

Evaluation Study on the Structural Behaviour of Fly Ash-Based Geopolymer at Elevated Temperatures - A Review

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Abstract. In this study, the published literatures on the mechanical properties and microstructure of the geopolymer concrete (GPC) after exposure to elevated temperatures have been investigated. Comparing the GPC and ordinary Portland cement (OPC) concrete specimens after exposure to high temperatures and tested for the mechanical properties and microstructure has been discussed. The findings of the previous studies indicate that the GPC has better compressive strength, develops minor cracks, and undergoes slight damage in the mass at the elevated temperature as compared to OPC concrete. For ambient-cured and heat-cured conditions, the experimental results of the GPC after exposure to high temperatures (400 °C onward) show almost the same mechanical properties, while the OPC concrete significantly loses the strength along with large cracks developed above 400 °C. Moreover, the scanning electron microscope test shows that the OPC concrete developed a lot of cracks and started losing the bonds between the matrix at 400 °C, while the GPC holds over its strength until 800 °C.

Keywords: Geopolymer concrete · Elevated temperature · Fly ash · Mechanical properties · Microstructure

1 Introduction

"Geopolymer concrete (GPC) has exhibited considerable potential as a sustainable alternative material system to ordinary Portland cement (OPC) concrete for various types of applications (Provis et al. [2015\)](#page-8-0). Owing to its inherent features of showing superior performance after exposure to the high temperatures above 600 °C, GPC has acquired particular concern from industry and academic world (Cao et al. [2017;](#page-7-0) Sarker et al. [2014;](#page-8-1) Saxena et al. [2017;](#page-8-2) Zhao and Sanjayan [2011\)](#page-9-0). On the contrary, OPC concrete suffers severe damages in the form of the spalling of concrete and strength reduction at elevated temperatures (Hertz [2005;](#page-7-1) Mendes et al. [2008\)](#page-8-3). The outstanding thermal performance

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of GPC is usually attributed to the modifications in the structure network of these materials when compared to that of OPC, which considerably loses strength when exposed to temperatures above 400 °C because of rapid loss of water during firing (Rivera et al. [2016\)](#page-8-4). The primary binding phase in fly ash-based GPC is an alkali-aluminosilicate hydrate (N-A-S-H) gel with composites (Myers et al. [2013;](#page-8-5) Richardson [2008\)](#page-8-6). The gel microstructure of GPC plays a significant role in fire resistance (Hassan et al. [2020c\)](#page-7-2). The structure of the pore network within GPC is dictated by gel microstructure. It has been revealed to control moisture at high temperatures, with decreased tortuosity and porosity increment in the pore network providing improved resistance to fire spalling attributable to the more accessible release of trapped water vapor and reduction of internal water vapour pressure (Barbosa and MacKenzie [2003;](#page-7-3) Kong et al. [2007;](#page-8-7) Lie et al. [1986\)](#page-8-8). The increase in aluminum content in the GPC mixture improves the strength and decreases micro cracking in GPC specimens exposed to elevated temperatures (Kashani et al. [2017\)](#page-8-9). Rashad and Zeedan [\(2011\)](#page-8-10) studied the strength of FA-based geopolymer paste at high temperatures and showed that FA based geopolymer pastes were more resistant to degradation than OPC paste when exposed to elevated temperature. Hassan et al. [\(2020e\)](#page-7-4) studied the effect of total aggregate content of GPC on the mechanical properties of GPC. The findings showed that 70% of the total aggregate content in the mixture give the best results in terms of mechanical properties of GPC. The authors also studied the effect of steel fibres of reinforced GPC beams on the structural behaviour. The results indicated that the addition of steel fibre could control the cracks propagation and width of cracks. However, the effect of steel fibres in the flexural strength is significantly small (Hassan et al. [2020d\)](#page-7-5).

