

## Effect of Curing Temperature and Curing Time on the Strength Development of Fly Ash-based Geopolymers Concrete

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**Abstract** Geopolymers concrete provides a sustainable alternative to the higher energy-intensive Portland cement concrete. However, to date, there is no standard mix design procedure for geopolymers concrete. An effective geopolymer concrete mix design procedure should include criteria for selecting the parameters that have a significant impact on its compressive strength, such as the alkaline activator solution content, binder content (e.g., fly ash), aggregate content, curing temperature, and curing duration. This study aims to investigate the effect of curing temperature and curing duration on the compressive strength and Pulse velocity of fly ash-based geopolymers concrete to build a mix design procedure that considers these parameters. Besides, an experimental investigation was carried out, as well as an extensive survey of related literature. This study showed that curing period and temperature have a significant effect on geopolymers' concrete compressive strength. Moreover, a curing activity index could be utilized to integrate the curing influence into the mix design procedure, which has the objective of scaling down the design required strength to arrive at the portion of the required design compressive strength relegated to a mix cured at ambient temperature.

**Keywords** Geopolymers Concrete, Fly Ash, Curing Temperature, Compressive Strength, Pulse velocity

### 1. Introduction

In the last few years, there has been a significant increase in the development of alternative methods for producing concrete that do not require the use of cement [1]. However, it is difficult to determine which parameters provide superior concrete performance. Several factors contribute to this complexity, including the use of different sources of materials (multiple sources of rock, sand, and cement), the replacement of cement by new cementitious materials (fly ash, ground granulated blast furnace slag, etc.), and the introduction of many new chemical additives that are claimed to produce a superior product [2-3].

The development of inorganic alumina-silicate polymer, known as geopolymers, is considered one of the attempts to produce more environmentally friendly concrete without utilizing cement. These polymers are synthesized from geological materials or by-product materials rich in silicon and aluminum, such as fly ash.

In Jordan, Fly ash is obtained from crushed, sieved, and burned of oil shale in Al-Lajjoun quarries (about 100km south of Amman, Jordan) [4]. Assa'ad and Asi had reported that the addition of Jordanian oil shale fly ash on the asphalt mixes enhanced the strength and water sensitivity properties. In general, the addition of fly ash to geopolymer concretes is influenced the workability

and the compressive strength properties [5].

Various studies have investigated the effect of curing conditions, curing temperature, and curing duration on the properties of geopolymers [6-12]. Alengaram et al. had reported in improved compressive strength of lightweight foamed geopolymer concrete containing palm oil fly ash. Lenoelli et al. had replaced sodium silicate with rice husk ash-NaOH solution to reduce the warming potential of geopolymers. Besides that, Huynh and Hwang have reported that the presence of rice husk ash in geopolymers concrete with 10 M of NaOH improved the compressive strength properties. Mirza et al. showed that curing at different temperatures for geopolymers containing palm oil fuel ash and fly ash will enhance the compressive strength and the microstructure properties of geopolymers. Consequently, these studies show that the curing temperature and curing time played a crucial role in determining the properties of geopolymer concrete.

The present literature lacked the behavior of oil shale fly ash-based geopolymer concretes considered to curing temperature and curing time parameters. This paper presents the effect of curing time and curing temperature on the compressive strength of Jordanian fly ash based geopolymer concrete. The compressive strength was investigated at different oven temperatures (60, 75, and 90 °C) for curing the geopolymers' concrete specimens.

## 2. Experimental Work Plan

### 2.1. Materials

The crushed stone coarse aggregate was obtained from Amman (Jordan) with a maximum size of 20 mm (ASTM Type-1 standards). Local sand from Amman (Jordan) with a specific gravity of 2.65 and water absorption of 3.5% was used as fine aggregates (ASTM Type-1 standards). The dry fly ash obtained from Al-Lajjoun quarries (about 100km south of Amman, Jordan) was used as the base material (ASTM C 618 Class F). The chemical composition of the fly ash is shown in Table 1.

A combination of sodium silicate solution and sodium hydroxide solution was chosen as the alkaline liquid. The sodium hydroxide solution was prepared by dissolving NaOH pellets in water. The concentration of sodium hydroxide solution was 12 molar (M) consisted of 480 grams (g) of NaOH pellets per liter of NaOH solution or 361 g of NaOH pellets per kg of NaOH solution. The chemical composition of the sodium silicate solution was Na<sub>2</sub>O=14.7%, SiO<sub>2</sub>=29.4%, and H<sub>2</sub>O=55.9% (by mass).

