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STRENGTH LOSS CHARACTERISTICS OF FLY ASH BASED GEOPOLYMER CONCRETE EXPOSED TO ELEVATED TEMPERATURES

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Abstract: Fly ash based geopolymer concrete, one of the environment friendly alternatives to conventional concrete, is expected to behave better at elevated temperatures. However limited information is available about its behaviour at elevated temperatures. This paper presents the engineering properties of fly ash based geopolymer concrete after exposure to elevated temperatures and compares the corresponding results with those of a conventional concrete having almost the same compressive strength of geopolymer concrete (at ambient temperature). The specimens were heated at a constant rate (5.5 ℃ /minute) to different set temperatures (200,400, 600 and 800 ℃). They were cooled to ambient temperature by air cooling and water cooling and then tested for their strength properties. It could be observed that, the fly ash based geopolymer concrete undergoes a higher rate of strength loss during its early heating period (up to 200°C) compared to OPC concrete. However, the residual strength properties of both the concrete are almost the same at 400°C temperature exposure and beyond 600°C, while OPC concrete loses its strength properties rapidly; geopolymer concrete improves its strength. Hence, it could be concluded that the fly ash based geopolymer concrete could be considered as a better sustainable material than conventional concrete under the situations where it may be exposed to temperatures beyond 400 ℃..
Keywords: Geopolymer, Fly ash, Concrete, High temperature, Engineering properties

INTRODUCTION

Construction industry needs materials and technologies that are environment friendly and sustainable. Most widely used construction material is cement. However, its manufacturing process leads to the production of greenhouse gas [1]. Fly ash, a waste product generated from thermal power stations causes environmental issues, unless disposed off properly [2]. Use of fly ash based geopolymer concrete as one of the construction materials will not only eliminate consumption of cement but also utilize industrial waste effectively. As a result, geopolymer (GP) concrete is emerging as one of the environment friendly alternatives to Ordinary Portland Cement (OPC) concrete

In GP concrete, a geopolymer binder is formed by alkali activation of amorphous alumina–silicate material under warm atmosphere. The result of geopolymerisation is the formation of a three dimensional structural framework which is formed after dissolution, hydrolysis and polycondensation reaction [3,4]. The effectiveness in the geopolymerisation process depends on type, particle size, and the degree of amorphous character and the chemical composition of alumino–silicate source materials [5–8]. Variables such as water to solid ratio, type and concentration of alkali, temperature of curing , period of curing, method of mixing etc. influence the properties of geopolymer mortar and concrete [9–11].

For normal applications, OPC concrete generally provides satisfactory thermal resistance up to a temperature exposure of about 400 °C. However, beyond this temperature, its strength properties decreases rapidly and wide spread cracking and subsequent spalling occurs [12–17]. Because of the low energy need for the production and expected better behaviour at elevated temperatures compared to OPC concrete, geopolymer compounds are being considered as sustainable fireproof building materials, heat insulators etc. However, most of the studies at elevated temperatures are on geopolymer paste and mortar [18–19]. Kong and Sanjayan [20], based on their study on fly ash based geopolymer concrete, have reported that the compressive strength of geopolymer concrete when exposed to elevated temperatures are influenced by the specimen size, size of coarse aggregate and type of aggregate. Kong et al. [19] observed a higher strength loss at elevated temperatures for metakaolin based geopolymer paste as against fly ash based geopolymer paste. At 800 °C, while metakaolin based geopolymer paste continued its strength loss, they observed a strength gain in fly ash based geopolymer.

Review of literature shows that, a systematic study on the engineering properties of geopolymer concrete exposed to elevated temperatures is still a gap area. Present paper focuses on an experimental investigation on the engineering properties of geopolymer concrete exposed to elevated temperatures and compares the corresponding behavior of a comparable OPC concrete.

EXPERIMENTAL PROGRAM

Fly ash based geopolymer (GP) concrete specimens were made and were exposed to a constant rate of temperature increase (5.5 °C/ minutes). The specimens were then cooled to ambient temperature by air cooling and water cooling. Specimens were then tested at ambient temperature to determine their various engineering properties. OPC concrete was designed in such a way that the cube compressive strength of both GP and OPC concrete are almost the same at ambient temperature so that their test result could be compared.