Fly-ash geopolymers are amorphous to the semi-crystalline equivalent of certain zeolitic materials with excellent properties, such as high fire and erosion resistance, and high strength materials. Recent works have shown that adding a moderate amount of minerals to a fly-ash geopolymer can give significant improvements in the fly-ash geopolymer structure and properties (Hu et al. [2009\)](#page-8-11). The alkaline activation of materials can be defined as a chemical process that provides a rapid change of some specific structures, partial or totally amorphous, into compact cemented frameworks (Hassan et al. [2019b,](#page-7-6) [2020a\)](#page-7-7). Some researchers described the alkali activation of fly ash as a physical-chemical process in which this powdery solid is mixed with a concentrated alkali solution in a suitable proportion to produce a workable and mouldable paste and stored at mild temperatures (*<*100 °C) for a short period to produce a material with good binding properties (Fernández-Jiménez and Palomo [2005\)](#page-7-8). At the end of this process, an amorphous alkaline aluminosilicate gel is formed as the main reaction product. Besides, Na- Herschelite-type zeolites and hydroxy-sodalite are formed as secondary reaction products (Cheng and Chiu [2003\)](#page-7-9). Many industrial by-products and other kinds of minerals can be used to produce the fly-ash geopolymers. The geopolymerisation reaction is susceptible to different raw materials (particle size and distribution, crystallization degree), nature of alkali-activators (Sodium/potassium hydroxide, Sodium/potassium silicate, the ratio of these two), Si/Al ratios, water/ash ratios, curing conditions (temperature, moisture degree, opening or healing condition, curing time) (Hassan et al. [2019a\)](#page-7-10)".

There are many cases where concrete is exposed to high temperatures like exposure from furnaces, fire exposure, nuclear exposure, exposure from thermal processes, etc. In such conditions, the proper understanding of the behaviour of concrete and structural elements exposed to high temperatures is essential (Bisby et al. [2013\)](#page-7-11). The desired information on the microstructure changes of fly ash based geopolymer concrete at elevated temperature is still lacking. Therefore, this review discusses the available data on mechanical properties and fire resistance of GPC for advancing the area of GPC concrete further.

1.1 Compressive Strength

"Hassan et al. $(2020b)$ reported that up to 800 °C, the test results indicated that fly-ash geopolymer concrete specimens retained about 50% of its strengths. In contrast, the OPC concrete specimens lost its total strength at 800 °C. The OPC concrete specimens retained about 52% of its strength until 400 °C; after that, it rapidly loses its strength. Also, the cracks developed after exposure to 800 °C temperature are much smaller and difficult to be noticed in GPC specimens as compared to the OPC concrete specimens, as shown in Fig. [1.](#page-2-0) The superiority performance of GPC at elevated temperatures has been proved from many other researches (Bakharev [2006;](#page-7-13) Hussin et al. [2015;](#page-8-12) Kong and Sanjayan [2008;](#page-8-13) Valencia Saavedra and Mejía de Gutiérrez [2017;](#page-8-14) Zhang et al. [2016\)](#page-9-1).

Fig. 1. Cracks of GPC and OPC concrete after exposure to 800 °C (Hassan et al. [2020b\)](#page-7-12)

Figure [2](#page-3-0) shows the prediction of the compressive strength of GPC and OPC concrete after exposure to different temperature levels based on the available equations proposed by (Eurocode [2005;](#page-7-14) Han et al. [2015;](#page-7-15) Li and Purkiss [2005\)](#page-8-15) for the strength prediction of OPC concrete. It could be seen from Fig. [2](#page-3-0) that, none of the equations proposed for OPC concrete is suitable for predicting the compressive strength of GPC after exposure to elevated temperatures. Hence, based on the experimental data, Hassan et al. [\(2020b\)](#page-7-12) proposed empirical relations for predicting the compressive strength of GPC after exposure

to various temperature levels. The proposed equations have developed by Hassan et al. [\(2020b\)](#page-7-12) were for both curing conditions of fly-ash geopolymer concrete; heat curing (GCT-H) and ambient curing (GCT-A), as shown in Eqs. [\(1\)](#page-3-1) and [\(2\)](#page-3-2).

$$
For GCT-H; f'_{CT} = (f'_{c} \times b + c \times T^{d})/(b + T^{d})
$$

where, $b = 20731.45$, $c = 11.76$, $d = 1.708$ (1)

$$
For GCT-A; f'_{CT} = (f'_c + b \times T - c \times T^2 + d \times T^3
$$

where, b = 0.0724, c = 0.000223, d = 1.55E – 07 (2)

where f'_{CT} is the residual compressive strength at '*T*' temperature (MPa), f'_{C} is the ultimate compressive strength of concrete (MPa) and '*T*' is the temperature in *°C.*

Fig. 2. Comparison of compressive strength of GPC and OPC concrete at high temperatures predicted from various published models (Eurocode [2005;](#page-7-14) Han et al. [2015;](#page-7-15) Hassan et al. [2020b;](#page-7-12) Li and Purkiss [2005\)](#page-8-15)

