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EFFECT OF SODIUM HYDROXIDE CONCENTRATION ON FRESH PROPERTIES AND COMPRESSIVE STRENGTH OF SELF-COMPACTING GEOPOLYMER CONCRETE

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Abstract

This paper reports the results of the laboratory tests conducted to investigate the effect of sodium hydroxide concentration on the fresh properties and compressive strength of self-compacting geopolymer concrete (SCGC). The experiments were conducted by varying the concentration of sodium hydroxide from 8 M to 14 M. Test methods such as Slump flow, V-Funnel, L-box and J-Ring were used to assess the workability characteristics of SCGC. The test specimens were cured at 70°C for a period of 48 hours and then kept in room temperature until the day of testing. Compressive strength test was carried out at the ages of 1, 3, 7 and 28 days. Test results indicate that concentration variation of sodium hydroxide had least effect on the fresh properties of SCGC. With the increase in sodium hydroxide concentration, the workability of fresh concrete was slightly reduced; however, the corresponding compressive strength was increased. Concrete samples with sodium hydroxide concentration of 12 M produced maximum compressive strength.

Keywords: Geopolymer concrete, Self-compacting geopolymer concrete, Workability, Compressive strength, Sodium hydroxide concentration

1. Introduction

With the increased use of cement in concrete, there have been environmental concerns both in terms of damage caused by the extraction of raw materials and emission of carbon dioxide during cement manufacture. This has brought pressures to reduce the cement consumption in the construction industry. An attempt in this regard is the development of geopolymer concrete. Geopolymer

Nomenclatures

Al_2O_3	Aluminium oxide
CaO	Calcium oxide
Fe_2O_3	Ferric oxide
K_2O	Potassium oxide
NaOH	Sodium hydroxide, M
Na_2O	Sodium oxide
P_2O_5	Phosphorus pentoxide
SiO_2	Silicon dioxide
SO_3	Sulphur trioxide
TiO_2	Titanium oxide

Abbreviations

ASTM	American Society of Testing and Materials
EFNARC	European federation of national trade associations representing producers and applicators of specialist building products
M	Molarity
SCC	Self-Compacting Concrete
SCGC	Self-Compacting Geopolymer Concrete
SSD	Surface Saturated Dry
Std. Dev.	Standard Deviation
XRF	X-Ray Fluorescence

concrete is an innovative binder material and can be produced by synthesizing from materials of geological origin or from by-product materials, which are rich in silicon and aluminium with highly alkaline solution [1].

In geopolymerisation, alkaline solution plays an important role. The alkaline solutions are from soluble alkali metals that are either Sodium or Potassium based [2, 3]. The most frequent alkaline solution used in geopolymerisation is a combination of sodium hydroxide or potassium hydroxide and sodium silicate or potassium silicate [2, 4-6]. The choice of the alkaline solution mainly depends upon the reactivity and the cost of the alkaline solutions. Previous studies [4, 7-9] indicate that sodium silicate solution in combination with sodium hydroxide is an effective alkaline activator.

Palomo et al. [4], in their study on fly ash-based geopolymers have reported that the type of alkaline solution was the significant factor affecting the mechanical strength of geopolymers. They found that the combination of sodium silicate and sodium hydroxide produced the highest compressive strength.

Xu and Van Deventer [7] studied a wide range of aluminosilicate minerals to make geopolymers. They used sodium or potassium hydroxide as an alkaline activator. They have reported that the addition of sodium silicate solution to the sodium hydroxide solution as an alkaline activator enhanced the reaction between the source material and the alkaline solution. They found that generally the sodium hydroxide solution caused a higher extent of dissolution of minerals than the potassium hydroxide solution.

Research conducted by Fernandez-Jimenez, et al. [8] on the effects of alkaline solution on the final product of geopolymer has shown that a combination of sodium hydroxide and sodium silicate produced a solid material almost without pores and has a strong bond between aggregate and geopolymer matrix.

In a research study conducted by Hardjito et al. [9] on geopolymer concrete manufactured from low-calcium fly ash activated with sodium silicate and sodium hydroxide solution, the authors have reported higher compressive strength and better durability of geopolymer concrete compared to Portland cement concrete. They have demonstrated that a combination of sodium hydroxide and sodium silicate solutions can be a good application for activator in fly ash-based geopolymer concrete.