Materials

Low calcium fly ash (ASTM Class F) obtained from a thermal power station (India) has been used for the present study. The chemical composition of fly ash is presented in Table1. The fly ash used had a specific gravity of 1.9. Partilce size distribution and XRD analysis are available in a publication [21].

A mixture of NaOH and Na_2SiO_3 solution (SiO_2 = 34.64%, Na_2O= 16.27%, water 49.09%) was used as alkali solution in the present investigation. NaOH pellets of 98% purity were used to make sodium hydroxide solution. The specific gravity of alkali liquid solution, having Na_2SiO_3/ NaOH (molarity 10) ratio 2.5 was 1.54.

Crushed granite aggregates of nominal size 20 mm was used as coarse aggregate. Natural river sand having fineness modulus of 2.36 was used as fine aggregate. The specific gravity of coarse and fine aggregate was 2.72 and 2.64 respectively. Ordinary Portland cement was used for making OPC concrete.

Table 1. Chemical composition of fly ash

Parameter	Content % by mass)
SiO_2	59.70
Al_2O_3	28.36
Fe_2O_3+Fe_2O_4	4.57
CaO	2.10
Na_2O	0.04
MgO	0.83
Mn_2O_3	0.04
TiO_2	1.82
SO_3	0.40
Loss of ignition	1.06

Mix proportioning

A preliminary study was carried out to arrive at the optimum proportion of the various constituents of GP concrete and it details are presented elsewhere [22]. Accordingly, the parameters that kept constant in the present investigation includes, aggregate content by volume (70%), the ratio of fine aggregate to total aggregate(0.35), ratio of alkali to fly ashy by mass (0.55), molarity of NaOH (10), ratio of Na_2SiO_3 to NaOH (2.5), ratio of water to geopolymer solid (0.25). The quantity of materials required to produce 1m^{3}of GP concrete based on the above proportions is given in Table 2.

Table 2. Quantity of materials required to produce 1m^{3}of GP and OPC concrete

Mix ID	Cemen (kg)	Flyash (kg)	Coarse aggregate (kg)	Fine Aggregate (kg)	NaOH Solution (kg)	Na2SO3 (kg)	Super Plasticizer (kg)
GP concr	--	310	1204	648	48.7	121	6.2
OPC concrete	475	---	1204	648	---	---	1.9

Mixing

The prepared NaOH solution was first mixed with calculated amount of Na_2SiO_3 liquid and kept for 24 hours before use. Coarse and fine aggregates in saturated surface dry conditions were thoroughly mixed with fly ash in a pan mixture. The alkali liquid and Naphthalene based superplastisizer (2% by weight of fly ash) were mixed together and then added to the dry mix. The whole mixture was then mixed together for 5 minutes.

Casting of specimens

Steel moulds of size150 mm x 150 mm x 150 mm, 100 mm x 100 mm x 500 mm, and 150 mm diameter, 300mm height were used for mechanical properties of both GP and OPC concrete. The fresh GP and OPC concrete filled in 3 layers and compacted with the help of a table vibrator. The OPC concrete specimens were kept in the mould for 24 hours under laboratory conditions and then they were demoulded and immersed in water for curing. GP concrete specimens were kept under laboratory condition for 60 minutes and then, after covering with thin steel plate, they were subjected to heat curing in an electric oven at 100 °C for a period of 24 hours. The curing temperature and period of curing were arrived at based on a preliminary study [22]. After temperature curing, GP concrete specimens were kept at room temperature till they were tested. Geopolymer paste was prepared with the same GP concrete and specimens were prepared for different microstructural analysis.

Heating and testing of specimens

OPC concrete specimens were taken out of curing tank on the 27th day, their surfaces dried with cloth and kept in the laboratory for 24 hours. The specimens were then kept in an oven for 1 hour at 60°C to remove surface moisture so that exposure spalling could be avoided during heating.

GP and OPC concrete specimens were heated in an electric furnace to 200˚C, 400°C, 600˚C and 800˚C. The rate of heating was kept at 5.5˚C /minute. After attaining the target temperature, specimens were kept at the same temperature for 1 hour to ensure that the specimens attain a uniform temperature throughout. The heated specimens were then cooled by two different methods namely air cooling and water cooling. Both GP and OPC concrete specimens were tested after they were cooled down to ambient temperature.

