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# Geopolymer concrete for ambient curing condition

P. Nath\*, P. K. Sarker\*

\* *Department of Civil Engineering, Curtin University, Kent Street, Bentley, WA, 6102, Australia  
(E-mail: pradip.nath@postgrad.curtin.edu.au; p.sarker@curtin.edu.au)*

## ABSTRACT

Recent studies on the various properties of heat-cured geopolymer concrete have shown its suitability for applications such as precast concrete members. Development of geopolymer concrete suitable for curing at ambient temperature will widen its application to concrete structures. This study aimed to achieve fly ash-based geopolymer concretes suitable for ambient curing condition. Class F fly ash was used as the base material which was reacted by sodium hydroxide and sodium silicate solutions. Ground blast furnace slag was added in different proportions to the mix to enhance the early age properties of concrete. Setting times of geopolymer pastes, workability of fresh concrete and compressive strength after curing at 20-23°C were investigated. Results were compared for the varying proportions of slag and alkaline activator in the mix. Inclusion of slag reduced the setting time to values comparable to that of ordinary Portland cement paste and improved the early-age compressive strength significantly. Setting time and compressive strength of geopolymers varied with the variation of alkaline activator to fly ash ratio and sodium silicate to sodium hydroxide ratio in the alkaline activator solution. Generally, fly ash and slag blend has improved the early-age mechanical properties of geopolymer concrete cured at ambient condition.

## KEYWORDS

Ambient curing; Fly ash; Geopolymer; Setting time.

## INTRODUCTION

The increasing demand of environment friendly construction has been the driving force for developing sustainable and economical building materials. The critical aspects influencing the development are performance of the materials under different and special user conditions, economic aspects as well as environmental impact aspects. Cement is an energy consuming and high green house gas emitting product. Geopolymers are gaining increased interest as binders with low CO<sub>2</sub>-emission in comparison to Portland cement. Geopolymers also exhibit similar or superior engineering properties compared to cement. Low-calcium fly ash-based geopolymer concrete has been reported to have excellent compressive strength, resistance to sulphate attack, and good acid resistance (Wallah and Rangan, 2006).

Geopolymer is an inorganic aluminosilicate polymer synthesized from alkaline activation of various aluminosilicate materials of geological origin or by-product materials like fly ash, metakaolin, blast furnace slag etc (Davidovits, 2008). The polymerisation process involves a substantially fast chemical reaction of aluminosilicate minerals under alkaline condition that results in a three dimensional polymeric chain. Three basic steps lead the chemical reaction: 1. dissolution of Si and Al atoms from the source material, 2. Orientation or condensation of precursor ions into monomers, and 3. setting or polycondensation of monomers into polymeric structures (Duxson *et al.* 2007). The final products of geopolymerisation are influenced by many factors regarding chemical composition of the source materials and alkaline activators (Diaz *et al.* 2010; Duxson *et al.* 2007; Yip *et al.* 2008).

The polymerisation process is generally accelerated in the higher temperature than ambient. Fly ash based geopolymers produced in ambient temperature achieve lower strength in the early days as compared to heat cured samples (Vijai *et al.* 2010). In this respect, the beneficial effect of calcium content in the source materials can be noted. The amount of calcium oxide (CaO) content of the fly ash precursor was found to have significant impact on the resulting hardened geopolymer. The additional CaO forms hydrated products such as calcium silicate hydrates (CSH), along with the alumino-silicate geopolymer network (Granizo *et al.* 2002; Yip *et al.* 2008). Increase in strength and decrease in setting time was observed with the increase of CaO content (Diaz *et al.* 2010). Properties of calcium sources and the alkalinity of the activating solution also influences the geopolymerisation process (Yip *et al.* 2008). Recently, the suitability of fly ash based geopolymers mixed with silica fume, metakaolin (Wu and Sun, 2010) and blast furnace slag (Guerrieri and Sanjayan, 2010) has been studied by several investigators. However, most of the findings are reported for mortar and paste samples initially cured in temperature higher than ambient for variable lengths of time.

Geopolymer concrete produced without using elevated heat for curing will widen its application to the areas beyond precast members. Hence this study aimed to produce geopolymer concrete suitable for ambient curing condition. Ground blast furnace slag was mixed with low calcium fly ash to study the setting time, workability and the strength properties of geopolymer concrete.

