Mechanical durability of an optimized polymer concrete under various thermal cyclic loadings – An experimental study


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ABSTRACT
Investigating the tensile strength ($\sigma_t$) and mode I fracture toughness ($K_{IC}$) of polymer concrete (PC) materials due to their quasi-brittle behavior is of great interest to engineers. In this paper, the mechanical durability of an optimized epoxy PC, focused on the two above properties, are experimentally investigated under three different freeze/thaw cycles. The diametrally compressed un-cracked Brazilian disc (BD) and the single edge notch bending (SENB) test configurations are used to measure the split tensile strength and fracture toughness, respectively. The thermal cycles; 25°C to 0°C (cycle-A), 25°C to 70°C (cycle-B) and 0°C to 70°C (cycle-C) applied for 7 days to the test specimens; are chosen according to the climate of Iran in different seasons. Experimental results show the noticeable influence of thermal cycles, especially cycle-B, on both fracture toughness and tensile strength. Heat-to-cool thermal cycle-A and thawing thermal cycle-B indicate the most increase and reduction, respectively on both $\sigma_t$ and $K_{IC}$ in comparison to ambient conditions. Also, it was shown that the fracture toughness and tensile strength of tested PC materials are reduced by increasing the mean temperature values of thermal cycles.

1. Introduction
Specific properties of polymer concrete (PC) materials such as high strength and low weight rather than ordinary Portland cement concretes [1,2], excellent bonding to other materials [3], ability to withstand corrosive environments and chemical attack [4], fast curing and very low permeability [5] made it a very attractive material in various industries. It has been widely used in construction industry and structural engineering applications such as bridge deck, pavement overlay, concrete crack repair [6], waste water pipe, hazardous waste containers, manholes and decorative construction panels [7]. For example as a practical application, authors have recently designed and manufactured drinking water filtration slabs for Tehran water distillation plant using PC [8]. However, it is necessary to characterize the properties of new materials (such as newly developed polymer concretes) introduced for novel applications according to the special conditions that they are encountered. Common mechanical and fracture properties such as modulus of elasticity, tensile strength, bending strength, compression strength, fracture toughness, and fracture energy have been investigated for different type of PC materials at room temperature [2,9–21]. PC is commonly composed of coarse aggregates, polyester or epoxy resin and chopped strand glass fibers. In order to develop the most economical PC, it is necessary to use minimum amount of polymer (which is the most expensive part...
of PC composition) and best method of curing since the polymer part plays the key role on the physical and mechanical properties of PC materials. Several researchers have investigated the influence of different percentage of resin used in the PC ingredients to obtain an optimum and economic PC. For example, Shokrieh et al. [2] proposed weight of resin 19%, Vipulanandan and Paul [20] used 10–20 wt% and Barbuta et al. [21] used 12.8–18.8 wt% for manufacturing an optimum PC.

The main problem of polymeric materials is related to visco-elastic properties of the polymer, which results in creep and high sensitivity to temperature. Mechanical properties of polymers, undergoing temperature variations, change considerably especially within the glass transition temperature range [22–24]. The glass transition takes place over a wide temperature range, which lies between 20 °C and 80 °C for many resins used in civil engineering. This means that the commercial and industrial polymers can experience the glass transition during their service life [24]. Therefore, in addition to the above-mentioned properties, mechanical durability, defined as the ability of a structure to retain its physique-chemical properties after a mechanical damage [25], is a key function for successful using of a new material which is used in severe environments. From this point of view, most of the available investigations were performed on the mechanical durability of PC at a specific temperature or cycle [22–26]. For example, Agavriloaie et al. [22] investigated the effect of 50 frost–thaw cycles in the temperature range from −15 °C to +20 °C, on the compressive strength of PC with epoxy polyurethane acryl. It had a loss of compressive strength of 11.58%. Soraru and Tassone [25] evaluated the mechanical durability and the strength degradation process of some classes of PC materials, using Vickers indentation data at various loads. Recently, the effect of temperature variations ranging from room temperature to 90 °C, was studied by Reis [19] on the flexural and compression strengths of epoxy and unsaturated polyester mortars.

