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Fracture properties of geopolymer paste and concrete

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Geopolymers are an emerging type of cementitious material purported to provide an environmentally friendly alternative to Portland cement-based concrete. This paper reports the results of experimental research on fracture properties (fracture energy and brittleness) of fly ash based geopolymer concrete and paste with various mix parameters. The characteristic length of the geopolymer concrete was approximately three times less than that of ordinary Portland cement (OPC) concrete, due to an increase in tensile splitting strength of about 28%, a decrease in elastic modulus of about 22% and a decrease in fracture energy of about 24%. The difference in characteristic length is similar to that reported between high-strength and normal-strength OPC concretes, indicating that the geopolymer concrete exhibits higher brittleness than its OPC counterpart. This trend was found to be consistent between pastes and concretes, implying that the difference between geopolymer and OPC concrete is due to the type of matrix formation (geopolymerisation or hydration). For geopolymer concretes made with different mix parameters, fracture properties are closely correlated to their compressive strength.

Introduction

General

Geopolymers are environmentally friendly cementitious materials that can be manufactured by using fly ash in combination with sodium silicate and sodium hydroxide solution (Hardjito and Rangan, 2005). After heat curing at 60°C for 24 h, they have been shown to possess optimum mechanical properties (Fernandez-Jimenez *et al.*, 2006; Rangan *et al.*, 2006). Initial studies of reinforced geopolymer concrete structural members showed that the load-carrying capacity of test members is similar to that of ordinary Portland cement (OPC) concrete (Sarker, 2009). Geopolymer concrete is thus believed to be suitable for structural applications.

With regard to safety assessment and design of concrete structures, it has been recognised that the fracture properties of concrete need to be considered in structural design (Hilsdorf and Brameshuber, 1991). For example, limits on minimum flexural and shear reinforcement are governed by the concrete brittleness (Roller and Russel, 1990). It is, therefore, necessary to evaluate the fracture properties of geopolymer concretes if the safety of structures built with such materials are to be assured. However, the fracture properties of geopolymer concretes have received little attention, although their mechanical properties (Fernandez-Jimenez *et al.*, 2006; Hardjito and Rangan, 2005) and structural behaviour (Sarker, 2009) have been studied to a certain degree.

This paper focuses on the study of the fracture properties of a geopolymer paste and concrete by measuring the splitting tensile strength, the modulus of elasticity and the fracture energy of the materials. Based on these properties, the degree of brittleness of both paste and concrete is calculated. For the purpose of benchmarking and comparisons, OPC counterparts were also prepared and tested.

Brittleness

Brittleness is commonly understood to be the tendency of a material to fracture abruptly before significant irreversible deformation occurs (Zhang *et al.*, 2000). To evaluate the degree of brittleness B of a concrete structure, Bache (1986) proposed:

$$B = \frac{Lf_t^2}{G_F E} \quad (1)$$

where G_F is the fracture energy, E is the modulus of elasticity, f_t is the tensile strength and L is the size of the test specimen. For test specimens of the same size, as in the present study, Equation 1 may be expressed as

$$l_{ch} = \frac{G_F E}{f_t^2} \quad (2)$$

where l_{ch} is defined as the characteristic length by Hillerborg *et al.* (1976) as a brittleness parameter in the fictitious crack model

(FCM) for concrete. The higher the degree of brittleness, the lower the value of l_{ch}.

Calculation of fracture energy

The fracture energy of paste was calculated by the stress intensity method. In this method, the stress intensity factor K was determined by performing a three-point bend test on a beam specimen with a notch on the tensile face of the beam, as shown in Figure 1. K is given by (Brown and Strawley, 1967):

$$K = Y\frac{6\boldsymbol{M}a^{1/2}}{bd^2} \tag{3}$$

where

$$Y = 1{\cdot}99 - 2{\cdot}47\left(\frac{a}{d}\right) + 12{\cdot}97\left(\frac{a}{d}\right)^2 - 23{\cdot}17\left(\frac{a}{d}\right)^3 + 24{\cdot}80\left(\frac{a}{d}\right)^4$$

a is the notch depth (m), d is the beam depth (m), $\boldsymbol{M}$ is the bending moment (kN/m) due to the maximum applied load and the self-weight of the beam, l is the beam span (m) and b is the beam width (m).

