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Effect of Curing Conditions on Strength of Fly ash-based Self-Compacting Geopolymer Concrete

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Abstract**—**This paper reports the results of an experimental work conducted to investigate the effect of curing conditions on the compressive strength of self-compacting geopolymer concrete prepared by using fly ash as base material and combination of sodium hydroxide and sodium silicate as alkaline activator. The experiments were conducted by varying the curing time and curing temperature in the range of 24-96 hours and 60-90°C respectively. The essential workability properties of freshly prepared Self-compacting Geopolymer concrete such as filling ability, passing ability and segregation resistance were evaluated by using Slump flow, V-funnel, L-box and J-ring test methods. The fundamental requirements of high flowability and resistance to segregation as specified by guidelines on Self-compacting Concrete by EFNARC were satisfied. Test results indicate that longer curing time and curing the concrete specimens at higher temperatures result in higher compressive strength. There was increase in compressive strength with the increase in curing time; however increase in compressive strength after 48 hours was not significant. Concrete specimens cured at 70°C produced the highest compressive strength as compared to specimens cured at 60°C, 80°C and 90°C.

Keywords**—**Geopolymer Concrete, Self-compacting Geopolymer concrete, Compressive strength, Curing time, Curing temperature

I. INTRODUCTION

DUE to growing environmental concerns of the cement industry, alternative cement technologies have become an area of increasing interest. It is now believed that new binders are indispensable for enhanced environmental and durability performance. In this aspect, geopolymer technology is one of the revolutionary developments related to novel materials as an alternative to Portland cement. The development of geopolymer concrete is an important step because of the potential application of geopolymers to a wide variety of industrial waste materials to produce added-value construction materials resulting in low-cost and environmentally friendly material with similar mechanical performance and appearance properties to those from Portland cement [1]. Geopolymer concrete is produced by activating different alumino-silicate based waste materials with highly alkaline solution. Curing of freshly prepared geopolymer concrete is the most crucial aspect and plays an important role in the geopolymerisation process [2]. Proper curing of concrete has a positive effect on the final properties of the geopolymer concrete. Curing of geopolymer concrete is mostly carried out at elevated temperatures [2], however, curing at ambient temperatures has also been carried out. At ambient temperatures; the reaction of fly ash-based geopolymeric materials is very slow and usually show a slower setting and strength development. It is believed that higher temperatures activate alumino-silicate phases in the fly ash, therefore, they are generally cured at elevated temperatures between 60^0C- 90^0C [3]-[8].

Previous research has shown that both curing time and curing temperature significantly influence the compressive strength of geopolymer concrete. Several researchers [4], [7], [9], [10] have investigated the effect of curing time and curing temperature on the properties of geopolymer concrete. Palomo et al. [4], in their study on fly ash-based geopolymers have reported that the curing temperature and curing time significantly affected the mechanical strength of fly ash-based geopolymers. They concluded that higher curing temperature and longer curing time proved to result in higher compressive strength.

van Jaarsveld et al. [7] studied the interrelationship of certain parameters that affected the properties of fly ash-based geopolymer. They have demonstrated that water content, curing time and curing temperature affected the properties of geopolymers; specifically the curing condition and calcining temperature influenced the compressive strength. They concluded that rapid curing and curing at high temperature reduced the compressive strength and caused a negative effect on the physical properties of the geopolymer.

In a study done by Xiaolu Guo et al. [9], on compressive strength and microstructural characteristics of class C fly ash geopolymer, the authors have reported that curing temperature had a significant effect on the compressive strength development. Compressive strength began to decrease after curing for a certain period of time at higher temperature. They revealed that prolonged curing can break down the granular structure of the geopolymer mixture.

Pavel Rovnanik [10], in his research study on effect of curing temperature on the development of hard structure of metakaolin-based geopolymer, has reported that curing

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temperature had significant effect on the setting and hardening of metakaolin-based geopolymer. The author has demonstrated that higher curing temperatures and longer curing time increase the early age compressive and flexural strengths. However, elevated temperature during early stage of hardening process results to the formation of larger pores consequently increases cumulative pore volume, which has a negative effect on the final mechanical properties of geopolymeric material.