In reality, a construction material is exposed to periodic environment loadings during its lifetime that can be simulated by thermal cycles [27]. Therefore, investigation of mechanical durability for these materials under freeze/thaw thermal cycles is of great interest for engineers. For example, Klemm and Marks [28] optimized the composition of PCs subjected to freezing and thawing cycles to design frost resistant PC materials. They employed two admixtures (methyl hydroxy ethyl cellulose – MHEC and polyvinyl acetate – PVA) with different percentages in the composition of PC material and experimentally obtained the values of compressive and flexural strengths after applying 300 freeze/thaw thermal cycles to the specimens. However, PC materials have shown a quasi-brittle behavior under different types of loadings such as compression and bending [2]. Therefore, it can be concluded that brittle fracture due to tensile cracking is the main cause of the overall failure of PC materials and it is important to evaluate the tensile strength (σt) and the fracture toughness (Kic) of this material. The available research studies that investigated the effect of thermal cycles on the fracture properties of PC materials are described here. Reis and Ferreira [29,11] studied the fracture properties of a typical fiber reinforced polymer concrete subjected to two environmental loadings, i.e., freeze/thaw thermal degradation and atmospheric exposure to evaluate only the stress intensity factor and the fracture energy. In another work [13], they evaluated the fracture properties of an epoxy concrete for two different exposures during one year, spring–summer and autumn–winter periods. The mentioned research reveal that only limited scientific works have been done on the strength properties of PC materials under thermal cycles and most of the available research papers are related to measure Kic at a constant or specific range of temperature or one specific thermal cycle. However, the deterioration of a material depends on how and to what extent it interacts with its surroundings. The environment a PC structure is exposed if considered in terms of sunshine, temperature, rainfall and wind, varies widely in duration, intensity and sequence. As far as the durability of materials is concerned, weight should be given to severe climatic conditions and depends on the confidence level required in the performance of the material, but in general it is the time-averaged climatic factors which should be considered [23].

In this study, the tensile cracking and crack growth resistance behavior of PC material is investigated experimentally under three different freeze/thaw thermal cycles (according to the weathering conditions and climate of Iran at different seasons). Using several diametrically compressed un-cracked Brazilian discs (BD) and single edge notch three-point bending (SENB) specimens made of an optimized polymer concrete, the effect of thermal cycles on both tensile strength and fracture toughness is investigated. It is shown that the amplitude and type of thermal cycles (i.e., cool-to-heat or vice versa cycles) have noticeable influences on the fracture behavior of tested PC material.

2. Experimental programs

Shokrieh and his coworkers [2] have recently studied the optimum percentage of the PC ingredients to obtain the maximum bending and compressive strengths, and also the interfacial shear strength between the PC and inner surface of a steel ring. Using the Taguchi method and performing a set of experimental tests, they found the following weight percentages for an optimized composition of PC: 48.3 wt% of coarse mineral aggregate (with 4–6 mm in size), 32.2 wt% of foundry sand filler (with 0.5–1.5 mm in size), 19 wt% of epoxy resin, and 0.5 wt% of E-glass chopped fibers of length 6 mm. In this study, the same composition was used to investigate the effects of freeze/thaw cycles on the tensile strength and fracture toughness of the optimized PC material. The epoxy resin based on bisphenol F with a polylamine hardener was used to fabricate PC materials. The low viscosity (160–180 cP at 25 °C) of this type of epoxy resin allows easy mixing and finally making an approximately homogenous mixture. The ingredients, especially sand, are attached to each other (agglomerated) if high viscosity resins are used for manufacturing the PC materials. The chopped glass fibers, sand fillers and epoxy resin with the above-mentioned weight fractions were mixed together inside a container to obtain a uniform mixture.

Several test specimens and experimental methods have been used in the past for evaluating the fracture toughness (Kic) and tensile strength (σt) of different quasi-brittle materials including PC materials [11,16,29–38]. A suitable test specimen should have simple geometry and testing set up. The ease of specimen preparation and convince of its testing using available apparatus are other primary requirements of a suitable test specimen. Moreover, since the PCs are among the brittle materials (i.e., weak against the tensile loads), it is preferred to test them under compressive loads rather than the direct tensile loads. Hence, the edge cracked rectangular beam subjected to symmetric three or four point bending [11,16,29], the center cracked circular disc under diametrical compression [30], the edge cracked semi-circular specimen subjected to three-point bend loading [14] are some of the previously used specimens for obtaining the fracture toughness of PC materials. For estimating the tensile strength of PC materials some test specimens such as Brazilian disc (BD) specimen subjected to diametral compression [30,33], rectangular beam subjected to flexural three or four point bend loading [32,33], the split tensile test specimen [35,36], the semi-circular bend (SCB) specimen [14] have also been used by the researchers. Although the loads applied to the mentioned tensile test methods are compressive, the stresses generated at certain locations in the test specimens (i.e., middle edge of bend specimens or the center of diametrically compressed disc specimens) become tensile. Thus, at some critical level of applied load, the specimen is split due to these tensile stresses and the corresponding value of σt can be obtained from the available formula using the splitting load of the specimen. These methods are so called indirect tensile strength determination which is often used for estimating of σt value for brittle and quasi-brittle materials such as rocks, ceramics, asphalt concretes, ordinary cement concretes and polymer concretes.