Concentration of sodium hydroxide is the most important factor for geopolymer synthesis [10]. The solubility of aluminosilicate increases with increase in hydroxide concentration [11]. The use of higher concentration of sodium hydroxide yield higher compressive strength of geopolymer concrete [9].

Hardjito et al. [12] conducted study on the effects of sodium hydroxide concentration on the compressive strength of fly ash-based geopolymer mortar. The authors have reported that alkaline concentration was proportionate to the compressive strength of geopolymer mortar. They have claimed that higher concentration of sodium hydroxide solution result in a higher compressive strength of geopolymer mortar.

Hongling Wang et al. [13], in their study on synthesis and mechanical properties of metakaolinite-based geopolymer have reported that higher concentration of sodium hydroxide solution provides better dissolving ability to metakaolinite and produces more reactive bond for the monomer, consequently increase inter-molecular bonding strength of the geopolymer. They have revealed that mechanical properties of the metakaolinite-based geopolymer activating metakaolinite with sodium hydroxide and sodium silicate solution were greatly dependent on the concentration of sodium hydroxide solution. With the increase of sodium hydroxide concentration, the compressive strength, flexural strength, and apparent density of the resulting geopolymer were increased.

Self-compacting Geopolymer concrete (SCGC) is relatively a new concept and can be regarded as the most revolutionary development in the field of concrete technology. SCGC is an innovative type of concrete that does not entail vibration for placing it and can be produced by complete elimination of ordinary Portland cement [14]. This research study aimed to investigate the effect of sodium hydroxide concentration on the fresh properties and compressive strength of SCGC made by using fly ash as source material and combination of sodium hydroxide and sodium silicate as alkaline activator.

2. Experimental Details

2.1. Materials

The materials used in this study were fly ash, fine and coarse aggregate, alkaline solution, superplasticizer and water.

2.1.1. Fly ash

Geopolymer concrete is produced by activating alumino-silicate based source material with an alkaline solution. Fly ash, which is rich in silica and alumina, has full potential to be used as one of the source material for geopolymer binder [15]. For this reason, fly ash has been chosen as a base material to synthesize geopolymer in order to better utilize this industrial waste by-product material.

In the present study, Low-calcium (ASTM Class F) fly ash obtained from Manjung Power Station, Perak, Malaysia was used as a source material for the synthesis of SCGC. The chemical composition of fly ash as determined by X-Ray Fluorescence (XRF) analysis is shown in Table 1.

Table 1. Chemical Composition of Fly Ash as determined by XRF.

Oxide	(%) by mass
Silicon dioxide (SiO_2)	51.3
Aluminium 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

2.1.2. Aggregates

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.

2.1.3. Alkaline solution

In geopolymerization, alkaline solution also plays an important role. The most common alkaline solution used in geopolymerisation is a combination of sodium hydroxide or potassium hydroxide and sodium silicate or potassium silicate [16]. In the present study, a combination of sodium hydroxide and sodium silicate was used as the alkaline solution.

Sodium hydroxide in pellets form with 99% purity, supplied by QuickLab Sdn Bhd, Malaysia and Sodium silicate solution (Grade A53 with SiO_2 = 29.43%, Na_2O = 14.26% and water = 56.31%) obtained from Malay-Sino Chemical Industries Sdn Bhd, Malaysia were used as the alkaline activators. In order to make sodium hydroxide solution, sodium hydroxide pellets were dissolved in potable water. Both the liquid solutions were then mixed together and alkaline solution was prepared.

2.1.4. Superplasticizer

To attain 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. The ordinary drinking water available in concrete laboratory was used for this purpose.

2.2. Mix proportions

In this experimental work, four different mixtures with the same content of fly ash (400 kg/m^3) were prepared to study the influence of sodium hydroxide concentration on fresh properties and compressive strength of SCGC. The details of the mix proportions are given in Table 2. Four levels of sodium hydroxide concentration i.e. 8 M, 10 M, 12 M and 14 M were used. Activation of aluminosilicate based materials with alkalis generally requires heat curing for the formation of alkali-activated binders. Concrete specimens were cured in the oven at 70°C for a period of 48 hours to complete geopolymerisation reaction. The temperature and curing time were selected based on the author's previous findings [14, 17]. In order to obtain the required workability characteristics of SCGC, a water content of 12% and superplasticizer dosage of 6% by mass of the fly ash was used for all mixes. For each mix, the alkaline solution-to-fly ash ratio was kept 0.5 whereas the ratio of sodium silicate to sodium hydroxide was kept 2.5.

