Behavior of high-strength polypropylene fiber-reinforced self-compacting concrete for thermal storing structures

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Abstract

The utilization of structural high-performance concrete reinforced with polypropylene fibers in applications demanding long exposure times to high temperatures, such as in case of thermal energy storage systems, is analyzed in this work. Different temperatures: 100, 300, 500 and 700°C (hot), as well as cooled-down states (cold) and different exposure times (6, 24 and 48 h) have been analyzed. The thermogravimetric analysis, fracture behavior, compressive strength, the Young's modulus and tensile strength of concrete were also experimentally determined and subsequently, a comprehensive analysis of the thermal and mechanical behavior of high-performance concrete under different thermal conditions has been carried out, broadening with longer exposure times the available results about the behavior of high-performance fiber reinforced concrete subjected to high-temperatures. When the temperature is increased, the mechanical properties of this concrete diminishes. The results shown that once thermal and moisture equilibriums are reached there is no influence of the exposure time. They provide useful information about the influence of different parameters of fiber-reinforced concrete subjected to high temperatures.

Keywords

high-strength concrete, polypropylene fibers, high temperatures, exposure time, fracture behavior

Notation

CA	Coarse aggregate	D1	PP fiber reinforced concrete with fibers of 6 mm length
CMOD	Crack mouth opening displacement	D2	PP fiber reinforced concrete with fibers of 24 mm length
D0	concrete without PP fiber reinforcement	DSC	Scanning calorimetric analysis

FA	Fine aggregate	S	Span of the fracture specimen: mm
FPZ	Fracture process zone	m	Mass of fracture specimen: kg
HO mm	Thickness of holder of clip gauge:	g m/s^2	Acceleration due to gravity: 9.8
LA	Limestone aggregate	P	Load: N
LVDT	Linear voltage displacement transducer	A	Remote tail constant
PC	Portland cement	C_i	Initial compliance: mm^2/N
PP	Polypropylene	E_c	Young's modulus: GPa
PPF	Polypropylene fiber	f_c	compressive strength: MPa
SCC	self-compacting concrete	f_{ct}	Tensile strength: MPa
SF	Silica fume	f_{cti}	Splitting tensile strength: MPa
SP	Superplasticizer	G_F	fracture energy: N/m
TGA	Thermo-gravimetric analyses	l_{ch}	Characteristic length: mm
W	Water	W_F	Total work of fracture: Nm
a mm	Notch depth of fracture specimen:	W_{nm} Nm	Non measured work of fracture:
B mm	Width of the fracture specimen:	α	Relative notch depth: mm
D mm	Depth of the fracture specimen:	δ	Displacement at midspan: mm
L mm	Length of the fracture specimen:	δ_u mm	ultimate displacement at midspan:
		$\mu(\theta)$	normalized bending moment

1. Introduction

The use of renewable energies as main energy source is a greatly topic of interest for countries that have not fossil energy sources and for those that are seriously committed on the reduction of CO₂ emissions into the atmosphere. For this reason, although renewable energy sources are being applied currently, their technologies go on being objective of researching [1–3]. Solar energy is one of the most commonly renewable energy established all over the world because it is abundant in many places and the prediction of the hours of solar radiation is very

easy compared to other renewable energy sources [2]. The technology for obtaining electricity by means of solar energy is the only one that allows its storage. The development of a solar energy storage system is then crucial to provide energy more uniformly and it would lead this renewable energy to be more competitive versus fossil energy systems. Storage system materials must bear not only loads but moreover effects of high temperature at variable time periods during plant operation. Other uses of concrete in thermal system of storage are the collector of vapor. These systems use directly the vapor as means of thermal energy storing. When the liquid water is heating up to obtain the vapor, this goes to the turbine or is stored in concrete tanks or basins to be used when the solar radiation is interrupted. In these cases, the concrete will stand subjected to high temperatures for long time being in direct contact with vapor. Thus, the stealing, calorific power and the expenses of the continent material are very important aspects. The concrete collector must hold a temperature range around 100°C and 300°C approximately.

Basically, the energy storage systems must be composed of two materials. One of them should be a material with high capabilities to storage the heat, such as mixtures of molten salts or phase-change materials [2,4–6], and the another one should be a structural material working as containment vessel of the former, having to be properly isolated by using refractory materials to guarantee that there is no damage in it by temperature. In this regards, concrete is emerging as a material with twofold application. On the one hand, concrete is one of the most employed structural materials due to its mechanical properties, workability on site, easy handling, availability in anywhere of the world and relatively low cost [2]. On the other hand, concrete has interesting heat storage capabilities because of its thermal properties [2]. In this sense, can be found in the literature heat storage technologies using concrete as a heat storage material, using a series of pipes containing water as heat transfer fluid inside a concrete block [2], containing phase-change materials inside [7,8].