This study aimed to analyze the effect of curing time and curing temperature on the compressive strength of fly ash-based self-compacting geopolymer concrete. Test results indicate that longer curing time and curing the concrete specimens at elevated temperature up to 70^0C result in higher compressive strength.

II. EXPERIMENTAL DETAILS

A. Materials

In the present study, Low-calcium (ASTM Class F) Fly ash was used as a source material for the synthesis of SCGC. Fly ash was obtained from Manjung power station, Lumut, Perak, Malaysia.The chemical composition of Fly ash as determined by X-Ray Fluorescence (XRF) analysis is shown in Table I.

Locally available crushed coarse aggregate of maximum size 14 mm having specific gravity of 2.66 was used in the preparation of all test specimens. The coarse aggregate was used in saturated surface dry (SSD) condition.

Natural Malaysian sand having specific gravity of 2.61 and the fineness modulus of 2.76 was used as fine aggregate. Fine aggregate was sieved for the size less than 5mm and used in dry condition.

For the alkaline activator, a combination of sodium hydroxide and sodium silicate solution was used. Sodium hydroxide in pellets form with 99% purity, supplied by QuickLab Sdn Bhd, Malaysia and Sodium silicate solution (Grade A53 with Na2O = 14.26%, SiO2 = 29.43% and water = 56.31%) obtained from Malay-Sino Chemical Industries Sdn Bhd, Malaysia were used. To prepare sodium hydroxide solution, sodium hydroxide pellets were dissolved in ordinary drinking water. Both the liquid solutions were mixed together and alkaline solution was prepared.

To achieve higher workability and required flowability of the fresh concrete, a commercially available superplasticizer (Sika Viscocrete-3430) supplied by Sika Kimia Sdn Bhd, Malaysia, and a specified amount of extra water (other than the water used for the preparation of sodium hydroxide solution) was also used.

TABLE I
CHEMICAL COMPOSITION OF FLY ASH AS DETERMINED BY XRF

Oxide	(%) by mass
Silicon dioxide (SiO_2)	51.3
Aluminum oxide (Al_2O_3)	30.1
Ferric oxide (Fe_2O_3)	4.57
Total $SiO_2 + Al_2O_3 + Fe_2O_3$	85.97
Calcium oxide (CaO)	8.73
Phosphorus pentoxide (P_2O_5)	1.6
Sulphur trioxide (SO_3)	1.4
Potassium oxide (K_2O)	1.56
Titanium dioxide (TiO_2)	0.698

B. Design of Mix Proportion

In this experimental work, a mix proportion with the content of 400 kg/m^3 of fly ash was designed to study the effect of curing time and curing temperature on the compressive strength of self-compacting geopolymer concrete. Four levels of curing time i.e. 24, 48, 72 and 96 hours and four ranges of curing temperature i.e. 60°C, 70°C, 80°C and 90°C were used. The details of the mix proportion are given in Table II. In order to attain required workability characteristics of self-compacting geopolymer concrete, a water content of 12% and superplasticizer dosage of 7% by mass of the fly ash were also used. The alkaline solution-to-Fly ash ratio was kept 0.5 whereas the ratio of sodium silicate to sodium hydroxide and concentration of sodium hydroxide were 2.5 and 12 M respectively.

TABLE II
DETAILS OF MIX PROPORTION

Fly Ash (Kg/m^3)	400
Fine Aggregate (Kg/m^3)	850
Coarse Aggregate (Kg/m^3)	950
Sodium Hydroxide (Kg/m^3)	57
Concentration of NaOH Solution (Molarity)	12
Sodium Silicate (Kg/m^3)	143
Super plasticizer (%)	7
Extra water (%)	12
Sodium Silicate/Sodium hydroxide by mass	2.5
Alkaline to Fly ash ratio	0.5
Curing time (hrs)	24-96
Curing temperature (°C)	60-90

C. Mixing Procedure

Mixing was carried out in two stages. Initially, Fly ash, Fine aggregate (in dry condition) and coarse aggregate (in saturated surface dry condition) were mixed in a pan mixture for about 2.5 minutes. At the end of this mixing, the liquid component of the geopolymer concrete mixture comprising alkaline solution, superplasticiser and the extra water, was added to the solid particles and the mixing continued for another 3 minutes. To ensure the mixture homogeneity, fresh concrete mix was hand mixed for further 2 to 3 minutes. The freshly prepared concrete mix was then assessed for the essential workability tests required for characterizing self-compacting concrete (SCC). Tests such as slump flow, slump flow at T_{50}, V-funnel, L-box, and J-ring were performed for this purpose.