A Polycarboxylate polymer was used as a superplasticizer, and the properties of this superplasticizer are shown in table 2.

**Table 1.** Chemical Compositions of Fly Ash

| Sample                         | Fly ash (%) |
|--------------------------------|-------------|
| SiO <sub>2</sub>               | 50          |
| Al <sub>2</sub> O <sub>3</sub> | 28.25       |
| Fe <sub>2</sub> O <sub>3</sub> | 13.5        |
| CaO                            | 1.79        |
| MgO                            | 0.89        |
| Na <sub>2</sub> O              | 0.32        |
| K <sub>2</sub> O               | 0.46        |
| SO <sub>3</sub>                | 0.38        |
| TiO <sub>2</sub>               | 1.54        |

**Table 2.** Properties of Polycarboxylate superplasticizer

|  |                     |
|--|---------------------|
| Appearance   | Light Yellow Liquid |
| Solid Content, %   | 50±1.0              |
| Density (23°C) (kg/m <sup>3</sup> )                        | 1.13±0.02           |
| PH   | 6.5-8.5             |
| Chloride Content,% ≤                                       | 0.1                 |
| Na <sub>2</sub> SO <sub>4</sub><br>(by solid content), % ≤ | 4.0                 |
| Water reducing ratio, %<br>≥                               | 25                  |

## 2.2. Geopolymer Concrete Mixture Proportion

A solution of Na<sub>2</sub>SiO<sub>3</sub> and NaOH (alkaline solution) was prepared one day before its use. The sand and coarse aggregates were mixed dry for 3 min. After 30 s, the fly ash alkaline solution and superplasticizer were added, then the mixing process was continued. A small amount of extra water was added to the mixer to achieve the desired workability. After all the ingredients were placed in the mixer, the materials were mixed for 5 min, followed by a 5-min rest period, and then another 5 min of final mixing. After 3 min of rest, cylindrical samples (100 X 200 mm) were prepared to determine the compressive strength of the geopolymer concrete mixtures. The proportions of geopolymer concrete mixtures used in the laboratory are given in Table (3) [13]. The geopolymer concrete mixture is shown in Figure (1).

## 2.3. Curing of Geopolymer Concrete Specimens

After casting, the curing of fly ash geopolymer concretes was taken place at different temperatures (60, 75, and 90 °C). The specimens were oven-cured for (8h, 16h, 18 h, and 24 h). After that, the geopolymer concrete specimens were left in the molds for at least six hours, and the specimens left for air-dry until the day of testing (28 days).

**Table 3.** Fly Ash Based Geopolymer Concrete Mixture Proportions (per m<sup>3</sup>)

| Materials   | Mass (kg/m <sup>3</sup> ) |
|---|---------------------------|
| Coarse aggregate  | 1300                      |
| sand  | 550                       |
| Fly ash   | 400                       |
| Sodium silicate solution<br>(SiO <sub>2</sub> /Na <sub>2</sub> O=2) | 103                       |
| Sodium hydroxide<br>solution  | 41 (12)molar              |
| Super Plasticizer   | 6                         |
| water   | 23                        |



**Figure 1.** Geopolymer Concrete Mixture

### 3. Test Results and Analysis

#### 3.1. Compressive Strength

The compressive strength of geopolymers concrete with 28 days' age is summarized in Table 4. All the results are expressed as the mean  $\pm$  standard deviations ( $n = 5$ ).

