

Investigation of the behaviour of geopolymer mortar after heating to elevated temperatures

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ABSTRACT

This paper presents experimental results on the colour change, weight reduction, flexural and compressive strength behaviours of geopolymer mortar after it has been heated up to 1000°C. The geopolymer mortar mixture was prepared using a normal concrete sand, pulverized fly ash and a solution of sodium silicate and sodium hydroxide as admixtures. Samples were heated to five levels of temperature, from 200°C to 1000°C in a furnace. After cooled down to ambient, the samples were tested to determine several properties. The results show that the strengths of geopolymer mortars increase dramatically when the temperature rises up to 300°C. After that, the strengths gradually reduce when temperature is increased to 1000°C. Interestingly, the strengths of geopolymer mortar samples are significantly higher than those of compared cementitious mortar samples at all temperatures. These experiments indicate that geopolymer mortar can be used as a fire resistant material, especially for linings for tunnel structures.

Keywords: Geopolymer, fire resistance, green materials, mortar, PFA

INTRODUCTION

Geopolymer (GP) is an alternative cementitious material produced by mixing pozzolanic compounds or rich aluminosilicate mineral materials (Si-Al) with catalysts, including sodium silicate solution (Na₂SiO₃) (or potassium silicate solution (K₂SiO₃)) and sodium hydroxide (NaOH) (or potassium hydroxide (KOH)). These compositions combine and react together resulting in an inorganic polymerization process. The result of the process is a three-dimensional (3D) polymer chain structure of Si-O-Al-O that is strong and durable (Joseph, 2002, 2008; Adward, 2008; Dao, 2009). After hardening, the performance of the polymer product is at least equivalent to traditional artificial stones.

Recently, polymer materials have been of interest to researchers as they have shown the potential to perhaps replace for traditional cementitious products. Investigations performed in several developed countries (Joseph, 2002, 2008; Adward, 2008) as well as in Viet Nam (Dao, 2009, 2010) confirm the outstanding mechanical and durability properties of GP material. The researches have also shown that it is much more suitable to utilise waste materials, which helps to reduce environmental pollution.

Investigations into the resistance of geopolymer binder and geopolymer concrete at elevated temperatures have been of particular interest and many promising results have been obtained (Joseph, 2002, 2008; Adward, 2008; Daniel et al., 2008; Cheng et al., 2003). The geopolymer binder has been confirmed to resist to temperatures up to 1200°C and is therefore seen as a suitable fire resistant material (Joseph, 2002, 2008; Adward, 2008). Geopolymer concrete, however, still loses its resistance at around 1000°C. This is possible mainly due to the large difference in the coefficients of expansion of the geopolymer mortar phase and the aggregates. This leads to damage (de-bonding) of the interface between the geopolymer binder and the surface of the aggregates, resulting in the disintegration of the geopolymer concrete (Daniel et al., 2008; Cheng et al., 2003). The effect of elevated temperature on the geopolymer mortar (GPM) (a mix of fine aggregate (like sand) and GP binder) has been paid little attention, hence this investigation to assess the resistance of GPM at elevated temperatures of GPM.

One of the requirements for safety when designing of construction structures, such as high rise buildings and tunnels, is the ability to resist elevated temperatures resulting from fires (British Tunnelling Society, 2004). In such cases, materials may be subjected to temperatures up to 1000°C or even higher. At these elevated temperatures, traditional cementitious concrete loses its strength and disintegrates, leading to the collapse process of the structure and the potential obstruction of any exits (Fig. 1). Due to this, the number of dead and injured is possibly increased. To reduce or to slow down the collapse process of the structures, the materials need to be able to work at temperatures greater than experienced during fire conditions, i.e. elevated temperatures. The quality of the materials needs to be maintained and for as long as possible, giving people sufficient time to exit of the dangerous location of the collapse (Theodor, 2008).

According to Eurocode 2 and 3 (EN 1992-1-2, 2004; EN 1993-1-2, 2005), the main reason for damage in concrete and reinforced concrete structures at elevated temperatures or during fires is the rapid reduction in the strength of the materials, especially the strength of concrete. The concrete and reinforced concrete layers are damaged mainly due to the following possible reasons: a) rapid release of concrete moisture leading to spalling; b) cracks created due to shrinkage; c) change in crystallization state and the crystals of the hardened cementitious mortar, and especially aggregates in concrete; d) the difference in strains between the phases of concrete and reinforcements leading to fire cracks.