## EXPERIMENTAL PROGRAM

### Materials

All geopolymer concretes were made with Class F fly ash (ASTM C 618). This fly ash was obtained from a Western Australian power plant. Commercially available ground blast furnace slag (GBFS) was used along with fly ash as a binder. The chemical compositions of fly ash and GBFS are shown in Table 1. The aluminosilicate binders were activated by a mixture of sodium hydroxide and sodium silicate solutions. Sodium hydroxide solution with desired concentration was prepared by mixing 97-98% pure pellets with tap water. Sodium silicate solution with SiO<sub>2</sub> to Na<sub>2</sub>O ratio by mass of 2.69 (SiO<sub>2</sub> = 30.7%, Na<sub>2</sub>O = 11.4% and water = 57.9%) was used. The fine aggregate used was natural sand having a nominal maximum size of 1.18 mm. Coarse aggregates were crushed granite with nominal maximum size of 7, 10 and 20 mm that met the Australian standard (AS 2758.1-1998) specifications. Physical properties and size distribution of aggregates are presented in Table 2.

### Manufacture of geopolymer concrete

*Mix proportioning.* The concrete mixtures were proportioned based on the previous works on fly ash based geopolymer concrete (Hardjito, 2005). Mix variables include the amounts of alkaline activator solution, the amount of fly ash replacement with slag and ratio of sodium silicate to sodium hydroxide solution (SS/SH). Fly ash was replaced by GBFS in the range of 10% to 30% of total binder. The SS/SH ratio was varied as 1.5, 2.0 and 2.5 and the concentration of sodium hydroxide solution was constant (14M) for every mixture. No extra water or superplasticiser was added.

The mixture proportions of eight mixes studied are shown in Table 3. Mix GC1, GC2 and GC3 were designed with varying alkaline activator content as 35%, 40% and 45% of total binder respectively. Each of these mixes had 10% slag content of the total binder and a constant SS/SH ratio of 2.5. Mix GC4, GC2, GC5 and GC6 were proportioned with fly ash replacing with slag as 0%, 10%, 20% and 30% respectively. In these mixes 40% alkaline activator was used with a

constant SS/SH ratio of 2.5. Another two mixes GC7 and GC8 were designed by varying SS/SH ratio as 1.5 and 2 respectively, while slag content and activator content were kept constant as 10% and 40% of the total binder respectively. The geopolymer mixtures were designated with their variable constituents in the mix. For example, A40 S10 R2.5 represents a geopolymer mixture having alkaline activator solution (A) as 40% of total binder, slag (S) as 10% of total binder and the ratio of sodium silicate and sodium hydroxide solution (R) as 2.5.

Setting time of geopolymer concrete mixes was tested on corresponding pastes. The mix proportions of the pastes are similar to those of the concrete mixes with the aggregates excluded.

**Table 1:** Chemical composition of fly ash and GBFS.

| Sample      | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | Fe <sub>2</sub> O <sub>3</sub> | CaO  | MgO  | Na <sub>2</sub> O | K <sub>2</sub> O | SO <sub>3</sub> | P <sub>2</sub> O <sub>5</sub> | TiO <sub>2</sub> | LOI <sup>a</sup> |
|-------------|------------------|--------------------------------|--------------------------------|------|------|-------------------|------------------|-----------------|-------------------------------|------------------|------------------|
| Fly ash (%) | 50.00            | 28.25                          | 13.5                           | 1.79 | 0.89 | 0.32              | 0.46             | 0.38            | 0.98                          | 1.54             | 0.64             |
| GBFS (%)    | 32.46            | 14.3                           | 0.61                           | 43.1 | 3.94 | 0.24              | 0.33             | 4.58            | 0.02                          | 0.55             | 0.09             |