Normally, the maximum serviceability temperature of the structural concrete is limited because of the potential damage caused by high temperatures. This damage consists of micro-cracks originated by the pore pressure inside the concrete matrix due to the evaporated water [9,10]. If the concrete has a proper matrix porosity as in case of normal strength concrete, the evaporated water could flow outside the concrete through the pore connection of the matrix. This effect, in combination with medium-high temperatures, could cause negligible effects of the pore pressure and therefore, would not provide damage in the concrete matrix [9,10]. In case of dense concrete mixes, such as high-strength concrete, or if the concrete is subjected to very high temperatures, the originated pore pressure can be very harmful for the concrete matrix, producing the failure of the material by spalling [9,10]. The only situation where the behavior of structural concrete under high temperatures is studied is in fire conditions, being the structural material analyzed as an accidental ultimate limit state according to the standards [EC-2, ACI]. The normalized tests of fire resistance of structural materials consist of a gradual heating of the material to a very high temperature (over 700°C). However, backing to the case of moderate temperatures, as the operating temperatures of the solar energy storage systems ($\leq 300^{\circ}\text{C}$), and if the porosity of the concrete is adequate, there is no an apparent reason to expect a dramatically decrease of the strength of concrete. Below 300°C, it should not be observed a great decomposition of the hydration products of the cement [10] and the damage it should be only produced by the pore pressure developed by the evaporated water inside concrete. However, these pore pressures could be not high enough to produce a

mechanical damage of concrete and the energy supplied to concrete by heating helps to the hydration of un-hydrated cement particles what counteract the effect of the thermal damage of concrete and even to produce an increase of its mechanical properties [11].

In these applications of the structural concrete for thermal storing, it is crucial the study of the cracking behavior of concrete to control the produced damage. Moreover, it is usually necessary to use high-performance concrete with high strength capacity, especially in tensile strength to prevent premature cracking because of the pore pressure. However, this kind of concrete when is exposed up to high temperatures is more degraded than normal concrete and is more affected by spalling and cracking [2]. The addition of PP fibers reduces these effects and improve the mechanical properties when they are working at high temperatures [3,4]. Fiber length is an important property to enhance the fracture behavior of concrete at room temperature since longer fibers have higher strength against pull-out failure [5]. Another important aspect to consider in these applications is the study of the behavior of concrete under long exposition time at a constant temperature and posterior cooling. Due to the usual limitation of the serviceability temperature of concrete there are few studies in the bibliography about this topic and the studied exposition time are very moderated [12]. Mechanical properties after cooling and exposure time are two essential aspects to be studied since in the applications described, the concrete is exposed to high temperatures during long time and alternatively to working and resting stages.

The use of self-compacting concrete is enormously extended due to its benefits on more efficient production and high performance of the final product. The pre-casting element industries [13] is one of the fields that use this sort of concrete due to its better fluidity that improves the final quality and reduce construction-time. Also is applied as high quality concretes, sometimes reinforced with PP or steel fibers, in buildings and industrial structures that may bear high temperatures during operation or in case of an accident fire [14][15].

In this work, the influence of different temperature on the mechanical behavior of high-strength self-compacting concrete mixes, plain and polypropylene fiber (6 mm and 24 mm of length) reinforced concrete, is analyzed for three exposure time (6, 24 and 48 hours) on hot specimens and (48 hours) on cold specimens (after heating). Special attention is paid on their fracture behavior. In this way, standardized tests to characterize the mechanical behavior of the different mixes are conducted for different temperatures and exposure times. The properties are measured directly on heated and cold concrete.

2. Experimental study

2.1 Materials and mix design

A summary about the materials used in the present study and their characteristics are shown below:

- a) Portland Cement (PC) Type II 32.5 B-L in accordance with EN 197-1 [16]. Its chemical composition is provided in Table 1. It is fundamentally SiO_2 and CaO .

- b) Fine aggregate (FA) with a maximum size of 2 mm. Its grading distribution is shown in Table 2. Its chemical composition is shown in Table 1. FA is fundamentally SiO_2 and Al_2O_3 .
- c) Limestone sand (LA) with a maximum size of 4 mm (Table 2). Its chemical composition is shown in Table 1. LA is composed fundamentally by SiO_2 and Al_2O_3 , and its chemical composition is very similar to FA
- d) Coarse aggregate (CA) with a maximum size of 10 mm (Table 2). Its chemical composition is shown in Table 1. CA is composed fundamentally by SiO_2 and Al_2O_3
- e) Silica fume (SF) from Sika model S92-D with 90% of particles below 0.1 μm and SiO_2 content over 90% (Table 1). SF is fundamentally SiO_2 .
- f) Superplasticizer (SP) (SIKA VISCOCRETE 20 HE) is specific for low water/binder ratio.
- g) Polypropylene (PP) fibers with 33 μm in diameter and two different lengths, 6 (PPF-6) and 24 mm (PPF-24). The tensile strength of the PP fibers was 450 MPa.

