

# On the Development of Fly Ash-Based Geopolymer Concrete

by Djwantoro Hardjito, Steenie E. Wallah, Dody M. J. Sumajouw, and B. Vijaya Rangan

*To reduce greenhouse gas emissions, efforts are needed to develop environmentally friendly construction materials. This paper presents the development of fly ash-based geopolymers concrete. In geopolymers concrete, a by-product material rich in silicon and aluminum, such as low-calcium (ASTM C 618 Class F) fly ash, is chemically activated by a high-alkaline solution to form a paste that binds the loose coarse and fine aggregates, and other unreacted materials in the mixture. The test results presented in this paper show the effects of various parameters on the properties of geopolymers concrete. The application of geopolymers concrete and future research needs are also identified.*

**Keywords:** compressive strength; creep; fly ash; shrinkage; sulfate resistance.

## INTRODUCTION

The contribution of ordinary portland cement (OPC) production worldwide to greenhouse gas emissions is estimated to be approximately 1.35 billion tons annually or approximately 7% of the total greenhouse gas emissions to the earth's atmosphere.<sup>1</sup> Also, it has been reported that many concrete structures, especially those built in corrosive environments, start to deteriorate after 20 to 30 years, even though they have been designed for more than 50 years of service life.<sup>2</sup>

The concrete industry has recognized these issues. For example, the U.S. Concrete Industry has developed plans to address these issues in "Vision 2030: A Vision for the U.S. Concrete Industry." In this document, strategies to retain concrete as a construction material of choice for infrastructure development, and at the same time to make it an environmentally friendly material for the future, have been outlined.<sup>3</sup>

To produce environmentally friendly concrete, Mehta<sup>4</sup> suggested the use of fewer natural resources, less energy, and to minimize carbon dioxide emissions. He categorized these short-term efforts as industrial ecology. The long-term goal of reducing the impact of unwanted by-products of industry can be attained by lowering the rate of material consumption.

In line with the above view, one of the efforts to produce more environmentally friendly concrete is to partially replace the amount of OPC in concrete with by-product materials such as fly ash. An important achievement in this regard is the development of high-volume fly ash (HVFA) concrete that uses only approximately 40% of OPC, and yet possesses excellent mechanical properties with enhanced durability performance. The test results show that HVFA concrete is more durable than OPC concrete.<sup>5</sup>

Another effort in this regard is the development of inorganic alumino-silicate polymer, called geopolymers, synthesized from materials of geological origin or by-product materials such as fly ash that are rich in silicon and aluminum.<sup>6</sup> The geopolymers paste can be used as a binder to produce concrete, instead of the cement paste.

Fly ash, one of the source materials for geopolymers binders, is available abundantly worldwide, and yet its use

to date is limited. From the 1998 estimation, the global coal ash production was more than 390 million tonnes annually, but its use was less than 15%.<sup>7</sup> In the future, fly ash production will increase, especially in countries such as China and India. From these two countries alone, it is estimated that by the year 2010 the amount of the fly ash produced will be 780 million tonnes annually.<sup>5</sup> Accordingly, efforts to use this by-product material in concrete manufacture are important to make concrete more environmentally friendly.

This paper presents the technology of making geopolymers concrete using low-calcium (Class F)<sup>8</sup> dry fly ash as its source material and presents the results of laboratory tests conducted on this material.

## RESEARCH SIGNIFICANCE

Most of the published research on geopolymers studied the behavior of pastes. The present study deals with the manufacture of geopolymers concrete and the influence of several parameters on the compressive strength. The research data presented in this paper are useful to understand the behavior of geopolymers concrete.

## PAST RESEARCH ON GEOPOLYMER PASTE OR MORTAR

The chemical compositions of geopolymers materials are similar to zeolite, but they reveal an amorphous microstructure.<sup>6</sup> High-alkaline solutions are used to induce the silicon and aluminum atoms in the source materials to dissolve and form the geopolymers paste. The polymerization process may be assisted by applied heat, followed by drying. The chemical reaction period is fast, and the required curing period may be within 24 to 48 h. Davidovits<sup>6</sup> reported that this material possesses excellent mechanical properties, does not dissolve in acidic solutions, and does not generate any deleterious alkali-aggregate reaction even in the presence of high alkalinity.