<sup>a</sup>Loss on ignition

**Table 2:** Properties of aggregates.

| Properties             | Coarse aggregate       |                 | Fine aggregate         |                 |
|------------------------|------------------------|-----------------|------------------------|-----------------|
|                        | Sieve (mm)             | Percent passing | Sieve (mm)             | Percent passing |
| Sieve analysis         | 19.00                  | 100.0           | 4.75                   | 100.0           |
|                        | 9.50                   | 50.0            | 2.36                   | 100.0           |
|                        | 4.75                   | 8.9             | 1.18                   | 99.7            |
|                        |                        |                 | 0.60                   | 77.2            |
|                        |                        |                 | 0.30                   | 24.2            |
|                        |                        |                 | 0.15                   | 2.7             |
|                        |                        |                 |                        |                 |
| Fineness modulus       | 6.40                   |                 | 1.96                   |                 |
| Specific gravity (SSD) | 2.67                   |                 | 2.62                   |                 |
| Absorption             | 0.40 %                 |                 | 0.99 %                 |                 |
| Unit weight            | 1645 kg/m <sup>3</sup> |                 | 1687 kg/m <sup>3</sup> |                 |

*Mixing and curing:* The alkaline activator was prepared in the laboratory by mixing the sodium hydroxide solution with sodium silicate solution about 30 minutes before actual concrete mixing to enhance reactivity of the solution. Concrete ingredients were mixed in a laboratory pan mixture. Aggregates, prepared in saturated surface dry condition, and the binders (fly ash and slag) were dry mixed thoroughly in the mixer. Premixed alkaline activator solution was then added gradually in the mixer. Mixing was continued for further 4-6 minutes depending on the consistency of the mixture. Cylindrical concrete moulds were filled with geopolymer concrete mixture in two layers and compacted on a vibrating table. The moulds were then cured in a controlled temperature of 20-23°C. Samples were demoulded 24 hours after casting and then left in 20-23°C for air curing until testing.

## Experiments

The workability of fresh geopolymer concrete mixtures was tested by slump test (AS 1012.3.1-1998). Compressive strength test was conducted at 3, 7 and 28 days. Cylinder specimens, 100 mm

in diameter and 200 mm in height, were tested at a loading rate of 0.33 MPa/s with a Controls MCC8 machine.

Setting time of geopolymer pastes was tested as per ASTM C 191-08. The paste was prepared by mixing the binders and the alkaline solutions manually in a bowl and cast into the conical mould of Vicat apparatus. Penetration of Vicat needle (1.00 ± 0.05 mm in diameter) in the paste was measured at regular intervals. The time for 25 mm penetration was determined by interpolation, which represents the initial setting time. The final setting time of the paste was also recorded when the needle left negligible mark on the paste surface. The test was conducted in a temperature of 21-23°C.

**Table 3:** Details of geopolymer concrete mix proportions (kg/m<sup>3</sup>).

| Mix no. | Designation  | CA <sup>a</sup> | Sand | Fly ash | GBFS | SS <sup>b</sup> | SH <sup>c</sup> | Na <sub>2</sub> O /SiO <sub>2</sub> | H <sub>2</sub> O /Na <sub>2</sub> O | Si/Al | w/s <sup>d</sup> |
|---------|--------------|-----------------|------|---------|------|-----------------|-----------------|-------------------------------------|-------------------------------------|-------|------------------|
| GC1     | A35 S10 R2.5 | 1209            | 651  | 360     | 40   | 100             | 40              | 0.11                                | 11.67                               | 1.77  | 0.18             |
| GC2     | A40 S10 R2.5 | 1209            | 651  | 360     | 40   | 114.3           | 45.7            | 0.12                                | 11.75                               | 1.80  | 0.20             |
| GC3     | A45 S10 R2.5 | 1209            | 651  | 360     | 40   | 128.5           | 51.5            | 0.13                                | 11.80                               | 1.84  | 0.22             |
| GC4     | A40 S00 R2.5 | 1209            | 651  | 400     | 0    | 114.3           | 45.7            | 0.12                                | 11.73                               | 1.77  | 0.20             |
| GC5     | A40 S20 R2.5 | 1209            | 651  | 320     | 80   | 114.3           | 45.7            | 0.13                                | 11.76                               | 1.84  | 0.20             |
| GC6     | A40 S30 R2.5 | 1209            | 651  | 280     | 120  | 114.3           | 45.7            | 0.13                                | 11.77                               | 1.89  | 0.20             |
| GC7     | A40 S10 R1.5 | 1209            | 651  | 360     | 40   | 96              | 64              | 0.14                                | 10.63                               | 1.76  | 0.20             |
| GC8     | A40 S10 R2.0 | 1209            | 651  | 360     | 40   | 106.7           | 53.3            | 0.13                                | 11.25                               | 1.78  | 0.20             |

<sup>a</sup>Coarse aggregate; <sup>b</sup>Sodium silicate solution; <sup>c</sup>Sodium hydroxide solution; <sup>d</sup>Water to solid ratio.