Table 1. Chemical composition of the materials

Chemical composition (%)	PC	FA	LA	CA	SF
SiO_2	20.69	86.50	86.97	83.35	95.63
Al_2O_3	5.24	5.83	5.56	4.50	0.43
Fe_2O_3	2.46	1.33	1.39	2.26	0.10
MnO	0.10	0.03	0.02	0.05	0.02
MgO	1.50	0.13	0.19	0.36	0.39
CaO	61.89	0.59	0.51	3.42	0.21
Na_2O	0.36	0.87	0.95	0.09	0.20
K_2O	0.73	2.37	1.97	1.04	0.79
TiO_2	0.28	0.13	0.15	0.23	<0.01
P_2O_5	0.17	0.07	0.02	0.05	0.08
SO_3	1.11	0.04	0.03	0.03	0.04
LOI	4.76	1.34	1.51	3.59	1.84
Specific Gravity	3.18	2.69	2.58	2.48	2.30

Table 2. Grading distribution

Size sieve (mm)	>16	10	8	4	2	1	0.5	0.25	0.125	0.063	<0.063
FA	0	0	0	0	0	22.20	35.82	26.15	10.33	3.74	1.10
LA	0	0	0	0	11.50	26.50	21.00	17.33	12.00	9.17	2.33
CA	0	19.30	40.13	38.31	2.26	0	0	0	0	0	0

Table 2 shows the grading distribution of FA, LA and CA. As it can be seen, FA is the finest, with an average particles size of 0.48 mm, LA presents average particle size of 0.66 mm and CA has 6.72 mm.

In this study three different mixes of self-compacting concrete were analyzed. The only difference between the mixes is the PP fiber content. The first one, called D0, is considered as the reference concrete and is not reinforced with PP fibers. The second and the third concrete mixes, called D1 and D2 respectively, has a PP fiber addition of 1.2 kg/m³. The length of the fibers is 6 mm for D1 concrete and 24 mm in case of D2 concrete. The concrete mix proportions are detailed in Table 3.

Table 3. Mix design of all concrete types

Type	Quantity (kg/m ³)							W/PC ratio
	PC	FA	LA	CA	SF	SP	PPF	
D0	657	867	139	1301	99	23	0	0.20
D1	657	867	139	1301	99	23	1.2 (PPF-6)	0.20
D2	657	867	139	1301	99	23	1.2 (PPF-24)	0.20

A laboratory mortar mixer was used to manufacture the different types of concrete. First, all dry constituents were poured in the mixer and rotating for 5 minutes. Afterwards, the PP fibers were added to the water along with the superplasticizer and added to the mixer. Finally, the mixer was rotating for 15 minutes until a homogenous and fluent mix was achieved. Slump flow tests were conducted on the self-compacting concrete (SCC) mixes according to EFNARC guidelines [17] with satisfactory results. The specimens were casted immediately, unmolded and inserted in a water bath after 24 hours, which was at 20°C for 28 days.

2.2. Heating test

As the influence of temperature on fracture behavior of PP fiber reinforced SCC is studied, experimental tests were performed on specimens at high temperatures (hot tests), as well as after cooling them down (cold tests). Additionally, a first batch of specimens of each mix (D0, D1, D2) were tested at room temperature without heating to be used as control results. The second batch of specimens, called hot tests, were heated at a rate of 10°C/min until five different temperatures (100, 300, 500 and 700°C), subjected to three different exposure times (6h, 24h and 48h) at each temperature and immediately tested. The third batch, called cold tests, were heated at the same rate and the same temperatures for 48 h and later cooled-down for 24h at ambient temperature, and after that, they were tested. The testing machine used in this work was designed to perform tests at high temperature, being possible insert the actuator, the specimen and the supports into the furnace.

2.3. Mass loss and structural effect

The effect of temperature on the concrete was monitored by thermal analysis and variable-temperature X-ray powder (VTXRD) analysis. VTXRD patterns were recorded at the CITIUS X-

ray laboratory (University of Seville, Spain) on a Bruker D8 Advance diffractometer (Bruker, Germany) fitted with a high-temperature camera (Anton Paar XRK 900, Austria) and a position-sensitive detector (Bruker Vantec PSD, Germany) in the temperature range 25–900 °C at a heating rate of 10 °C·min⁻¹ (target: Cu; voltage: 40 kV; current: 40 mA; θ : θ geometry combining divergence Göbel mirrors configuration; detector: radial Soller slits; time scans: 5 min).

Simultaneous TG/DTA measurements were performed at the CITIUS Functional Characterization Service (University of Seville, Spain) on a TA (model STD-Q600) instrument, with alumina as reference. The samples were placed into Pt crucibles and maintained at air throughout the heating period. The temperature was increased at a constant rate of 10° C/min.

Differential scanning calorimetric analysis (DSC) was carried out. The samples, 5 mm in diameter and 3 mm thick, were placed in non-hermetic aluminum containers and subjected to a heating program of 2°C/min in a TA Instruments 2920 DSC from 40°C to 300°C in, using nitrogen as purging gas [19].