Figure 1. Damage of lining tunnel structure due to fire in Holland, New York State, US, 1949 (Google).

Viet Nam is currently developing its high rise building infrastructure. It is also in the first stages of tunnel projects. As a results, investigations into novel materials that can be made

able to better resist elevated temperatures, especially for tunnel design is necessary. However, Viet Nam has no guidance for incorporating non-standard materials into structures, particularly when considering elevated temperatures and currently, tunnel structures are being lined with traditional Portland cement materials (PC).

This paper presents the results of a laboratory investigation looking at heating geopolymer mortar (GPM) up to 1000°C in order to determine the changes in colour and weight, and the flexural and compressive strength behaviour. The results of the GPM samples were compared to those obtained for traditional Portland cement mortar (PCM) specimens. The results clearly support the possible application of GPM material in structures, especially for the lining of tunnel structures and, therefore, its use as a replacement for traditional PCM.

SAMPLE PREPARATION

Materials and proportions

Results presented here are taken from a series of tests performed on geopolymer binders and geopolymer materials. This investigation used class F pulverized fuel ash (PFA) from Phalai power plant, Haiduong province, Viet Nam. This PFA was obtained by using air classification technology. Other materials are Portland cement (PC), sodium hydroxide (NaOH) and potassium silicate (K_2SiO_3) those are similar to the materials used in previous published investigations (Dao, 2009, 2010). Details of the composition and fineness of the class F PFA are presented in Table 1 and Table 2. Sodium hydroxide solution was diluted from solid sodium hydroxide flakes of 98% purity. 560 gram solid NaOH diluted in water was used to get one litre of NaOH solution with a molarity of 14M. The K_2SiO_3 solution had a ratio of $K_2O/SiO_2/H_2O$ of 14.7/29.4/55.9 % in quantity. Modulus of the K_2SiO_3 is 3.

Table 1. Chemical composition of the PFA using in this investigation (% in mass)

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	TiO ₂	SO ₃	LOI*
51.74	24.53	5.59	0.81	1.95	4.42	0.11	0.76	0.31	8.98

*) Loss on ignition

Table 2. Gradation of the PFA used in this investigation

Size (µm)	30	20	10	5
Passing content (%)	95	51.67	33.06	16.77

PFA has an average dimension of 9.6 µm and surface area of 8169 cm²/cm³. The aggregate used to prepare the GPM mixes was a natural sand with a modulus, $M_k = 2.6$. To prepare the PCM mixes, a standard sand (TCVN 6227, 1996) was used.

Table 3. Materials, proportions and curing conditions to manufacture samples

No	Samples	Materials and proportions (mass)						Curing conditions
		Sand	$\frac{PC}{Sand}$	$\frac{W}{PC}$	$\frac{PFA}{Sand}$	$\frac{NaOH^*}{PFA}$	$\frac{K_2SiO_3}{PFA}$	
1	PCM	1**	0.33	0.5				In water, room condition***
2	GPM	1			0.33	0.157	0.393	60°C/24 hrs + room condition

*) NaOH solution 14M; **) standard sand; ***) room condition ($t = 25 \pm 3^\circ C$; humidity = $70 \pm 15\%$).

The water (W) used was tap water. Materials, proportions and curing conditions to manufacture the samples are presented in Table 3.

Mixing

Materials were mixed using a Holbat Mixer. Control mixes (PCM) using cementitious binder PCB30 were mixed following the TCVN 6016-95 standard (1995). The mixing process is in accordance with the guidelines reported in a previous paper (Dao, 2010).

Sample preparation

Following mixing, the materials were cast in prism moulds to produce samples of 40×40×160 mm. The sample preparation process was in accordance with the guidelines reported in a previous paper (Dao, 2010).

Curing regime

The curing regimes were as follows:

The control samples were cured in accordance with TCVN 6016-95 (1995).