RESULTS

Compressive strength

Table 3 gives the compressive strength of GP and OPC concrete after exposed to elevated temperature. From the Table 3, it could be observed that, for almost the same compressive strengths of both GP and OPC specimens at ambient temperature, there is a higher strength loss for GP concrete during the early stages of the temperature rise. In the present study, at 200˚C , while air cooled and water cooled OPC concrete had a strength loss of about 0.4 % and 9% respectively, the corresponding loss of strength of GP concrete is about 26 % and 31% respectively.

Table 3. Cube compressive strength of GP and OPC specimens after exposed to elevated temperatures

Exposure Temperature (°C)	GP Concrete				OPC Concrete			
	Air cooled		Water cooled		Air cooled		Water cooled	
	Comp. strength (MPa)	SD*	Comp. strength (MPa)	SD*	Com. strength (MPa)	SD*	Comp strength (MPa)	SD*
Ambient (28)	57.30	0.45	–	–	59.85	0.68	–	–
200	42.52	0.68	39.40	0.68	59.60	1.17	54.37	0.88
400	37.33	0.45	35.85	0.44	42.66	0.44	39.85	1.14
600	30.82	0.67	28.00	0.94	32.44	0.84	31.48	0.54
800	32.88	0.38	31.30	0.31	21.00	0.31	19.55	0.62

* – SD – Standard deviation

The FTIR spectrum analysis of GP paste exposed to elevated temperatures is presented in figure 1[23], which shows a shift in the wave number and a substantial reduction of the peak in Si–O–Al (alumino silicate) and Si–O–Si regions (wave number 460cm^{-1} to 1088cm^{-1}) at a temperature exposure of 200˚C, indicating a reduction in their bonding force and decrease in chain length [6]. Also, the bands representing water molecule (hydroxyl groups) in GP paste showed a marked decrease in their peak at a temperature exposure of 200˚C and further increase in exposure temperature did not cause any further decrease in these peaks(wave number 3440 cm^{-1}). This means that, most of the weakly bound water molecules that were either adsorbed on the surface or trapped in the large cavities between the geopolymeric products get expelled at about 200˚C. The combined result of the above may lead to a higher strength reduction in GP concrete compared to OPC concrete during the initial heating process.

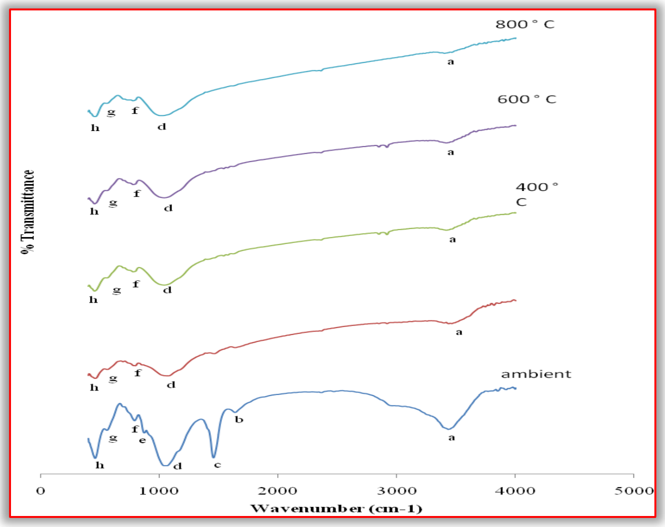


Figure 1. FTIR of Geopolymer paste exposed at different temperature (air cooled)
ambient : $a= 3430\ cm^{-1}$, $b=1635\ cm^{-1}$, $c=1453.13\ cm^{-1}$, $d=1045\ cm^{-1}$, $e=870.26\ cm^{-1}$
$f=788\ cm^{-1}$, $g=555\ cm^{-1}$ and $h=455\ cm^{-1}$
200 ℃: $a_1=3436\ cm^{-1}$ $d_1=1062\ cm^{-1}$ $f_1=795\ cm^{-1}$ $h_1=458\ cm^{-1}$
400 ℃: $a_2=3399\ cm^{-1}$ $d_2=1046\ cm^{-1}$ $f_2=788\ cm^{-1}$ $h_2=446\ cm^{-1}$
600 ℃ : $a_3=3419\ cm^{-1}$ $d_3=1039\ cm^{-1}$ $f_3=776\ cm^{-1}$ $h_3=451\ cm^{-1}$
800 ℃: $a_4=3399\ cm^{-1}$ $d_4=1006\ cm^{-1}$ $f_4=775\ cm^{-1}$ $h_4=453\ cm^{-1}$

Even though the free water in concrete gets removed during the initial heating of OPC, the strength gained due to the hydration of unreacted cement particles compensates the strength loss due to other parameters in concrete when heated up to about 200 ˚C; a behaviour well accepted by many researchers [13,24].