2.4. Pore size distribution

Pore size was determined using a mercury intrusion porosimeter with a measuring pressure range from 1.02×10^{-2} to 2.04×10^2 MPa. The contact angle selected was 141°, so the measurable pore size ranged from 0.007 to 144 μ m. Samples used had the shape of pellets about 5 mm in size. All the samples were previously heated at 105°C until the moisture was eliminated.

2.5. Compressive strength (f_c)

Compressive strength tests, for control concrete, on hot and cold specimens, were carried out in accordance with EN 12390-3 [20] in cubes 50×50×50 mm. These cubes were obtained by sawing off the prisms casted of 100×100×440 mm in size. All prisms were made with the same concrete matrix to avoid differences in the material properties. The specimens of each batch were simultaneously heated in the same furnace to ensure that all of them were subjected to the same thermal cycle. The load was increased continuously at a rate of approximately 0.5 MPa/s until the entire rupture of the specimen. Four cube specimens were tested for each case.

2.6. Tensile strength (f_{ct})

The tensile strength of concrete is an important mechanical property to analyze its fracture behavior. Cylindrical specimens 100×200 mm (diameter × height) were manufactured for each concrete mix. Due to complexity of the direct tension test of concrete, indirect tensile strength tests were used, namely Brazilian split-cylinder strength tests [21]. The experimental tests were carried out for specimens at ambient temperature (control concrete and cold specimens). The tensile strength for hot tests, was estimated from the maximum load value on three-point bending tests, knowing that the aforementioned load is related to that strength as indicated by Wang et al. [22] in their procedure to determine the tensile strength and fracture toughness of concrete or stated by Murthy et al. [23] by means of equation (1). This procedure

was checked using specimens at ambient temperature (control concrete and cold specimens) obtaining tensile strength values in agreement with their corresponding experimental results. So its application to estimate this material property on hot tests, in which is very difficult do it experimentally, is a reasonable way to calculate it.

$$P = \frac{2f_{ct}(D-a)^2 B}{3L} \mu(\theta) \quad (1)$$

The Brazilian tests were carried out following the procedures recommended by EN-12390-6 [24] on the 100×200 mm cylinders. The specimens were loaded through plywood strips 15 mm wide × 4 mm thick. The velocity of displacement of the machine actuator was 2.8 kN/s. Four specimens were tested for each case.

2.7. Determination of fracture energy (G_F)

Notched beams 50×50×220 mm (width × depth × length = $B \times D \times L$) were employed for hot and cold fracture energy determination. Fracture energy was assessed through stable three-point bending tests according to the RILEM work of fracture method [25]. Some modifications were considered, such as the recommendations proposed by Guinea et al. [26–28], in order to obtain a size-independent value. Four specimens were tested for each case. Specimen length was $L = 220$ mm, so specimen span length was $S = 200$ mm and equal to four times the depth of the cross section and therefore, the influence of the span/depth ratio [29] is avoid. Initial notch depth was $a = 25$ mm, so relative notch depth was $\alpha = 0.5$ for all cases. Notches were prepared using a diamond saw before heating.

Three-point bending tests were performed in a ± 300 kN dynamic machine as illustrated in Figure 1. The vertical displacement at midpoint was measured for specimens at ambient temperature (control concrete and cold specimens) by means of a linear voltage displacement transducer (LVDT) mounted on a rigid frame fixed to the specimen. However, this measurement was obtained for hot specimens from the actuator position record because the high temperature achieved on the heating tests did impossible to use any measuring instrument. Loading was conducted according to actuator control, at a rate of 0.01 mm/min until the peak load was reached; after this, velocity was increased to 0.05 mm/min during the post-peak branch of the P - δ curve. Supports were mounted on steel ball bearings to allow them to rotate about the beam's major axis.

From the P - δ curve, the fracture energy of concrete was calculated by using Equation (2), specified in RILEM [25] and with the corrections corresponding to the adjustment of the tail of the load-displacement curve of Guinea et al [26].

$$G_F = \frac{W_F}{B(D-a)} \quad (2)$$

where G_F is the fracture energy (N/m), W_F is the total work of fracture and $B(D-a_0)$ is the ligament area. The total work of fracture is given by:

$$W_F = W_m + W_{nm} \quad (3)$$

W_m is the measured work of fracture corresponding to the total area below the $P-\delta$ curve recorded until the end of the test at δ_u . W_{nm} is the non-measured work of fracture energy, obtained by means of the remote tail constant, A , taking into account the influence of cutting the $P-\delta$ curve [28].

$$W_{nm} = \frac{2A}{\delta_u} \quad (4)$$

Figure 1. Schematic view of three-point bending test on notched specimen

2.8. Young's modulus (E_c)

Young's modulus for specimens tested at ambient temperature (control concrete and cold specimens) was calculated experimentally according to EN 12390-13:2014 [30] by gradually loading a cylindrical specimen in compression to approximately a third of its failure load and measuring the corresponding strain using 30 mm strain gauges. By contrast, it was impossible to use measurement instruments on hot tests and subsequently, the Young's modulus was calculated based on the compliance method of Jenq and Shah [31] for these specimens. This method requires to know the initial slope of the load-crack mouth opening displacement curve (P-CMOD) which was not measured for hot specimens. The initial slope of the P-CMOD curve is related to Young's modulus as shown in equation (5) and therefore, the initial slope of the load-displacement curve (P- δ) is also related to the material stiffness so it is possible to obtain an initial slope of the P-CMOD curves and establish their relationship with the P- δ curves. The P-CMOD curve for hot tests were estimated from load-actuator-position curves applying the aforementioned rate. The P-CMOD estimated was used to calculate the Young's modulus by means of the compliance method of Jenq and Shah [31].

