



Water Absorption, Density, Mechanical Strengths and High-temperature Resistance of Metakaolin-based Geopolymer Concrete Reinforced with Hybrid Polyolefin and Simple Polypropylene Fibers

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ABSTRACT

In recent years, geopolymer has been introduced as a novel and green alternative to the Portland cement. On the other hand, in terms of technical characteristics, concrete has some disadvantages, most notably low tensile strength and consequently low ductility. Therefore, the use of different fibers in the concrete mixture is considered as an appropriate solution to eliminate these defects. In this experimental study, two types of polymer fibers, including simple polypropylene and 4-element polyolefin hybrid fibers, were used to manufacture fiber reinforced geopolymer concrete specimens. In this regard, fiber reinforced and non-fiber specimens were made and associated tests including: density, water absorption, compressive, indirect tensile and flexural strengths, were performed. Also, to study effect of fibers on high-temperature resistance of metakaolin-based geopolymer concrete, specimens weight and compressive strength loss percentage after exposure to high temperatures up to 800 °C, were measured. The obtained results indicated that using fibers in geopolymer concrete mixture, result in increasing compressive, indirect tensile and flexural strengths and also decreasing in density and water absorption. Further, the use of hybrid fibers due to their ability to inhibit the cracking process from both micro and macro levels, significantly improved compressive, indirect tensile and flexural strengths compared to simple fibers. In term of high-temperature resistance, although the polymer fibers reduced the risk of the explosive sapling of specimens, resulting in less weight loss than non-fiber specimen, but overall, it can be concluded that these fibers did not have a significant effect on the high-temperature resistance of geopolymer concrete.

Keywords:

Fiber reinforced geopolymer concrete, Hybrid fibers, Polyolefin fibers, Mechanical strengths, High-temperature resistance.



1. Introduction

Concrete is the most consumed construction material after water, due to its special features including formability and availability of raw materials [1]. But production process of Ordinary Portland Cement (OPC) as the main constituent of conventional concrete has major environmental disadvantages, including high energy consumption and carbon dioxide (CO₂) emissions [2]. On the other hand, climate change due to global warming is currently one of the most significant environmental challenges. Greenhouse gas emissions is the main contributing factor to global warming, with CO₂ having the greatest share (65%) among other greenhouse gases [3]. The production process of Ordinary Portland Cement (OPC) is identified as one of the major sources of CO₂ emission, i.e. 1-ton CO₂ release to produce 1-ton OPC [4, 5]. Furthermore, OPC production process is accounted for 7 to 10% of global CO₂ emissions [6-8]. Therefore, developing an appropriate and workable substitution for OPC is of great importance. In recent years, geopolymers have been introduced as environmentally friendly cementitious materials capable of reducing the negative environmental impacts associated with OPC [9]. Geopolymers were first developed by Davidovits, as a new family of binders of inorganic origin [10]. Regarding civil engineering applications, Geopolymer Concrete (GPC) has showed enhanced physical and mechanical properties over OPC, e.g. higher mechanical strength and rapid concrete hardening [11–14], higher resistance to elevated temperatures and fire [15–17], enhanced durability [18], lower permeability, improved resistance to solvents and acids [19], and lower creep effects [20, 21]. Geopolymers are inorganic Alumina-silicate substances comprised of SiO₂ and Al₂O₃ enriched raw materials, and an active alkali solution [22]. The geopolymerization process involves a substantially fast chemical reaction under alkaline condition on Si-Al minerals, that results in a three-dimensional polymeric chain and ring structure consisting of Si-O-Al bonds [23-24]. The raw material, depending on required characteristics, cost and availability, can be of natural origin (e.g. Zeolite), synthetic (e.g. metakaolin) or waste materials (e.g. fly ash or blast furnace slag). Metakaolin is a raw material obtained from calcinating kaolin at 600–800 °C. The alkaline activator solution is one of the two main constituents of geopolymers which plays a significant role in the formation of Al and Si crystals, and is normally chosen based on Na and K (solvent alkali metals) solutions. The most common alkali solution used in geopolymerization is a compound solution of NaOH or KOH and Na₂SiO₃ or K₂SiO₃ [25]. In addition to its advantages, concrete also has disadvantages. Low tensile strength and consequent low ductility and high brittleness are some of the major disadvantages of concrete. Introduction of fibers in the concrete mix is a solution developed in the past decades to overcome this issue. Usage of fibers to enhance brittle composites dates back to thousands of years [26], e.g. adding horse tail hair to reinforce mortar and plaster [27]. Application of different types and geometries of fibers in concrete, commonly known as “Fiber Reinforced Concrete (FRC)”, has shown to effectively control crack propagation and improve physical and mechanical properties of the concrete composite in various structural applications, e.g. tensile and flexural strength, resistance to impact and extreme temperatures, seismic effects, etc. In recent years, application of 2-part and multipart hybrid fibers to enhance various properties of the concrete composite has gained significant interest. In these hybrid fibers, fibers of different lengths made from the same material are combined together. Fiber Reinforced Geopolymer Concrete (FRGPC) is a new type of GPC which has been the subject of many recent studies to investigate its potential pros and cons [28-30].