The value of G_F is then calculated from:

$$K = (G_F E)^{1/2} \tag{4}$$

The stress intensity factor method is not effective for a heterogeneous material such as mortar or concrete in which substantial energy dissipation occurs during fracture, resulting in non-linear behaviour during loading. Therefore, a method based on the FCM (RILEM, 1985) was used in the current investigation because of its simplicity and the availability of extensive test data for OPC concrete for benchmarking purposes.

The test setup used for the calculation of G_F is the same as that in the stress intensity method (Figure 1). The fracture energy is calculated as:

$$G_F = \frac{W_0 + \boldsymbol{mg}\delta_0}{A_{lig}} \tag{5}$$

where W_0 is the area under the load–deformation curve (N/m) or (J/m^2), $\boldsymbol{mg}$ is the self-weight of the beam between support (kg), δ_0 is the deformation at final fracture and A_{lig} is the ligament area ($= b(d - a)$) (m^2).

Experimental work

Materials

OPC conforming to the requirements of ASTM type I was used for making the OPC concrete. The fly ash used for making geopolymers in this investigation was dry ASTM type F (low calcium) fly ash. The chemical composition of the binders, as determined by X-ray fluorescence (XRF) analysis, is summarised in Table 1.

Constituent: %	OPC	Fly ash
Al_2O_3	4·7	30·5
SiO_2	19·9	48·3
CaO	63·9	2·8
Fe_2O_3	3·4	12·1
K_2O	0·5	0·4
MgO	1·3	1·2
Na_2O	0·2	0·2
SO_3	2·6	0·3
Loss on ignition	3·0	1·7

Table 1. Chemical composition of binders

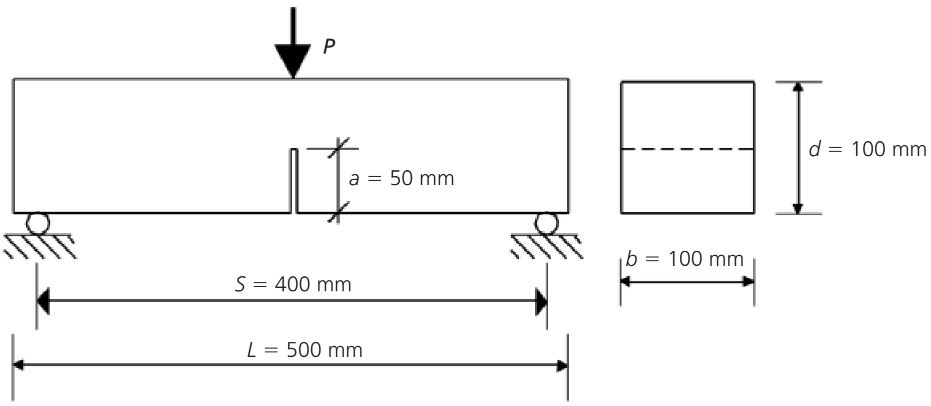

Figure 1. Test arrangement for three-point bend test

Coarse aggregate was crushed old basalt quarried from the Readymix Mount Shamrock quarry. Maximum aggregate size was 14 mm. The fine aggregate was locally available river sand.

The alkaline liquid used in geopolymers consisted of a mixture of commercially available grade D sodium silicate solution with a specific gravity of 1·53 and a modulus ratio (M_s) of 2 ($M_s = SiO_2/Na_2O$ ($Na_2O = 14{\cdot}7\%$ and $SiO_2 = 29{\cdot}4\%$ by mass)) and sodium hydroxide solution. The sodium hydroxide solution was prepared by dissolving the commercial-grade sodium hydroxide (NaOH) pellets (98% purity) in distilled water. Both alkaline solutions were prepared and mixed together 1 day prior to usage.