D. Casting and Curing of Test Specimens

After assessing the necessary workability properties as guided by EFNARC [11], the fresh concrete was placed in steel moulds of dimensions 100 x 100 x 100 mm and allowed to fill all the spaces of the moulds by its own weight. Three cubes were prepared for each test variable. After casting the moulds, without any delay, they were kept in the oven at a specified temperature for a specified period of time in accordance with the test variables selected. At the end of the curing period, the moulds were taken out from the oven and left undisturbed for about 15 minutes. The test specimens were removed from the moulds and left to air dry in the room temperature conditions until tested for direct compression at the specified age.

III. RESULTS AND DISCUSSION

A. Fresh Properties of SCGC

The properties of fresh SCC differ significantly from that of conventional fresh concrete. There are three distinct fresh properties of SCC which are fundamental to its performance both in fresh and hardened state. According to EFNARC [11], a concrete mix can only be classified as SCC if the requirements for all the three workability properties are fulfilled. The three essential fresh properties required by SCC are filling ability, passing ability and resistance to segregation. To accomplish the workability properties, tests such as slump flow, slump flow at T50, V-funnel, L-box, and J-ring were carried out. All the tests were performed by following the European Guidelines for SCC. The test results of fresh properties of SCGC are presented in Table III. The results of the quantitative measurements and visual observations showed that freshly prepared concrete mix had good flow, filling & passing ability, and produced desired results and were within the EFNARC range of SCC.

TABLE III
WORKABILITY TEST RESTULTS

Workability Test Results		Acceptance Criteria for SCC As per EFNARC [11]	
		Minimum	Maximum
Slump flow (mm)	710	650 mm	800 mm
$T_{50\ cm}$ Slump flow (sec.)	4.0	2 sec.	5 sec.
V-Funnel Flow time (sec.)	7	6 sec.	12 sec.
L-Box (H_2/H_1)	0.96	0.8	1.0
J-Ring (mm)	5	0 mm	10 mm

B. Compressive Strength of SCGC

Compressive strength is one of the most common measures used to evaluate the quality of hardened concrete and is considered as the characteristic material value for the classification of concrete. Many researchers have used compressive strengths measurements as a tool to assess the success of geopolymerisation process [12]. Compressive strength test was performed in accordance with BS EN 12390-3:2002 using 2000 KN Digital Compressive & Flexural Testing Machine. At the end of specified oven curing period, a set of three cubes for each test variable was tested at the ages of 1, 3, 7, and 28 days. The compressive strength test results are presented in Table IV. The reported compressive strength is the average strength of three specimens.

TABLE IV
COMPRESSIVE STRENGTH TEST RESTULTS

Mix	Compressive Strength Test Results			
	1-Day	3-Day	7-Day	28-Day
	(MPa)			
S1	45.01	45.85	46.94	48.53
S2	51.03	51.98	52.26	53.80
S3	51.41	52.20	52.69	53.92
S4	51.68	52.33	52.72	53.99
S5	44.81	45.64	45.98	47.54
S6	51.03	51.98	52.26	53.80
S7	48.56	49.22	49.80	50.77
S8	47.99	48.83	49.67	50.42

1. Effect of Curing Time on Compressive Strength

Fig. 1 shows the influence of curing time on the compressive strength of self-compacting geopolymer concrete. The test specimens were cured in the oven at a temperature of 70°C. The curing time varied from 24 hours to 96 hours (4 days). It is believed that longer curing time improved the geopolymerisation process resulting in higher compressive strength. The test results shown in Fig. 1 indicate that the compressive strength increases with increase in curing time. The test specimens cured at 70°C for a period of 96 hours produced the highest compressive strength at all ages. The rate of increase in strength was rapid up to 48 hours of curing time; however, the gain in strength beyond 48 hours is not significant. The results shown in Fig. 1 clearly demonstrate that longer curing time does not produce weaker material as claimed by van Jaarsveld et al [7]. The trend of these test results is similar to those observed by Hardjito et al. [13] in their study on Fly ash-based geopolymer concrete.