Gao et al [31] conducted research on GPC reinforced with 6 and 12 mm long steel fibers and showed that the shorter fiber is more effective in controlling micro cracks, while the longer fiber provides ductility at extensive cracking scenarios. A hybrid fiber configuration showed to yield optimal crack control features. Asrani et al [32] investigated slag-based FRGPC using Polypropylene or PP (13 mm long), glass (15 mm long) and 3D-steel (60 mm long) fibers of 0.3, 0.3 and 1.6% volume content, respectively, and as single and hybrid fiber GPC configurations. The results showed that incorporating only PP fiber results in a significant increase (about 108%) of flexural strength over plain GPC. Hybridization showed to further improve strength characteristics, e.g. a hybrid PP and steel FRGPC composite displayed 30 and 200% growth in compressive and flexural strength over plain GPC, respectively. Alberti et al [33] studied the properties of Polyolefin fiber-reinforced concrete enhanced with steel fibers in low ratios and concluded that the use of polyolefin fibers improves mechanical strength and provides considerable ductility and flexural toughness. Han et al [34] investigated the effect of Polyolefin fibers on the specifications of concrete containing silica fume. The results showed that using these fibers resulted in a 13% increase in flexural strength as well as a 70% decrease in crack propagation. Additionally, the specimens containing Polyolefin fibers exhibited 2 times higher impact resistance than those containing steel fibers and 14 times more than the control (no fiber) specimens. Deng et al [35] also studied the effect of macro-Polyolefin fibers on the concrete properties. The researchers observed the positive effects of Polyolefin fibers in preventing crack propagation by increasing the fiber content. Celik et al [29] studied the effect of different fiber types for FRGPC, on its resistance to elevated temperatures. Polyolefin, Basalt, Modified Polyamide and PVA fibers with lengths 10, 12, 10 and 8 mm, respectively, and incorporated as non-hybrid composites were considered. The results showed that fiber content had a small effect on changing the compressive and flexural strengths and specimen weights of the FRGPC composites when subjected to elevated temperatures (300–900 °C). In this paper, the mechanical and physical properties of GPC reinforced with different fiber types (simple PP and 4-part polyolefin) and volume content (0.15, 0.2 and 0.25%) is investigated. Specific density, water absorption capacity, compressive, tensile and flexural strengths and resistance to elevated temperatures (200–800 °C) of the various FRGPC composites is investigated using standard relevant testing procedures and the results are then compared to each other and to that of plain GPC (no fibers). This study takes a step in better understating the different properties of FRGPC composites and to form a basis for its potential structural applications.

2. Materials and Methods

2.1. Materials

The X-Ray Fluorescence (XRF) chemical analysis of the metakaolin used in this study is illustrated in Table 1. NaOH with 98% purity and liquid Na₂SiO₃ with SiO₂/Na₂O molar ratio of 2 were used to prepare the alkaline activator solution. Table 2 represents the chemical analysis of the Na₂SiO₃ and NaOH substances. Aggregates with granular sizes of 7-10 mm was used as coarse aggregate (sand) and < 4 mm sized aggregates were used as fine aggregate. SSD specific gravity and water absorption tests were conducted on the coarse and fine aggregates using the ASTM C127 [36] and ASTM C128 [37] procedures, respectively, illustrated in Table 3. The fineness modulus (using ASTM C136 [38]) and sand equivalent (using ASTM D2419 [39]) values of the fine



aggregates were measured equal to 3.01 and 73, respectively. To reduce water content and improve workability of concrete, polycarboxylate-based Super Plasticizer (SP) was incorporated.