Very limited research data are available in the literature. Most of the past research on the behavior of geopolymers material was based on the binder paste or mortar using small size samples. In addition, some of the conclusions are contradictory. Based on the laboratory tests on fly ash-based geopolymers binder, Palomo, Grutzeck, and Blanco<sup>9</sup> have shown that the curing temperature, the curing time, and the type of activator affected the compressive strength, while the solution-to-fly ash ratio was not a relevant parameter. Increase in the curing temperature increased the compressive

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strength. The type of alkaline activator that contained soluble silicates resulted in a higher reaction rate than when hydroxides were used as the only activator.

While van Jaarsveld, van Deventer, and Lukey<sup>10</sup> confirmed the importance of curing at elevated temperature for fly ash-based geopolymers material, they found that curing for a longer period of time at elevated temperature weakened the microstructure. Barbosa, MacKenzie, and Thaumaturgo<sup>11</sup> stated that the water content played an important role on the properties of geopolymers binders, besides the chemical composition of the oxides used as activators.

## WHAT IS GEOPOLYMER CONCRETE?

In the authors' experimental work, geopolymers are used as the binder, instead of cement paste, to produce concrete. The geopolymers paste binds the loose coarse aggregates, fine aggregates and other unreacted materials together to form the geopolymers concrete. The manufacture of geopolymers concrete is carried out using the usual concrete technology methods.

As in the portland cement concrete, the aggregates occupy the largest volume, that is, approximately 75 to 80% by mass, in geopolymers concrete. The silicon and the aluminum in the fly ash are activated by a combination of sodium hydroxide and sodium silicate solutions to form the geopolymers paste that binds the aggregates and other unreacted materials.

## EXPERIMENTAL WORK

In the present experimental work, low-calcium (Class F)<sup>8</sup> dry fly ash obtained from the silos at a local power station was used as the base material. The chemical composition of the fly ash, as determined by x-ray fluorescence (XRF) analysis, is given in Table 1.

Analytical grade sodium hydroxide in flake form (NaOH with 98% purity) and sodium silicate solutions (Na<sub>2</sub>O = 14.7%, SiO<sub>2</sub> = 29.4% and water = 55.9% by mass), were used as the alkaline activators. To avoid the effect of unknown contaminants in the mixing water, the sodium hydroxide flakes were dissolved in distilled water. The activator solution was prepared at least one day prior to its use. To improve the workability of fresh concrete, a commercially available naphthalene-based high-range water-reducing admixture was used. Four types of locally available aggregates, that is, 20, 14, and 7 mm aggregate, and fine sand, in saturated surface

**Table 1—Composition of fly ash as determined by XRF (mass %)**

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	MgO	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	LOI*
53.36	26.49	10.86	1.34	0.37	0.80	1.47	0.77	1.43	1.70	1.39

\*Loss on ignition.

dry condition, were mixed together. The grading of this combined aggregate had a fineness modulus of 5.0.

The aggregates and the fly ash were mixed dry in a pan mixer for 3 min. The alkaline solutions and the high-range water-reducing admixture were mixed together, then added to the solid particles and mixed for another 3 to 5 min. The fresh concrete had a stiff consistency and was glossy in appearance. The mixture was cast in 100 x 200 mm cylinder steel molds in three layers. Each layer received 60 manual strokes and vibrated for 10 s on a vibrating table. Five cylinders were prepared for each test variable.

Immediately after casting, the samples were covered by a film to avoid the loss of water due to evaporation during curing at an elevated temperature. After being left in room temperature for 30 to 60 min, specimens were cured in an oven at a specified temperature for a period of time in accordance with the test variables selected.

At the end of the curing period, the 100 x 200 mm test cylinders were removed from the molds and kept in the plastic bag for 6 h to avoid a drastic change of the environmental conditions. The specimens were then left to air dry at room temperature until loaded in compression at the specified age in a universal test machine. Before testing, the specimens were weighed to determine the density of the material. The loading rate and other test procedures used were in accordance with the details specified in the relevant Australian Standard for testing OPC concrete.<sup>12</sup>

Numerous trial mixtures of geopolymers concrete were made and tested in the laboratory. The data collected from these studies indicated that the salient parameters affecting the compressive strength of geopolymers concrete are as listed below:

- Silicon oxide (SiO<sub>2</sub>)-to-aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) ratio by mass of the source material (fly ash); this ratio should preferably be in the range of 2.0 to 3.5 to make good concrete (Table 1);
- Activator liquids-to-source material (fly ash) ratio by mass;
- Concentration of sodium hydroxide (NaOH) liquid measured in terms of Molarity (M), in the range of 8 to 16 M;
- Sodium silicate-to-sodium hydroxide liquid ratio by mass; the effect of this parameter depends on the composition of the sodium silicate solution;
- Curing temperature in the range of 30 to 90 °C;
- Curing time in the range of 6 to 96 h; and
- Water content in the mixture.

