x 800 mm and 100 (diameter) x 200 (height) mm concrete specimens were casted. Once the specimens were casted, all specimens were wrapped with polythene sheets to avoid moisture evaporation. After 24 hours of curing, the specimens were de-moulded and put in a curing tank filled with water for 28 days. When the prism specimens were cured as designed, they were cut into a prism with dimensions of 100 x 100 x 50 mm and 100 x 100 x 200 mm. For both sizes of specimens, one surface with dimensions of 100 x 100 mm was treated for having too much roughness. Following our previous studies [9, 13], the sandblasting method was adopted in this study for roughing, which was considered as the best method for substrate surface treatment. It can obtain a uniform and clean rough surface since it introduced no further damage to the substrate concrete [22]. The specimens were treated until the exposure of coarse aggregate to reach the same roughness level because the surface roughness is essential to the bond performance. The roughness of the treated surface was measured quantitatively by a three dimensional shape measurement apparatus. Peaks and valleys were measured from the apparatus and arithmetic mean value was taken as the roughness coefficient (Ra). Thirty samples were used for quantification of Ra and the average value was 0.67 mm, which was similar to the concrete surface (CSP) No. 6 or No. 7 as provided by the International Concrete Repair Institute [23]. The roughness values were very close to each other based on the standardized sandblasting method.

After the treated concrete prisms were again immersed in water for 24 hours for saturation, they were put in moulds by exposing the treated surface, which was dried by towel. The PCM was overlaid on the concrete, which was prepared in a laboratory by simply adding clean water at a temperature of 20 °C. Composite specimens of two geometry types were compared; (1) 100 mm cube, and (2) 100 x 100 x 400 mm prism. The bulk specimens of the PCM were also prepared with dimensions of a 100 mm cube and a 100 x 100 x 400 mm prism. Composite specimens and bulk PCM specimens were cured for 28 days, including 7 days of wet curing

and 21 days dry curing to achieve the high strength of PCM [2]. After curing, the material tests were conducted at the designed temperature conditions, and the test results are shown in Table 5.

2.2 Exposure Conditions

According to the environmental conditions in sub-tropical regions, four series tests were designed as follows; (1) Series-I: bulk and composite specimens were exposed to 60 °C in an oven for 24 hours. The influence of the high temperature on the PCM and PCM repaired concrete structures were studied; this was denoted by “T_{SD}” (Thermal behaviour for Short Duration). Additionally, the testing condition was also at 60 °C, for which an environmental chamber was established surrounding the spilt test specimen during loading (Fig. 1(a)). To maintain the temperature during testing, an insulation box was designed to cover the specimen ((Fig. 1(b)). The temperature during testing was monitored by a thermocouple, which was embedded at the interface during casting of the composite specimens. Almost the same temperature level was established during testing, but there might be some strength recovery during transporting of the specimen from environmental chamber to the testing machine and the actual mechanical degradation could be more severe. To obtain the actual strength recovery value, the transporting period of specimens should be very short to make sure that the temperature of the specimen is not decreased. (2) Series-II: The temperature duration was extended from 24 hours to 30 days to simulate the influence of the summer season of the sub-tropical region on the PCM and composite specimens; the acronym used for this was “T_{MD}” (Thermal behaviour for Moderate Duration). Both types of specimens were tested at a high temperature as well as after cooling down at room temperature (20 °C). (3) Series-III: Temperature variation during the day and night was incorporated by exposing the specimen to 60 °C for 12 hours and then exposing it to 30 °C for another 12 hours. One day is required to complete one cycle and the specimens were mechanically tested after 30 cycles of exposure. This series is denoted by “T_{DN}” (Thermal behaviour for Day Night variation) (4) Series-IV: Seasonal variation was designed by putting specimens in an oven at 60 °C for 1 day, in water for another day, at 5 °C for another day and finally at 25 °C to simulate the effects of the summer, a rainy season, winter and spring seasons of many regions of the world. Four days are needed to complete 1 cycle and the specimens were tested after the 10th cycle exposure. This is denoted by “T_{SV}” (Thermal behaviour for Seasonal Variation) in this study. By considering the cyclic conditions, behaviour of strengthened structures can be investigated appropriately. A better environmental reduction factor can be proposed for design purpose. A summary of the exposure conditions is presented in Table 2.

Table 2. Summary of the exposure conditions.

Series No	Description	Notation
Series-I	Short duration (24 hours) temperature exposure at 60 °C	T _{SD}
Series-II	Moderate duration (30 days) constant temperature exposure at 60 °C	T _{MD}
Series-III	Cyclic temperature condition; 12 hours at 60 °C and 12 hours at 30 °C to simulate the influence of day night variation.	T _{DN}

Series-IV	Cyclic temperature exposure given by 24 hours at 60 °C, 24 hours at 20 °C in water, 24 hours at 5 °C and finally 24 hours at 25 °C.	T _{sv}
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2.3 Testing

The mechanical performances of the PCM and PCM-concrete composite specimens were experimentally evaluated. The polymers were extracted from the PCM and its properties were also assessed. According to an ASTM guideline [24], an unconfined uniaxial compression test was conducted on the concrete and PCM cylinder specimens. The cylinder size was 100 mm in diameter and 200 mm in length. The compressive strength was obtained by using the average value of the three specimens. The tensile strength of the PCM and PCM-concrete composite specimens was measured by performing split tensile and flexural tests. The split tensile test was performed on a 100 mm cubical specimen by following the ASTM standards (see Fig. 1(a)) [25], using Eq. (1) to determine the split tensile strength (f_{st}). The same amount of tensile stress generated at the middle of the specimens in either the cylinder or cube was considered in the tests [26]. Since the size of the strip has an influence on the stress distribution during loading, the split tensile strength can be corrected by incorporating the ratio of the width of the strip (10 mm in this study) to the height of the specimen (β), as presented in the Eq. (2) [26].

$$f_{st} = \frac{2P_u}{\pi A} \quad (1)$$

$$f_{st}(\beta) = \frac{2P_u}{\pi A} [(1 - \beta^2)^{5/3} - 0.0115] \quad (2)$$

where f_{st} is the split tensile strength (MPa), $f_{st}(\beta)$ is the corrected split tensile strength considering the effect of the strip (MPa), P_u is the ultimate load (kN), A is the area of the specimen interface (m²), and β is the ratio of the width of the strip to the height of the specimen, which is 0.1 in this study.

According to a JCI standard (JCI-S-001-2003) [27], the flexural tests (three-point bending) were conducted on notched beam specimens with a size of 100 x 100 x 400 mm, in which the size of the notch was 100 x 30 x 5 mm, as shown in Fig. 1(b) [9]. The interfacial flexural strength (f_{ft}) was calculated by Eq. (3).

$$f_{ft} = \frac{3}{2} \cdot \frac{\left(P_u + \frac{mg}{2}\right)L}{b(d - a_o)^2} \quad (3)$$

where mg is the weight of the specimen, L is the span of the specimen (340 mm), b is the width of specimen (100 mm), d is the depth of specimen (100 mm) and a_o is the depth of the notch (30 mm).