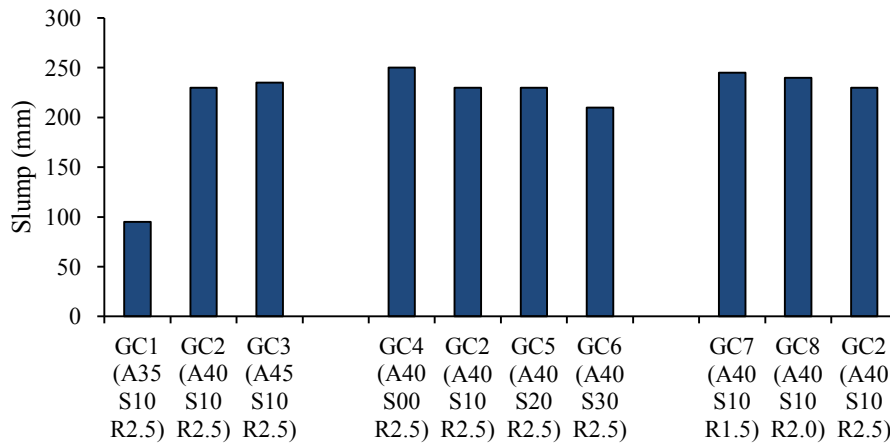
## RESULTS AND DISCUSSION

A total of eight mixtures were designed to study the effect of various parameters on the compressive strength and setting time of geopolymer concrete at ambient temperature. The results in the following figures are plotted in three groups: first groups (GC1, GC2 and GC3) represents the effect of the ratio of alkaline activator solution to binder content, second group (GC4, GC2, GC5 and GC6) represents the effect of slag content and third group (GC7, GC8 and GC2) represents the effect of the SS/SH ratio in the mix.

### Workability of fresh concrete

Slump test was conducted immediately after mixing the geopolymer concrete. Figure 1 shows the average slump values of different mixtures. Generally concrete mixtures had ‘collapse’ slump due to its sticky and viscous nature in fresh state. Although no superplasticiser or extra water was added, every mix with 40% or higher activator solution slumped more than 200 mm. Water content is an important parameter that affects workability. All the mixtures in this study have a H<sub>2</sub>O/Na<sub>2</sub>O ratio in the range of 10-14, which is observed as workable geopolymer concrete by Hardjito (2005). However, slump value may be affected by some other factors such as moisture content of aggregates, variation of ambient temperature, mixing time and degree of condensation reaction between binder and alkaline solution.

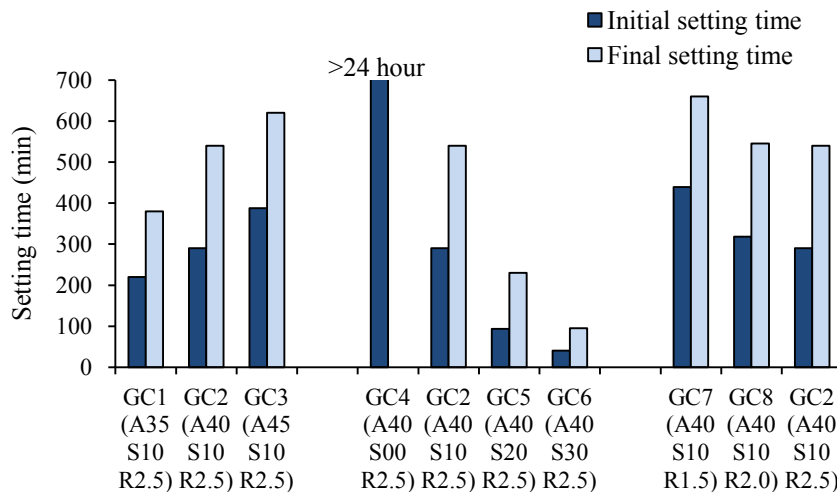
Generally, slump increased with the increase of activator solution content in the mix. Slump was influenced by the presence of slag in addition to fly ash. As the content of slag increased the slump value decreased to some extent. Geopolymer mix GC4 that contains no slag in the mixture slumped the most. The consistency of geopolymer concrete also varied due to the variation of SS/SH ratio in the mix. With the increase of SS/SH ratio from 1.5 to 2.5, slump value decreased slightly.