In this study, the single edge notch bending (SENB) and the un-cracked circular disc subjected to diametral compression (Brazilian disc) were therefore used to obtain the fracture toughness (Kic) and tensile strength (σt), respectively. Fig. 1 schematically shows the geometry and loading configuration of the SENB and SCB test samples. Obviously, it can be seen from this figure that both specimens have simple geometry such that their preparation needs only very simple cubic and cylindrical casts. Moreover, they can be easily tested using conventional fixtures and testing machines and the type of applied loading is compressive for both specimens.
To prepare the test specimens, the mixture of PC was cast into the cubic metal mold with dimensions of $L = 180$ mm, $W = 75.4 \pm 1.06$ mm, $B = 36 \pm 1.59$ mm and also inside a steel ring with diameter of $D = 75$ mm and height of $B = 21$ mm. Accordingly, several SENB and BD specimens were manufactured from the PC material. Before casting, the inner surfaces of the molds were coated with release film to provide de-molding of the PC specimens. The prepared PC samples were cured at room temperature for 7 days and post-cured for 2 h at $80^\circ$C. Also, for preparation of an edge crack in the prismatic beam, a very thin high speed rotary diamond saw with thickness of 0.35 mm was used to introduce a very narrow notch of 20 mm length. Thus, the crack length to width ratio ($a/W$) was about 0.27 in the SENB specimens.

2.2. Test procedure

The prepared SENB and BD specimens were then exposed to three thermal cycles which were selected according to Iran climate in different seasons [2] as follows:

- Cycle-A: $25^\circ$C to $-30^\circ$C for 7 days, half of each cycle takes 12 h.
- Cycle-B: $25^\circ$C to $70^\circ$C for 7 days, half of each cycle takes 6 h.
- Cycle-C: $30^\circ$C to $70^\circ$C for 7 days, half of each cycle takes 12 h.

Fig. 2 shows the profiles of the applied thermal cycles. Thermal cycles were performed by a thermal chamber with a minimum cooling capacity of $-35^\circ$C and a maximum heating capacity of $70^\circ$C. The temperature stability rate is $10^\circ$C/min for the cooling system. But this rate is different for the heating system. For temperatures in the range of $0$ to $30^\circ$C, $0$ to $0^\circ$C and $30$ to $10^\circ$C, the stability rates are 4, 1 and 0.5 $^\circ$C/min, respectively. It should be noted that thermal cycling involves repeatedly cycling a specimen between two temperatures with a sufficient dwell time at either extreme to allow thermal equilibrium to be attained.

The cyclic heat treated samples were then loaded using a servo hydraulic tension/compression test machine. The SENB samples were loaded inside a three-point bend fixture with span of $S = 140$ mm and the un-cracked circular Brazilian discs were compressed diametrally between two flat platens. The tests were carried out at room temperature and under displacement control conditions with a constant cross head speed of 0.5 mm/min. Fig. 3 shows test setup and the fixtures used for the $K_c$ and $\sigma_t$ experiments on the PC material. For conducting the fracture toughness and tensile strength tests, the specimens were loaded monotonically until the final fracture and splitting the samples in two same halves. The complete load–displacement data were recorded during the tests using a computerized data logger. Accordingly, a total number of 36 SENB and 12 BD specimens heat treated at three thermal cycles-A, -B and -C were tested successfully.

3. Results and discussion

Some typical curves of load ($P$) versus mid-span deflection ($\delta$) are presented in Fig. 4 for SENB specimens under thermal cycle A. The load–deflection curves were corrected for possible nonlinearities at initial low loads. These curves were nearly linear for all the samples, showing the brittle failure behavior of the tested materials. Therefore, the fracture toughness and the tensile strength of the tested PC material were determined from the maximum peak load recorded for each test. The scatter may be seen in the results comes from non-uniformity of PC material especially at the crack tip. Similarly, Fig. 5 shows also some load–displacement curves obtained for BD test specimens under cycle B.
The critical mode-I stress intensity factor ($K_{IC}$) for the SENB specimen loaded in three-point bending using the linear elastic fracture mechanics is obtained from [15]:

$$K_{IC} = \frac{3P_{\text{max}}S}{2BW^{\frac{3}{2}}} \left[ 1.99(\frac{a}{W})^{\frac{1}{2}} - 2.15(\frac{a}{W})^{3/2} + 6.1(\frac{a}{W})^{5/2} - 6.65(\frac{a}{W})^{7/2} + 2.7(\frac{a}{W})^{9/2} \right] \left( 1 + \frac{a}{W} \right)^{1/2} \left( 1 - \frac{a}{W} \right)^{1/2},$$

where $P$ is the applied load, $B$, $a$, $W$ and $S$ are the specimen thickness, the crack length, the specimen width and the span of three-point bend test, respectively. The obtained fracture loads and fracture toughness results for three thermal cycles-A, -B and -C are presented in Tables 1–3. The effects of three freeze/thaw thermal cycles on the average value of $K_{IC}$ has been compared in Fig. 6 with the value of $K_{IC}$ at ambient conditions. The value of fracture toughness at ambient condition is provided from Ref. [16] that is equal to 1.8 ± 0.2 MPa$\sqrt{m}$. It is found that thermal cycles-A and -C have approximately the same influence on the fracture toughness of PC and increase it about 11%. While the thermal cycle B decreases the $K_{IC}$ about 16.7% in comparison with the value of fracture toughness at ambient condition. This phenomenon shows that the fracture toughness of PC is affected significantly by thawing cycles because this kind of cycles may affect the transient glass temperature of polymer and subsequently change its properties. Also, for the investigated freeze/thaw thermal cycles the mode I fracture toughness value of the tested PC may vary between upper limit (i.e., cycle-A) and the lower limit (i.e., cycle-B) about 33%.

The indirect tensile strength of the tested PC materials can be determined using the un-cracked BD specimens from the following equation [39]:

$$\sigma_T(\text{BD}) = \frac{P_{\text{split}}}{\pi R^2} \left[ 0.156 \left( \frac{R}{L} \right) + 0.964 \right]$$

where $P_{\text{split}}$ is the critical splitting compressive load. The average values of tensile strength of PC for cycles-A, -B and -C are compared with $\sigma_T$ of the same PC tested at the ambient conditions (with no heat treatment) in Fig. 7. Similar to behavior observed for the fracture toughness data, it is seen from this figure that cycle-A and

![Fig. 4. Load-displacement curves of SENB specimens under thermal cycle-A.](image)

![Fig. 5. Load-displacement curves of BD specimens under thermal cycle-B.](image)

![Table 1](image)

<table>
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<tr>
<th>Specimen no.</th>
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1.994 (±0.2)
cycle-B provide upper and lower tensile strength limits, respectively for the tested PC material. It means that the tensile strength of the tested PC material may vary about 24% due to the type and amplitude of thermal cycles applied to this material. Meanwhile, based on the results shown in Figs. 6 and 7, the discrepancy between the ambient condition and heat treated condition becomes more pronounced when the PC material is subjected to cycle-B (i.e., freeze-to-thaw cycle and only heating). It also reveals that tensile type cracking of PC materials might be affected more under cool-to-heat environments such as cycle-A and -C.

**Table 2**

<table>
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<tr>
<th>Specimen no.</th>
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<th>( K_{\text{IC}} ) (MPa m(^{1/2}))</th>
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**Table 3**

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<td>1.985 (±0.15)</td>
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Fig. 6. Comparison of fracture toughness of PC under three thermal cycles and ambient conditions.

Fig. 7. Tensile strength of PC material tested under ambient conditions and three thermal cycles-A, -B and -C.

Fig. 8. Typical fracture trajectory observed for a BD specimen made of PC material.
25 °C to 70 °C) and -C (i.e., from −30 °C to 70 °C) have not positive influence on the mechanical strength properties of the PC material and increase the risk of tensile rupture and sudden crack growth. Hence, the use of such materials is recommended for those regions having moderate climates in different seasons of a year (typically with the temperature amplitude of ±25 °C). Furthermore, it is seen from the experimental results that the tensile strength and fracture toughness values can be attributed to the mean value of temperature defined as follows for each cycle:

$$T_m = \frac{T_{\text{max}} + T_{\text{min}}}{2}$$

(3)