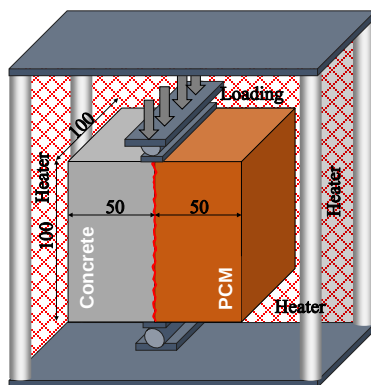
In addition to the flexural strength, the load-displacement in the mid-span of specimen can also be obtained by performing three-point bending tests. Based on the results, the fracture energy was calculated from Eq. (4).

$$G_f = \frac{W_0 + W_1}{A_{lig}} \quad (4)$$

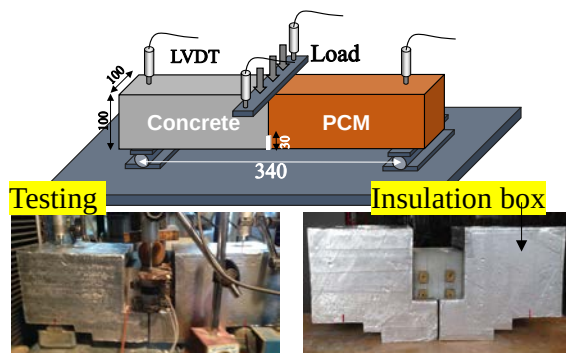
$$W_1 = 0.75 \left[\frac{L}{L_0} m_1 + 2m_2 \right] g \cdot CMOD_c \quad (5)$$

where G_f is the fracture energy (N/m), W_0 is the area below the load-displacement curve up to the rupture of the specimen (N.m), W_1 is the contribution by the dead weight of the specimen (Eq. (5)) and loading jig (N.m), A_{lig} is the area of the broken ligament (m²), L_0 is the total length of the specimen (m), m_1 is the mass of the specimen (kg), m_2 is the jig placed on the specimen (kg), g is the gravitational acceleration (m/s²) and $CMOD_c$ is the crack mouth opening displacement at the time of rupture (m).

The polymers were extracted from the PCM after conducting the mechanical tests. Large size pieces of PCM were ground into a fine powder, which can pass through a 150 µm sieve. The fine powder was then put into a container and three solvents were used to extract the polymers. After 24 hours of treatment, the mixture was filtered, and the filtrate was evaporated to obtain the polymers. Details of the solvent used and the amount of extracted polymers are presented in Table 3. Then, the extracted polymers were tested to investigate the glass transition temperature (T_g), melting point (T_m) and molecular weight (M_n). The state of the polymers transits from a glassy or crystalline phase to a rubbery phase after T_g , whereas it shifts to a viscous phase after T_m . Both, T_g and T_m , are the intrinsic properties of the polymer and the change in such properties can change the mechanical behaviour of the polymer. A DSC test was performed following the ASTM guidelines [28]. T_g was observed from the DSC curve as a midpoint of the tangent between the extrapolated baseline before and after the transition, while an endo-thermal peak represents the T_m of the polymers. The DSC energy was used against the temperature from -50 to 150 °C at the rate of -10 °C/min, in which T_g and T_m were measured in the second cycle of heating. In addition, the M_n of the polymer was measured by conducting a GPC test, which is a widely used methodology [12].



(a) Split tensile strength



(b) Flexural strength

Fig. 1. Geometry details and schematic diagrams of the composite specimens for bond test evaluation (all units are in mm).

Table 3. Polymers extracted in mg using different solvents.

Solvent	PCM exposed to several exposure conditions				
	Ref.	T _{SD}	T _{MD}	T _{DN}	T _{SV}
Chloroform (CHCl ₃)	770	138	73	175	215
Tetrahydro Furan (THF)	246	58	12	10	24
Methanol (MeOH)	12	47	17	5	0

Table 4 presents the summary of the different tests conducted and the number of specimens used under each exposure condition. The reference specimen was not exposed to any environmental condition and tested at 25 °C. Fig. 2 presents the comprehensive summary of the experimentation.

Table 4. Summary of the test and number of specimens corresponding to each test

Exposure Conditions		T _{SD}		T _{MD}			T _{DN}			T _{SV}	
Testing temperature (°C)		20	60	20	60	20	30	60	60	25	5
PCM	Compression	3	3	---	---	---	---	---	---	---	---
	Split	3	3	3	3	3	---	---	3	3	3
	Flexure	3	3	3	3	---	---	---	---	---	---
Composite	Split	3	3	3	3	3	3	3	3	3	3
	Flexure	3	3	3	3	---	---	---	---	---	---
Polymers	T_g	2		2			2			2	
	T_m	2		2			2			2	
	M_n	1		1			1			1	

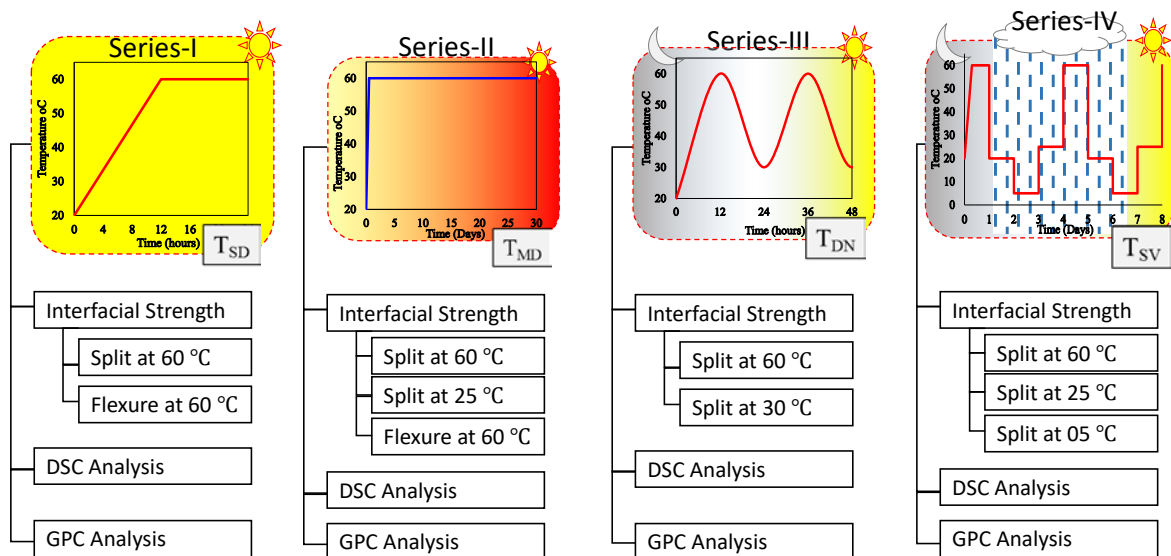


Fig. 2. Summary of the experimentation and exposure conditions.