Table 2. 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 sodium hydroxide solution (Molarity)	8-14
Sodium silicate (kg/m^3)	143
Superplasticizer (%)	6
Extra water (%)	12
Curing time (hrs)	48
Curing temperature (°C)	70

2.3. Mixing, casting and curing of SCGC

Initially, fly ash, dry fine aggregate and coarse aggregate in SSD condition were mixed in 100 litre capacity pan mixer for about 2.5 minutes. After dry mixing, a liquid mixture containing alkaline solution, superplasticizer and extra water was added in the mixer and the wet mixing was done for another 3 minutes. The freshly prepared concrete was then assessed for the essential workability tests required for characterizing self-compacting concrete (SCC). After ensuring the necessary workability requirements as guided by EFNARC [18], the fresh concrete was then cast in 100×100×100 mm steel moulds. Three cubes were prepared for each test variable. After casting the moulds, without any delay, they were kept in the oven and cured at a temperature of 70°C for a period of 48 hours.

At the end of the curing period, moulds were demoulded and test specimens were left to air-dry at room temperature until the day of testing.

3. Testing of Specimens

3.1. Fresh properties

The functional requirements on a fresh SCC are different from those on a vibrated fresh concrete. According to EFNARC [18], a concrete mixture can only be classified as SCC, if the requirements for its three key characteristics viz: filling ability, passing ability, and resistance to segregation are fulfilled. In the present study, the workability-related fresh properties of various SCGC mixes were measured using Slump flow, $T_{50\ cm}$ slump flow, V-funnel, L-box and J-Ring test methods. All the tests were performed by following the European guidelines for SCC [18].

3.2. Compressive Strength

Compressive strength is one of the most important parameters of 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 geopolymerization process [19]. In this study, compressive strength test was performed on 100×100×100 mm cubical specimens in accordance with BS EN 12390-3:2002 using 2000 KN Digital Compressive & Flexural Testing Machine. The test cube was subjected to a compressive force at the rate of 3.0 kN/s until it failed. At the end of specified oven curing period, a set of three cubes for each test variable were tested at the age of 1, 3, 7 and 28 days.

4. Results and Discussion

To study the effect of sodium hydroxide concentration on the fresh properties as well as on compressive strength of SCGC, four concrete mixtures M_1 (8M), M_2 (10M), M_3 (12M) and M_4 (14M) were prepared. All the other test parameters were kept constant. The experimental results of various fresh properties and compressive strength test are given in Tables 3 and 4, respectively.

4.1. Effect of sodium hydroxide concentration on fresh properties

The results of fresh properties of various SCGC mixes containing different proportions of sodium hydroxide are presented in Table 3 and shown in Figs. 1-5. Test results show that concentration variation in sodium hydroxide between 8 M to 14 M had least effect on the fresh properties of SCGC. An increase in the concentration of sodium hydroxide increased the viscosity of the solution. It was observed that concrete mixes containing higher concentration of sodium hydroxide were more cohesive and fluidity and flowability of SCGC mixes was reduced when the proportion of sodium hydroxide was increased. This is in line with the results of the study conducted by konda et al. [20]. Their study indicated that workability of freshly prepared low-calcium fly ash-based geopolymer concrete was decreased with the increase in the concentration of sodium hydroxide from 10 M to 16 M.

Table 3. Fresh Properties.

Mix ID	Slump flow (mm)	$T_{50\,cm}$ Slump flow (sec.)	V-Funnel flow time (sec.)	L-Box (H_2/H_1) ratio	J-Ring Blocking step (mm)
M_1 (8M)	700	4.0	9.5	0.96	5
M_2 (10M)	690	4.0	10	0.95	6
M_3 (12M)	690	4.5	10	0.94	7
M_4 (14M)	675	5.0	12	0.90	9
Acceptance Criteria for SCC as per EFNARC [18]					
Min.	650 mm	2 sec.	6 sec.	0.8	0 mm
Max.	800 mm	5 sec.	12 sec.	1.0	10 mm

Table 4. Compressive Strength Test Results.