Specimen preparation and curing regime

Two paste and four concrete mixes were used in the investigation. For all the concrete mixes, the binder content was fixed at 24% of the total mass of dry materials; the remainder consisted of aggregates. The coarse and fine aggregate proportion was fixed at 2·34. The mix proportions and other mix-design variables are summarised in Table 2.

The mixing procedures used in the manufacture of OPC and geopolymer materials were similar. The binders (cement or fly ash) and the liquid component (water or alkaline liquid) were mixed in a conventional pan mixer for 5 min. The cement and aggregates or the fly ash and aggregates were dry mixed for 3 min. The water or alkaline liquid then was added and wet mixing was carried out for 4 min. The mixture was poured into different types of moulds (depending on the test for which they were intended) in three equal layers. Each layer was vibrated for 15–30 s on a vibration table.

Curing for OPC and geopolymer-based materials was done in different ways.

(*a*) The OPC paste and concrete specimens were cured under polyethylene sheets for 24 h in a laboratory environment. They were then removed from the moulds and transferred to a tank of saturated limewater at 23 ± 2°C as the moist-curing regime to satisfy ASTM C192 requirements (ASTM, 2007). Specimens were cured for 28 days.

(*b*) The geopolymer paste and concrete specimens were kept in the moulds and covered by polyethylene sheet and placed immediately in a preheated oven at 60°C. The curing times are given in Table 2. The specimens were then demoulded.

After curing, both OPC and geopolymer specimens were stored in a controlled environment kept at relative humidity 50 ± 3% and temperature 23 ± 2°C. This environment meets the International Organization Standardization (ISO) requirements as a standard atmosphere for conditioning and testing of materials known to be sensitive to variations in temperature or relative humidity. All compression specimens were sulphur capped to satisfy ASTM C617 requirements (ASTM, 2009). Specimens were tested after 28 days. For the same type of tests, both OPC and geopolymer specimens were tested on the same day.

Testing

Compressive strength

The compression tests were performed on 100 × 200 mm cylinders in accordance with AS1012.9 (SA, 1999).

Tensile splitting strength

The tensile strength was measured by performing the cylinder splitting test on 150 × 300 mm cylinders in accordance with AS1012.10 (SA, 2000).

Static modulus of elasticity and Poisson's ratio

The compression tests were performed on 100 × 200 mm cylinders in accordance with AS1012.17 (SA, 1997).

	Mass: kg/m^3					
	Mix 1	Mix 2	Mix 3	Mix 4	Mix 5	Mix 6
14 mm aggregate	—	—	1294	1294	1294	1294
Fine sand	—	—	554	554	554	554
Cement	1279	—	394	—	—	—
Fly ash	—	1249	—	381	381	381
Water	512	—	158	—	—	—
Sodium hydroxide solution (8M)	—	160	—	49	49	106
Sodium silicate solution	—	402	—	122	140	117
Mass ratios						
Water/cement	0·4	—	0·4	—	—	—
Alkali liquid/fly ash	—	0·45	—	0·45	0·5	0·65
$Na_2SiO_3/NaOH$	—	2·5	—	2·5	2·9	1·1
Curing time of geopolymers at 60°C: h	—	18	—	18	72	168

Table 2. Mix designs

Fracture energy

Three-point bending tests were performed on $100 \times 100 \times 500$ mm beams for concretes and pastes. The displacement control rate was 0·5 mm/min so that the maximum load for any specimen was achieved within the first 30–60 s. The fracture energy of the concretes and pastes was calculated as described earlier.

Results and discussion

Density and compressive strength

The mass and dimensions of two cylinders per mix were measured in order to calculate the density of the material; a third specimen was measured if the initial two densities differed by more than 5%. Three specimens were tested to determine compressive strength. The standard deviation for all the results was below 5·0% except for a standard deviation of 7·8% measured for mix 2. The density and compressive strength values of each mix are summarised in Table 3, which shows that there were no significant differences between the density of OPC paste (mix 1) and that of geopolymer paste (mix 2). The compressive strength results are also similar: 71·2 MPa for the geopolymer paste and 68·2 MPa for the OPC paste.