3. RESULTS AND DATA DISCUSSIONS

3.1 Mechanical Strength

3.1.1 Influence of short temperature duration

To study the influence of a short duration, the specimens were exposed to 60 °C for 24 hours. The mechanical properties of the specimens were obtained by conducting compressive, split tensile and flexural tests before and after exposing the specimens at an elevated temperature. Table 5 presents the mechanical properties of the bulk specimens of concrete and PCM. The values in the parenthesis indicate the standard deviation among the three specimens. The compressive and tensile strength reductions of concrete were 15.97 and 17.68% at 60 °C, respectively, compared to strengths at 20 °C. Meanwhile, the PCM mechanical strengths reduction was more distressing compared to concrete strengths reduction, as shown in Fig. 3. More than a 20% reduction in the compressive and flexural strength was observed at an elevated temperature. The mechanical reduction of concrete was due to the difference in the thermal expansion coefficients between the aggregate and cement paste, which generated high internal stresses, ultimately resulting in micro-cracks and cracks forming at the interfacial transition zone (ITZ). The cracks at ITZ degrade the bond between the aggregate and cement paste, which deteriorate the concrete, hence the specimen was tested at an elevated temperature. The strength reduction may also be due to the porosity increase of the concrete at an elevated temperature, as serious damages were generated at the microstructural level when concrete was dried in the oven at 60 °C [29]. During drying, some of the fine pores collapsed from the stress from the surface tension of the receding water menisci. Ultimately, this process resulted in larger pores, reducing the mechanical strength of the concrete with an increase in porosity [30]. PCM is also a cementitious material with a high cement content and a significant reduction in the mechanical strength with temperature is obvious. The cohesive mechanism of the PCM is the formation of polymer films, which surround the hydrated products and result in a strong ITZ [2]. The polymer films may be damaged by the high temperature due to the high temperature sensitivity of polymers, resulting in the deterioration of the PCM [31, 32]. Therefore, a detrimental influence due to short duration temperature on the mechanical properties of concrete and PCM was observed and the mechanical degradation of the PCM was more severe than that of concrete.

Table 5. Mechanical properties of concrete and PCM under T_{SD} .

Strength	Material	Temperature (°C)				Reduction in Strength (%)
		20 °C		60 °C		
Compressive	Concrete	38.20	(0.86)	32.10	(1.10)	15.97
	PCM	42.91	(1.15)	33.85	(0.53)	21.12
Split	Concrete	2.88	(0.23)	2.37	(0.44)	17.68
	PCM	3.31	(0.13)	2.84	(0.05)	14.19
Flexural	PCM	4.22	(0.19)	3.30	(0.01)	21.65

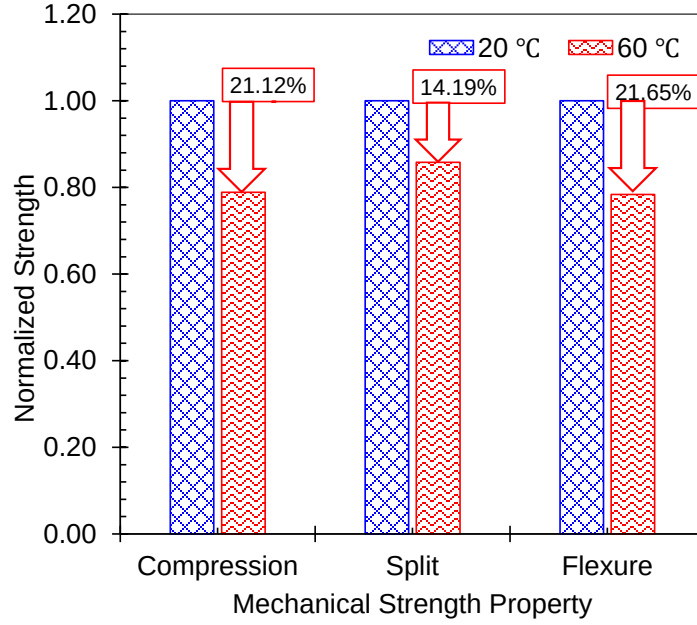


Fig. 3. Normalized mechanical strengths of the PCM bulk specimens under T_{SD} .

The tensile strengths of the bulk and composite specimens under the short duration temperature exposure condition (T_{SD}) is presented in Fig. 4. It can be seen that the composite specimens have a lower tensile strength compared to the bulk specimen, even at a normal temperature (20 °C). For the split and flexural tensile strengths, the reductions were respectively 21.70 and 14.37% that of the corresponding bulk PCM strength at 20 °C. The strength reduction of the composite specimen was due to the weak interface between the two constituents. Although adequate roughness was provided on the substrate concrete and the PCM has excellent adhesive properties, the interface is still the weakest zone. At an elevated temperature, further reductions of 36.20 and 18.93% in the composite specimens were observed for the split and flexural strength, respectively. The governing factors for the mechanical performance of the composite specimens are the interface condition and the strength of the constituents. It was observed that mechanical strength of the constituents reduced with temperature. The interface is the most porous layer compared to the rest of the specimens, and the high porosity leads to a reduction in strength. The porosity further increases at an elevated temperature, which may lead to a further reduction in the strength of the composite specimens. Both concrete and PCM have different thermal expansion coefficients, so the thermal stresses are generated at the interface that cause the deterioration, resulting in a weak bond strength with a change in temperature. Thus, the reduction in tensile strength with a temperature increase was higher for the composite specimens compared to the bulk specimens.

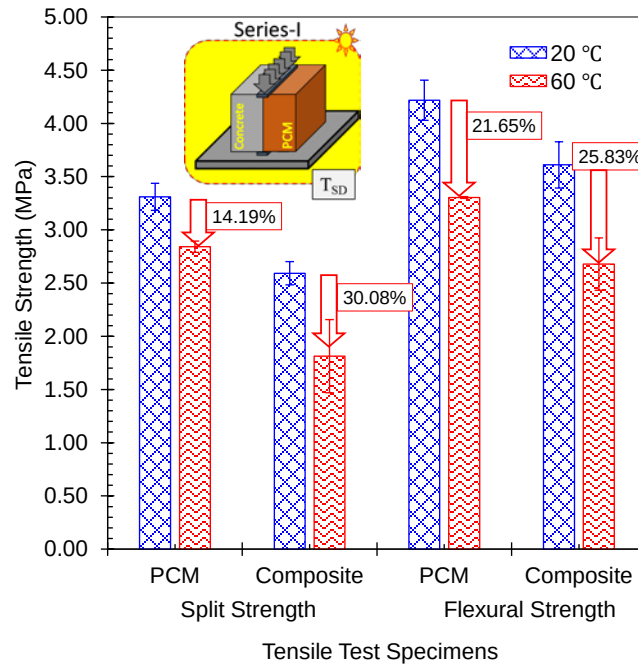


Fig. 4. Tensile strength of PCM and its composite under T_{SD} .

After the mechanical tests, the failure modes of the specimens were obtained. The failure modes of the composite specimen include adhesive failure of the interface, cohesive failure of the concrete or PCM and partial adhesive and partial cohesive failure of the materials. The possible failure modes of the composite specimens are classified in Fig. 5 along with an explanation of all abbreviations used to describe the failure modes. As shown in Fig. 6, the failure mode of the control specimens (tested before any exposure condition) was adhesive failure (Fig. 6(a)), whereas at an elevated temperature a hybrid type of failure mode was observed as most of the PCM was attached to the concrete side (Fig. 6(b)). The attached amount of PCM was calculated by importing the image in the Autodesk software (AutoCAD version 2014). The boundary was marked around the attached part and the area of the boundary was measured. For the control specimen, the failure mode was adhesive interface failure, with approximately 90% separation between the concrete and PCM observed. However, at an elevated temperature, 80% of the PCM was attached to the concrete side and a 20% interface can be seen, while the concrete cohesion is completely absent.