It must be noted that only the binder (which usually occupy approximately 20 to 25% of the total mass) is different in geopolymers concrete when compared to OPC concrete. Therefore, the effects of properties and grading of aggregates were not investigated in this study.

## EFFECT OF PARAMETERS

In this section, we present the influence of various parameters on the compressive strength of geopolymers concrete as observed in the laboratory tests. In the later part of the paper, the test results on drying shrinkage, creep, and sulfate resistance are presented. Each of the test data points plotted

in various graphs corresponds to the mean value of the compressive strengths of five test cylinders in a series. The standard deviations are plotted on the test data points as the error bar.

In the experimental work, the activator liquids-to-fly ash ratio by mass was kept constant at approximately 0.35. The coarse and fine aggregates constituted approximately 77% by mass in all mixtures.

Table 2 gives the composition of four different mixtures and the 7-day compressive strengths of 100 x 200 mm test cylinders cured at 60 °C for 24 h. In Table 2, the second column gives the concentration of NaOH liquid in terms of molarity (M). The third column is the ratio of sodium silicate/NaOH by mass in liquid form. The last column gives the mean 7-day compressive strengths of test cylinders.

### Concentration of sodium hydroxide

In Table 2, the only difference between the Mixtures A-1 and A-3 is the concentration of sodium hydroxide as measured by Molarity (second column). Mixture A-3 with higher concentration of NaOH yielded higher compressive strength than Mixture A-1. A similar trend is also observed for the Mixtures A-2 and A-4.

### Sodium silicate-to-sodium hydroxide liquid ratio

The effect of sodium silicate-to-NaOH ratio in liquid form on compressive strength can be seen by comparing the compressive strengths of Mixtures A-1 and A-2 as well as A-3 and A-4 (Table 2). For Mixtures A-1 and A-2, although the concentration of NaOH liquid (in terms of molarity) is the same, in Mixture A-2 the sodium silicate/NaOH ratio is higher than that of Mixture A-1. This produced a higher compressive strength in Mixture A-2 than Mixture A-1. A similar trend is also observed in the results of Mixtures A-3 and A-4.

The results given in Table 2 reveal that the interrelation of various oxides contained in the mixture composition affects the compressive strength.

### Curing temperature

Figure 1 shows the effect of curing temperature on the compressive strength for Mixtures A-2 and A-4. All other test variables were held constant. Higher curing temperature resulted in larger compressive strength, even though an increase in the curing temperature beyond 60 °C did not increase the compressive strength substantially.

### Curing time

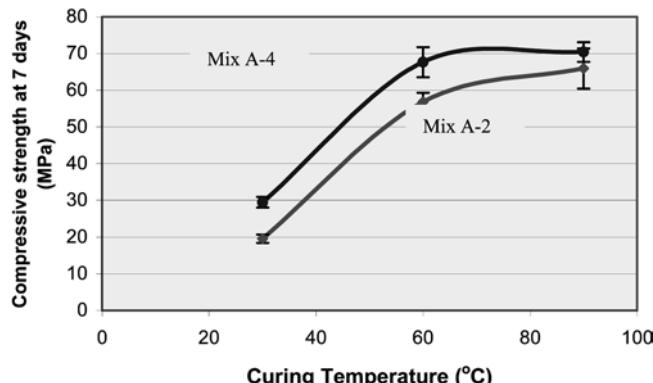
Figure 2 shows the influence of curing time on the compressive strength for Mixture A-2. Longer curing time improved the polymerization process resulting in higher compressive strength. The results indicate that a longer curing time at 60 °C does not produce weaker material as claimed by van Jaarsveld, van Deventer, and Lukey.<sup>10</sup> However, the increase in strength for curing periods beyond 48 h is not significant.

