Strength properties of Fly Ash based Geopolymer Concrete cured at different temperatures.

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ABSTRACT: This paper presents the results of an experimental study of the effect of curing temperature on the compressive strength and split tensile strength of Geopolymer concrete (GPC) which is made using fly ash produced at Norochcholai Coal Power Plant, Sri Lanka. A relationship between splitting tensile strength and compressive strength is also developed using test data. The results are then compared with some expressions published in international literature. A mixture of Sodium Silicate and Sodium Hydroxide solutions was used to activate low calcium class F fly ash to form Geopolymers. All specimens were oven cured for 48hours where curing temperature was varied from ambient temperature to 80ºC at 20ºC intervals. One day of rest period was given to every sample before curing. Test results showed that curing temperature has a significant effect on rate of strength gaining of GPC. The output of this study has provided a better understanding of the correlation between splitting and compressive strengths of GPC, which has therefore helped to generate a new expression with better accuracy for GPC prepared using locally available fly ash.

Keywords: Geopolymer concrete, fly ash, curing temperature, oven curing, compressive strength, split tensile strength

1. INTRODUCTION

With the modern concept of sustainable development in the construction industry, attention was driven to the alternatives to Ordinary Portland Cement Concrete (OPC) in order to reduce greenhouse emissions. As a solution, Geopolymer Concrete (GPC) was introduced which is an innovative, sustainable construction material in which cement is replaced by alumina –silicate rich material like fly ash and chemically activated by alkali solutions. In Sri Lanka, a considerable amount of fly ash is produced annually from Norochcholai coal power plant as a by-product and is disposed in landfills. Therefore, this novel topic of Geopolymer gained the interest of many researchers in Sri Lanka to utilise fly ash waste to produce GPC.

Compressive strength and tensile strength are the two most common measures of the structural quality of concrete which can be related to each other, depending on the nature of the concrete. The empirical formula relating f (split) and fc' had been suggested by researchers having the formulae type;

$$f(split) = k(fc')^n \qquad (1)$$

Where *k* and *n* are constant coefficients; *f(split)* is the split tensile strength and fc' is the compressive strength of the concrete cylinder.

Different experimental values for the coefficients *k* and *n* were proposed by other researchers are indicated in Table 01.

Table 01. Coefficients *k* and *n*

Source	*k*	*n*
1.(ACI-Committee-318, 2014)	0.56	0.5
2.(ACI-Committee-363, 2005)	0.59	0.5
3.(Arioglu et al., 2006)	0.387	0.63
4.(JCI, 2008)	0.13	0.85
5.(JSCE, 2007)	0.23	0.67
6.CEB-FIB (Muller & Hilsdorf, 1991)	0.3	0.67
7.(Raphael , 1984)	0.313	0.667
8.(Ahamad & Shah, 1985)	0.462	0.55
9.(Oluokun et al., 1991)	0.294	0.69

Some studies suggest that ACI-318 coefficients underestimate the splitting tensile strength for high strength concrete and overestimate it for low strength concrete (Arioglu et al., 2006)

2. BACKGROUND OF GPC

The alumina-silicate compounds of fly ash dissolve in alkali activators and form a binder which is known as Geopolymer. The process of Geopolymerisation can be simply introduced in three steps namely dissolution of alumina–silicate under a strong alkali activator solution, reorganisation of free iron clusters and polycondensation of alumina-silicates. This process results in a three-dimensional polymeric chain and ring structure termed as "Geopolymer backbone" consisting of Si-O-Al-O (poly sialate bonds.) (Chung et al., 2013)

Geopolymers can be produced from alumina-silicate rich materials such as metakaolin, fly ash, bottom ash, Ground Granulated Blast Furnaces Slag (GGBS), silica fumes and rice husk ash which are also often referred as alkali-activated cements or inorganic polymer cements. (Duxon et al., 2007)

During combustion of ground or powder coal in an electric power generating plant, the impurities in coal are fused in suspension and float out of the combustion chamber with external exhaust gases which are then solidified after cooling down. These particles are known as fly ash and contain cementitious properties. ASTM Class C fly ash has a higher calcium percentage than ASTM class F. Since calcium interferes with the polymerisation process to alter microstructure, it will result in a reduction of strength. (Gourley, 2003)