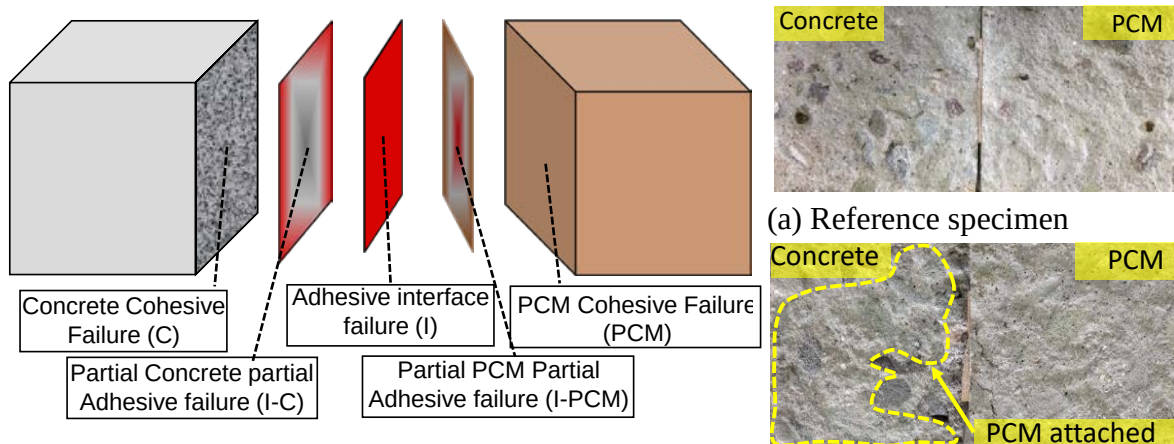
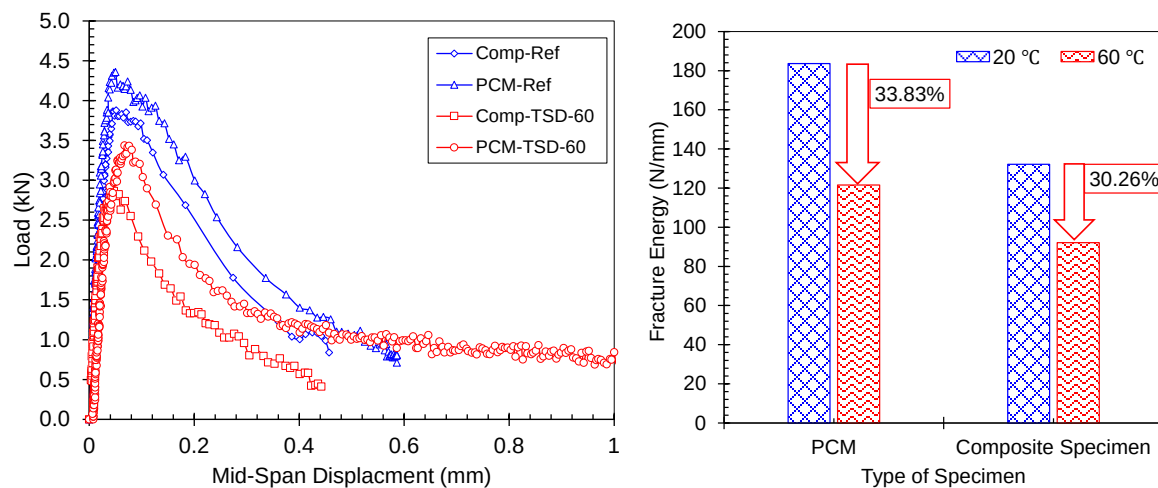


Fig. 5. Classification of failure modes of composite specimens.

(b) Specimen tested at 60°C
Fig. 6. Failure mode of split specimen tested under T_{SD} .

From the three-point bending test, the load-displacement relationships were obtained. Based on the load-displacement curve, the fracture energy was also calculated based on Eqs. (4) and (5). The results for the PCM and PCM-concrete composites at 20 °C and 60 °C were compared, as shown in Fig. 7. It is clear that the ultimate load, slope at the elastic stage and the area below the load-displacement curve all reduced dramatically after exposure to 60 °C. There is a clear tendency about the mechanical reduction of the PCM and PCM-concrete composites. The load-displacement curve can be clearly seen as two stages: ascending and descending. As observed from the ascending stage, both the flexural strength and elastic modulus reduced with the elevated temperature. Although the flexural behaviour of the bulk PCM specimen is generally superior to the PCM-concrete composites specimen due to the weak point of PCM-concrete interface, it seems that the fracture energy reduction for bulk PCM specimens was more severe than the PCM-concrete composite specimen.



(a) Load displacement relationship

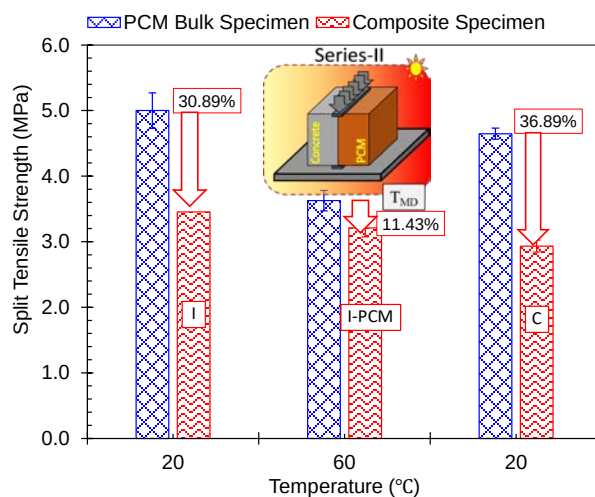
(b) Fracture energy

Fig. 7. Three point bending test on the PCM and its composites under T_{SD} .

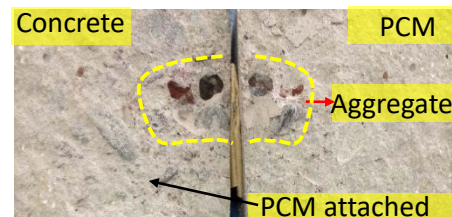
3.1.2 Influence of moderate temperature duration

A moderate temperature duration was considered to simulate one summer season (almost three months) in a tropical region where the temperature may rise to 60 °C for few hours during the day. This duration was accelerated in a laboratory by exposing the specimen in an oven at 60 °C for 30 days. The specimens were mechanically tested at elevated temperature as well as after cooling down. The results of the split tensile strength are presented in Fig. 8(a) and the strength degradation can be clearly observed. For the PCM bulk specimen, the strength reduction was more severe at a moderate duration exposure (27.49%) compared to short duration exposure (14.19%) when tested at an elevated temperature. Although the split tensile strength recovery of the PCM was also observed when tested after cooling until room temperature, the tensile strength was still less than that of the control specimen. The increase

in the tensile strength after cooling was 21.99% that of the elevated temperature and was less than the control specimen by 7.05%. For a composite specimen, the bond strength reduction was also observed with temperature and a further reduction was observed after the specimen was cooled, as shown in Fig. 8(a). The reductions in the bond strength were 7.08 and 15.12% at elevated temperature and after cooling, respectively, compared to control specimen. The bond strength reduction was relatively low (15.12%) in the moderate duration exposure compared to the reduction in short duration (30.08%) when tested at an elevated temperature. The smaller reduction during moderate duration exposure was due to the enhancement behaviour of the concrete at a high temperature. Continuous drying of concrete causes an increase in the Van der Waals forces of attraction in the hydrated products, which results in an improved microstructure of cement paste and ultimately results in improved mechanical strength [33]. Additionally, the fact that porosity increases parabolically with moderate temperature and continuous exposure may reduce the porosity, resulting in mechanical strength improvement of concrete [30]. The behaviour of the composite specimens was also discussed in light of the failure mode, as presented in Fig. 8(b). At an elevated temperature, the I-PCM failure mode was again observed, similar to the short duration temperature case, whereas concrete cohesive failure was observed when the specimen was tested after cooling. The PCM strength recovered after cooling, which may also be the result of strong adhesion between the concrete and PCM. Hence, the weakest zone is the concrete compared to the PCM and PCM-concrete interface, which resulted in concrete cohesive failure.