Xu and Deventer [\(2003\)](#page-9-2) studied the effect of the structural and surface properties of source materials on geopolymerisation. In their work, kaolinite, albite, and fly ash were chosen as alumino-silicate source materials. Alkaline potassium and silicate solutions were used for the study. Specimens were cured for 24 h at 40 °C. X-ray diffraction, Xray fluorescence, X-ray photoelectron spectroscopy, and scanning electron microscopy (SEM) were used to investigate the influence of source materials on the geopolymerisation process. They observed that the fly ash that has an amorphous structure and possesses the lowest binding energies in its structure showed the highest reactivity during geopolymerisation and thereby more compressive strength. The content of K and Ca in the gel also influences the geopolymerisation and the compressive strength. They observed a higher geopolymerisation in mixtures of two or three source materials (both aluminosilicate and alkalis) as compared to that of the single source material.

Moreover, the increase in the molarity of alkaline solution leads to a reduction in the thermal resistance and compressive strength. Kong and Sanjayan [\(2008\)](#page-8-13) studied the thermal damage of geopolymer composites; their findings showed that the strength of GPC increased by approximately 53% with heat curing as compared with ambient curing. They noted that the coarse aggregates also expanded with heat. Up to 800 \degree C, the expansion of aggregates reached up to 2.5%. Zhang et al. [\(2016\)](#page-9-1) studied the compressive strength of fly-ash geopolymer composites at elevated temperature and showed that the fly-ash geopolymer composite with coarse aggregates greater than 12 mm exhibited higher strength for both heat and ambient curing. Kong and Sanjayan [\(2008\)](#page-8-13) found that the compressive strength of GPC is constant at temperatures 800–1000 °C, while for the OPC concrete, the compressive strength falls to zero at 800 °C".

2 Microstructure of Fly-Ash Geopolymer Concrete After Exposure to High Temperatures

"Diaz et al. (2010) studied the behaviour of fly-ash geopolymer paste using two types of class F fly ash and three class C fly ash obtained from different sources. NaOH and Na₂SiO₃ solution were used as alkali. Samples were cured for three days at 60 °C. Chemical, X-ray diffraction (XRD) and particle size distribution (PSD) analyses were performed on the fly ash samples. Fly-ash geopolymer paste was analyzed using XRD and Raman spectroscopy. In addition, setting time and compressive strength tests were performed on fly-ash geopolymer concrete specimens. NaOH solution and $Na₂SiO₃$ solution with a 1:1 ratio were used for the study. It was reported that of the behaviour of fresh mixture and the mechanical properties of the hardened matrix were mainly influenced by three factors; the chemical, crystallographic and physical properties of the fly ash. They observed a positive influence of CaO content on the compressive strength. However, high CaO content causes rapid setting (less than 3 min).

Xu and Deventer [\(2002\)](#page-9-3) studied the effect of the structural and surface properties of source materials on geopolymerisation. In their study, kaolinite, albite, and fly ash were chosen as alumino-silicate source materials. Alkaline potassium and silicate solutions were used for the study. Samples were cured for 24 h at 40 °C. X-ray diffraction, Xray fluorescence, X-ray photoelectron spectroscopy and scanning electron microscopy (SEM) were used to study the influence of source materials on geopolymerisation. They observed that the fly ash that has an amorphous structure and possesses the lowest binding energies in its structure showed the highest reactivity during geopolymerisation and thereby more compressive strength. The content of K and Ca in the gel also influences the geopolymerisation and the compressive strength. They observed a higher geopolymerisation in mixtures of two or three source materials (both alumino- silicate and alkalis) as compared to the single source material.