Mechanical properties of GPC depend on various factors which include the source of alumina-silicate material, alkaline activator properties, ratio of alkaline liquid to fly ash, ratio of water to geopolymer solid ratio, curing temperature, curing time, handling time, addition of superplasticizer, rest period, water content of mixture, method of curing and the age of concrete. (Hardjito & Rangan, 2005)

Activation solution provides the required alkalinity which is usually preferred to be a combination of NaOH and Na_2SiO_3 due to its low cost, high availability, and high efficiency. The NaOH has the ability to extract alumina- silicates from the source material (Van Jaarsveld et al., 2003)

whereas Na_2SiO_3 improves the rate of polymerisation. Abdulla investigated the effect of the Na_2SiO_3/NaOH ratio on the compressive strength of GPC. (Abdulla et al., 2012) In his study, various Na_2SiO_3/NaOH ratios (0.5, 1.0, 1.5, 2.0, 2.5, and 3) were used to determine the highest compressive strength while keeping the molarity of NaOH at a constant of 10M. His study suggests that the highest strength is achieved when the ratio is 2.5. In addition, the strength of GPC highly depends on the concentration of NaOH. Many researchers have found that the strength is increased with the increasing concentration of NaOH. (Chung et al., 2013), (Budh & Warhade, 2014). (Shivaji & Hamane, 2015)

Higher water to geopolymer solid ratio results in low strength but high workability. This is the ratio of the total mass of water in the mixture (sum of water in NaOH solution, Na_2SiO_3 solution, and extra water added) to total Geopolymer solid mass (the sum of fly ash mass, NaOH solids mass, Na_2SiO_3 solid mass). (Lioyd & Rangan, 2010). D. Hardjito studied the effect of activator to fly ash by mass ratio on compressive strength and observed that the optimum ratio is 0.4. (Hardjito et al., 2004)

OPC shows a higher strength at lower temperatures but not in elevated temperatures above 400°C (Shaikh & Vimonsatit, 2014) whereas GPC is more stable at higher temperatures. At ambient temperature, the reaction of fly ash is extremely slow (Puerats et al., 2000) and it results in low compressive strength. Higher strengths can be achieved by oven curing when compared to wet curing at similar temperatures. (Junaid et al., 2015). The rest period is simply the interval between the end of casting and the start of oven curing of the samples.

3. EXPERIMENTAL PROGRAM

3.1 Materials used

In this research ASTM class F fly ash which was produced at Norochcholai coal power plant, Puttalam was obtained from Holcim Lanka (Pvt) Ltd, as the alumina-silicate material. Table 02 shows the results of the X-ray Fluorescence (XRF) analysis of fly ash. The fineness of fly ash was obtained as 85% passing through a 45 µm sieve and specific gravity was measured as 2.14.

Table 02: X-ray Fluorescence (XRF) analysis of fly ash

Chemical Properties	Percentage (%)
Loss on ignition	4.4
CaO	7.8
SO_3	0
$SiO_2+Al_2O_3+Fe_2O_3$	83.9
Total alkalis	1.9
MgO	1.8
Cl-	0

Local aggregates, comprising a maximum aggregate size of 14mm coarse aggregates and fine aggregates, in saturated surface dry conditions, were used. The coarse aggregates were crushed granite-type aggregates and the fine aggregates were fine river sand. The particle size distribution of fly ash was analysed using a sieve analysis test in the laboratory.

The chemical composition of the industrial-grade 'D' sodium silicate was given by the supplier as $Na_2O=17.2\%$ by mass, $SiO_2=34.4\%$ by mass, and water 48.4% by mass. The specific gravity of sodium silicate was measured as 1.65. The sodium hydroxide (NaOH) pellets were with 97%-98% purity.

3.2 Mix Design Procedure

In Junaid's study, the mix design procedure of GPC is described in a few steps which is based on already developed G-Graphs. (Junaid et al., 2015). The proposed method is based on comparing the strength, workability, and cost as same as OPC mix designs. Initially, target 7 days' average compressive strength is set and then located on the relevant G-graph developed for particular molarity of NaOH, curing temperature and curing duration to obtain values for activator/Fly Ash ratio and Water/Geopolymer solid ratio. The proposed values for different parameters obtain from reviewing the literature are detailed in Table 03.

In this study, the amount of fly ash was assumed as 370kg/m³.This value should lie between 360kg/m³ and 380kg/m³ for a strength requirement between 25MPa and 40MPa. The density of GPC was assumed as 2300kg/m³. Mix proportions were obtained from the equations 2, 3 and 4. The results obtained are tabulated in Table 04.