(a) Split tensile strength



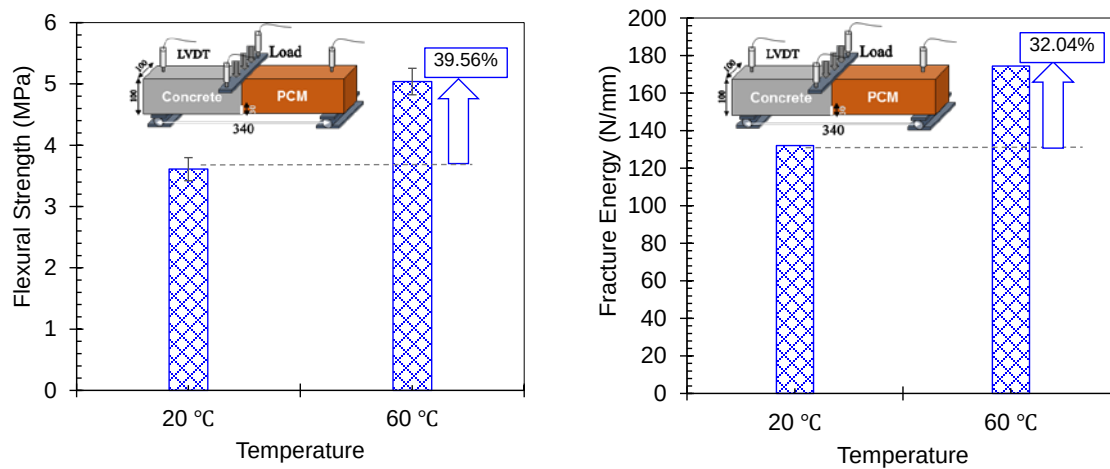
(b) I-PCM failure for T_{MD} at 60 °C



(c) Cohesive concrete failure after cooling

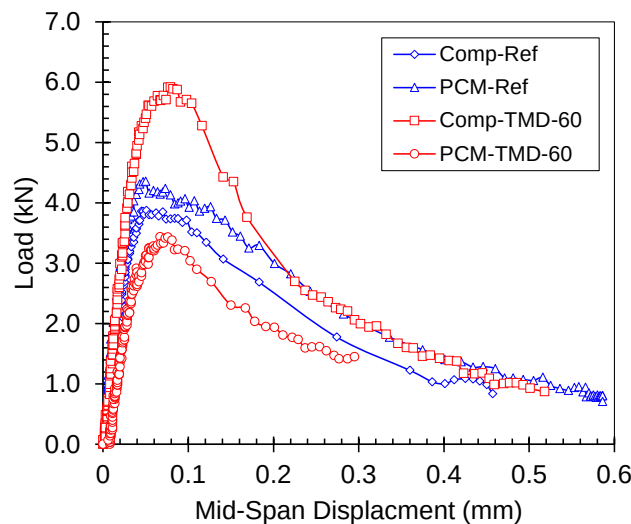
Fig. 8. Tensile strength evaluation under T_{MD} along with the failure modes.

The three point bending test was conducted to evaluate the load displacement relationship, flexural strength and fracture energy under the moderate duration exposure condition (T_{MD}), as the results presented in Fig. 9. The exposure period was 45 days instead of 30 days since there was less influence of moderate duration compared to the short duration exposure condition on composite specimens. The flexural strength of the bulk PCM specimen was also measured and a 21.50% reduction in the flexural strength was observed at an elevated temperature. Since the concrete strength at an elevated temperature during a long exposure condition can increase, Fig. 9(a) presented 39.56% increase in flexure strength with temperature. A similar trend for the fracture energy was also observed and a 32.04% increase in the fracture energy was found (see Fig. 9(b)). The mechanical variation tendency can also be seen in the load-displacement relationship, as shown in Fig. 9(c).



(a) Interfacial flexural strength

(b) Interfacial fracture energy



(c) Load displacement relationship

Fig. 9. Three point bending test on the PCM and its composites under T_{MD} .

3.1.3 Influence of temperature cycles

Cyclic temperature conditions were applied by simulating the day-night variation of summer and a seasonal variation of the tropical region. For the day-night variation case, 60 and

30 °C were set as the day and night temperature, respectively. For the day-night exposure condition (T_{DN}), the interfacial split tensile strength was evaluated at both temperature levels after exposure to 30 cycles, with the results presented in Fig. 10(a). A detrimental influence on the bond strength at an elevated temperature and recovery after cooling down was also observed by conducting tests at different temperatures. The reduction of the bond strength at an elevated temperature is consistent with the results of short and moderate duration exposures. The maximum bond strength reduction was observed for a short duration and the least reduction was observed for moderate duration, whereas the day-night cyclic influence was close to the short duration influence. Since the temperature was cyclic in the day-night variation condition, the PCM deteriorates at an elevated temperature and may restructure itself after cooling. The mechanical strength of concrete may also be improved by the cyclic temperature condition as explored in the previous study [34], in which the concrete was exposed to thermal cycles and the temperature level was also moderate (65, 75 and 90 °C). The bond strength increase was 18.91% from testing at 60 to 30 °C, but the cooled strength is still 13.27% less than that of the control specimen. The variation in the bond strength with temperature can also be revealed by the failure mode, as presented in Fig. 10(b). The control specimen underwent failure by adhesive debonding, whereas the failure mode shifted to PCM cohesive failure due to the deterioration of the PCM with temperature, as shown in Fig. 10(b). However, when the composite specimen was tested at 30 °C, as presented in Fig. 10(c), the failure mode again shifted to the adhesive interface failure due to the improvement of the PCM and concrete strength at a low temperature condition. It can be concluded that bond strength reduces with temperature and is recovered when tested at a low temperature.