Figure 2 shows the residual compressive strength of test specimen (in percentage of strength at ambient temperature) after the exposure to different temperatures and tested after cooling by air and water cooling methods. From Fig 2, it could be observed that, the air cooled OPC specimen does not experiences much strength reduction up to 200˚C and beyond this, there is more or less a constant rate of strength reduction up to 800˚C.

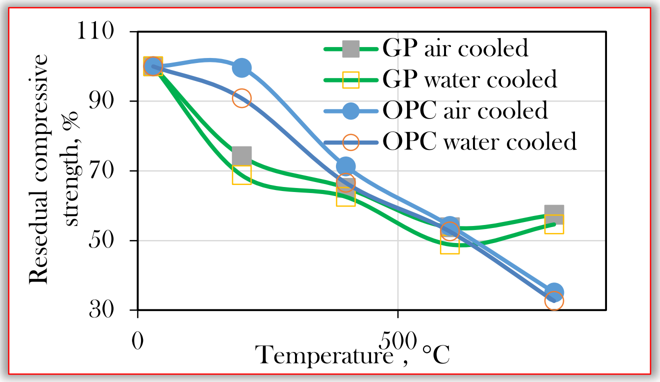


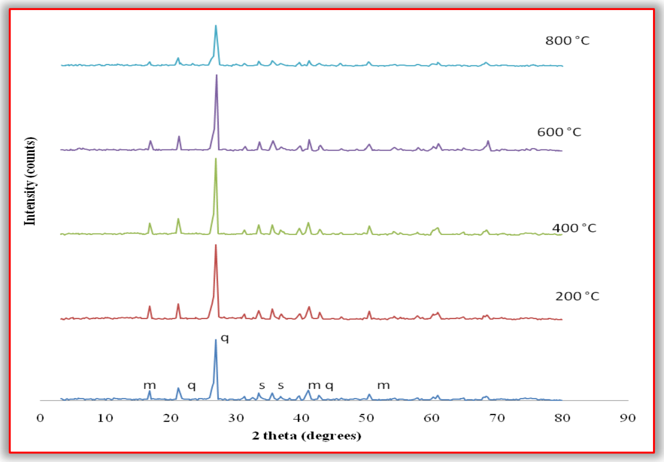
Figure 2. Residual cube compressive strength of GP and OPC concrete after exposure to elevated temperatures

Compared to air cooled OPC specimen, the rate of strength reduction between 200°C and 400°C is less for GP concrete and the percentage residual strength is almost same for both the types of concrete at 400°C. It may further be noted that, while the rate of strength loss between 400°C and 600°C is almost the same for both the types of concrete. However GP concrete shows a strength gain beyond 600°C, while OPC concrete continues to lose its strength.

From the XRD analysis (figure 3) of GP paste[23], an additional polymerization of GP concrete could be observed for a temperature exposure after 600°C, which is evident from the increased glass phase content above 600°C, as against the 90% glass phase content up to 600°C. Also, while the FTIR spectrum of GP paste showed only marginal reduction in the peak intensities over the Si–O–Al and Si–O–Si region for the temperature exposure between 200°C and 600°C, the peak intensity corresponding to Si–O–Si linkage increases slightly beyond 600°C, confirming the polymerization of initially unreacteted materials beyond 600°C.

It could be observed that, water cooled OPC specimens showed a lower strength at all exposure temperatures in the range between 3% and 9% compared to the strength of air cooled specimens primarily due to the thermal shock induced due to sudden cooling. Similar behaviour has been reported by other investigators also [25–27]. The water cooled GP concrete specimens also shows a lower strength compared to air cooled GP specimens (4% to 9% lower strength) and its behaviour is similar to that of OPC concrete when exposed to water cooling.