Table 03: Proposed values for different parameters

Parameter	Proposed values
Activator/fly ash	0.4
Water/geo-polymer solids	0.25
Sodium Silicate/Sodium Hydroxide	2.5
Molarity of NaOH	12M
Rest period	1day
Curing temperature	28°C(ambient), 40°C, 60°C, 80°C
Duration of curing	48 hours

$$\frac{Na_2SiO_3(aq)+NaOH(aq)}{FA} = 0.4 \qquad (2)$$

$$\frac{Na_2SiO_3(aq)}{NaOH(aq)} = 2.5 \qquad (3)$$

$$\frac{W_{free}+W_{OH}+W_{Si}}{FA+Solids_{OH}+Solids_{Si}} = 0.25 \qquad (4)$$

Where, W_{free}= Free water amount in the system; W_{OH} = Water content in hydroxide; W_{Si} = Water content in silicate solution; $Solids_{OH}$ = Solid content in hydroxide solution; $Solids_{Si}$ = Solid content in silicate solution.; FA = Fly ash content in the mix.

Table 04: Mix proportions used in the study

Material	Amount (kg/m³)
NaOH	42.3
Na_2SiO_3	105.7
Fly ash	370
Coarse aggregates (14mm)	876
Fine aggregates (Sand)	584
Free water	22

3.3 Specimen Casting and Testing

Initially, the 480g of NaOH pellets were dissolved in one litre of water to achieve 12M which was then mixed with Na_2SiO_3 to produce an alkali activator. This solution was mixed 24 hours prior to casting. Then fly ash, fine aggregates, and coarse aggregates were mixed together in a mechanical mixture for few minutes before mixing it with the alkaline activator. Once a homogeneous mix was achieved, fresh concrete was cast into moulds. Concrete cubes of 100mm *100mm *100mm were casted to perform compression strength test and cylinders of 100mm diameter and 200mm height were casted to perform split tensile strength test.

In this research, a set of samples were cured at ambient temperature and others were oven cured for 40^oC, 60^oC, and 80^oC for 48 hours after one day of a rest period. Samples were wrapped in Aluminum foil before being placed in the oven as in Figure 1. After the curing period, all test samples were placed at the ambient temperature until the testing were carried out.

Figure 1. Oven curing of samples

Cubes were used to determine the compressive strength of GPC with a stress control loading at 0.5MPa per second as in Figure 2. To determine the split tensile strength on GPC cylinders, the load is applied diametrically and uniformly along the length of the cylinder. To allow the uniform distribution of this applied load and to reduce the magnitude of the high compressive stresses near the points of application of this load, strips of hard cardboard were pasted along with the specimen as in Figure 3.

Figure 2: Compression strength testing

Figure 3: Split tensile strength testing

Assuming concrete specimen behaves as an elastic body, a uniform lateral tensile stress of *f (split)* acting along the vertical plane causes the failure of the specimen, which can be calculated from the equation 5. (ASTM C496/C496M-11, 2011),

$$F(split) = 2P/\pi DL \qquad (5)$$

Where, *P*=Compressive load at failure; *L*=length of the cylinder; *D* =Diameter of the cylinder

4. RESULTS AND DISCUSSION

The results of the compressive strength of cubes f(cu) cured at different temperature are tabulated in Table 05 and Table 06 at 7-day and 28-day respectively. For each and every curing temperature, 3 specimens have been tested. Equal cylindrical strength (fc') is derived using 0.8 multiplying factor as suggested by BS Standards. (BS1881-part 116, 1989).

Table 05. Compressive strength results at 7-day

| Curing Temperature (°C) | Sample Number | 7day | | | |
		Weight (g)	Compressive Strength ,fc (MPa)	Cylinder strength, fc' (MPa)	Average Strength (MPa)
28	C7-1-28	2359.9	21.28	17.02	16.36
	C7-2-28	2357.6	19.63	15.70	
	C7-3-28	2358.9	20.45	16.36	
40	C7-1-40	2367.4	34.2	27.36	24.43
	C7-2-40	2311.2	24.54	19.63	
	C7-3-40	2355.3	32.88	26.30	
60	C7-1-60	2274.7	58.11	46.49	54.87
	C7-2-60	2307.3	78.64	62.91	
	C7-3-60	2296.4	69.02	55.22	
80	C7-1-80	2286.7	66.84	53.47	48.04
	C7-2-80	2248.5	54.75	43.80	
	C7-3-80	2264.9	58.56	46.85	