**Figure 1.** Effect of different parameters on the slump of the geopolymer mixtures.

### Setting time of geopolymer pastes

Figure 2 depicts the variation of setting time due to variation of different parameters. The setting time tests were carried out in a controlled temperature of 21-23°C. In this condition, fly ash based geopolymer generally takes a long time to set due to slow rate of chemical reaction at low temperature. In this study, mix GC4 which was designed with only fly ash as a binder required more than 24 hours before showing any sign of setting. Setting time of geopolymer pastes improved significantly when slag was incorporated in the mix as a binder. Both initial and final setting time decreased with the increase of slag content. Mix GC2 having 10% slag of total binder achieved initial setting time of 290 minutes, which decreased to 94 and 41 minutes for inclusion of 20% and 30% slag in mix GC5 and GC6 respectively. The difference between initial and final setting time also reduced with the increase of slag content in the paste. It indicates that the higher the slag content in the paste the quicker is the rate of setting. The results establish that slag as a part of fly ash binder is effective to accelerate setting time of geopolymer concrete in ambient condition.



**Figure 2.** Effect of different parameters on the setting time of the geopolymer pastes.

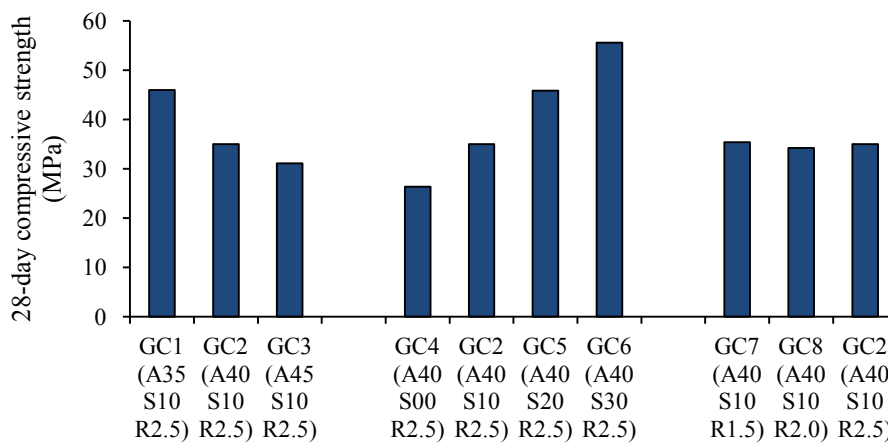
Setting time is also influenced by the alkaline activator solution to binder ratio as well as SS/SH ratio of the alkaline solution as shown in Figure 2. Geopolymer pastes required longer time to set when alkaline solution content is higher in the mix. Mix GC1, GC2 and GC3, which have 10% slag, showed an increasing trend of setting time for 35%, 40% and 45% alkaline liquid content respectively. Mix GC2, having 5% more alkaline liquid than mix GC1, required 70 minutes more

for initial setting and mix GC3 required 98 minutes more than mix GC2 for 5% increase of alkaline liquid. Increasing alkaline liquid content caused higher w/s ratio and decelerated of the condensation process of geopolymerisation.

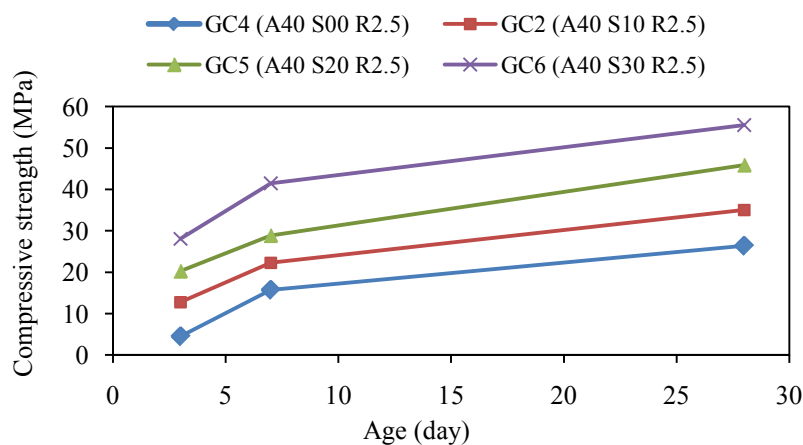
The effect of SS/SH ratio on setting time was studied by paste mix GC7, GC8 and GC2. Increasing SS/SH ratio of alkaline solution with constant amount of alkaline activator solution and slag (40% and 10% of total binder respectively) decreased setting time. Mix GC7 with least amount of sodium silicate (SS/SH ratio of 1.5) took longer time to set compared to GC8 and GC2. This is because of the reduced value of Si/Al ratio which indicates the proportion of Si present in the mix. As the amount of Si decreases in the paste, polymerisation process slow down to some extent.