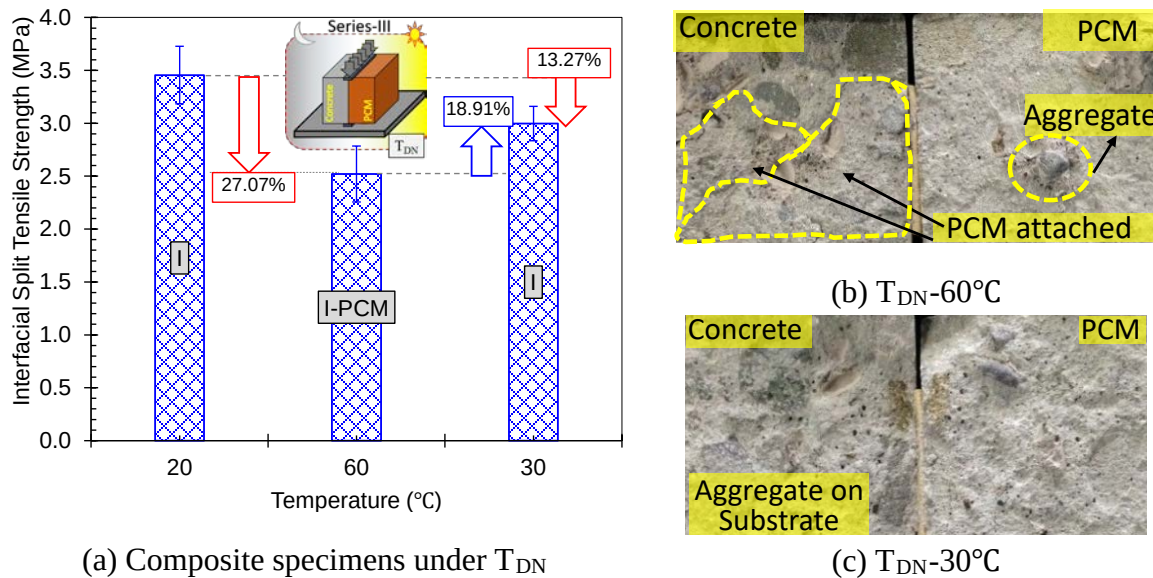
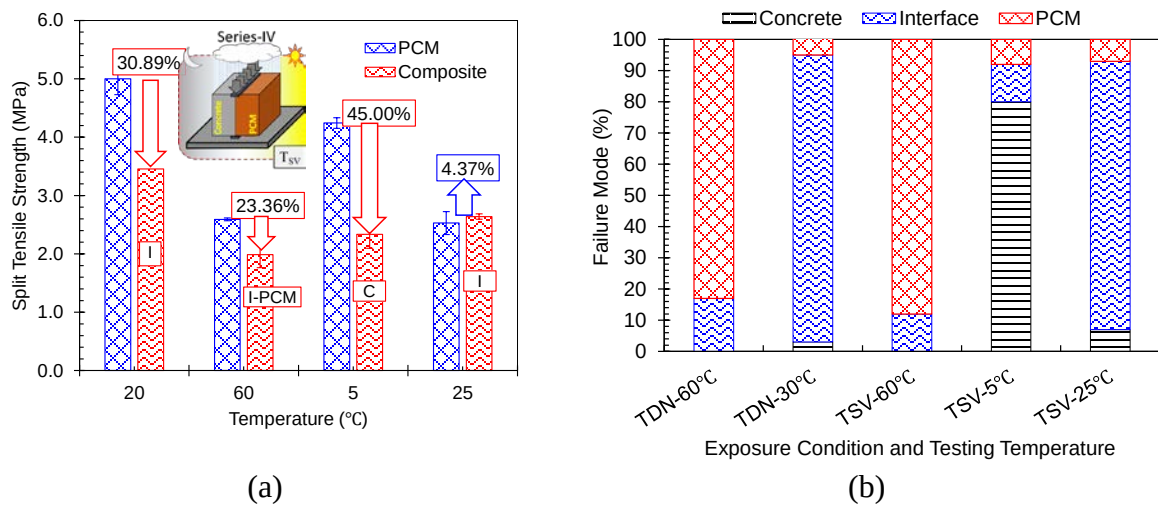


Fig. 10. Split tensile strength and failure mode under T_{DN} .

The seasonal variation of summer, rain, winter and spring was simulated by exposing the specimen to 60 °C, immersion in water, approximately 5 °C, and 25 °C, respectively. One season was represented by exposing the specimen for 1 day, with four days needed to complete one

cycle of the seasonal variation exposure condition (T_{SV}). Mechanical tests were performed after 10 cycles of exposure at each temperature. The results of the tensile strength of the PCM and bond strength of the composite specimens are presented in Fig. 11(a). The PCM strength was reduced when tested at 60, 5 and 25 °C by 48.16, 15.15 and 49.42%, respectively, compared to the control specimen. A significant improvement of approximately 63.70% was observed when the specimen tested close to the T_g temperature, compared to the specimen tested at 60 °C, which agrees with the findings from other studies [2, 32]. Due to the cyclic conditions, the polymers in the PCM may degrade and cannot recover fully. This may be the main reason that there was marginal difference between the PCM tensile strength tested at 60 and 25 °C.

For the composite specimens, the bond strength reduction was observed under all exposure conditions compared to the control specimens (see Fig. 11(a)). The reduction of the bond strength at an elevated temperature was again the maximum among all conditions and the strength was 42.52% less than that of the control specimen. At 5 and 25 °C, the bond strength reductions were 32.47 and 23.61%, respectively, compared to the control specimen. The recovery in the bond strength from an elevated temperature was also observed at 17.48 and 32.88% when tested at 5 and 25 °C, respectively. In all cases, the flexural strength of the composite specimen was less than the bulk PCM specimen, with the exception of the specimen tested at 25 °C. Although the bond strength increase due to cooling was marginal (4.37%), the failure mode was adhesive failure and the concrete substrate was also attached to the PCM side. The failure modes of all specimens of cyclic temperature conditions are explained quantitatively in Fig. 11(b) and a pictorial view of the failure surfaces under T_{SV} are mentioned in Fig. 11(c-e). It can be seen from Fig. 11(b) that at an elevated temperature, most of the PCM (approximately 80%) were attached to the concrete side under both cyclic conditions (T_{DN} -60 °C and T_{SV} -60 °C), whereas adhesive failure was observed at a normal temperature condition (T_{DN} -30 °C and T_{SV} -25 °C). Concrete cohesive failure was observed when the specimen was tested close to T_g . In the pictorial views of the failure surfaces, the attachment of the material was marked, making it clear that most of the PCM is attached to the concrete side at elevated temperature, which verifies the degradation of the PCM at an elevated temperature.



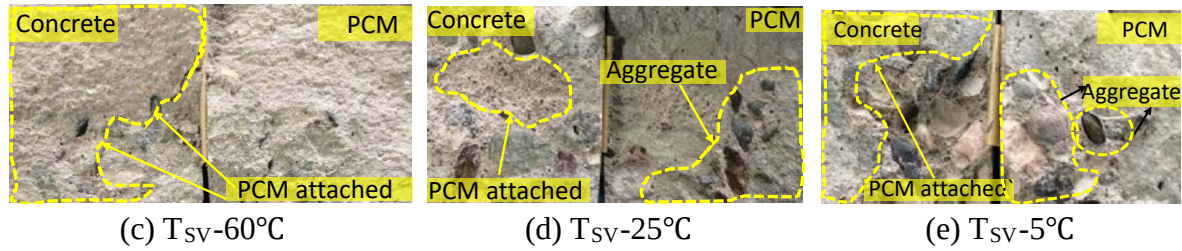


Fig. 11. Split tensile strength and failure mode under T_{SVs} .

3.2 Polymer Properties

3.2.1 Glass transition and melting point

DSC tests were conducted, and the results are plotted to investigate the degradation or decomposition in the physical properties of polymers, as shown in Fig. 12. The glass transition temperature (T_g) and melting point (T_m) are considered as the two basic properties of the polymers and the behaviour of the polymer significantly varies at different temperature levels. Due to different exposure condition, the design value of T_g could be changed, which indicates the variation in the structure of polymer. This change can cause the deterioration of polymer film and ultimately damage the PCM and the adhesive layer. Plasticization of the polymers occurs after T_g , which may degrade the adhesive property of the composite specimen. Since the polymer film could penetrate into concrete substrate and contribute to the bond performance, the degradation of mechanical properties of polymer can affect the bond strength significantly. Generally, epoxies and adhesive have polymers with a T_g of more than 50 °C and it is adequate for application in most of the regions. However, in the case of PCM, T_g of polymer is set below 10 °C to make it a soft and flexible material. Due to the rubbery phase, the polymer can be easily mixed with other constituents of the PCM; cement, additives and aggregate, etc.. After T_m , polymer turns to viscous phase which may totally lose the strength, as well as the part of bond strength contributed by the polymer film.