The GPM samples, after 24 hours in the mould, were cured in an oven at 60°C for 24 hours. They were then demoulded and stored in laboratory condition until required for testing.

RESULTS AND DISCUSSION

There were 36 GPM samples and 18 control (PCM) samples prepared and tested. As mentioned above, after curing the samples were stored under laboratory conditions until they achieved constant mass. This was an attempt to ensure that the samples possess a similar internal humidity before being fired in the furnace. The furnace used in this investigation was made in Germany and was a VMK1600 with a maximum firing temperature of 1600 °C (Figure 2).



Figure 2. Furnace VMK1600



Figure 3. GPM samples before and after firing at 1000 °C

Five sets of the samples (each set includes 6 GPM and 3 PCM samples) were fired at five different temperatures; 200, 400, 600, 800 and 1000 °C. The rate of heat increase was 5 °C/minute. After reaching the target temperature, the temperature was maintained for 2 further hours and then the furnace was switched off. The samples were kept in the furnace for a further 24 hours and allowed to cool down to laboratory conditions. The samples were

then tested to assess several key properties. The results of the fired samples were compared to the control samples which did not undergo firing. This testing method replicates the testing process presented in ASTM E119 (2000) and ISO 834 (1985). Additionally, this method enhances the adverse conditions experienced by the samples as they were fired in the furnace chamber at the elevated temperatures for a long time.

Colour change

After firing at the defined temperatures the samples were assessed visually to determine colour change and cracks.

In terms of cracks on the surface of the samples after firing, it was shown that the PCM samples possessed a greater number of cracks and cracks that were wider when compared with the GPM samples. This was possibly because the free water content in the PCM samples was higher than that of the GPM samples (Table 3). The evaporation of the free water results in cracking.

In terms of colour change, the PCM samples exhibited a fairly uniform change in colour across the cross section. This suggests that the heat transferred uniformly to the sample core. However, the change in colour of the GPM samples was definitely not uniform across the sample; the outer layers were a brick red colour, and the core area was a much darker colour (Fig. 3, 4). This is possibly due to the GPM samples having a lower heat transfer coefficient. As a result, the core of the GPM samples was potentially subjected to a lower temperature than the surface or to a similar temperature but for a reduced duration. The outer layer, under the direct effect of the high temperature, changed into ceramic material but the core area remained as GP material. The low heat transfer coefficient of the GPM protects the core layers of structures subjected to fire, which would help to maintain the load bearing ability of GPM structures.



Figure 4. Colour change in the cross section of samples fired at 1000 °C
a) PCM samples, b) GPM samples

Mass change

All the samples were weighed before and after firing to an accuracy of ± 0.1 gram. The test results are shown in Figure 5.

As shown in Figure 5, the mass of all samples reduces after firing; the higher the firing temperature, the higher the mass loss. As the temperature increased from 200 to 600 °C, there was a steady linear increase in mass loss (i.e. 1.4%, 3.04% and 4.21% at 200, 400 and 600 degrees C, respectively). However, at temperatures of 800 and 1000 °C, the mass loss was negligible and stable at 4.24%.

The trend of mass loss of the PCM samples was similar to the GPM samples, but significantly higher. At the firing temperatures of 200, 400, 600 and 800 °C, the mass of

PCM samples reduced 3.66%, 5.46%, 6.91% and 8.04%, respectively. After 800 °C, the mass loss was stable at 8.04%.