The strength reduction in OPC concrete when exposed to high temperatures is primarily due to the decomposition of the cement paste and the corresponding loss of adhesion [28].



q=quartz, m=mullite s=siliminate

Figure 3. XRD of Fly ash and Geopolymer binder paste exposed to elevated temperatures (air cooled)

Further, the reason for a lower compressive strength of water cooled OPC specimen compared to air cooled specimen is due to the micro cracks developed subsequent to the induced thermal shock [17,21]. The free calcium hydroxide present in hydrated Portland cement decomposes into calcium oxide at high temperature. If this calcium oxide is wetted after being cooled, it transforms into calcium hydroxide again, causing a volume change (may be up to about 40%) and this may also result to the formation of micro cracks in concrete

Tensile strength

Tables 4 and 5 shows the split tensile strength and flexural strength of GP and OPC specimen respectively, tested after exposure to elevated temperatures. The plots of these residual strengths in terms of percentage initial strength are given in the fig 5 and 6 respectively.

From Tables 4 and 5 as well as from figs.4 and 5, it could be observed that, both split and tensile strength of GP concrete is slightly lower than the corresponding values of OPC concrete up to a temperature of 400°C. However, beyond this temperature, GP concrete behaves better. Further, similar to the compressive strength, beyond 600°C, there is a strength gain for GP concrete in both split and flexural strength. Similar behaviour has been observed by other investigators [29]. In the present investigation, the residual split tensile strength of air cooled GP concrete exposed to 600 °C is 32.3% and that in OPC concrete is 27.0%. However, the corresponding values at 800°C exposure temperature are respectively 35.6% and 19.3%.

Further, the rate of strength reduction of both OPC and GP concrete is more or less the same up to 600°C in the case of split tensile strength as well as flexural strength.

Table 4. Split tensile strength of GP and OPC specimens after exposed to elevated temperatures

Exposure Temperature (°C)	GP Concrete				OPC Concrete			
	Air cooled		Water cooled		Air cooled		Water cooled	
	Split tensile strength (MPa)	SD*	Split tensile strength (MPa)	SD*	Split tensile strength (MPa)	SD*	Split tensile strength (MPa)	SD*
Ambient (27)	5.44	0.76	–	–	5.47	0.46	–	–
200	4.17	0.38	3.89	0.49	4.45	0.63	4.30	0.92
400	2.61	0.89	2.47	0.69	3.04	0.87	2.89	0.43
600	1.76	0.86	1.37	0.55	1.48	0.64	1.45	0.78
800	1.94	0.75	1.58	0.69	1.06	0.57	0.95	1.10

* SD – Standard deviation

Table 5. Flexural strength of GP and OPC specimens after exposed to elevated temperatures

Exposure Temperature (°C)	GP Concrete				OPC Concrete			
	Air cooled		Water cooled		Air cooled		Water cooled	
	Flexural strength (MPa)	SD*	Flexural strength (MPa)	SD*	Flexural strength (MPa))	SD*	Flexural strength (MPa))	SD*
Ambient (28)	5.30	0.68	–	–	5.44	0.45	–	–
200	4.23	0.53	4.10	0.68	4.53	0.67	4.40	0.38
400	2.89	1.21	2.61	0.86	3.36	0.77	3.19	0.82
600	1.86	0.87	1.52	0.58	1.72	0.83	1.49	0.89
800	1.90	0.47	1.65	0.96	0.72	0.66	0.61	0.87

* SD – Standard deviation

Modulus of elasticity

The slope of secant drawn at one third of the characteristic compressive strength of concrete has been considered as the modulus of elasticity of concrete. Standard cylinder specimens have been used to determine the modulus of elasticity.

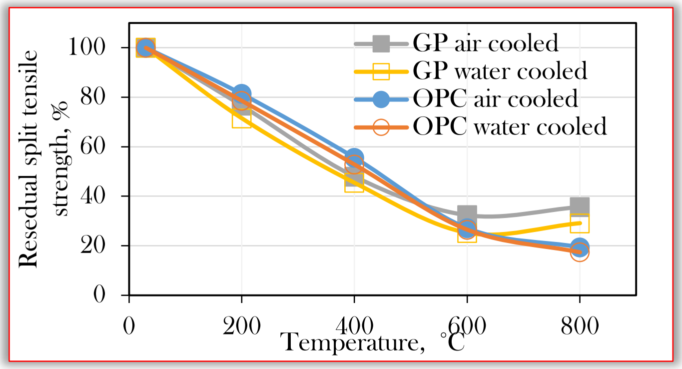


Figure 4. Residual split tensile strength of GP and OPC concrete after exposure to elevated temperatures