### Compressive strength of geopolymer concrete

Compressive strength development of different geopolymer concrete mixtures was determined up to 28 days of ambient curing (23°C). Figure 3 shows the 28-day compressive strength variation of concretes due to the variation of mix parameters. The results shown in the following figures are the average value of three specimens tested at the test age.



**Figure 3.** Variation of 28-day compressive strength of the geopolymer concretes.



**Figure 4.** Development of compressive strength of the geopolymer concretes having different percentage of GBFS.

Geopolymer concrete with fly ash only as a binder (mix GC4), when cured in ambient condition (20-23°C), reacted slowly to develop strength. When GBFS was incorporated in the mixture as a part of total binder with constant alkaline activator (40%) and SS/SH ratio of 2.5, the strength increased significantly. As shown in Figure 4, the strength increased from the early age of 3 days with the increase of slag content in the concretes. At 28 days, mix GC2, GC5 and GC6, having 10%, 20% and 30% fly ash replaced with GBFS respectively, achieved 33%, 74% and 110% higher strength as compared to strength of control geopolymer mix GC4 (no slag) respectively. In other words, 28-day compressive strength increased up to 10 MPa for every 10% increment of slag content from control concrete. The improvement of strength of fly ash and slag blended geopolymer concrete is probably due to the increase of calcium bearing compound in the dissolved binder which produced reaction product from both alkali activated fly ash and slag. However higher Si/Al ratio of slag incorporated mixes also contributed to harden fast and develop strength.

Compressive strength was influenced by the amount of alkaline activator in the mixture. Comparing the results of mix GC1, GC2 and GC3 it can be seen that, increasing the activator content from 35% to up to 45% of total binder reduced the strength. Though the ratio of Si/Al increases for higher alkali activator content with respect to constant total binder, it is the ratio of water to solid (w/s) that affects the strength. Strength decreased when w/s ratio increased, i.e. water content increased.

The variation of compressive strength was not significant when ratio of SS/SH was varied with constant alkaline activator (40% of total binder) and slag content (10% of total binder) in the mixture. However, the mix with SS/SH ratio of 1.5 (GC7) achieved slightly higher 28-day strength than the mixes with higher SS/SH ratio of 2 and 2.5. Vargas *et al.* (2011) found increased strength with fly ash based geopolymer paste with the increase of Na<sub>2</sub>O/Si<sub>2</sub>O ratio in the mix. The findings resemble the current study, however, the mixes in this study contains 10% slag along with 90% fly ash which might have affected the behaviour of the geopolymer binder.

## CONCLUSION

Eight geopolymer concrete mixtures were designed with fly ash and slag as the binder source materials. Setting time, workability and compressive strength of geopolymers cured in ambient temperature were compared for the variation of slag content, total alkaline activator solution content and ratio of sodium silicate to sodium hydroxide in the solution. The results can be summarised as follows:

- Inclusion of slag in the fly ash based geopolymer mixture decreased the setting time and increased the compressive strength. Adding slag up to 30% of the total binder achieved compressive strength up to 55 MPa at 28 days. Setting time reduced rapidly with the increasing amount of slag in the mixture. Slump of fresh concrete decreased slightly as slag content increased.
- With the increase of alkaline activator solution in the mix from 35% to 45% of total binder, the setting time increased and compressive strength decreased. Slump of fresh concrete also increased with the increase of alkaline solution in the mixture.
- Alkaline activator solution with SS/SH ratio of 2.5 achieved lesser slump and setting time than those with 1.5 and 2. Compressive strength variation of concretes was not significant due to variation of SS/SH ratio in the mix.

## ACKNOWLEDGEMENT

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