The density of concrete primarily depends on the density of aggregates used in the mix. Since the type of aggregates in all the concrete mixes were the same, the density of the concrete mixes varied only marginally, between 2445 and 2555 kg/m^3. The compressive strength of the concrete mixes made with OPC was slightly lower than that of the geopolymer concrete.

Figure 2 shows the effect of the composition of the alkaline liquid on the compressive strength of geopolymer concrete. Among all the geopolymer concretes, the fastest strength development was observed for mix 4, which has a sodium silicate solution to sodium hydroxide solution ratio of 2·5. A similar test trend was also reported earlier by Hardjito and Rangan (2005). The presence of soluble silicate species leads to fast strength development due to an increase in the extent of dissolution of aluminium and silica in the fly ash and hence enhanced geopolymerisation (Palomo *et al.*, 1999). The effect of soluble

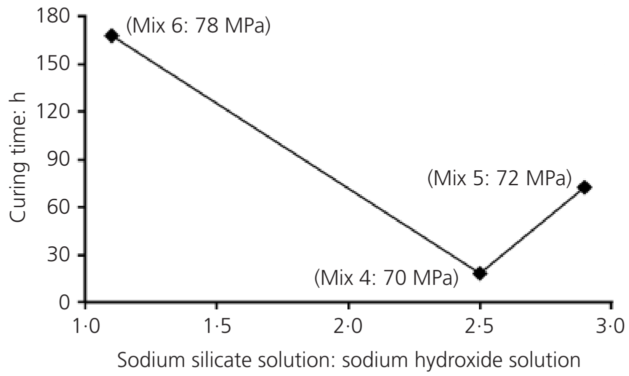

Figure 2. Effect of ratio of sodium silicate solution to sodium hydroxide solution on compressive strength

silicate content on the compressive strength of geopolymers has been comprehensively investigated and well explained in the literature (Duxson *et al.*, 2007).

Mechanical and fracture properties of pastes

The mechanical properties of concrete vary according to its constituents. A comparison of the properties of geopolymer and OPC paste is presented in Figure 3: the geopolymer paste shows a higher tensile splitting strength but lower modulus of elasticity and fracture energy. Characteristic length was calculated using Equation 2. The characteristic length of the geopolymer paste was found to be one third of its OPC counterpart; this indicates that geopolymer paste possesses a significantly higher brittleness than OPC paste.

Tensile splitting strength of concrete

To measure the tensile splitting strength, at least two specimens were tested for each mix. The results are plotted in Figure 4. Similar to the paste results, the tensile splitting strength of geopolymer concrete was found to be higher than its OPC counterpart.

Tensile strength is commonly expressed in terms of the compressive strength for OPC concrete using empirical relations. Australian standard AS 3600 (SA, 2009) gives the tensile strength as:

	Density: kg/m^3	Compressive strength: MPa	Tensile strength: MPa	Modulus of elasticity: GPa	Poisson's ratio	Fracture energy: N/mm	Characteristic length: mm
Mix 1	1876	68·2	2·8	15·3	—	15·2	29
Mix 2	2031	71·2	3·3	11·2	—	9·1	10
Mix 3	2530	65·1	3·9	45·2	0·16	98·9	294
Mix 4	2555	69·8	5·0	35·5	0·19	80·1	114
Mix 5	2496	72·1	4·9	36·1	0·15	69·8	105
Mix 6	2445	77·9	5·1	41·2	0·16	65·2	103

Table 3. Properties of mixes

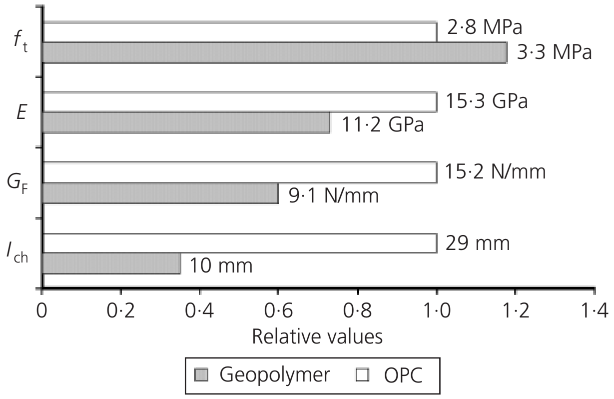

Figure 3. Comparison of properties of geopolymer and OPC pastes

6. $$f_t = 0{\cdot}4 f_c^{1/2} \text{ for } f_c \leqslant 65 \text{ MPa}$$

where f_c is the compressive strength of concrete. EN 1992-1-1 (Eurocode 2) (CEN, 2004) expresses the tensile strength f_t as