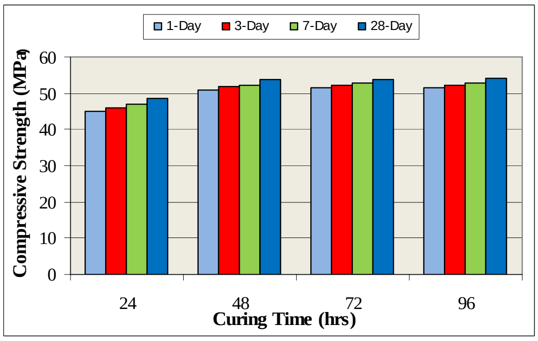

Fig. 1 Effect of Curing Time on Compressive Strength

2. Effect of Curing Temperature on Compressive Strength

Curing temperature plays a significant role in the setting and hardening of the geopolymer concrete [2]. Hardjito et al. [14], in their study on low-calcium Fly ash-based geopolymer mortar have reported that curing temperature plays an important role in the geopolymerization process of Fly ash-based geopolymer. They have concluded that higher the curing temperature, higher will be the rate of geopolymerization process, which eventually accelerates the hardening of geopolymer mortar.

The effect of curing temperature on the compressive strength is illustrated by the test data shown in Fig. 2. As curing of fly ash-based geopolymer concrete is usually carried out at an elevated temperature in the range of 60-90°C; therefore, in this experimental study the curing temperature was varied from 60 to 90°C. The test specimens were cured in the oven at a temperature of 60, 70, 80 and 90°C for a period of 48 hours. All the other test parameters were kept constant.

Compressive strength results shown in Fig. 2 indicate that higher curing temperature does not ensure higher compressive strengths as claimed by Hardjito et al. [15] in their study on Fly ash-based geopolymer concrete. Test results show that an increase in the curing temperature from 60°C to 70°C increases the compressive strength of the concrete. However, increasing the curing temperature beyond 70°C decreases the compressive strength of self-compacting geopolymer concrete.

It is believed that increase in the curing temperature from 60°C to 70°C increased the rate and extent of reaction through an increase in the heat of reaction; consequently increased the compressive strength of the concrete.

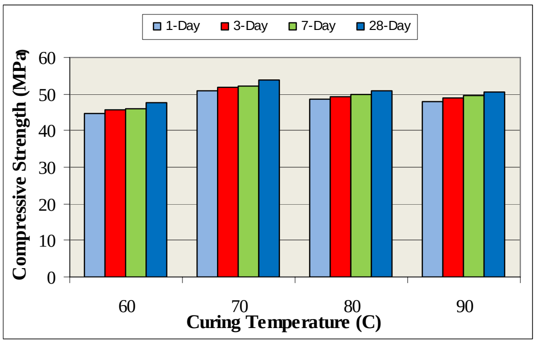

Fig. 2 Effect of Curing Temperature on Compressive Strength

V. CONCLUSION

In this experimental work, the effect of curing conditions on the compressive strength of fly ash-based self-compacting geopolymer concrete was investigated. Test results indicate that curing time and curing temperature significantly affect the compressive strength of hardened concrete. Based on the test results reported here, the following conclusions can be drawn.

1. Longer curing time improves the geopolymerisation process resulting in higher compressive strength. Increase in compressive strength was observed with increase in curing time. The compressive strength was highest when the specimens were cured for a period of 96 hours; however, the increase in strength after 48 hours was not significant.
2. Compressive strength of concrete increased with the increase in curing temperature from 60°C to 70°C; however an increase in the curing temperature beyond 70°C decreased the compressive strength of self-compacting geopolymer concrete.

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