Hou et al. (2009) investigated the influence of $(SiO₂/Na₂O)$ concentration and curing conditions on the phase composition, microstructure of geopolymer paste prepared using Class F fly ash. The FTIR spectra of alkali-activated fly ash samples showed an increase in chain length and more alumino-silicate gel formation for the sample pre-cured for one day before temperature curing. From the XRD of fly-ash geopolymer, cured in different conditions, they observed that the geopolymers prepared using Class F fly ash and sodium silicate solutions were amorphous. However, the crystalline compounds initially present in the fly ash like Quartz, mullite, and hematite have not undergone dissolution process during the reaction. Reaction kinetics and mechanism of fly-ash geopolymers were studied by Rahier et al. [\(2007\)](#page-8-16). Sindhunata et al. [\(2008\)](#page-8-17) studied the leaching, pore network alteration and gel crystallization of geopolymers. They used Class F fly ash as alumino-silicate material. Activating solutions were prepared by mixing potassium hydroxide or sodium hydroxide with water and commercial silicate solutions. They observed that in alkaline hydroxide or carbonate solutions with up to pH 14 have little effect in terms of leaching of Si and/or Al species, pore network alteration, or gel crystallization. More concentrated hydroxide solutions lead to a more significant extent of leaching, as well as the collapse of the geopolymer gel structure and the formation of detectable quantities of crystalline zeolites. Immersion in water does not show significant leaching of Si or Al species".

Hassan et al. [\(2020b\)](#page-7-12) investigated the microstructure of GPC specimens for ambient and heat curing at various temperatures (room temp., $200 \degree C$, $400 \degree C$, $600 \degree C$, and $800 \degree C$) and compared with the microstructure of OPC concrete specimens. Figure [3](#page-5-0) (a) shows the microstructure of ambient-cured GPC specimens at room temperature condition; it can be seen that the specimens have a lot of unreacted fly ash particles since the reaction between the alkaline solution and fly ash particles needs in case of ambient-cured GPC specimens. However, the fly ash particles of heat-cured GPC specimens totally destroyed by alkaline solution, as shown in Fig. [3](#page-5-0) (b). Moreover, the SEM image of OPC concrete specimen at a normal condition is shown in Fig. 3 (c). The image shows the formation of hydrates and un-hydrate in the sample.

For both curing conditions, i.e., ambient and heat curing, the microstructure of GPC specimens after exposure to 800 °C shows that some voids and cracks produced resulting in some loss in the strength of geopolymeric gel and then compressive strength of GPC, as shown in Fig. [4](#page-6-0) (a) $\&$ (b). While Fig. 4 (c) shows the microstructure of OPC concrete specimens after exposure to 800 °C, and it can be seen that the OPC concrete gets burnt, and the crystal CH is formed. The un-hydrated cavities formed over hydrated give the dense strength of concrete, resulting in the reduction of strength.

Fig. 3. SEM micrographs of GPC and OPC concrete specimens for normal (room) temperature (Hassan et al. [2020b\)](#page-7-12)

Fig. 4. SEM micrographs of GPC and OPC concrete specimens after exposure to 800 °C (Hassan et al. [2020b\)](#page-7-12)

3 Future Scope of the Work

Many studies have been reported on the behaviour of concrete exposed to elevated temperatures. While considering fly-ash geopolymer concrete as an alternate material for cement concrete, information on the behaviour of fly-ash geopolymer concrete at elevated temperatures is also vital, particularly when they are exposed to such conditions. However, a systematic study addressing the behaviour of fly-ash geopolymer concrete exposed to elevated temperatures is lacking at present. Further, structural members exposed to elevated temperatures behave differently compared with the behaviour of materials at elevated temperatures. Therefore, it is also important to know the structural behaviour of members made from fly-ash geopolymer concrete after exposure to elevated temperatures. Hence, such a study needs to be well-investigated for refining and conform the available information in this matter before introducing this technology to the construction industry.

4 Conclusions

Fly-ash geopolymer composites are commonly assumed to give decent fire resistance due to their microstructure and chemical composition. This literature review presents the results and information reported on the previous studies regarding the influence of high temperatures on the fly-ash geopolymer material. The previous studies indicated that the fly-ash geopolymer concrete showed better structural stability than conventional concrete after exposure to high temperatures owing to more stable crosslinked aluminosilicate polymer framework. The teeny cracks were developed between 600 ºC and 800 ºC in GPC while they appeared on the specimens of conventional concrete at the temperature of 200 C. Moreover, GPC had better thermal resistance as compared to conventional concrete, as proven by thermal behaviour, increase in compressive strength, minimal changes of the bands in FTIR spectra and compact microstructure as evidenced by SEM images. The literature review also showed that the strength loss in GPC at high temperatures (800 ºC onward) might be attributed to the thermal mismatch between the fly-ash geopolymer mixture and the aggregates. Finally, the published studies on the structural behaviour of GPC reported that it is more feasible to use fly-ash geopolymer concrete than conventional concrete as source material for producing fire-resistant concrete. This could be utilized as structural elements requiring fire-resistant performance.

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