Table 1. XRF chemical analysis of metakaolin.

Chemical substance	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	Na ₂ O	K ₂ O	ZrO ₂	MnO	TiO ₂	LOI
Weight %	54	31.7	1.13	4.8	2.3	4.1	0.1	0.11	1.41	1.41

Table 2. Chemical analysis of NaOH, KOH, and Na₂SiO₃ solutions.

NaOH			Na ₂ SiO ₃		
Chemical substance	Result	Unit	Chemical substance	Result	Unit
NaOH	98	%	SiO ₂	30	%
Na ₂ CO ₃	1	%	Na ₂ O	14.5	%
NaCl	200	ppm	Water	55.5	%
Fe	6	ppm			
SiO ₂	15.7	ppm			

Table 3. Specific gravity and Water absorption of aggregates.

Material	SSD Specific gravity (gr/cm ³)	Water absorption (%)
Coarse aggregates	2.62	1.3
Fine aggregates	2.59	3.2

Two types of fibers were used: PP and 4-part hybrid (comprised of micro and macro mesh fibers, sinusoidal fibers and single strand fiber with high strength and elastic modulus properties) Polyolefin. The main properties of the fibers are presented in Table 4. Figure. 1 depicts the two fibers used in this study.

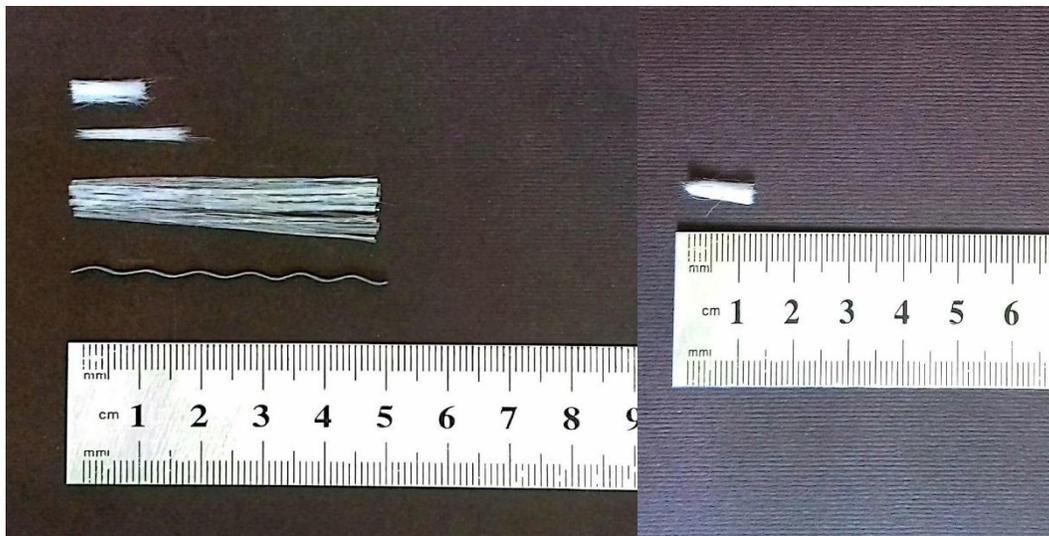


Figure 1: 4-part hybrid Polyolefin and PP fibers



Table 4. Properties of fibers.

Parameters	PP	4-part hybrid Polyolefin
Length (mm)	12	12,20,45,50
Density (gr/cm ³)	0.91	0.91-0.93
Tensile strength (MPa)	400	570-700
Modulus of elasticity (GPa)	3.5	4.5-6.5
Water absorbency	No	No
Alkaline and acid resistant	Excellent	Excellent
Melting point	160	160-170