Mix ID	Compressive Strength (MPa)							
	1-Day	Std. Dev.	3-Day	Std. Dev.	7-Day	Std. Dev.	28-Day	Std. Dev.
M_1 (8M)	41.45	1.32	42.14	1.71	43.62	1.21	44.87	1.40
M_2 (10M)	45.19	1.07	46.02	0.38	47.32	1.05	49.28	0.94
M_3 (12M)	47.83	1.35	48.52	1.01	49.44	0.78	51.52	0.72
M_4 (14M)	46.96	1.03	47.64	0.82	48.98	1.19	50.46	0.73

The effect of sodium hydroxide concentration on individual fresh property tests is discussed in the subsequent paragraphs.

4.1.1. Slump flow test

This is the most commonly used test and gives a good assessment of filling ability. This test was performed to assess the horizontal free flow of fresh concrete. The results of the slump flow test are shown in Fig. 1. All the four mixtures showed almost good flowability and displayed good resistance to segregation. The test results shown in Fig. 1 indicate that the slump flow for all the mixes was within the EFNARC range of 650-800 mm [18]. A maximum slump flow value of 700 mm was achieved for a mix having sodium hydroxide molarity as 8 M. With the increase in concentration of sodium hydroxide between 8 M to 14 M, the viscosity of the solution was increased. As a result, the flow of the concrete was decreased.

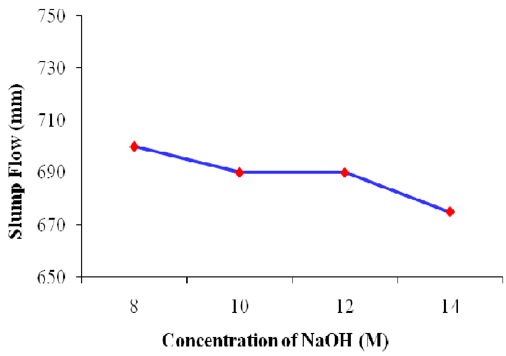

Fig. 1. Effect of Sodium Hydroxide Concentration on Slump Flow.

4.1.2. $T_{50\ cm}$ slump flow

This test gives an indication of the relative viscosity and provides a relative assessment of the unconfined flow rate of the SCC mixture. Figure 2 shows the results of the $T_{50\ cm}$ Slump flow. Test results of $T_{50\ cm}$ Slump flow shows that all the four mixes qualified the permissible limits (2-5 seconds) given by EFNARC [18]. A lowest slump flow time of 4 seconds was recorded for mixes containing sodium hydroxide molarity as 8 and 10. An increase in the quantity of sodium hydroxide increased the viscosity and reduced the fluidity of concrete which in turn resulted to the increase in T_{50} time.

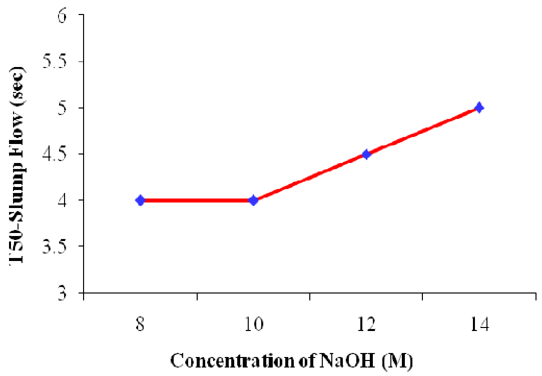

Fig. 2. Effect of Sodium Hydroxide Concentration on T_{50}-Slump Flow.

4.1.3. V-funnel test

This test is primarily used to measure the filling ability (flowability) of SCC and can also be used to evaluate the ability of concrete to flow through a continuously reducing section without segregation and blocking. To measure the filling ability and assess the segregation resistance of the freshly prepared concrete, all the four mixes with varying concentration of sodium hydroxide were tested by V-funnel test. Figure 3 illustrates the results of the V-funnel test. Although the results of V-funnel test for all the mixes were within the permissible limits given by EFNARC [18], however, most of the results remained towards the upper limit of 12 seconds. A minimum flow time of 9.5 seconds was recorded for mix with sodium hydroxide concentration as 8 M. With the increase in concentration of sodium hydroxide, the fluidity and flowability of concrete was decreased. Consequently, the V-funnel flow time was increased.