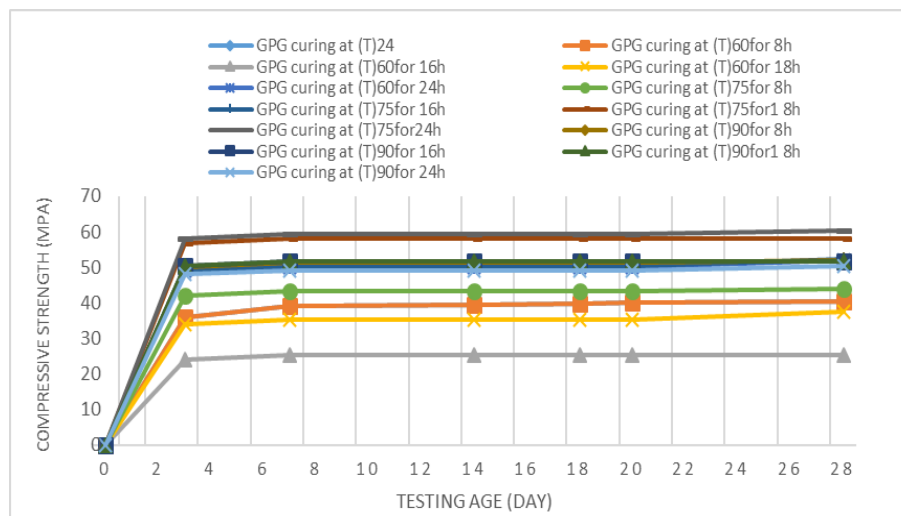
The influences of curing temperature on the 28-day compressive strengths of geopolymers concrete are shown in (Figure 2). The compressive strength of the geopolymers concretes increases with the increase of curing temperature (from 60 to 90 °C). This increment is due to the higher geopolymerization degree at higher temperatures, which results in the formation of a higher amount of reaction products [14]. Regardless of the curing regime adopted, some of the alumina silicate source materials will remain unreacted in the hardened geopolymers concrete. However, the amount of these unreacted solids will be reduced by applying a higher curing temperature. Specifically, during the geopolymerization process, the fly ash grains are not completely dissolved, and the reaction occurs at the surface layer of the solid particles to form primary geopolymer gel [15].

The further geopolymerization rate is then primarily controlled by the diffusion of hydroxide and silicate ions through the primary geopolymer gel, which is mainly affected by curing temperature early in the reaction. However, for long periods of heat curing (18 h and 24 h), the optimum strength achieved at 75 °C. This finding is conformed with previous studies [16,17], reporting that curing geopolymers concrete for long periods and at elevated temperatures (e.g., 90 °C) leads to deteriorate its microstructure, resulting in a lower compressive strength. Among all the different curing durations and temperatures, the geopolymers concrete cured at 75 °C for 24 h shows the highest compressive strength (at 28 days) of 60.28 MPa [13]. However, reducing the heat curing duration from 24 to 18 h, does not lead to a significant reduction in strength (around 3% reduction). As a result, 18 h heat curing at 75°C could be considered as the optimum heat curing condition for fly ash-based geopolymers concrete tested in this study.

The geopolymer concrete samples curing at 75°C for 18 h attained a low strength after one day, as shown in Figure (2). Their strength increased to 56.3 MPa after 7 days and reached 58.2 MPa after 28 days. This finding highlights the importance of heat curing for geopolymers concrete.

**Table 4.** Compressive Strength of Fly Ash Based Geopolymer Concrete 28 Days Age.

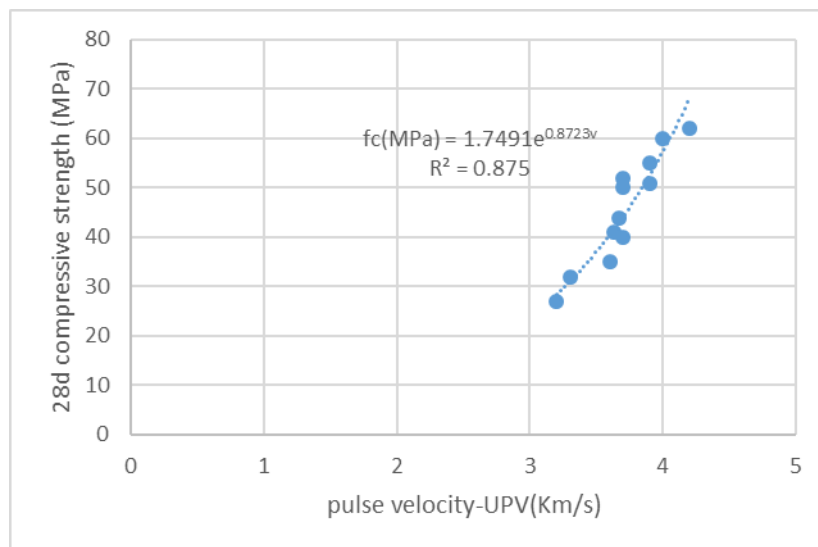
| Mix designation | Curing temperature (°C) | Heat curing duration(h) | fc,28 (MPa) |
|-----------------|-------------------------|-------------------------|-------------|
| GPC-SAC         | 25                      | N.A                     | 40.1±1.6    |
| GPC-60D-8h      | 60                      | 8                       | 25.2±1.4    |
| GPC-60D-16h     | 60                      | 16                      | 37.4±1.2    |
| GPC-60D-18h     | 60                      | 18                      | 43.4±1.2    |
| GPC-60D-24h     | 60                      | 24                      | 51.8±0.18   |
| GPC-75D-8h      | 75                      | 8                       | 44.±0.1     |
| GPC-75D-16h     | 75                      | 16                      | 52.2±1.6    |
| GPC-75D-18h     | 75                      | 18                      | 58.0±0.6    |
| GPC-75D-24h     | 75                      | 24                      | 60.1±0.8    |
| GPC-90D-8h      | 90                      | 8                       | 52±0.01     |
| GPC-90D-16h     | 90                      | 16                      | 51.6±0.2    |
| GPC-90D-18h     | 90                      | 18                      | 51.6±0.2    |
| GPC-90D-24h     | 90                      | 24                      | 50.2±0.13   |



