Investigation into the Effect of the Duration of Exposure on the Behaviour of GPC at Elevated Temperatures

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Abstract. Ordinary Portland Cement (OPC) Concrete has long been used in the construction industry as a primary material owing to its versatility, superior performance, low cost, easy workability and availability of accepted standards of practice. The readily available raw materials for the manufacture of cement, and subsequently for concrete itself, have been a driving force for the acceptance of concrete as a construction material worldwide. Recently however, OPC concrete has come under scrutiny over its large carbon footprint. This is largely due to the energy intensive manufacturing process of cement and the extensive use of virgin material in cement production. Focus is therefore shifting to engineer new construction materials that offer similar advantages to that of OPC concrete while being environmentally friendly. Geopolymer Concrete (GPC) is such a material. It has emerged during the last decades, and has been found to possess excellent engineering properties as well as enormous benefits on the sustainability front. The current study is conducted to investigate the compressive strength of GPC up to temperatures of 1000°C for varying duration of exposure time. It was found that when tested at temperatures of 600°C, 800°C and 1000°C, the GPC samples exhibited a higher compressive strength (8-18%). However, the samples tested after cooling recorded a residual compressive strength 25-50% lower than the ambient strength. Yet, the residual strength of GPC is significantly higher than that of OPC. The duration of exposure time was found to have an insignificant effect on the strength properties of GPC, especially at higher temperatures. Scanning Electron Microscopy (SEM) was used to reveal the changes to the micro-structure that took place after exposure to high temperatures and to get a useful insight into the behaviour of geopolymers.

1 Introduction

Alkali liquids (usually a soluble metal hydro-oxide and/or alkali silicate) can be used to react with silica (SiO2) and alumina (Al2O3) rich natural materials, like metakaolin or with industrial by-products, like Fly Ash (FA), Silica Fume (SF), Rice Husk Ash (RHA) or Slag to produce binders [1-3]. Though the details are still debated, many researchers agree that the basic reaction mechanism takes place in three stages; namely: dissolution of Si and Al from the source material, hydrolysis or gelation, and condensation forming a 3D network of silico-aluminates also termed as the ‘geopolymer backbone’ [1, 2, 4-8]. Davidovits [2] describes geopolymerization as an exothermic reaction, and has schematised it as follows:

\[
\text{Si-Al source} + \text{Silicates} + \text{Water} + \text{Alkaline Liquid} \rightarrow \text{Geopolymer Precursor}
\]

GPC does not require any hydraulic binder. The role of fly ash (FA) in GPC is entirely different from that it plays when used as a cement replacement material in OPC concrete to enhance certain properties such as workability or to reduce the heat of hydration. In such cases FA has no pronounced effect on the strength of concrete [9] especially early strength. In GPC, however, FA is the sole source of aluminosilicates for reaction with the alkaline solution to form the binder, and is thus a critical factor in strength development.
1.1 Changes at High Temperatures to OPC and GPC

Owing to its very different chemistry as compared to OPC, GPC behaves very differently when exposed to high temperatures. It is widely accepted [10] that OPC loses over 80% of strength when the temperature exceeds 800°C. This is due to inherent physical stresses caused by the introduction of heat energy, and the chemical breakdown of the hydrated phases in the cement paste [11]. There have been a number of general findings published in the literature that cover the effects of high temperatures. It is widely accepted [10] that OPC, GPC behaves very differently when exposed to high temperatures of 100°C, 350°C, 600°C, 800°C and 1000°C. The heating rate was set at 4.5°C/min for all cases, which results in very high thermal expansion.

2 Materials and Testing

The GPC samples were made using Grade D Sodium Silicate and 12M Sodium Hydroxide prepared from 98% pure flakes as alkaline solutions; while ASTM Class F Fly Ash was used as the main aluminium and silicate source. Cylinders with nominal measurements of 75mm diameter and 150mm height were mixed and cast using the procedure described in [19]. The samples, after an initial rest period, were cured for 72 hours at 80°C. They were then placed in an environmental room with a relative humidity of 50% and a temperature of 23°C till the time of testing. All testing was done at 7 days after mixing. The composition of mix M-1 and the ambient mean compressive strength \( f'c \) is given in Table 1.

### Table 1: Composition and Mean Compressive Strength \( f'c \) of the GPC Samples

<table>
<thead>
<tr>
<th></th>
<th>M-1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fly Ash</td>
<td>420</td>
</tr>
<tr>
<td>NaOH</td>
<td>44</td>
</tr>
<tr>
<td>Na$_2$SiO$_3$</td>
<td>110</td>
</tr>
<tr>
<td>Coarse</td>
<td>1120</td>
</tr>
<tr>
<td>Fine</td>
<td>650</td>
</tr>
<tr>
<td>Free Water</td>
<td>28.5</td>
</tr>
<tr>
<td>SP*+VM^</td>
<td>7.2</td>
</tr>
<tr>
<td>( f'c )</td>
<td>44</td>
</tr>
</tbody>
</table>

*Superplasticiser, ^Viscosity Modifier

The samples were grinded, measured and weighed in accordance with Australian Standards [20] to establish the ambient compressive strength. The grinded samples were then put in a furnace and heated to desired temperatures of 100°C, 350°C, 600°C, 800°C and 1000°C. The heating rate was set at 4.5°C/min for all cases, which is consistent with RILEM 129-MHT standard recommendations [21]. The time required for the samples to reach equilibrium was established by embedding K-Type thermo couples into the sample core as well as on the surface and in the air. This data was collected and plotted against time. It was found that irrespective of the target temperature, the time required to reach thermal equilibrium was around 90 min as seen in Figure1. The target temperature was maintained for 2 and 6 hours respectively after equilibrium was established to study the effect of duration of exposure on strength of GPC. At this time the samples were removed from the furnace. Hot strength was determined by testing the sample immediately after removing it from the furnace. As the testing duration was rather short (under 2 mins), it was established that the heat loss during testing was insignificant to affect the results in any way. Samples to be tested for residual strength were removed from the furnace and kept in an identical switched off furnace to cool down to ambient temperature overnight. These samples were then weighed, measured and tested as per standards to obtain their residual strengths. After strength...
testing, the samples were collected and marked in air
tight containers to undergo Scanning Electron
Microscopy (SEM) for changes to their microstructure. A
Hitachi TM3000 instrument was used for this purpose.