2.2. Experimental Program

In this part, 7 mix designs were defined, as illustrated in Table 5. To optimize the fiber volume content, different values of fiber volume content were added to the GPC specimens: 0.15%, 0.2% and 0.25%. Initially, the alkaline activator solution, constituting of NaOH (14M), Na₂SiO₃, SP and the extra water (according to each mix design) are combined and allowed to cool for 24 hrs. In the mixing process, the aggregates, metakaolin and fibers were first dry mixed in the mixer for 3 minutes. Next, the alkaline activator solution was added and the concrete was mixed for a further 2 minutes. Subsequently, compressive (100×100×100 mm cubes), tensile (200×100 mm cylinders) and flexural (100×100×500 mm beams) specimens were molded and vibrated for 10 seconds on a vibrating table. The specimens were cured in the oven (80 °C) for 24hrs. After the curing process, the specimens were allowed to rest at laboratory ambient temperature. The specimens were subjected to the 7- and 28-day compressive, tensile (Brazilian) and 3-point flexural strength tests, as well as water absorption capacity and specific density tests.

Table 5. Fiber Reinforced GPC mix designs.

Mix ID	Metakaolin (Kg/m ³)	NaOH 14M (Kg/m ³)	Na ₂ SiO ₃ (Kg/m ³)	Coarse aggregates (Kg/m ³)	Fine aggregates (Kg/m ³)	SP (Kg/m ³)	Extra water (Kg/m ³)	Fiber content (%)
Control	350	140	210	840	840	14	40	0
MP-0.15	350	140	210	840	840	14	40	0.15
MP-0.2	350	140	210	840	840	14	40	0.2
MP-0.25	350	140	210	840	840	14	40	0.25
M4-0.15	350	140	210	840	840	14	40	0.15
M4-0.2	350	140	210	840	840	14	40	0.2
M4-0.25	350	140	210	840	840	14	40	0.25

The various tests were conducted according to standard testing procedures: compressive strength test according to the BS1881: Part116 [40], indirect tensile strength test according to ASTM C496 [41], 3-point flexural strength test according to ASTM C293 [42] and ASTM C1018 [43], water absorption capacity and specific density tests according to ASTM C642 [44]. Three 100×100×100 mm cube specimens for each of the following fiber reinforced GPC mix designs (which displayed the best mechanical strength results) were considered to evaluate FRGPC



resistance to elevated temperatures: M4-0.2, MP-0.2 and control (no-fibers specimen) as a basis for comparison. For elevated temperature testing, the 28-day specimens were placed in the oven subject to various elevated temperatures: 200, 400, 600 and 800 °C. Fifteen cube specimens were produced for each of the considered mix designs, where 12 specimens were subjected to elevated temperatures (3 specimens for each aforementioned temperature level) and 3 specimens were considered as control specimens (not subjected to elevated temperatures). The oven temperature was raised with a constant rate of 1 °C/minute. After reaching the considered temperature, the specimens were kept in the oven for 3 h and then the oven was turned off to gradually cool down to ambient temperatures. The specimens were then removed from the oven, weighed and subjected to compressive testing.

3. Results and Discussions

3.1. Water Absorption and Specific Density

Figures. 2 and 3 illustrate the specific density and water absorption capacity of the FRGPC and unreinforced GPC specimens, respectively. The specific density of the control specimen (unreinforced GPC) is calculated equal to 2335 kg/m³. Usage of fibers in the GPC shows to reduce specific density with fiber volume content. Also, increasing the fiber content resulted in a further reduction in the specific density of the specimens, so that in the FRGPC containing 0.25% fibers, almost 3% of the specific density was reduced. This can be explained to the lower specific density of the fibers compared to the GPC matrix. The specific density of the fibers used is in the range of 0.91-0.93 gr/cm³ (equivalent to 910-930 kg/m³), thus reducing the specific density of the FRGPC specimens compared to the control specimen. The water absorption capacity of the control specimen is around 4.3%. Water absorption capacity of the FRGPC composites reduces with fiber content. The random dispersion of the micro and macro size fibers allow them to stitch micro cracks and prevent development of new ones in the geopolymer matrix. This mechanism results in the higher density of the geopolymeric matrix structure and consequent reduction in water absorption capacity [45].