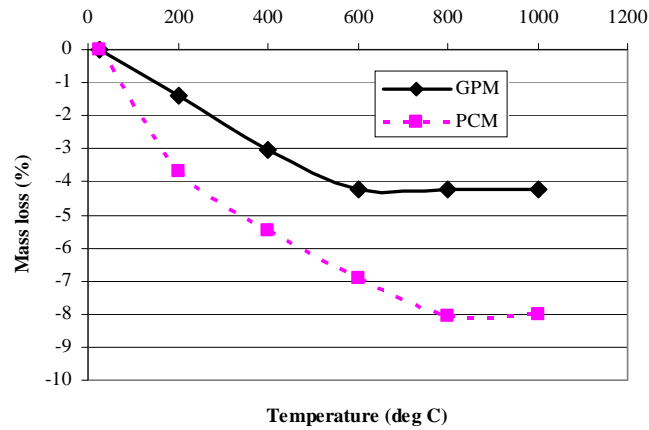


Figure 5. Mass loss due to elevated fire

It was predicted from the results that when the temperature is increased, this leads to the free water, adsorbed water and chemical bound water in the samples being evaporated in that order. However, due to the fact that the water content in the GPM samples was less than that of in the PCM samples, the mass loss of the GPM samples was lower than that of the PCM samples. This allows us to hypothesise that shrinkage of the GPM samples would be significantly lower than that of the PCM samples. These results suggest a potential reduction in the damage of GPM structures in comparison with structures using the traditional Portland cement when subjected to fire or elevated temperatures.

Bending test

The flexural tests were run in accordance with TCVN 6016-95 (1995). To improve the sensitivity of the tests, the three-point bending frame was integrated into the C.B.R soil test equipment (Dao, 2010). Flexural strength results of the samples after firing at 200, 400, 600, 800 and 1000 °C were statically evaluated (ACI 214R, 2002). Average flexural strength (R_{ku}^{tb}), standard deviation (σ) and coefficient of variation (C_v) are presented in Table 4 and Figure 6.

Compared to the unfired samples, all samples fired at both 200 and 400 °C possessed increased flexural strength. The increase in the GPM samples was greater in comparison to that of the PCM samples. The GPM samples improved by approximately 60% at 200 °C, from 10.74 MPa up to 17 MPa, while the PCM samples only increased 15%, from 6.64 MPa up to 7.94 MPa. This result possibly indicates that by increasing the temperature up to 400 °C polymerization of the inorganic compounds is promoted leading to improved flexural strength.

The flexural strength of both the GPM and PCM samples reduced when the temperature was higher than 600 °C. However, at 1000 °C the PCM samples decreased to a value of 0.3 MPa while the GPM samples only reduced to a strength of 3 MPa (ten times higher than that of the PCM samples). This results confirm why structures using PCM materials collapse at around 1000 °C. However, the results also suggest that structures prepared using GPM materials would maintain about 30% of their load bearing capacity. In short, the flexural

strength of GPM materials in this investigation is higher than that of PCM materials at a range of temperatures up to 1000 °C, from two times at room temperature (Dao, 2010) up to a factor of 10 at 1000 °C. This suggests that the fire resistance of GPM materials is significant.

Table 4. Flexural behaviour

Temp.	GPM			PCM
	R_{ku}^{tb}	σ	C_v	R_{ku}^{tb}
°C	MPa	MPa	%	MPa
25	10.74			6.64
200	17.00	1.19	6.98	7.94
400	12.15	0.71	5.88	7.00
600	3.95	0.48	12.10	1.66
800	3.02	0.21	6.88	0.69
1000	3.01	0.16	5.17	0.31

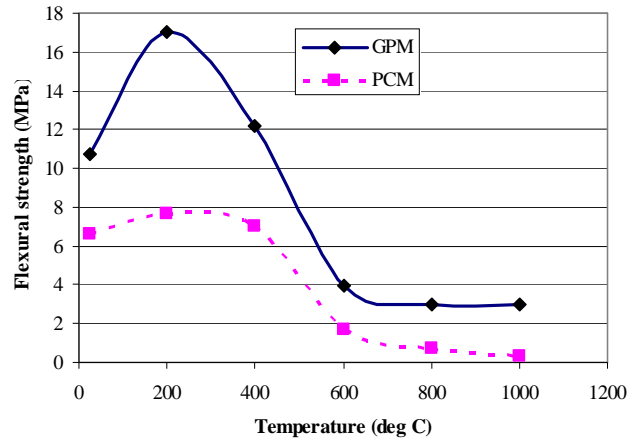


Figure 6. Flexural strength at different temperatures

Compressive test

Compressive strengths were obtained in accordance with TCVN 6016-95 (1995). Compressive strengths of the samples after firing at 200, 400, 600, 800 and 1000 °C were statically evaluated (ACI 214R, 2002). Average compressive strength (R_n^{tb}), standard deviation (σ) and coefficient of variation (C_v) are presented in Table 5 and Figure 7.