### High-range water-reducing admixture

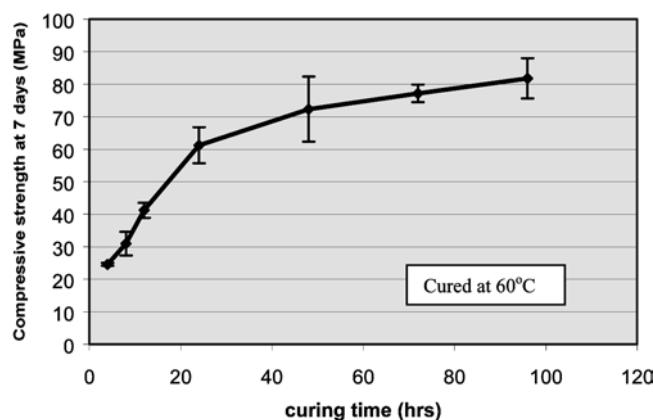
In fresh state, the geopolymers concrete has a stiff consistency. Although adequate compaction was achievable, an improvement in the workability was considered as desirable. A series of tests were therefore performed on Mixture A-2 (Table 2) to study the effect of adding commercially available naphthalene-based high-range water-reducing admixture.

**Table 2—Effect of parameters on compressive strength**

Mixture	Concentration of NaOH liquid in molarity (M)	Sodium silicate/NaOH liquids ratio by mass	7-day compressive strength after curing at 60 °C for 24 h, MPa
A-1	8M	0.4	17.3
A-2	8M	2.5	56.8
A-3	14M	0.4	47.9
A-4	14M	2.5	67.6



*Fig. 1—Effect of curing temperature on compressive strength.*



*Fig. 2—Influence of curing time on compressive strength for Mixture A-2.*

The results of these tests are shown in Fig. 3. The addition of high-range water-reducing admixture improved the workability of the fresh concrete, but has very little effect on the compressive strength up to approximately 2% of this admixture to the amount of fly ash by mass. Beyond this value, the compressive strength decreased from 56.5 MPa for 2% of high-range water-reducing admixture to 46 MPa for 3.5% of this admixture.

Figure 3 shows two sets of data. In one set, the test cylinders were allowed to rest for 60 min after casting and then placed in the oven for curing at 60 °C for 24 h. In the other set, there was no rest period and the test cylinders were placed in the oven immediately after casting.

The results plotted in Fig. 3 show very little difference between the strengths of the two sets of specimens. This is an important outcome in practical applications of geopolymers concrete. For instance, when geopolymers concrete is used in precast concrete industry, the results in Fig. 3 indicate that there is sufficient time available between casting of products and sending them to the curing room.

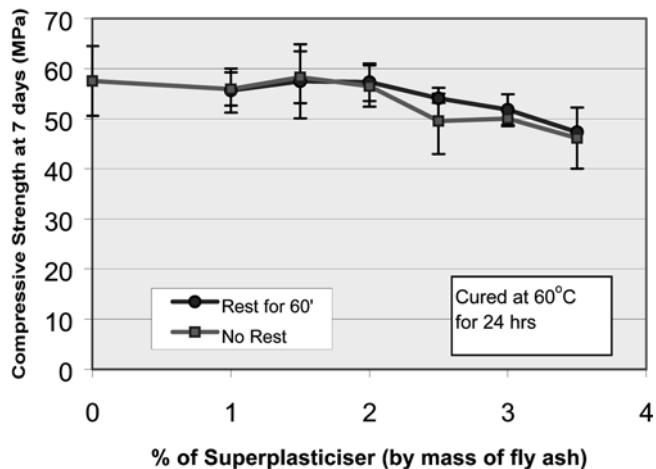


Fig. 3—Effect of high-range water-reducing admixture addition on compressive strength for Mixture A-2.