Table 6. Compressive Strength results at 28 day

| Curing Temperature (°C) | Sample Number | 28day | | | |
		Weight (g)	Compressive Strength,fc (MPa)	Cylinder Strength,fc' (MPa)	Average Strength (MPa)
28	C28-1-28	2346.5	37.09	34.72	31.21
	C28-2-28	2334.6	43.4	29.25	
	C28-3-28	2312.7	36.56	29.82	
40	C28-1-40	2238.6	37.28	28.81	32.29
	C28-2-40	2263.9	36.01	38.24	
	C28-3-40	2341.9	47.8	52.34	
60	C28-1-60	2264	65.43	56.82	57.34
	C28-2-60	2297.6	71.02	62.85	
	C28-3-60	2303.5	78.56	50.25	
80	C28-1-80	2292.8	62.81	47.90	48.90
	C28-2-80	2288.3	59.87	48.54	
	C28-3-80	2297.1	60.68	34.72	

Figure 4 depicts the variation of cylindrical strength fc' with the curing temperature. This shows that curing temperature has a significant effect on both 7-day and 28-day compressive strength of GPC. The optimum curing temperature of fly ash-based GPC is 60°C and there is no strength gain after the optimum temperature.
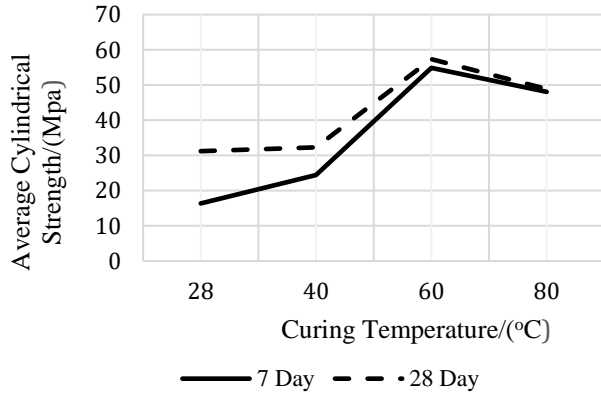
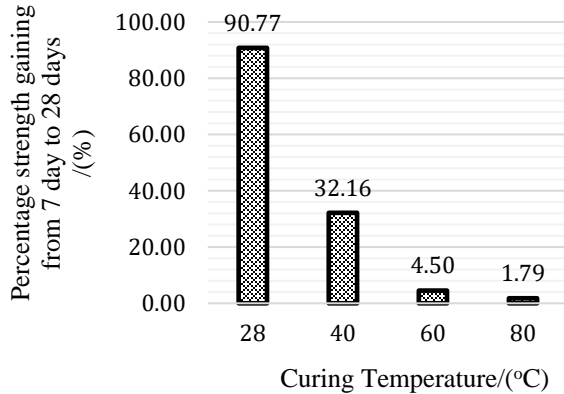Figure 4. Cylindrical Compressive strength for 7 days and 28 days at different curing temperatures

Figure 5. Compressive strength gaining percentage from 7day to 28 day with curing temperature.

Figure 5 shows that the percentage strength gain from 7 days to 28 days has drastically decreased with the curing temperature. Even though the compressive strength achievement is low at the ambient temperature, the percentage of strength gain from 7 days to 28 days is almost double as 7-day compressive strength. This may be due to the low rate of Geo-polymerization process at early stages of strength gaining at ambient temperature.

At 80°C the percentage strength gain from 7 days to 28 days has become negligible. This indicates that at higher curing temperatures, GPC achieves its target strength at early ages whereas at ambient temperature strength gaining rate is low at early ages.

Table 7. Split tensile strength results at 7day

Curing Temperature /(°C)	Samples	Weight/ (g)	7 day Split Tensile Strength /(MPa)	Average Strength/ (MPa)
28	Cy7-1-28	3774.7	1.34	1.31
	Cy7-2-28	3741.9	1.27	
	Cy7-3-28	3754.7	1.31	
40	Cy7-1-40	3809.4	2.61	2.24
	Cy7-2-40	3737.2	1.77	
	Cy7-3-40	3789.4	2.35	
60	Cy7-1-60	3702.7	4.31	3.98
	Cy7-2-60	3699.2	3.85	
	Cy7-3-60	3696.5	3.79	
80	Cy7-1-80	3710.8	4.09	3.54
	Cy7-2-80	3699.6	2.97	
	Cy7-3-80	3704.4	3.56	

The figure 6 shows the relationship between cylindrical strength and derived splitting tensile strength from different *k, n* values given in the references mentioned in Table 01. Relationship between split tensile strength and cylindrical strength values from the experimental data is also included to compare the results.