Table 5. Compressive behaviour

Temp.	GPM			PCM
	R_n^{tb}	σ	C_v	R_n^{tb}
°C	MPa	MPa	%	MPa
25	41.98			40.71
200	71.01	9.51	13.39	38.05
400	68.53	5.75	8.39	35.21
600	47.42	5.22	11.01	29.35
800	31.77	3.96	12.47	12.79
1000	26.36	4.10	15.54	5.91

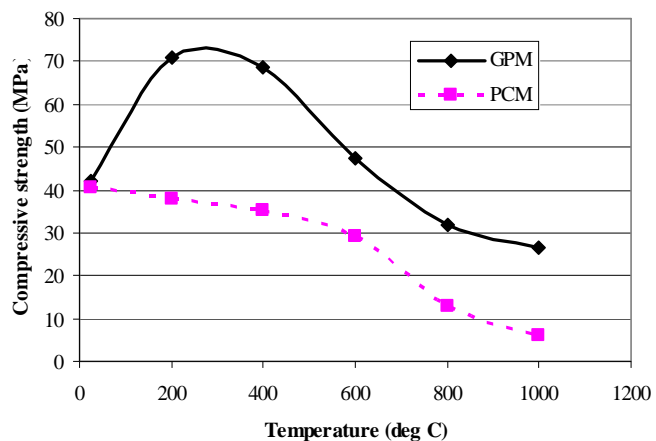


Figure 7. Compressive strength at different temperatures

At room temperature, the compressive strength of the GPM samples were around 42 MPa, which was similar that of the PCM samples (see Fig. 7). These results agreed previous researches into GP materials (Dao, 2009, 2010). When the temperature was risen up to 600 °C, the compressive strength of the PCM samples gradually reduced from 40.71 MPa to values of 38, 35 and 29 MPa (7, 14 and 18 %, respectively, in comparison the value at room temperature). Conversely, the GPM samples increased in strength, when the temperature increased to 600 °C, showing increases of 70, 63 and 13 %, respectively, in comparison with the control samples cured at room temperature. Again, it is thought that the increase in temperature up to 600 °C could promote polymerization of the inorganic compounds in the GPM.

When the temperature was increased up to 1000 °C, the compressive strength of the PCM samples dramatically reduced to 6 MPa, equivalent to only 14% of the strength at room temperature. Similarly, the GPM samples also decreased in strength but the strength still remained over 26 MPa, equivalent to 63% of that strength achieved at room temperature. These results show that whilst there is a significant deterioration of the PCM samples at 1000 °C, the GPM materials retain significant strength and therefore load capacity .

The dramatic reduction in compressive strength of the PCM ties in with the mass loss analysis discribed earlier. When free water, adsorbed water and chemical bound water is lost, the microstructure of the hardened cement paste is damaged, leading to a loss in the load bearing capacity of PCM matrix. Conversely, the 3D microstructure of GPM material is clearly much more robust making it far more suitable to resist elevated temperatures.

CONCLUSION

From the experimental results obtained in this investigation, the following conclusions can be drawn:

- ❖ After firing at 1000 °C, the PCM samples almost disintegrated while the GPM samples were converted into a ceramic material and still maintained good load bearing capacity.
- ❖ After firing at 1000 °C, the mass loss of GPM samples was approximately 50% of that of the PCM samples.
- ❖ Flexural strength of the GPM samples increased with a rise in temperature from room temperature to 400 °C. After firing at 1000 °C, the flexural strength of the GPM samples was ten times greater than that of the PCM samples.
- ❖ The compressive strength of the PCM samples doubled after firing at 400 °C. After firing at 1000 °C, the compressive strength of the PCM samples reduced to approximately 14% of that achieved at room temperature, whilst the GPM samples retained approximately 63% of that measured at room temperature.
- ❖ Further investigations into the behaviour of geopolymer mortar materials fired at temperatures higher than 1000 °C are still required. These further investigations should also consider large structural elements. The research presented here helps to suggest that the use of GPM should definitely be considered in construction, particularly for tunnel linings where fire is a significant load case.

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