7. $$f_{t,0{\cdot}05} = 0{\cdot}7 \times 0{\cdot}3 \times f_c^{2/3} \text{ 5\% fractile,} \quad \text{for } f_c \leqslant 50 \text{ MPa}$$

and

8. $$f_{t,0{\cdot}05} = 0{\cdot}7 \times 2{\cdot}12 \times \ln[1 + (f_c/10)] \text{ 5\% fractile,} \quad \text{for } f_c > 50 \text{ MPa}$$

The characteristic tensile strength calculated using Equations 6–8 is compared with the experimental results in Figure 4.

Modulus of elasticity and Poisson's ratio

Similar to the tensile strength, the modulus of elasticity is also expressed as a function of the compressive strength. ACI 318-08 (ACI, 2008) recommends the following equation for normal-density concrete of strength greater than 21 MPa:

9. $$E = 4700(f_c)^{1/2}$$

For the modulus of elasticity of concrete for strengths up to 65 MPa, AS 3600 (SA, 2009) recommends:

10. $$E = 0{\cdot}043\rho^{3/2}(f_c)^{1/2}$$

where ρ is the density of concrete.

The measured static modulus of elasticity of concretes tested in the current and previous studies (Hardjito and Rangan, 2005) is compared to the ACI 318-08 (ACI, 2008) and AS 3600 (SA, 2009) formulas in Figure 5, which shows that the measured values of geopolymer concrete were consistently lower than the values of OPC concrete calculated using Equations 9 or 10.

The test results shown in Figure 5 demonstrate that the elastic modulus for geopolymer concrete is approximately 23% lower than that of its OPC counterpart. The modulus of elasticity is sensitive to the chosen aggregates and the testing conditions, and in these tests the aggregates and test methods were kept the same for both geopolymer and OPC concretes. Figure 5 is in agreement