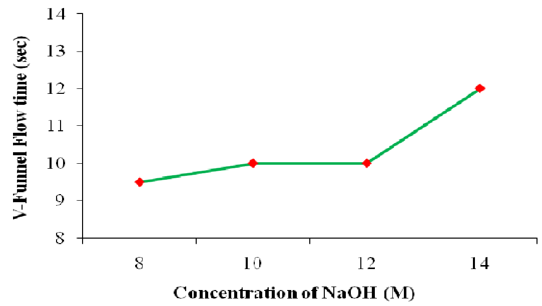

Fig. 3. Effect of Sodium Hydroxide Concentration on V-Funnel Flow Time.

4.1.4. L-box test

This test is used to assess the filling and passing ability of SCC. Figure 4 shows the results of the L-box test. While assessing the fresh concrete for passing ability, it was observed that all the four mixes passed through the bars of L-box very easily and no blockage was seen in any of the mixes. Test results indicate that all the four mixes produced desired results and were within the EFNARC range of 0.8-1 [18]. The results of L-box test show that the blocking ratio (H_2/H_1) was gradually decreased with the increase in the concentration of sodium hydroxide. The same reasons and mechanism mentioned for slump flow and V-funnel test results are also commanding the explanations for the results of L-box test.

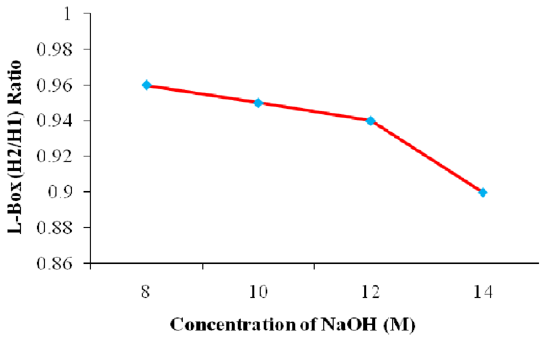

Fig. 4. Effect of Sodium Hydroxide Concentration on L-Box (H_2/H_1) Ratio.

4.1.5. J-ring test

This test is used to determine the passing ability of the SCC. Figure 5 shows the results of the J-Ring test. The results of the quantitative measurements and visual observations showed that all the four mixes had good passing ability and the J-Ring value for all the mixes was within the permissible limits of 0-10 mm given by EFNARC [18]. A lowest value of 5 mm was achieved for the mix containing 8M sodium hydroxide concentration. With the increase in concentration of sodium hydroxide, the flowability and passing ability of fresh concrete was reduced. As a result, J-Ring value was also increased.

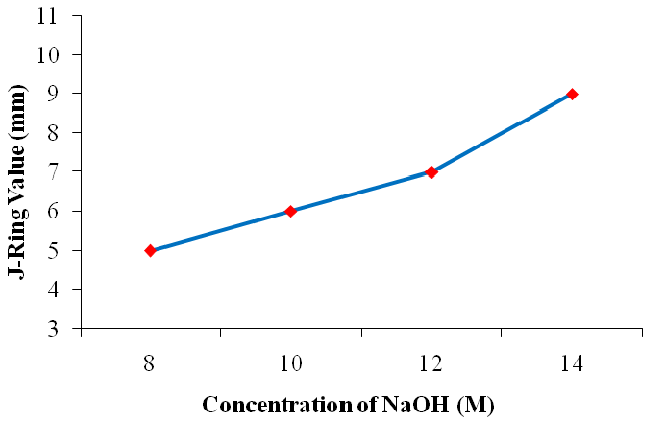

Fig. 5. Effect of Sodium Hydroxide Concentration on J-Ring Value.

4.2. Effect of sodium hydroxide concentration on compressive strength

In geopolymer synthesis, concentration of sodium hydroxide significantly affects both the compressive strength and micro structure of geopolymers [21]. The sodium hydroxide concentration in the aqueous phase of the geopolymeric system acts on the dissolution process, as well as on the bonding of solid particles in the final structure [22]. The use of high concentration of sodium hydroxide leads to greater dissolution of the initial solid materials and increases geopolymerization reaction and hence higher compressive strength is achieved [23].