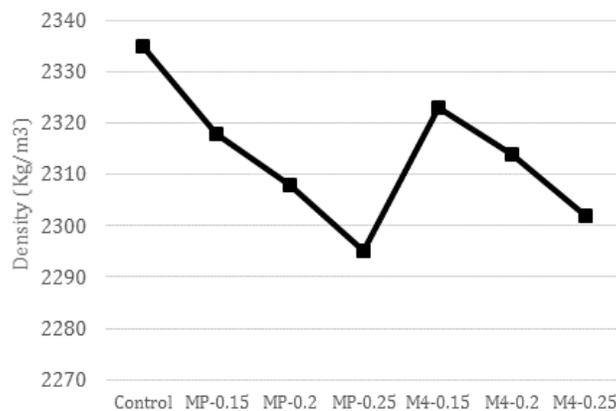


Figure 2. Specific density of the FRGPC composites

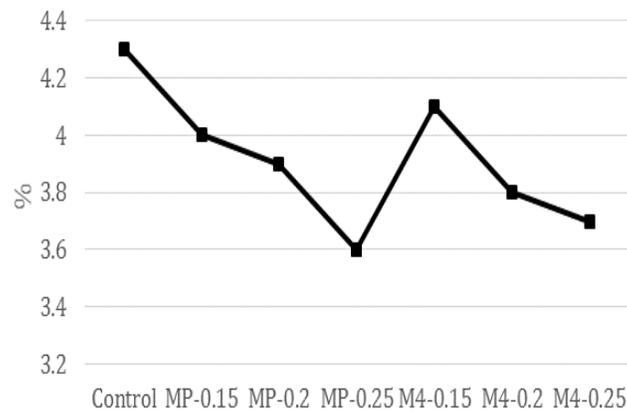


Figure 3. Water absorption of the FRGPC composites.

3.2. Compressive Strength

The 7- and 28-day compressive strengths of the FRGPC composites are illustrated in Figure 4. By observing the results of the PP fiber reinforced GPCs, the compressive strengths decrease for 0.15 and 0.25% fiber content compared to the control specimen (around 3.1 and 1.5% respectively), and increase for 0.2% fiber content (about 1.2%). From the results obtained, it can be concluded that the PP fibers generally have a nonsignificant yet negative effect on compressive strength of GPC composites. As for the 4-part hybrid FRGPC composites, the same fiber contents yield to approximately 1.9, 5.5 and 3.29% increase in compressive strengths compared to the control specimen, respectively. Unlike the PP fibers, 4-part hybrid fibers displayed a positive influence on GPC compressive strength. Previous studies on the influence of PP fibers on GPC compressive strength shows inconsistency in the results. In line with the obtained results herein, Noushini et al [46] attributed the negative effect of PP fibers (18, 19 and 51 mm long, 0.5% volume content) on GPC compressive strength to the low tensile strength and air pocket development around these fibers. Conversely, Asrani et al [32] showed that usage of 0.3% volume content PP fibers (13 mm long) results in about 6% increase in GPC compressive strength. In all fiber types, the 0.2% fiber content showed to be the optimal fiber content with regard to compressive strength. Further increase of fiber content up to 2.5% results in decrease in GPC compressive strength. This issue may be due to the reinforcement of the concrete matrix by hybrid fibers and the improvement of the Interfacial Transition Zone (ITZ). The ITZ, is the boundary area between the cement paste and the surface of aggregates, fibers or rebar which plays an important role in permeability, durability and strength of concrete. The microstructures of the ITZ and the cement paste are different and compared to the cement paste, the ITZ microstructure has more porosity and micro cracks. The thickness of the ITZ depends on parameters such as fiber type, cement type, pozzolan type, etc. Using fibers in optimal ratio, can strengthen the ITZ and improve the mechanical properties of concrete. But on the other hand, with increasing fiber content from 0.2 to 0.25%, the compressive strength decreased slightly. All fibers used were of polymeric materials, which due to high flexibility, can result in fiber balling in the concrete mix at high fiber contents. This phenomenon leads to perforations in the mortar matrix and subsequent internal flaws in interfacial transition zones and thus reduction in GPC compressive strength [47, 48].