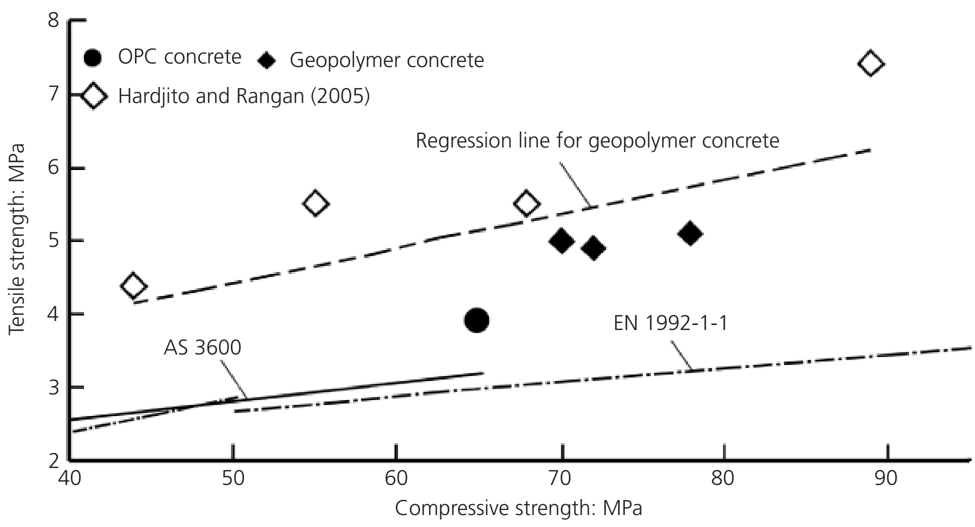

Figure 4. Comparison of tensile strength of geopolymer and OPC concretes

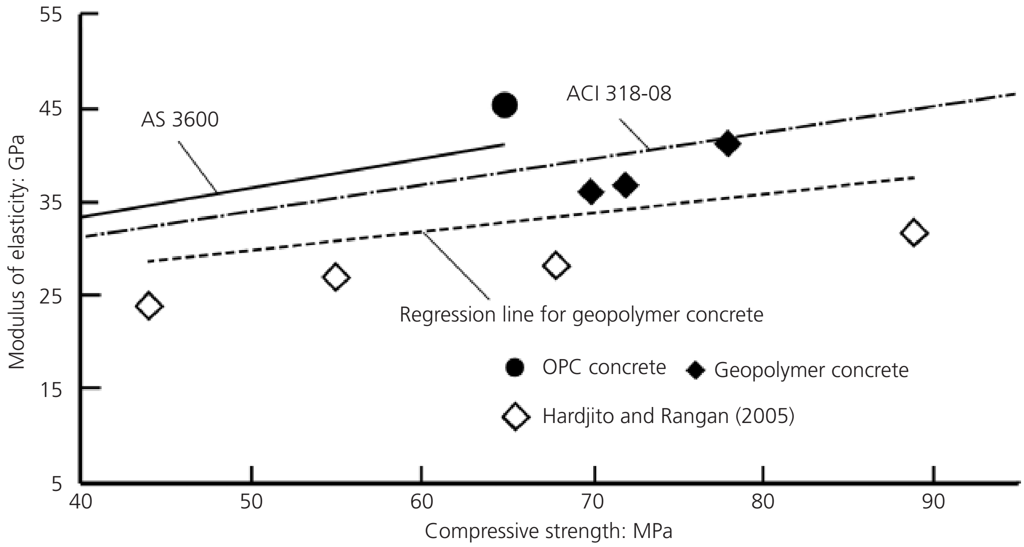

Figure 5. Comparison of modulus of elasticity of geopolymer and OPC concretes

with the data presented by Fernandez-Jimenez *et al.* (2006). For a similar strength level, Fernandez-Jimenez *et al.* (2006) found that the elastic modulus of geopolymer concrete was approximately one half of that of a comparable OPC concrete. The low modulus of elasticity of the geopolymer concrete must have resulted from the lower modulus of elasticity of the geopolymer paste as compared with the OPC paste. The values of elastic modulus presented in Figure 3 confirm this hypothesis.

Test results for Poisson's ratio measured in the test programme as well as from the literature (Hardjito and Rangan, 2005) are presented in Figure 6; the results show a mean of 0·17 and a standard deviation of 2·2%. The mean is close to the value of 0·16 measured for OPC concrete. It is obvious from Figure 6 that the Poisson's ratio of geopolymer concrete is not correlated to its compressive strength.

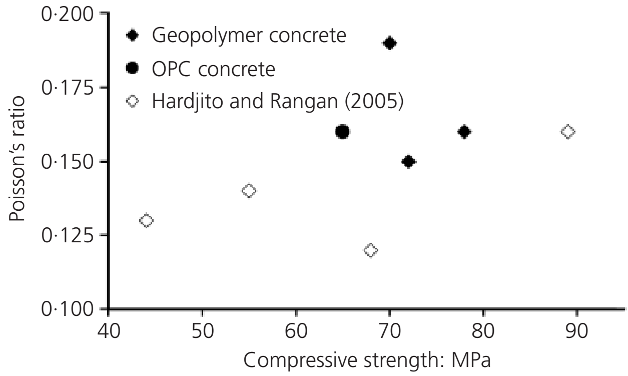

Figure 6. Comparison of Poisson's ratio of geopolymer and OPC concretes

Fracture energy

For the three-point bend test (Figure 1), four beams were tested and the average value of the measured fracture energy of the four samples was taken to ensure repeatability. The variability in the data can be seen in Figure 7; the results include error bars, showing 90% double-side confidence intervals for the mean value of G_F.

CEB-FIP (Hilsdorf and Brameshuber, 1991) recommends a simple empirical formula relating G_F to the mean compressive strength of OPC concrete:

$$11. \quad G_F = \alpha_F f_c^{0 \cdot 7}$$

in which α_F is an empirical coefficient depending on the maximum aggregate size (Table 4).