According to these indications, after obtaining the initial compliance (C_i) of the P-CMOD curves for all hot specimens, the elastic modulus of concrete is obtained with the following expression:

$$E_c = \frac{6SaV_1(\alpha')}{C_iBD^2} \quad (5)$$

where $V_1(\alpha')$ is a geometric function given by:

$$V_1(\alpha') = 0.8 - 1.7\alpha' + 2.4\alpha'^2 + \frac{0.66}{(1-\alpha')^2} + \frac{4D}{S}(-0.04 - 0.58\alpha' + 1.47\alpha'^2 - 2.04\alpha'^3) \quad (6)$$

$$\alpha' = \frac{a + HO}{D + HO}$$

HO is the clip gauge holder thickness. In this case, HO was 1 mm and there was no accuracy loss if $\alpha' \approx \alpha$.

Obviously, according to expression (5), materials with higher initial P - $CMOD$ compliance curves have lower elastic modulus values.

3. Results and discussion

3.1 Mass loss

Figure 2. TG of concrete: D0 (without PP fibers)

Thermogravimetric analysis of D0 from ambient temperature to 1000°C after 28 days is shown in Figure 2. As it can be seen, D0 shows three different weight losses. The first was observed at 100°C, it is a 4.2%wt and is due to the moisture present in D0. The second, between 200-500°C, presented only a mass loss of 1.5 %wt and was the result of dehydration of chemically bounded water of several hydrate compounds in concrete (calcium silicate, carbo-aluminates and ettringite), although it was mainly due to the dehydration of calcium silicate hydrates at 450°C. The third one was observed between 650°C and 750°C, presented a 3.5 % wt and was caused by the de-carbonation of calcium carbonate from the clinker and the filler [32].

Figure 3. Mineral composition of D0

The mineral composition of a D0 specimen portion at different temperatures (from ambient temperature to 900°C) after 28 days is shown in Figure 3. Quartz, PDF 01-085-0335, (q), Calcite: CaCO_3 , PDF 01-083-0578, (c), C_3S : Ca_3SiO_5 , PDF 00-055-0738, (s) and Albite: $\text{NaAlSi}_3\text{O}_8$, PDF 01-089-6427, (a) are the principal mineralogical compounds at ambient temperature. When the temperature of D0 is 700°C, calcite disappears, because it is decomposed in CO_2 and CaO , as it can be seen in Figure 2.

Figure 4. DSC of PP fibers

Figure 4 shows the DSC of polypropylene fibers. The melting process took place over a range of temperatures between 145°C and 180°C, as can be seen from the width of the melting endotherm. The melting point was 165°C (the peak of the melting endotherm).

Figure 5: TGA of PP fibers

As shown in Figure 5, fibers have only one mass loss at 460°C, because they are turned into water and CO₂.

3.2. Fracture energy

The values of fracture energy measured for each hot and cold test at different exposure time are plotted in Figure 6. In D0 hot tests (Figure 6.a), the fracture energy has a decrease trend at 100°C. In that case, concrete matrix has not till relevant micro-cracking and thus, vapor cannot be freely evacuated. This leads to high moisture gradients in the matrix that reduces the fracture energy [9]. After 100°C, fracture energy rises up to 500°C in order to the area under load-deflection curves (Figure 7.a-d) is increased with the temperature increment [33]. Although the maximum load decrease due to micro-cracking by spalling, the deflection increase [9]. At 700°C, a significant amount of micro-cracking is produced due to the decarbonation of calcium carbonate (Figure 2-3) reducing much more the material resistance [10] and decreasing the fracture energy drastically.

In D1 and D2 hot tests, PP fiber-reinforced mixes (Figure 6.b and Figure 6.c respectively), the fracture energy is slightly higher at 25°C and 100°C because fibers compensate the effect of high pressure and moisture gradient inside the concrete [10][34]. At low temperatures, 25°C and 100°C, the PP fibers conditions have not been affected by temperature (Figure 4) and its role is the sewing of cracks. At aforementioned temperatures, the fibers become concrete more ductile, as it can be deduced from higher fracture energy values (Figure 6.b and c) and area under load-deflection curves (Figure 8.a and 9.a). The values of fracture energy increases in D1 and D2 hot tests for 300°C because fibers are partially melted (Figure 4). Thus, internal vapor is evacuated more easily and the area under load-deflection curves raise (Figure 8.b and Figure 9.b). At 500°C, fracture energy is still high due to the same motive that D0 (Figure 8.c and Figure 9.d). At 700°C, additional to decarbonation, PP fibers are completely turned in gaseous water and CO₂ and the pore-level in the concrete matrix is significantly raised at 33 μm (the diameter of the fibers), as is shown in Figure 10. This can be seen in so reduced area under load-deflection curves and peak load (Figure 8.d and Figure 9.d). The addition of longer fibers increase the ductility of material as can be deduced from higher fracture energy values in Figure 6.c than 6.b. Furthermore, longer fibers generate more network of channels through which steam disappeared more efficiently, decreasing concrete damage and increasing fracture energy [10].