Figure 6 illustrates the effect of sodium hydroxide concentration on the compressive strength of concrete. The test results shown in Fig. 6 demonstrate that the compressive strength of SCGC increases with the increase in the concentration of sodium hydroxide. Compressive strength of concrete specimens increases as sodium hydroxide concentration in the aqueous phase increases from 8 to 12 M; however, it decreases with the further increase in sodium hydroxide concentration. It is accepted that an increase in alkali concentration enhanced geopolymerization process resulting to an increase in the compressive strength of SCGC. However, excess hydroxide ion concentration caused aluminosilicate gel precipitation at the very early stages, and subsequent geopolymerization was hindered, resulting in lower strength [24]. This is in line with the results of the study conducted by Alonso and Palomo [25]. Their study indicated that when activator concentration increased above 10 M, a lower rate of polymer formation was produced resulting in the decrease of mechanical strength.

Similar pattern of results is reported by Mustafa et al. [26], who investigated the effect of six different concentration of sodium hydroxide (6M, 8M, 10M, 12M, 14M, 16M) on fly ash-based geopolymer paste. The test samples were cured at 70°C for a period of 24 hours. The authors have reported that test specimens with sodium hydroxide concentration of 12 M produced maximum compressive strength. However, Hardjito et al. [12] have reported that alkaline concentration was proportionate to the compressive strength of geopolymer mortar. They have stated that higher concentration of sodium hydroxide solution resulted in a higher compressive strength of samples.

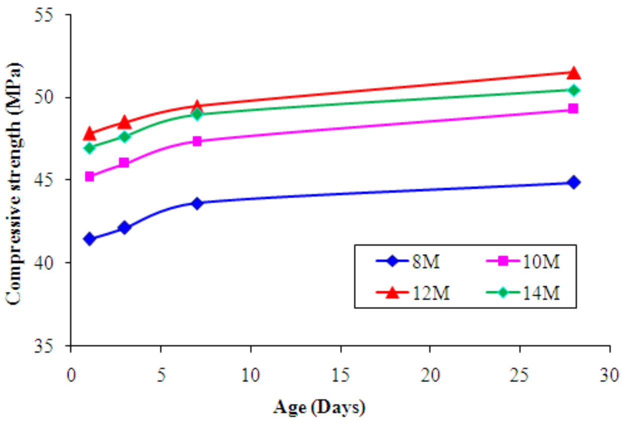

Fig. 6. Effect of Sodium Hydroxide Concentration on Compressive Strength.

4. Conclusions

In this experimental work, the effect of sodium hydroxide concentration on fresh properties and compressive strength of fly ash-based SCGC was investigated. The results of the quantitative measurements and visual observations showed that concentration variation in sodium hydroxide had least effect on the fresh properties of SCGC. Addition of sodium hydroxide from 8 M to 14 M in various SCGC mixes resulted in only 3.6% reduction in the slump flow (from 700 mm to 675mm). An increase in the concentration of sodium hydroxide from 8 M to 14 M, although increased the viscosity and cohesiveness of concrete and reduced the fluidity and flowability of various SCGC mixes, nevertheless, all the four concrete mixes still met the requirements of SCC suggested by EFNARC. In contrast, the concentration of sodium hydroxide was found to have a substantial effect on the compressive strength of SCGC. Based on the test results reported here, it can be concluded that the increased sodium hydroxide concentration in the aqueous phase would have positive, as well as negative effects on the mechanical properties of the SCGC. A higher concentration of sodium hydroxide in the aqueous phase proved to have positive effect on geopolymerization process and this is revealed by the improved compressive strength of SCGC. However, an increase in the sodium hydroxide concentrations beyond 12 M caused negative effect on the geopolymerization resulting in lower the compressive strength of SCGC.

Test results indicated that compressive strength of SCGC is not a monotonous function of sodium hydroxide concentration. Compressive strength of SCGC increased as sodium hydroxide concentration in the aqueous phase was increased from 8 to 12 M. Further increase of sodium hydroxide concentration decreased the compressive strength of SCGC. At 28-day, mixture M_1 (8M) achieved a compressive strength of 44.87 MPa, whereas mixtures M_2 (10M), M_3 (12M) and M_4 (14M) achieved a compressive strength of 49.28, 51.52, and 50.46 MPa, respectively; an increase of 9.8%, 14.8% and 12.4% in comparison with the strength of mixture M_1 (8M).

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