**Figure 2.** Compressive strength development of Geopolymer concrete

## 3.2 Ultrasonic pulses velocity test

The ultrasonic pulses velocity test method describes how to measure the travel velocity of ultrasonic pulses through a material. The ultrasonic pulses velocity of geopolymers concretes (28 days age) is plotted against the 28-day compressive strength, as shown in Figure (3). The pulse velocity of geopolymers concretes samples is ranging between 3200 and 4100m/s (Figure 3). Moreover, the pulse velocity in geopolymers concrete is increasing with an increase in the compressive strength. The compressive strength and ultrasonic pulses velocity of geopolymers concretes have a high correlation coefficient ( $R^2 = 0.875$ ). The measurements of ultrasonic pulse velocity provide good characteristics for the compressive strength of geopolymers concretes.



**Figure 3.** Relationship between ultrasonic pulses velocity and compressive strength of geopolymer concretes at 28 days of age.

## 4. Conclusion

This paper has presented the influence of curing temperatures on the compressive strength of fly ash-based geopolymers concrete. The fly ash was obtained from burned oil shale and used as a base material. The geopolymer gel formed from the activation of fly ash by using the mixture solution of  $\text{Na}_2\text{SiO}_3$  and  $\text{NaOH}$ . The geopolymer gel combines the sand and fine aggregates and the other materials to establish the fly ash-based geopolymer concrete. Based on the experimental work reported in this study, the following conclusions are drawn:

1. Depending on the heat curing regime, fly ash-based geopolymer concrete samples developed compressive strengths ranged between 25.34 and 60.28 MPa at 28 days. The compressive strength increased with the increase in the curing temperature up to  $75^\circ\text{C}$  and curing duration up to 24 hours.
2. The longer curing time, in the range of 18 to 24 hours, results in higher compressive strength of fly ash-based geopolymers concrete.
3. An increase in curing temperature from  $60^\circ\text{C}$  to  $75^\circ\text{C}$  for specimens curing for 18 hours causes the increase in compressive strength of fly ash-based geopolymers concrete by 4%.
4. An increase in curing temperature from  $75^\circ\text{C}$  to  $90^\circ\text{C}$  for specimens curing for 18 hours causes a decrease in compressive strength of fly ash-based geopolymers concrete by 5%.
5. An increase in curing temperature from  $60^\circ\text{C}$  to  $75^\circ\text{C}$  for specimens curing for 24 hours causes the increase in compressive strength of fly ash-based geopolymers concrete by 2%.

6. An increase in curing temperature from 75°C to 90°C for specimens curing for 24 hours causes a decrease in compressive strength of fly ash-based geopolymers concrete by 11%.
7. The compressive strength of geopolymers concretes can be estimated by using measurements of ultrasonic pulses velocity.

Based on the presented studies, it can be concluded that the curing temperature and curing time improved the compressive strength of geopolymer concrete. Further works are required to study the effect of curing temperature and curing duration on surface and bulk resistivity of the geopolymers concrete

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## Conflicts of Interest

The author declares no conflict of interest.

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