Figure 6: Evolution of fracture energy with temperature for each exposure time: (a) D0 hot tests, (b) D1 hot tests, (c) D2 hot tests and (d) cold tests.

As for D0 cold test, there is a similar tendency with a difference in comparison to D0 hot test due to the cooling process. In that process, there is an additional micro-cracking that become further damaged matrix earlier (Figure 6.d) [9,10].

Comparing D1 with D0 cold test, at 100°C the addition of PP fibers increases the ductility of material, as it can be seen in the growing of deflection in Figure 11.a and b. At 300°C, the maximum load noticeable decreased because PP fibers are partially melted (Figure 4). The deflection increased because the matrix has more pores due to fiber melting and that rise the ductility (Figure 10 and 11) [10]. Both effects, a light decreasing of maximum load and increment of deflection, leads to rise the fracture energy Figure 6.d. At 500°C and 700°C, the tendency in load-deflection curves are the same that at 300°C (Figure 11.a and b). The main difference is that fibers are completely melted and consequently, the harmful level is higher (Figure 5). Thus, the fracture energy is decrease (Figure 11.b).

Regarding to D1 cold test (Figure 6.d) in comparison with D1 hot test, the maximum load increases at 100°C since two effects happen simultaneously, the temperature affects to the cement paste improving its hydration [10][9] and the reduction of internal pressure because there is more time to be evacuated during cooling [9]. As a consequence, the fracture energy is

higher than D1 hot (Figure 6.b). At higher temperatures (300, 500 and 700°C), the pore-level and micro-cracking rise in the cement paste owing of heating and the additional effect of the thermal gradient during cooling [9,10]. Therefore, concrete becomes weaker, as shown in load-deflections curves (less maximum load and more deflection), Figure 11.b.

The addition of longer fibers weakens the bearing capacity at room temperature and 100°C, as it can be seen from the comparison between D1 and D2 on hot and cold tests (Figure 6.b-c and Figure 6.d respectively). At 300°C, when fibers are partially melted, the network of channels in matrix is larger, so spalling is reduced noticeably. As a consequence, the peak load on load-deflection curves are higher (Figure 8-9.b and Figure 11.b-c). At 500°C and 700°C, with fibers completely melted, the harmful is similar to D1 or D2 cases as is clear from Figure 8-9 c-d and Figure 11. b-c.

The exposure time has no influence on the fracture energy because the core of the samples reaches the exposure time in less than 6 hours leading to the specimens (6h, 24h and 48 h) to similar thermal effects.

Figure 7: Load-deflection curves in D0 hot tests: (a) 100 °C, (b) 300 °C, (c) 500 °C and (d) 700 °C.

Figure 8: Load-deflection curves in D1 hot tests: (a) 100 °C, (b) 300 °C, (c) 500 °C and (d) 700 °C.

Figure 9: Load-deflection curves in D2 hot tests: (a) 100 °C, (b) 300 °C, (c) 500 °C and (d) 700 °C.

Figure 10. Pore size distribution of concrete D1

Figure 11: Load-deflection curves in cold tests: (a) D0, (b) D1 and (c) D2.

3.3 Compressive strength

For all mixes, the compressive strength is shown in Figure 12. The effect of temperature, cooling process and exposure time on the specimens with different PP fiber length in hot and cold tests can be analyzed from these results.

As observed in Figure 12.a, b and c (hot tests), a decrease-increase-decrease trend is shown for every concrete (D0, D1 and D2 mixes) at the three exposure times (6, 24 and 48 hours). This behavior is similar to that exposed in previous studies by Zhang and Bicanic [9]. For D0 hot mixes (Figure 12.a), when the specimens are heated up to 100°C, the compressive strength decreases owing to vapor pressure in the concrete matrix [10][9]. Between 100°C and 300°C, temperature affects to the cement paste improving its hydration and leading to an increase of the compressive strength [10][9]. At temperatures higher than 300°C, there is considerable micro-cracking in the matrix and hence, the compressive strength is weakened with the increment of temperature [10][9].

In D1 and D2 hot tests (Figure 12.b and c), in which the specimens were reinforced with PP fibers (short and long length respectively), the results were qualitatively and quantitatively similar to that obtained in D0 (Figure 12.a). These fibers do not provide any improvement on

compressive mechanical properties [35]. The inclusion of fibers in matrix reduces compressive strength when those are completely melted at 700°C.