According to Figure 06, experimental values do not match with any of the equations given in the literature. Therefore, correlation between the split tensile strength *f (split)* and compressive strength of GPC is analysed and the empirical relation can be expressed as:
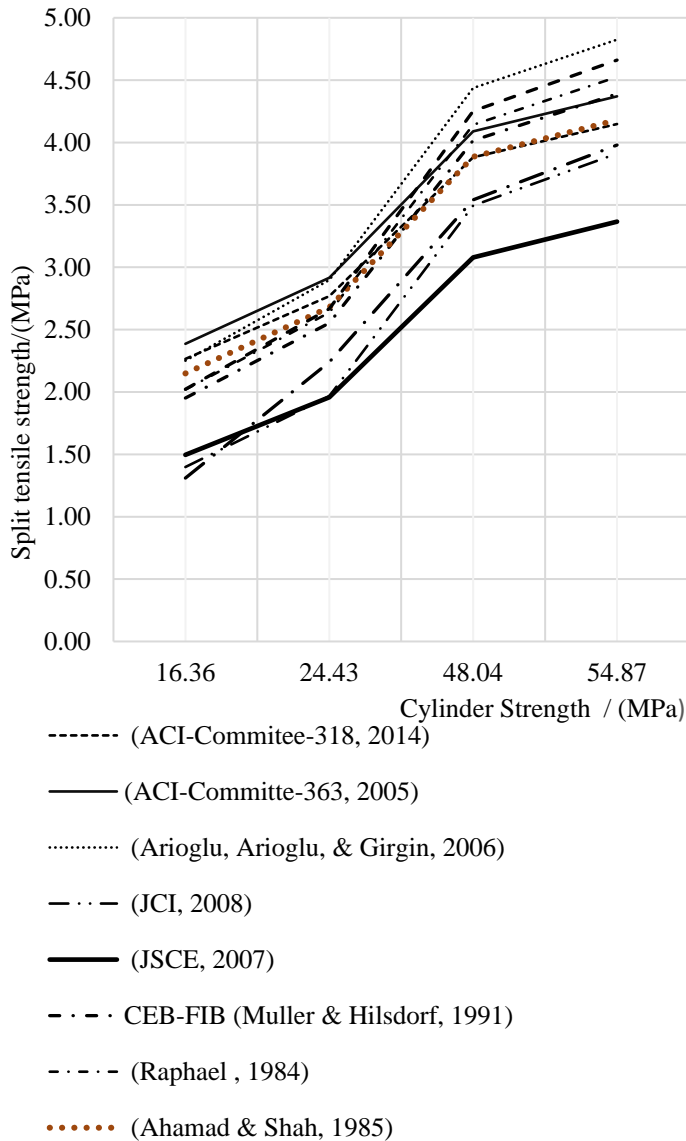
$$f\ (split) = 0.27(fc')^{0.67} \qquad (6)$$

Figure 06. Comparison of experimental and theoretical Split tensile strength to compressive strength ratio

Legend:
- ------- (ACI-Commitee-318, 2014)
- ——— (ACI-Committe-363, 2005)
- ············ (Arioglu, Arioglu, & Girgin, 2006)
- — ·· — (JCI, 2008)
- ——— (JSCE, 2007)
- — · — · CEB-FIB (Muller & Hilsdorf, 1991)
- — · — · — (Raphael , 1984)
- ••••• (Ahamad & Shah, 1985)

5.CONCLUSION

1. The GPC attains its target strength much faster under heat cured condition compared to ambient cured condition.

2. The optimum curing temperature of fly ash-based GPC is 60°C and there is no strength gain after the optimum curing temperature.

3. At ambient curing temperature, the strength gain percentage from 7 days and 28 days is considerably high and as curing temperature increases, it has reduced drastically.

4. Equations given in the literature to estimate splitting tensile strength from compressive strength were not accurate for GPC concrete according to the study. The relationship between the compressive and splitting tensile strength of GPCs mixes was found to be :

$$f(split) = 0.27(fc')^{0.67}$$

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