As shown in Fig. 12, the T_g value of the reference polymer was 8.84 °C and a small variation in T_g was observed under different exposure conditions. Its value decreases to 7.74 °C when the polymer was exposed to a short duration temperature (T_{SD}), and further reduced to 7.27 °C when exposed to the moderate temperature duration (T_{MD}), as shown in Fig. 12(a) and Fig. 12(b), respectively. Fig. 12(c) presents the DSC curves of polymers exposed to cyclic temperature condition along with the reference polymer. The value of T_g decreases to 6.42 °C for the T_{DN} exposure condition, whereas an increase in T_g was observed when the polymer was exposed to T_{SV} , compared with reference polymer. The reduction of the T_g value was consistent with short and moderate duration exposure conditions. It can be concluded that the elevated temperature induced the polymer deterioration and the damage is partially irreversible. The increase in T_g may be due to the moisture condition (immersion in water for 24 hours) [11]. A change in the glass transition temperature with different temperature levels were also observed [35]. From Fig. 12 and the discussion in Section 3.1, it can also be concluded that there will be reduction in the mechanical strength of the PCM if the T_g value changes from the manufactured designed value. In contrast, the melting point (T_m) remained almost constant under all designed exposure conditions except T_{MD} (Fig. 12). It may be concluded that severe exposure conditions

change the T_g of polymers, whereas T_m is unaffected. The change in T_g resulted in the deterioration of PCM.

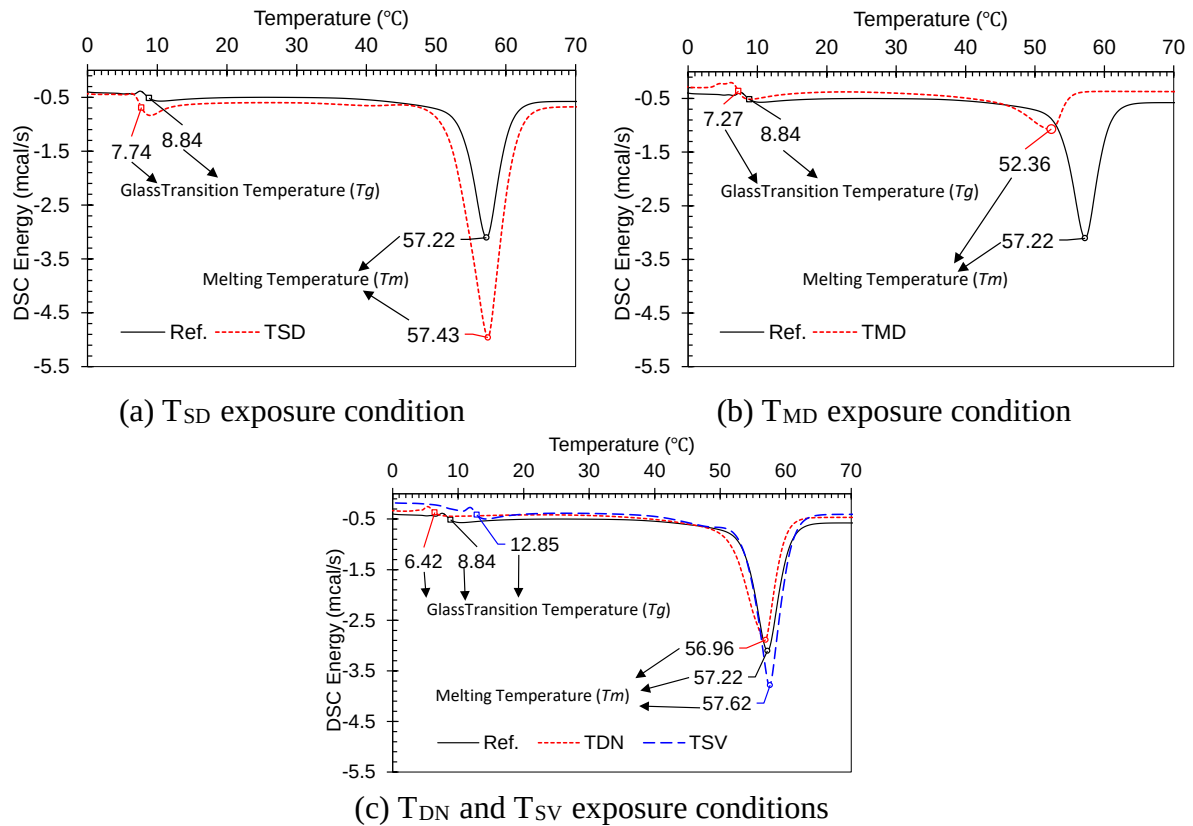


Fig. 12. DSC curve of the polymer after designed exposure conditions.

3.2.2 Molecular weight

Polymerization is a process in which polymer chains form and M_n increases. When the degree of polymerization increases, the mechanical strength of polymer modified cement mortar also increases [36]. Impregnation of polymers in cement mortar with a high degree of polymerization results in an increase in the mechanical strength of the mortar and vice versa [37]. M_n is an important property of the polymer and its evaluation may be beneficial for evaluating the degree of polymerization, decomposition or degradation of the polymers.

The M_n of the polymers was evaluated after being exposed to designed exposure conditions, with the GPC results shown in Fig. 13. It can be observed from the first peak of the GPC curve that the broadness and M_n remain the same in the range of 60,000 to 110,000. The second peak of the GPC curves indicates the oligomers amount. The ratio of the area of the oligomer peak to the area of the M_n peak was calculated at 0.56 for the reference polymer. The increase of the ratio implies an increase in the amount of oligomers and a reduction of the M_n . The ratio increases to 0.68 and 0.86 for the polymers exposed to T_{SD} and T_{MD} , respectively, compared to the reference polymer. A significant increase of the ratio (4.84) was observed under the cyclic temperature condition of seasonal variation. The increase in the ratio was attributed towards the degradation in the polymers, which ultimately influenced the mechanical strength of the PCM. The significant increase in the ratio means there was a significant reduction in the mechanical strength of the PCM. This mechanism was verified by the results

of interfacial split tensile strength under the T_{SV} condition. It was concluded that the GPC results are consistent with the mechanical strength reduction of the PCM under concerned exposure conditions. Therefore, the degradation of the polymers could be attributed to an increase in the amount of oligomers.