In regard to D0 cold tests (Figure 12.d), the evolution of results has an decrease-increase-decrease trend comparable to that observed in previous studies [9]. At 100°C, the compressive strength is slightly greater than hot test because this temperature not produce a high vapor pressure. At 300°C, the micro-cracking caused by heating and cooling process avoid the increment of resistance, that occurs in hot tests (see Figure 12.a), driving to the decrease of compressive strength [36]. As for 500°C, the compressive strength decreases because the harm generated during heating process and cooling process, are more significant because of so high achieved temperatures [9]. At 700°C, the huge temperature originate the processes of decomposition of the cement paste [9,10] joined to micro-cracking during cooling process [9].

For PP fiber reinforced specimens, D1 and D2 (Figure 12.d), the compressive strength is similar than D0 at 100°C, even it can be reduced when the amount of fibers is very high, as it can be seen in Figure 12.d, because of worm effect of fibers that have originated more pores in concrete matrix [35]. At 300°C, the fibers are partially melted, fiber channels are not completely free and the vapor has more difficulties to be evacuated, generating spalling so the concrete matrix contains further damage in comparison with room temperature and the same mixes in hot tests. At 500°C and 700°C, fibers are melted and the behavior is similar to that explain for D0 cold tests.

The exposure time has no influence on the compressive strength because the core of the samples reaches the exposure time in less than 6 hours leading to the specimens (6h, 24h and 48 h) to similar thermal effects.

Figure 12: Evolution of compressive strength with temperature for each exposure time: (a) D0 hot tests, (b) D1 hot tests, (c) D2 hot tests and (d) cold tests.

3.4. Young's modulus

Hot and cold tests have a decrease trend from 25°C to 700°C, so stiffness is always reduced. The decrease in E_c with temperature in all mixes was due to the micro-cracking of the cement paste caused by the procedure of heating (in hot tests, Figure 13.a, b and c) and the additional effect of cooling process (in cold tests, Figure 13.d) [10]. In reference to hot tests (Figure 13.b and c), the addition of PP fibers improves the Young's modulus of material at 25°C [34][37]. Nevertheless, there is a sharp drop of 50 % approximately, at 100°C, which show as the vapor pressure quickly affects to this property. However, from 100°C to 700°C the evolution of E_c is slighter and almost linear [9,10]. As the temperature increases, it is generated an increment of the thermal micro-cracking which therefore reduces the stiffness of concrete [10]. The effect of the fibers is barely evidenced at temperatures below 500°C. The greater values are obtained with PP fiber reinforced concrete [10].

Figure 13: Evolution of Young's modulus with temperature for each exposure time: (a) D0 hot tests, (b) D1 hot tests, (c) D2 hot tests and (d) cold tests.

The effect of PP fibers is slightly remarkable but it is worth noting as D2 (reinforced with long fibers) and D1 mixtures (reinforced with short fibers) attain better results. This is because the addition of PP fibers increases the ductility of the material and this effect is more relevant when PP fibers are longer [37]. When fibers are melted, they reduce internal vapor pressure by means of the canal connections, generated by melting PP fibers so vapor pressure can be evacuated more efficiently [10].

3.5. Splitting tensile strength

Figure 14: Evolution of splitting tensile strength with temperature for each exposure time: (a) D0 hot tests, (b) D1 hot tests, (c) D2 hot tests and (d) cold tests.

The trend observed with the temperature in hot tests (Figure 14.a, b and c) responds to a three-stages decrease-increase-decrease trend accord to that shown by Zhang and Bicanic [9].

The effect of PP fibers is not very noticeable on splitting tensile strength (Figure 14. b and c) for hot tests at 25°C [35]. For all mixes in hot tests (D0, D1 and D2), the tensile strength decreases when mixes attain 100°C, like occurred with the compressive strength tendency [9,10]. When the temperature is higher than 100°C, the tensile strength of concrete is enhanced because of the effect of hydration of cement paste by the temperature. In fibers reinforced mixes, from 500°C to 700°C, more remarkable descent took place because of the dehydration, decomposition of cement paste [9,10] and the melting of fibers, generating more voids in the concrete matrix than D0 case [10,38,39].

In order to look into the evolution of results in cold tests (Figure 14.d), these have an decrease trend like can be seen in the previous studies [9][10]. The splitting tensile strength results are better than hot tests at 100°C because vapor pressure has more time to be evacuated during cooling process. At temperatures higher than 100°C, the concrete matrix, which was damaged

by the previous heating, raise its internal damage due to the cooling process that generates more micro-cracks and propagates those which already existed [9].

The effect of PP fibers on splitting tensile strength was more noticeable than the effect on residual compressive strength but the behavior is similar. The additions of PP fibers to the concrete matrix generates worm effect that increases the amount of pores. This effect is more remarkable when the amount of fibers and length rises. When concrete mixes achieve temperatures higher than 300°C, the fibers are partially or completely melted and the voids in the concrete matrix is significantly higher. So the splitting tensile strength is reduced. This effect is more remarkable for mixes reinforced with longer fibers (D2, Figure 14.d).