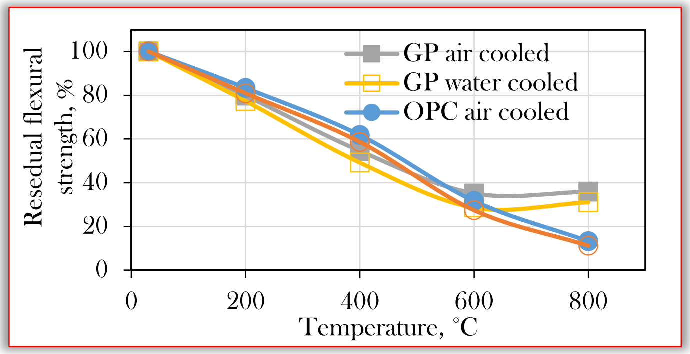


Figure 5. Residual flexural strength of GP and OPC concrete after exposure to elevated temperatures

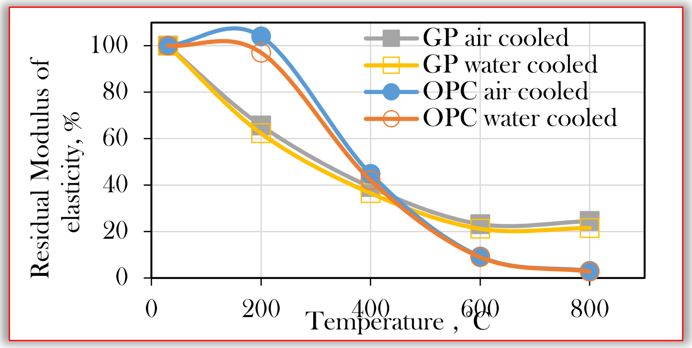


Figure 6. Modulus of elasticity of GP and OPC concrete after exposure to elevated temperatures

Figure 6 shows the variation of the modulus of elasticity (%) of GP and OPC concrete after exposure to elevated temperatures.

It could be observed that, compared to OPC concrete, while GP concrete shows a lower residual modulus of elasticity up to about 450°C, and higher values for exposure temperatures above 450°C. Also, unlike OPC concrete, GP concrete does not undergo further reduction in modulus of elasticity beyond 600°C, a behaviour similar to that of compressive strength of GP concrete. For the present study, at 600°C air cooled GP had a residual modulus of elasticity of 23.2% as against 9.2% in the case of OPC concrete. Further, at 800°C, air cooled GP concrete had a residual modulus of elasticity of 24.5% and the corresponding value of air cooled OPC concrete is only 2.9%.

CONCLUSIONS

Following conclusions could be derived from the study conducted on the fly ash based concrete.

- Fly ash based geopolymer (GP) concrete undergoes a high rate of strength loss (compressive strength, tensile strength and modulus of elasticity) during its early heating period (up to 200°C) compared to OPC concrete.
- The high rate of strength loss in GP concrete at its early heating period is contributed primarily due to the chemical restructuring of Si–O–Al (alumino silicate) and Si–O–Si compound and due to the formation of micro crack as a result of the removal of water (weakly bound and free water) from the geopolymer matrix.
- At a temperature exposure beyond 600°C, the unreacted crystalline materials in GP concrete get transformed into amorphous state and undergo polymerization. As a result, there is no further strength loss (compressive strength, tensile strength and modulus of elasticity) in GP concrete, whereas, OPC concrete continues to lose its strength properties at a

faster rate beyond a temperature exposure of 600°C.

- For the present study, after a temperature exposure of 600°C, both air cooled GP and OPC concrete had about 54% residual cube compressive strength (compared to the strength at ambient temperature, which is almost the same for both GP and OPC concrete). However, at 800°C, while GP concrete slightly gained its residual strength (to 57%), while OPC concrete continue to lose its strength (35% residual strength).
- Effect of thermal shock due to water cooling on GP and OPC concrete after exposed to elevated temperatures is more or less similar. For the present study, both GP and OPC concrete had a maximum strength loss of about 10% due to water cooling.

Hence, it could be concluded that the fly ash based geopolymer concrete could be considered as a better sustainable material than conventional concrete under the situations where it may be exposed to temperatures beyond 400 °C.

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