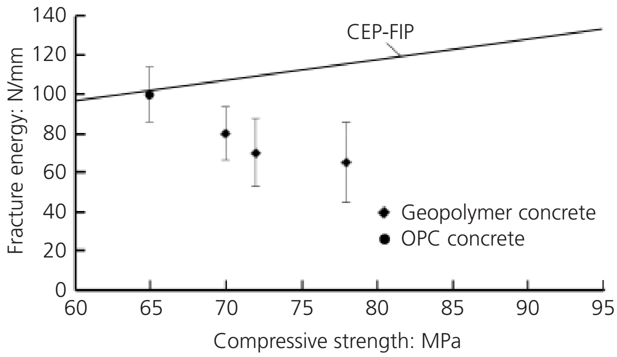

Figure 7. Comparison of fracture energy of geopolymer and OPC concretes

Maximum aggregate size, *g*: mm	α_F
8	4
16	6
32	10

Table 4. Coefficient α_F in Equation 11

Figure 7 shows that the geopolymer concrete possesses a lower fracture energy than the OPC concrete, and the measured G_F values fall below the value predicted by the CEB-FIP relationship (Equation 11). The fracture energy of concrete is generally believed to be governed by the microstructure of the paste and the size, texture and angularity of the coarse aggregates. In the current investigation, the first factor seems to be the most prominent cause of the different fracture energies of the geopolymer and OPC concretes because the other factors were kept the same for both concretes. This is further supported by the fact that the geopolymer paste exhibited a lower fracture energy than the OPC paste (Figure 3).

Struble *et al.* (1989) investigated microstructural aspects of the fracture of hardened cement paste using scanning electron microscopy (SEM). They found that cracks pass around unhydrated cement grains rather than passing through them. This provides experimental evidence to support the claim by Baldie (1985) that unhydrated cement grains behave as strong inclusions in hardened cement paste. When a crack is forced to grow around a particle, high energy is required for crack propagation in the tortuous path created by the particle's angularity. This is generally the case in OPC concrete, where concrete made with crushed river gravel was found to have a higher fracture energy than concrete made with round river gravel (Nallathambi *et al.*, 1984).

In a geopolymer paste, unreacted ash particles are also found to be embedded in the binder, and show a range of degree of bonding to the gel (Lloyd *et al.*, 2009). The morphology of fly ash is different to that of cement. As shown in Figure 8, fly ash particles are globular solids whereas cement particles are angular. It is thereby hypothesised that the morphology of the raw material might be the reason for the different G_F values of the geopolymer and OPC pastes. In the matrix, a crack will deviate from a path of less energy in order to bypass a tough unhydrated particle. Crack propagation in a geopolymer is likely to be less tortuous and therefore consumes less energy when compared with that in OPC paste.

Typical load–displacement plots of the three-point bend tests are shown in Figure 9. The area under the curve represents the work of fracture, which is proportional to G_F. The steep descending branch in the post-ultimate range indicates that geopolymer concrete is brittle.