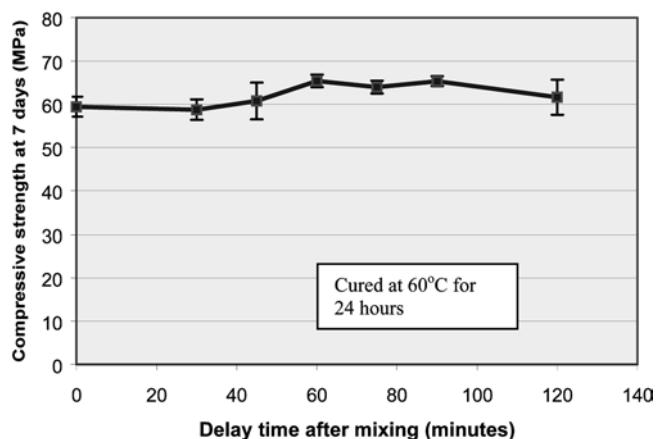


Fig. 4—Influence of delay time on compressive strength for Mixture A-2.

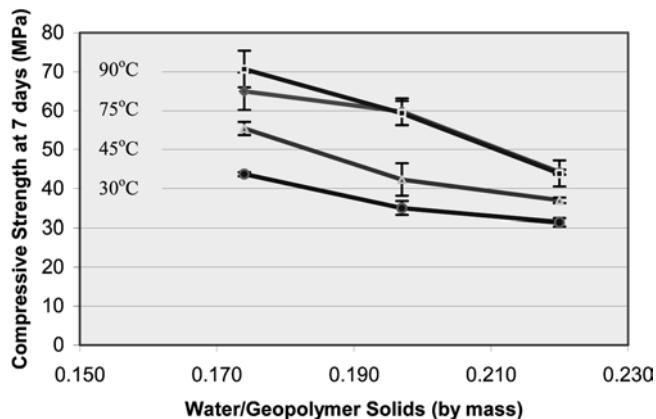


Fig. 5—Effect of the water-to-geopolymer solids ratio on compressive strength.

### Handling time

Due to lack of a suitable method to determine the initial setting time of geopolymer concrete, the setting time of the fresh concrete could not be measured. The laboratory experience by the authors, however, showed that the fresh concrete could be handled up to 120 min after mixing without any sign of setting and without any degradation in the compressive strength (Fig. 4).

To obtain the data plotted in Fig. 4, the fresh concrete was allowed to stand at room temperature after mixing and prior to being placed in molds for a certain period of time. This time is referred to as the delay time in Fig. 4. For instance, a delay time of 60 min means that the fresh concrete was cast in the molds 60 min after mixing, and a zero delay time indicates that casting took place immediately after mixing. The slight variation in the compressive strengths plotted in Fig. 4 is probably due to minor changes in the laboratory room temperature and humidity.

### Water content in the mixture

Previous research by Barbosa, MacKenzie, and Thaumaturgo<sup>11</sup> on geopolymers pastes showed that the water content in the mixture played an important role on the properties of geopolymers binders. To study the effect of water content on the compressive strength of geopolymers concrete, several tests were performed. The basic mixture used in this series of tests was Mixture A-4 (Table 2). The other details of the mixtures were the same as those used in the earlier part of this paper. The percentage of the high-range water-reducing admixture to the mass of fly ash was 1.5%, the delay time was 30 min, and there was no rest period.

The effect of water content is illustrated in Fig. 5 by plotting the compressive strength versus water-to-geopolymer solids ratio by mass. For a given geopolymers concrete, the total mass of water in the mixture is taken as the sum of the mass of water in the sodium silicate solution, the mass of water in the sodium hydroxide solution, and the mass of extra water, if any, added to the mixture. The mass of geopolymers solids is the sum of the mass of fly ash, the mass of sodium hydroxide flakes, and the mass of sodium silicate solids (that is, the mass of Na<sub>2</sub>O and SiO<sub>2</sub> in sodium silicate solution).

To vary the water-to-geopolymer solids ratio, water was added to Mixture A-4 (Table 2). The water-to-geopolymer solids ratio of Mixture A-4 was 0.174. By adding extra water of 10.6 kg/m<sup>3</sup>, the water-to-geopolymer solids ratio became 0.197, and by adding extra water of 21.2 kg/m<sup>3</sup>, this ratio was 0.220. The 7-day compressive strengths of geopolymers concrete cylinders produced from these mixtures are plotted in Fig. 5 for different curing temperatures.