where $T_{\text{max}}$ and $T_{\text{min}}$ are the maximum and minimum values of temperature in each thermal cycle. Accordingly, the corresponding values of $T_m$ for cycles-A, -B and -C is equal to −2.5 °C, 47.5 °C and 20 °C, respectively. In Figs. 10 and 11, the variations of $K_{ic}$ and $\sigma_t$ versus $T_m$ have been plotted for the investigated PC materials. It is seen from these figures that both fracture toughness and tensile strength are reduced by increasing the mean temperature value. Empirical polynomial curves (i.e., Eqs. (4) and (5)) have also been fitted to the experimental results to obtain an empirical prediction for fracture toughness and the tensile strengths for other mean temperature values. When the PC material is subjected to a thermal cycle with subzero mean temperature (such as cycle-A), it is expected to see an increase in the overall stiffness of PC mixture, because of higher rigidity and stiffness of resin at low temperature. Hence, greater fracture loads might be obtained at the onset of fracture or crack initiation stage of tested PC materials at low temperatures. Similar observation has been used for low temperature cracking and failure behavior of other viscosity affected materials such as bitumen and binder of asphalt mixtures. For example, Aliha et al. [40] recently showed that the low temperature fracture toughness of an asphalt concrete containing stiffer bitumen (60/70 with PG 64-22) is more than the fracture toughness of those asphalt mixtures made from softer bitumen (85/100 with PG 58-28). However, by increasing the mean temperature to higher than zero values (such the case of cycle-B investigated in this research) the stiffness of resin used in the mixture of PC is reduced more in comparison with the subzero or ambient temperatures and the load bearing capacity and the resistance of material against tensile type cracking becomes smaller. Moreover, the obtained empirical curves reveal that while the tensile strength of tested PC materials is more sensitive to the low mean temperatures (i.e., in the typical range of −2.5 °C and 20 °C), the variations of $K_{ic}$ value is more pronounced for higher mean temperature ranges (typically $T_m$ greater than 20 °C). It means that the risk of tensile type failure of un-cracked PC materials should be high during winter but the probability of unstable fracture growth of pre-existing cracks becomes more in hot seasons of the year.

$$K_{ic} = -3.46e^{-4}T_m^2 + 5.36e^{-3}T_m + 2.011$$

(4)

$$\sigma_t = 1.247e^{-3}T_m^2 - 9.915e^{-2}T_m + 8.78$$

(5)

Also, thawing cycle-B indicates the most reduction (about 17%) in both tensile strength and fracture toughness. This is why the temperature in each cycle reaches to glass transition temperature of bisphenol-F epoxy resin ($T_g$ = 80 °C) used in PC material. By increasing the temperature up to the $T_g$, the resin begins to lose its crystalline structure and the stress–strain behavior of polymer change from Hookean brittle behavior to nonlinear behavior with tearing in fracture [41]. It is finally noted that the main scope of
this research was the experimental study of freeze/thaw cycles on the tensile type failure resistance of PC material. More comprehensive and theoretical based studies should also be performed in the future to understand the mechanisms of experimental findings and theoretical prediction of the test observations on the durability of polymeric concretes.

4. Conclusions

The mechanical durability of a polymer concrete (PC) is of great interest due to two reasons: (1) a polymer as a binder and key part of PC has high sensitivity to temperature change; (2) PC has shown a quasi-brittle behavior under different mechanical loadings. Therefore, the effect of different freeze/thaw cycles on the tensile strength (σt) and mode I fracture toughness (Kc) of an optimized PC material were investigated experimentally. The lower and upper range of each thermal cycle is selected in accordance with weathering conditions and climate of Iran in various seasons at different locations of country. The following points are concluded from the experimental results:

- For the whole thermal cycles, tensile type failure was observed and freeze/thaw cycles did not change the failure modes of PC.
- Noticeable influence of thermal cycles observed on both σt and Kc of the tested PC material. For example, thawing cycle-B and heat-to-cool cycle-A had the highest reduction and increase on both properties, respectively.
- Cycles-A and -B provided respectively upper and lower limits for both Kc and σt, whereas cycle-C had an intermediate effect between cycles-A and -B.
- Comparison of thawing and freezing cycles showed that tensile type cracking of PC materials might be affected more under cool-to-heat environments such as cycle-A and C (without attention to decrease or increase) rather than heat-to-cool one.
- Heat-to-cool thermal cycles increase the load bearing capacity and durability of the tested PC material, while the cool-to-heat thermal cycles may considerably increase the risk of brittle tensile type fracture.
- Both Kc and σt were reduced by increasing the mean temperature (Tm) of the investigated thermal cycles. According to the experimental findings of this research variation of Kc was more sensitive to the higher Tm values and conversely the tensile strength value of tested PC material was more influenced by the lower mean temperature values of freeze/thaw thermal cycles.

References