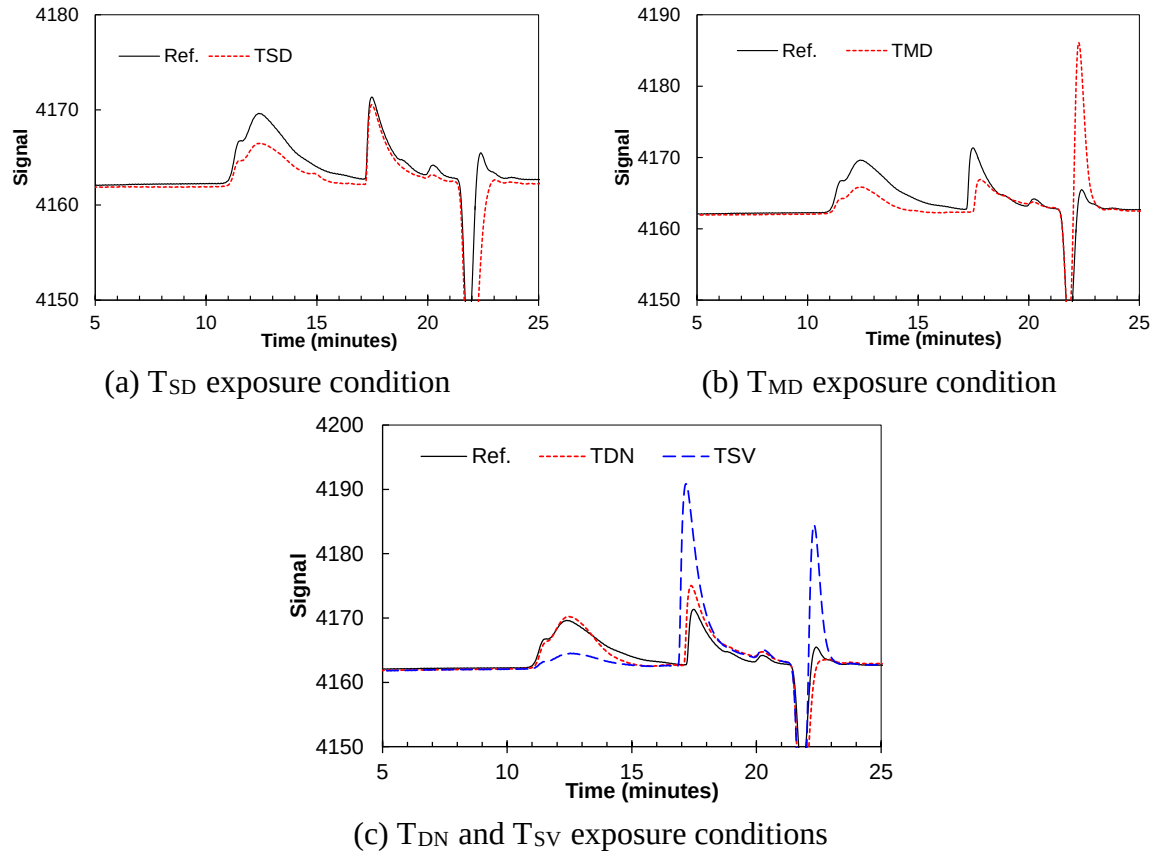


Fig. 13. GPC analysis of polymer after designed exposure condition.

4. CONCLUSIONS

The mechanical performance of PCM and its composite with concrete under different hygrothermal conditions were investigated in this study. By conducting the splitting tensile and flexural tests on composite specimens, a degradation in the bond strength via tension was observed. Four exposure conditions with a maximum temperature level of 60 °C were designed to simulate the influence of short duration (T_{SD}), moderate durations (T_{MD}) and cyclic condition. The cyclic conditions included two cases, a day-night variation (T_{DN}) and a seasonal variation (T_{SV}). The polymers were also extracted after conducting a mechanical test, and the glass transition (T_g) and melting temperature (T_m) were assessed by DSC analysis. The molecular weight (M_n) was measured via GPC analysis for each exposure condition and following conclusions were extracted;

- 1) Compressive, split tensile and flexural strengths of PCM were reduced at elevated temperature. Additionally, the PCM tensile strength was further reduced with an increase in the temperature duration (14.19 to 27.49% for T_{SD} to T_{MD} compared to the reference specimen) and a maximum reduction of 48.16% was observed under the cyclic temperature condition. Recovery in the tensile strength was also observed when it was tested after cooling. For the moderate duration case, the increase in tensile

strength after cooling was 21.99% that of testing under elevated temperature, and it was less than the control specimen by 7.05%.

- 2) The bond strength of the PCM-concrete interface was also reduced at an elevated temperature but the reduction under the T_{MD} condition was less (7.08%) than the reduction under T_{SD} (30.08%) due to the strength improvement of the concrete. For a further increase in the duration, the increase in the flexural strength and fracture energy was observed.
- 3) Cyclic temperature conditions have a detrimental influence on the interfacial split tensile strength and the degradation under T_{DN} was 27.07% and under T_{SV} it was 42.52% when tested at an elevated temperature (60 °C). A recovery in strength was observed if the specimen was tested after cooling. The maximum recovery was observed when the testing temperature was less than T_g . A significant improvement of 63.7% was observed when the specimen was tested close to T_g compared to the specimen tested at 60 °C.
- 4) The failure mode of all composite specimens at the macro level was adhesive failure. However, at the meso-level, the hybrid failure mode was observed and at elevated temperature the failure mode shifted from adhesive failure to partial adhesive and partial PCM cohesive failure. Quantitatively, 80% of the PCM was attached to the substrate concrete when tested at 60 °C under all exposure conditions.
- 5) A change in glass transition (T_g) temperature from the designed value has a detrimental influence on the mechanical strength of PCM. The T_g values vary from the reference polymer by 12.44 and 17.66%, while the PCM split tensile strength reduces by 14.19 and 27.49 from the reference PCM strength when exposed to T_{SD} and T_{MD} exposure conditions, respectively. Similarly, under the cyclic condition of seasonal variation, the T_g value increased by 45.36% from the reference polymer and a reduction in the PCM tensile strength was also significant (48.16%). The T_m value was almost constant under all designed exposure conditions and may have a marginal influence on the mechanical properties of PCM.
- 6) As the temperature duration increases, the ratio of the area regarding the amount of oligomers to the area regarding the molecular weight of the GPC curve was observed, which is consistent with the results of the tensile strength of PCM. The maximum value of the ratio was observed for the T_{SV} condition and a maximum reduction in the tensile strength was also observed under this condition.

ACKNOWLEDGMENT

The authors would like to acknowledge the contribution of Prof. Toshifumi Satoh for his guidance regarding the extraction of polymers and conducting the DSC and GPC tests. The authors are also grateful to Denka Company Limited for providing the polymer cement mortar. This work is supported by the National Natural Science Foundation of China through Grant (Project No. 51708133) and China Postdoctoral Science Foundation (Project No.2017 M622633).

LIST OF NOTATIONS

PCM	Polymer cement mortar
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T_g	Glass transition temperature
T_m	Melting point temperature
DSC	Differential scanning calorimetry
GPC	Gel permeation chromatography
M_n	Molecular weight
R_a	Roughness coefficient
f_{st}	Split tensile strength
$f_{st}(\beta)$	Corrected split tensile strength
P_u	Ultimate load
A	Area of interface
f_{ft}	Flexural strength
d	Depth of specimen
a_o	Depth of notch
G_f	Fracture energy
W_0	Area below the load-displacement curve
W_1	Contribution by the dead weight of the specimen
A_{lig}	Area of the broken ligament
$CMOD_c$	Crack mouth opening displacement
T_{SD}	Short Duration (Series-I)
T_{MD}	Moderate Duration (Series-II)
T_{DN}	Cyclic Temperature; Day-Night variation. (Series-III)
T_{SV}	Cyclic Temperature; Seasonal Variation (Series-IV)
C	Concrete cohesive failure
I	Adhesive interface failure
PCM failure	PCM cohesive failure
I-C	Partial concrete partial adhesive failure
I-PCM	Partial PCM partial adhesive failure

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