As expected, the addition of water improved the workability of the mixtures. The test data shown in Fig. 5 demonstrate that the compressive strength of geopolymers concrete decreases as the ratio of water-to-geopolymer solids by mass increases. The trends of these test results are similar to those observed by Barbosa, MacKenzie, and Thaumaturgo<sup>11</sup> for their tests on geopolymers pastes. The test trends are somewhat analogous to the well-known effect of water-to-cement ratio on the compressive strength of OPC concrete, although the chemical processes involved in the formation of the binders of both these types of concretes are entirely different.

The results shown in Fig. 5 also confirm that an increase in the curing temperature increased the concrete compressive strength. However, increasing the curing temperature from 75 to 90 °C did not show any significant gain in the compressive strength. Similar results were reported by previous researchers working on geopolymers binders<sup>9,10</sup> using various source materials.

### Age of concrete

Figure 6 shows the effect of age of concrete on the compressive strength. The concrete specimens for this purpose

were prepared without adding any high-range water-reducing admixture, and there was no delay time and rest period.

Because the chemical reaction of the geopolymer paste is a fast polymerization process, the compressive strength does not vary with the age of concrete, when cured for 24 h. This observation is in contrast to the well-known behavior of OPC concrete, which undergoes a hydration process and hence gains strength over time.<sup>13</sup>

### Unit weight

The unit weight of concrete primarily depends on the unit weights of aggregates used in the mixture. Because the type of aggregates in all mixtures did not vary, the unit weight of the concrete varied only marginally between 2330 to 2430 kg/m<sup>3</sup>.

### DRYING SHRINKAGE, CREEP, AND SULFATE RESISTANCE

Figure 7 shows the measured drying shrinkage and creep strains of geopolymer concrete produced using Mixture A-2. The test specimens were cured for 24 h at 60 °C. The percentage of high-range water-reducing admixture by mass of fly ash was 1.5%. There was no rest period before casting, and the delay time after mixing was 30 min. The 7-day compressive strength was 53.7 MPa. The creep specimens were loaded on the seventh day to produce a sustained stress of 22 MPa (approximately 40% of the compressive strength). The details of test specimens and test procedure for creep tests were in accordance with the relevant Australian Standard for OPC concrete.

Figure 7 shows that the drying shrinkage strains are extremely small indeed. The ratio of creep strain-to-elastic strain (that is, creep factor) reached a value of approximately 0.30 in approximately 6 weeks. Beyond this time, the creep factor increased only marginally.

To evaluate the resistance of geopolymer concrete to sulfate attack, a series of tests were performed. The test specimens were soaked in a 5% sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>) solution for periods of time. The test results reported elsewhere<sup>14</sup> showed that after 12 weeks of exposure, there were no significant changes in the compressive strength, the mass, and the length of test specimens.

### APPLICATIONS AND ECONOMICS OF GEOPOLYMER CONCRETE

The authors have used conventional methods, similar to those used in the case of portland cement concrete, for mixing and placing of geopolymer concrete. For a specified compressive strength, the required workability of the concrete, in terms of slump, can be obtained by adjusting the water content and the concentration (in terms of molar) of sodium hydroxide in the mixture. For instance, the authors have successfully manufactured many mixtures with a compressive strength in the range of 30 to 80 MPa and the slump of concrete varying from 100 to 250 mm depending on the molar of sodium hydroxide and the mass of extra water added to the mixture.

A series of 175 x 175 x 1500 mm reinforced geopolymer concrete columns were also manufactured and tested in the laboratory. The compressive strength of the concrete in these structural columns ranged between 42 to 66 MPa, and the slump was approximately 240 mm. The details of this research will be presented in forthcoming papers. With the current state of knowledge, the authors believe that geopolymer concrete is ideally suitable for the manufacture of precast concrete (both

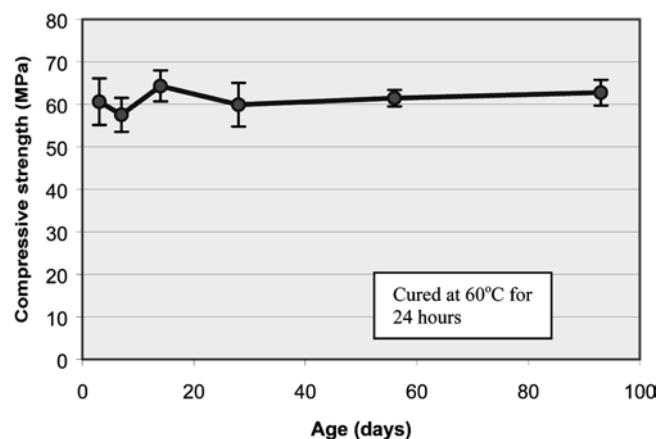


Fig. 6—Compressive strength at different ages for Mixture A-2.