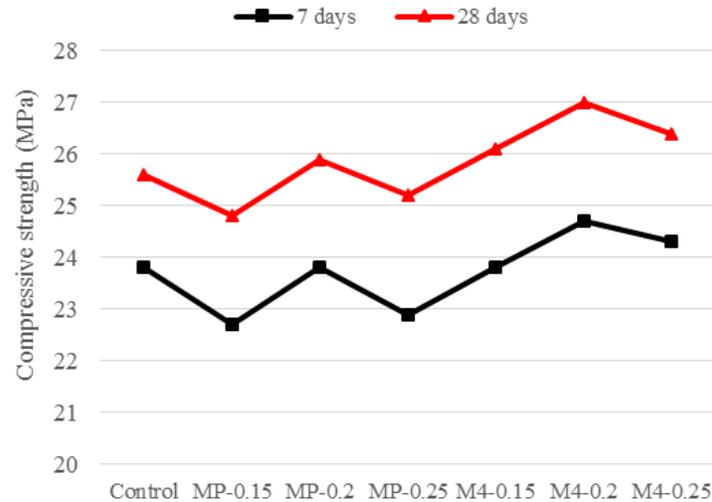


Figure 4. Compressive strength of the FRGPC composites.

3.3. Tensile and Flexural Strengths

The 7- and 28-day tensile and flexural strengths of the FRGPC and control specimens are illustrated in Figures 5 and 6, respectively. According to Figure 5, The lowest 7- and 28- day tensile strength were measured in the control specimen (1.44 and 1.72 MPa, respectively), and the 0.2% hybrid FRGPC specimen (M4-0.2) showed the highest 7- and 28-day tensile strengths (2.09 and 2.31 MPa, respectively) among all FRGPC specimens. Using 0.15, 0.2 and 0.25% of PP fiber results in approximately 15.1, 23.2 and 19.1% improvement in tensile strengths compared to the control specimen. In the hybrid FRGPC composites, 0.15, 0.2 and 0.25% fiber content results in approximately 21.1, 34.3 and 32.7% improvement in tensile strengths compared to the control specimen, respectively. The obtained results indicated that using fibers improved tensile strength values compared to the control specimen furthermore, better performance of the hybrid fibers over the PP fiber. Optimal results are achieved at 0.2% fiber content. Further increase in fiber content from 0.2 to 0.25% caused a slight decrease in tensile strength compared to the optimum value (0.2%). The 7- and 28-day flexural strengths of the control specimen were 2.62 and 3.02 MPa, respectively. Using 0.15, 0.2 and 0.25% of PP fiber results in approximately 30.4, 46 and 42.1% improvement in flexural strengths compared to the control specimen. In the hybrid FRGPC composites, 0.15, 0.2 and 0.25% fiber content results in approximately 84.2, 69.8 and 78.7% improvement in flexural strengths compared to the control specimen, respectively. The optimal PP and hybrid fibers content for maximum flexural strength improvement was 0.15%. However, the flexural strength decreases from 0.15 to 0.2% fiber content, and increases from 0.2 to 0.25%.

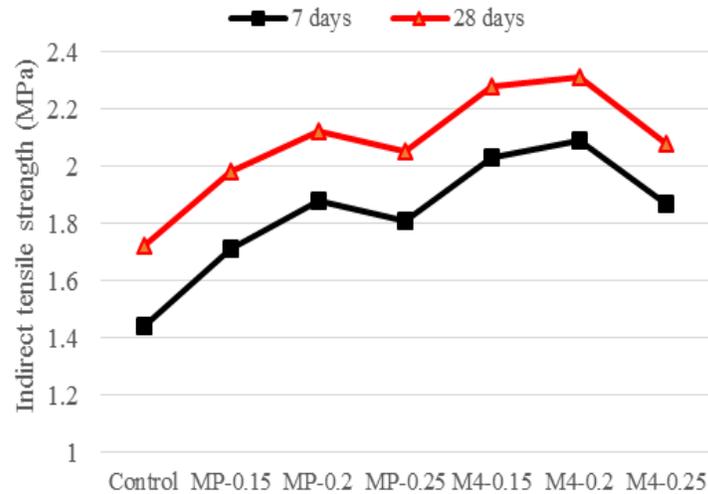


Figure 5. Tensile strength of the FRGPC composites.