At 350°C the rate of strength loss is much slower as
compared to 100°C heating. This can be attributed to the
fact that the temperature is high enough for considerable
amount of unreacted FA particles to undergo
gopolymerization. The strength gained during this
additional geopolymerization offsets the strength lost due
to escaping water.

At much higher temperatures (600°C and 800°C) GPC gains 10-15% strength throughout the heating
period. This gain may be a result of complete
gopolymerization in the matrix as well as additional
molecular changes in the system and are discussed further
in the subsequent section. This gain however, flattens out
after the first thermal equilibrium is reached and no
further strength gain is observed with continued heating.

3 Results and Discussion

3.1 Hot and Residual Strength

The measured strength was normalized against the
ambient strength. The normalized hot strength is plotted
in Figure 2. It can be noticed that for the lower
temperatures of 100°C and 350°C, GPC looses 15-25% of
its strength in the first 2 hours of heating. After this initial
loss the retained strength flattens out to about 80% of
ambient at the 6 hour mark. The early loss in strength,
especially at 100°C, can be attributed to the loss of free
water in the GPC system. The escaping water leaves
behind pores and voids compromising the integrity of the
matrix. Moreover, the temperature is not high enough, or
maintained long enough, for chemical changes to occur
within the matrix, which results in strength gains, as is
the case for the high temperature tests. However, a slight
increase in strength (around 5-8%) is noticed as the
heating reaches the 6 hour mark. This indicates that the
sustained heat has facilitated the reaction between the
unreacted FA particles and the alkaline solution resulting
is a moderate strength gain.

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The normalized residual strength of GPC samples
are shown in Figure 3. All GPC samples experienced a
loss in strength when tested after cooling. Depending on the
exposure temperature the percentage of strength lost
varies between 25-50%. At higher temperatures of 600°C,
800°C and 1000°C the strength loss is between 40-50%
down from a 15% increase when tested hot. Even at
relatively lower temperatures of 100°C and 350°C the
residual strength is around 10% lower than the hot
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Figure 1: Two Step Temperature Rise to Establish Time for
Core Equilibrium

Figure 2: Normalized Hot Strength of GPC Samples

Figure 3: Normalized Residual Strength of GPC Samples

Figure 4: Hot and Residual Strength for Each Individual Temperature
aggregates). During cooling the stresses caused by the thermal incompatibilities of these materials induce cracking resulting in lower strengths.

3.2 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) was used to obtain the micrographs of the GPC samples before and after exposure to high temperatures. The micrographs clearly show a significant change in the structure of the GPC matrix and gives valuable insight into the strength behaviour of these samples. Figures 5 (a and b) show that there is no considerable change in the structure of the GPC, with several unreacted FA spheres still scattered throughout the sample. Moreover, in Figure 5 b, several micro-cracks can be seen in the sample which may be a result of thermal incompatibilities between the matrix and aggregates. They may also have been caused by the stress experienced during testing. Also noticeable in the micrographs are voids, which may have been caused by escaping water or lack of compaction. After the samples have been exposed to 350°C (Figure 5c), it is apparent that the FA particles have collapsed with extensive pitting visible throughout the micrograph. This supports the hypothesis that the increase in temperature facilitated the geopolymerization within the matrix. However, at this temperature several unreacted FA spheres are still visible.

The pitting, diffusion and collapsing of FA spheres is widespread when the GPC samples are exposed to 600°C. There are no unreacted FA spheres visible at this temperature while the matrix resembles a solidified mass of near molten matter. Also visible is the near-monolithic phase in GPC, although at this temperature these are far and few in between. This clearly shows a change in the material behaviour of GPC at this temperature and points to a viscous-flow-type phenomenon generally attributed to glass and metals and is reported by Pan [17]. As the temperature is increased further to 800°C and then to 1000°C, the near-monolithic phase in GPC is found more frequently through the matrix. This signifies the sintering of the fly ash particles and explains why at these high temperatures GPC exhibits higher compressive strength. However, upon cooling, due to thermal shock or incompatibilities within the concrete, extensive cracking is evident in the samples. This accounts for the considerable loss of strength observed between hot and residual strength of GPC at higher temperatures. It was also observed that the duration of exposure has no effect on the microstructure of the GPC samples.

4 Conclusions

1. Most changes in strength and microstructure of GPC samples occur in the first 2 hours of exposure to high temperatures.
2. After the initial changes have taken place the duration of exposure has an insignificant effect on the strength of GPC samples.

3. Microstructural changes in GPC occur rather quickly over a short period of time after equilibrium temperature is reached.

4. At relatively low exposure temperatures the movement of water within or from the GPC system has a considerable negative effect on its strength. However, as the temperature is increased further, the microstructural changes offset this strength loss caused by movement of water and GPC experiences a gain in strength.

5. For all tests conducted the residual strength of GPC is always less than the hot strength irrespective of exposure temperature. However, this difference is magnified at high temperature ranges.

References