3.6. Characteristic length

Characteristic length was evaluated for hot and cold mixes. This fracture parameter provides information about the intrinsic brittleness of cohesive material [40][41]. It is directly related with the fracture process zone (FPZ) because although this parameter is not exactly the length of the FPZ but there is a relationship between them [42]. Therefore, a material with higher characteristic length will have greater FPZ length and this leads to higher ductility [43].

As all specimens had the same size, no exists size effect and consequently, it was possible to analyze the ductility of mixes by means of the characteristic length values directly. The characteristic length is defined as:

$$l_{ch} = \frac{E_c G_F}{f_t^2} \quad (7)$$

Where f_t is the tensile strength and, in this case, it was considered equal to splitting tensile strength f_{ti} .

For hot tests, Figure 15.a, b and c, there is a decrease-increase trend where the minimum point is at 300°C. At 100°C, vapor pressure cannot be evacuated freely because the conducts or voids within concrete matrix are not enough. Thus, fracture energy for D0, D1 and D2 (Figure 6.a, b and c) and elasticity modulus (Figure 14.a, b and c) decrease. The decrease was more noticeable on elasticity modulus which can attained up to 50% less than its value at 25°C. If temperature is increased at 300°C, this affects to the cement paste improving its hydration and leading to an increase of the splitting tensile strength (Figure 14.a, b and c) [10][9] and decreasing the characteristic length. From 500°C to 700°C, the pore level, is very significant, as can be seen in Figure 7, and the softening is increased. The post peak softening is significantly raised, as can be seen in Figure 8.c, 8.d, 9.c, 9.d, 10.c and 10.d. and all this leads to obtain characteristic length values and FPZ higher. In PP fiber-reinforced hot mixes (Figure 15.b and c), the characteristic length is higher than no-reinforced specimens (Figure 15.a) because this is increased when PP fibers are exposed to temperatures below its melting point and when they are partially or completely melted since both situations provide a softer matrix. Although this effect is more noticeable at temperatures higher than 500°C since pore level is higher (Figure 7).

According to cold tests (Figure 15.d), at 100°C, the tendency is similar to those for hot tests with the difference that the minimum values of the characteristic length are attained. The damage generated during cooling process, is not significant, and so it is shown on the values of fracture energy, Young's modulus and tensile strength (Figure 6.d, 13.d and 14.d), which lightly descend the characteristic length. At this temperature, the maximum capacity load and less ductility is achieved for any cold mixes (Figure 11.a, b and c), thus characteristic length is decreased.

At 300°C, the matrix of cold mixes is more damaged than hot tests at the same temperature because have the additional micro-cracking during cooling process. In Figure 12.a, b and c can be observed there is a significant increase on the deflection and reduction of the maximum load of load-deflections curves for all cold mixes. This leads to have more ductile material with higher characteristic length.

For temperatures higher than 300°C, the level of damage in the matrix is increased (Figure 7) and the PP fibers are melted. This even help to have more voids inside concrete. Subsequently, the characteristic length is higher, although its loading capacity is lower (Figure 12.a, b and c). The addition of fibers increases the ductility at any temperature, as hot tests. This effect is more remarkable for longer fibers.

Figure 15: Evolution of characteristic length with temperature for each exposure time: (a) D0 hot tests, (b) D1 hot tests, (c) D2 hot tests and (d) cold tests

4. Conclusions

This study explored the effect of temperature on fracture behavior and other mechanical properties of high-strength polypropylene fiber-reinforced self-compacting concrete at a range of temperatures up to 700°C and after cooling at three different exposure time. From results, the following conclusions can be drawn:

- The non-reversible process of weight loss increases with higher temperatures. This process follows a moisture dehydration at low temperature, a dehydration of chemically bounded water at medium-high temperature and a decarbonation of calcium carbonate at highest temperatures.
- The addition of fibers generates a network of channels in matrix that reduces internal pressure damage and spalling effects. Nevertheless, it reduces material strength at room temperature due to the increment of pores by trapped air.
- Fibers produce a bridge effect on crack front that sew micro-cracks while are not degraded. This improves strength properties and ductility at hot and cold conditions.

- Longer fibers create a major network of channels to evaporate internal pressure and reduce spalling effect but increase the number of voids in matrix that reduces mechanical properties and produces a softening of the material.
- Cooling process generates micro-cracking due to thermal gradient that is more remarkable from medium-high temperatures, when thermal gradient is more abrupt and fibers are partially melted. These harmful effects are added to those generated during heating process.
- When the thermal gradient is narrow, cooling process does not produce significant damage and fibers help to sew micro-cracking. This provide higher mechanical properties.
- Exposure time does not affect when the temperature is uniformly achieved in all matrix. From that moment, concrete behavior will be the same independently if specimens are warmed for longer time.

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