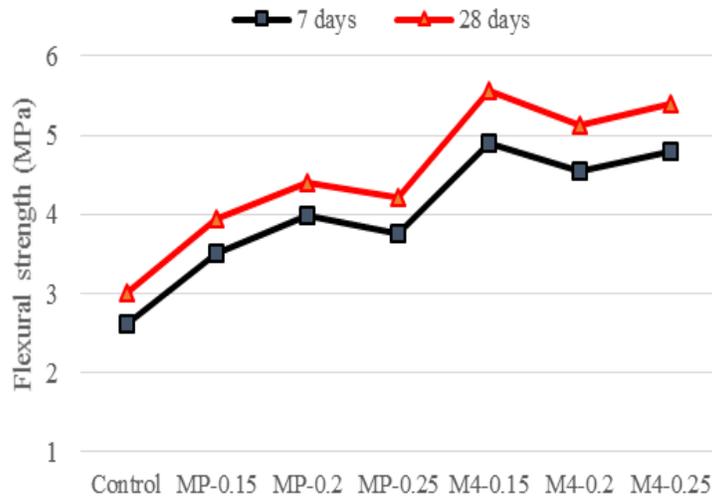


Figure 6. Flexural strength of the FRGPC composites.

The advantageous effect of fibers on the tensile and flexural strengths of GPC composites leads to enhanced ductility characteristics over plain (no fiber) GPC. The polymeric fibers improve the geopolymeric matrix of the composites in terms of formation and/or redistribution of cracks [49]. The basic geopolymer structure includes the formed amorphous geopolymeric gel, residual unreacted raw material particles and varied pores [49-52]. Fibers can offer a bridging effect over the pores or cracks by embedding its two thrams in the cementitious matrix, resulting in increased toughness and strength of the geopolymeric matrix [49]. As a result, higher tensile and flexural strengths were observed in FRGPC specimens than non-fiber ones. On the other hand, fibers used in this study were hybridized using short and long lengths. Hybridization of fibers in terms of size and type results in the synergistic effect of fibers. The positive synergetic effects of hybridization stems from the different mechanisms of short and long fibers in the GPC matrix. Shorter fibers are



more effective against smaller minor cracks, while longer fibers are mainly activated at higher loading scenarios to prevent formation and opening of major cracks. Therefore, the simultaneous usage of different fiber geometries develops positive features in the GPC composite for different levels of exerted loads [31, 53].

3.4. Resistance to Elevated Temperatures

Figure 7 illustrates the mean compressive strength of the FRGPC and unreinforced (control) specimens after subjected to various elevated temperatures. Changes in compressive strength of the heat subjected specimens compared to corresponding nonexposed ones are illustrated in Table 6.

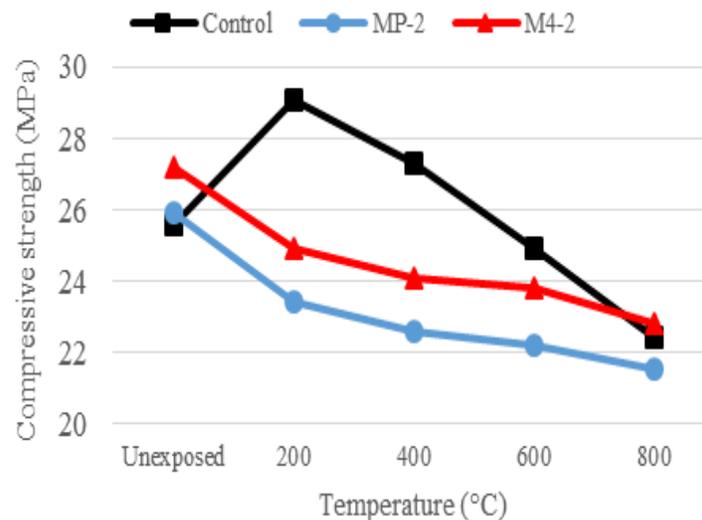


Figure 7. Mean compressive strength of FRGPC & unreinforced specimens subjected to elevated temperatures.

Table 6. Change in specimen compressive strength (%) subject to elevated temperatures.