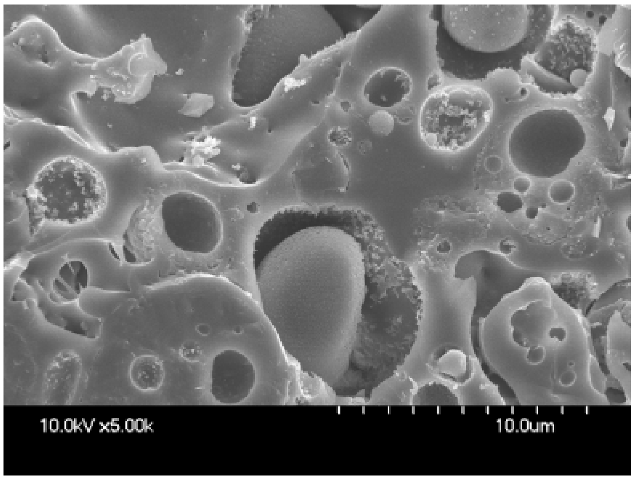

Figure 8. SEM image of geopolymer showing unreacted fly ash particles

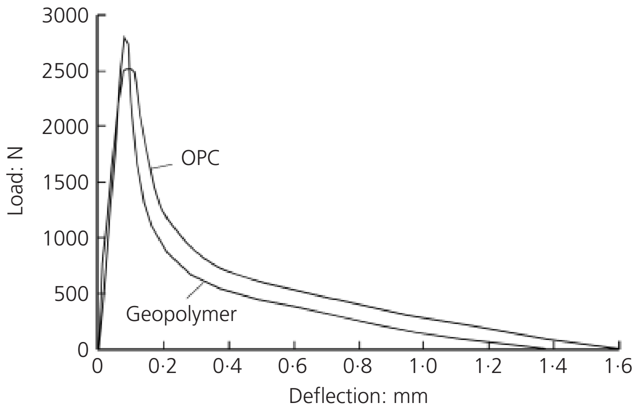

Figure 9. Typical load–deflection plot of OPC and geopolymer concrete beam specimens

Brittleness

In order to quantify the brittleness of the material, the characteristic length l_{ch} was calculated using Equation 2. The results for the concrete mixes in this investigation (Figure 10) indicate that l_{ch} of OPC is approximately three times larger than that of the geopolymer concrete.

If all the differences in properties of the geopolymer and OPC concretes are compared together (as was done for the pastes in Figure 3), the difference in brittleness between the two materials is obvious. From a chemical point of view, the intrinsic brittleness of geopolymer-based materials might be due to their highly cross-linked framework (Davidovits, 1991). In general, highly cross-linked materials are brittle in nature.

In the current investigation, geopolymer concretes were prepared

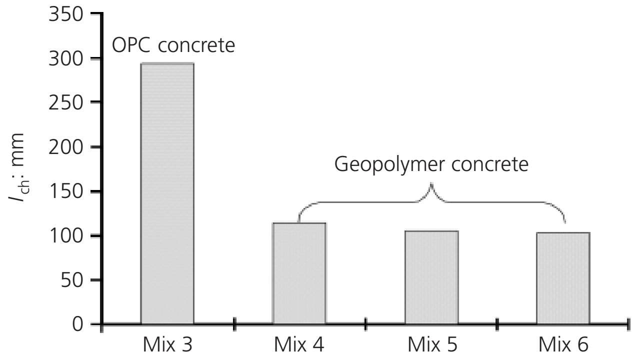

Figure 10. Characteristic length of OPC and geopolymer concretes

using alkali liquids with different soluble silicate contents. The similar compressive strengths of the geopolymer concretes were developed by adjusting the curing regimes (Table 2). The test results show that geopolymer concretes with a similar compressive strength level also exhibit a similar l_{ch}. This indicates that the brittleness of geopolymer concretes is primarily related to the compressive strength rather than the other parameters investigated in this study.

It is noteworthy that the difference in l_{ch} between geopolymer and OPC concretes is similar to that reported between high-strength and normal-strength OPC concretes (Gettu *et al.*, 1990). The care and consideration given to high-strength concrete structures in structural design with regards to brittleness should, therefore, be extended to geopolymer concretes.

Conclusions

Based on the experimental work on geopolymer and Portland cement-based paste and concrete reported here, the following conclusions are drawn.

(*a*) For a given strength level, geopolymer paste and concrete have a higher brittleness than the equivalent OPC paste and concrete.
(*b*) The differences in properties between geopolymer and OPC pastes are consistent with the differences between geopolymer and OPC concretes.
(*c*) Similarly to OPC concrete, the mechanical properties (tensile strength, modulus of elasticity and fracture energy) of geopolymer concrete can be empirically related to its compressive strength.
(*d*) The brittleness of geopolymer concrete is primarily related to its compressive strength rather than the other parameters investigated in this study.
(*e*) The fracture energy and elastic modulus of geopolymer paste and concrete are lower than those of OPC paste and concrete.
(*f*) The tensile strength of geopolymer paste and concrete is higher than that of OPC paste and concrete.

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