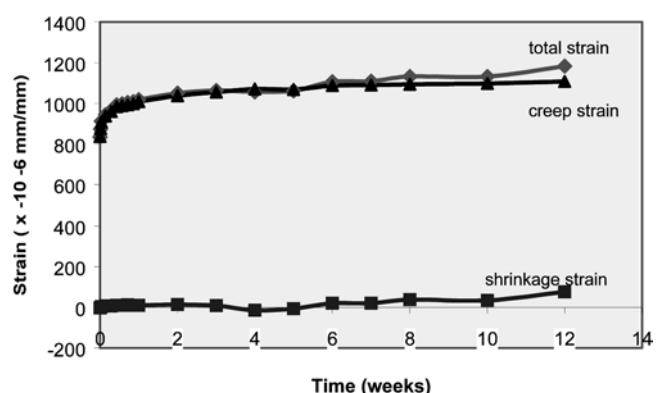


Fig. 7—Drying shrinkage and creep strains for Mixture A-2.

reinforced and prestressed) elements and other products needed for infrastructures.

Based on their laboratory experience, the authors found that the cost of geopolymer concrete per cubic meter is approximately the same as that of portland-cement concrete. If one considers the impact of the possible carbon dioxide tax on the price of cement and the environmental advantage of utilization of fly ash, the geopolymer concrete may prove to be economically advantageous.

### FUTURE RESEARCH NEEDS

As in the case of portland cement concrete, the properties of constituent materials in the geopolymer concrete mixture will influence the physical properties of the hardened concrete. It is therefore necessary to collect experimental data on various properties and use these data to formulate appropriate codes of practice. Future research should also focus on the fundamental science of geopolymers to determine the mechanism of chemical reaction during setting and hardening.

### CONCLUSIONS

This paper presented the development of geopolymer concrete. The binder in this concrete, the geopolymer paste, is formed by activating by-product materials, such as low-calcium (Class F) fly ash, that are rich in silicon and aluminum.

In the experimental work, the fly ash (Table 1) from a local power generation plant was used as the source material. A combination of sodium silicate solution and sodium hydroxide solution was used as the activator. The geopolymer paste binds the loose coarse and fine aggregates

and any unreacted materials to form the geopolymers concrete. Based on the experimental work reported in this paper, the following conclusions are drawn:

1. Higher concentration (in terms of molar) of sodium hydroxide solution results in a higher compressive strength of geopolymers concrete (Table 2);
2. Higher the ratio of sodium silicate-to-sodium hydroxide liquid ratio by mass, higher is the compressive strength of geopolymers concrete (Table 2);
3. As the curing temperature in the range of 30 to 90 °C increases, the compressive strength of geopolymers concrete also increases (Fig. 1);
4. Longer curing time, in the range of 6 to 96 h (4 days), produces larger compressive strength of geopolymers concrete. However, the increase in strength beyond 48 h is not significant (Fig. 4);
5. The addition of high-range water-reducing admixture, up to approximately 2% of fly ash by mass, improved the workability of fresh geopolymers concrete with very little effect on the compressive strength of hardened concrete (Fig. 3);
6. The rest period between casting of specimens and the commencement of curing up to 60 min has no effect on the compressive strength of geopolymers concrete (Fig. 3);
7. The fresh geopolymers concrete is easily handled up to 120 min without any sign of setting and without any degradation in the compressive strength (Fig. 4);
8. As the ratio of water-to geopolymers solids by mass increases, the compressive strength of the concrete decreases (Fig. 5);
9. The compressive strength of geopolymers concrete cured for 24 h at 60 °C does not depend on the age (Fig. 6); and
10. The geopolymers concrete undergoes very little drying shrinkage and low creep (Fig. 7). The resistance of geopolymers concrete against sodium sulfate is excellent.<sup>14</sup>

This paper also identified the applications of geopolymers concrete and future research needs.

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