Mix ID	Temperature			
	200°C	400°C	600°C	800°C
Control	+13.67±0.2	+6.64±0.1	-2.73±0.3	-12.5±0.3
MP-2	-9.61±0.1	-12.73±0.3	-14.56±0.2	-17.1±0.1
M4-2	-8.26±0.1	-11.17±0.4	-12.41±0.2	-16.13±0.4

The compressive strength of the unreinforced GPC composite (Control) shows increase at 200 and 400 °C, i.e. around 13.6 and 6.6% respectively, and decrease at 600 and 800 °C (around 2.7 and 12.5% respectively) compared to corresponding unexposed specimens. As observed in past research [54, 55], GPC composites tend to show compressive strength increase when subject to temperatures of around 200–400 °C, due to dissolve and polycondensation of unreacted aluminosilicate compounds, resulting in higher matrix density and subsequent rise in compressive strength. Geopolymers have shown to maintain molecular stability up to 600 °C [54], but at 800 °C, difference in thermal resistance of the aggregates and geopolymeric matrix at contact regions, results in the formation and propagation of micro cracks and thus reduction of compressive strength



[56]. Unlike the unreinforced GPC case, the FRGPC composites displayed gradual strength reduction with increasing elevated temperatures, reaching a maximum approximate 16.13% reduction compared to the unexposed cases for all FRGPC cases. The melting point of the different fibers used in this study is around 160– 170 °C, therefore at temperatures above 200 °C, a considerable number of the fibers tend to melt and develop micro channels within the concrete structure. The resulting perforations would result in reduction of concrete matrix density and thus compressive strength decline [57]. By comparing the fiber reinforced GPC composites and Control, although the unreinforced composite displayed higher strengths at 200 °C, yet both FRGPC and Control displayed relatively close compressive strengths at the extreme temperature level of 800 °C. In summary, the 4-part hybrid fibers displayed a better thermal performance over PP fibers, yet, the polymeric fibers generally showed no significant effect on the resistance of GPC composites subject to elevated temperatures. The amount of change in specimen weight subjected to elevated temperatures is summarized in Table 7. It can be observed that all specimens experience gradual weight loss with elevated temperature increase. The unreinforced Control mix undergoes a steeper weight loss than the fiber reinforced cases, i.e. from 0.4% at 200 °C to 14.46% at 800 °C. This can be explained by crack propagation, spalling and laminating of the concrete under elevated temperatures. In the FRGPC composites, a higher weight loss compared to Control is seen at 200 °C, but a slight decline is observed at 400 °C. At the ultimate 800 °C, an average 10% reduction in weight compared to the unexposed specimens is observed in FRGPC cases which is less than the unreinforced composite. The melting of fibers and formation of micro channels in the matrix leads to reduction of internal pressures in the concrete and thus reduction of spalling and laminating effects compared to the unreinforced case [57].

Table 7. Weight loss (%) of specimens subjected to elevated temperatures.

Mix ID	Temperature			
	200°C	400°C	600°C	800°C
MR1.5	-0.4±0.1	-1.32±0.1	-8.61±0.4	-14.46±0.5
MP-2	-3.45±0.2	-3.94±0.2	-5.57±0.1	-10.05±0.2
M4-2	-3.09±0.3	-3.35±0.4	-5.11±0.3	-9.43±0.4

4. Conclusion

In this paper, the mechanical and physical properties of GPC reinforced with different fiber types (Polypropylene or PP and 4-part polyolefin) and volume content (0.15, 0.2 and 0.25%) is investigated. The main results drawn from this study, with regard to its limitations, can be summarized as below:

1- Presence of fibers cause reduction of specific density and water absorption of GPC composites, due to micro-structure enhancement and increased density of the matrix. These effects displayed a direct relation with fiber content and independent of fiber type.

2- Fibers slightly improve the compressive strength of GPC, with the hybrid fibers showing a greater influence due to hybridization effects. The 4-part polyolefin hybrid fiber displayed the most advantageous performance in this regard, i.e. around 5.5% increase compared to the unreinforced GPC.



3- Due to the crack-bridging mechanism of the fibers, the fiber reinforced GPC composites displayed significant improvement in tensile and flexural strength over unreinforced GPC. The hybrid fibers displayed better results compared to the PP fiber case. The 4-part hybrid fiber GPC composites showed a 34.3% increase in tensile strength, and an 84% improvement of flexural over unreinforced GPC, respectively.

4- The 4-part hybrid fibers displayed a better thermal performance over other fibers, yet, the fibers generally showed no significant effect on the resistance of GPC composites subject to elevated temperatures.

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