

Effect of Superplasticizer and NaOH Molarity on Workability, Compressive Strength and Microstructure Properties of Self-Compacting Geopolymer Concrete

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Abstract—The research investigates the effects of superplasticizer and molarity of sodium hydroxide alkaline solution on the workability, microstructure and compressive strength of self-compacting geopolymer concrete (SCGC). SCGC is an improved way of concreting execution that does not require compaction and is made by complete elimination of ordinary Portland cement content. The parameters studied were superplasticizer (SP) dosage and molarity of NaOH solution. SCGC were synthesized from low calcium fly ash, activated by combinations of sodium hydroxide and sodium silicate solutions, and by incorporation of superplasticizer for self compactability. The workability properties such as filling ability, passing ability and resistance to segregation were assessed using slump flow, T-50, V-funnel, L-Box and J-ring test methods. It was found that the essential workability requirements for self compactability according to EFNARC were satisfied. Results showed that the workability and compressive strength improved with the increase in superplasticizer dosage. An increase in strength and a decrease in workability of these concrete samples were observed with the increase in molarity of NaOH solution from 8M to 14M. Improvement of interfacial transition zone (ITZ) and micro structure with the increase of SP and increase of concentration from 8M to 12M were also identified.

Keywords—Compressive strength, Fly ash, Geopolymer concrete, Workability

I. INTRODUCTION

CONCRETE is one of the vital materials for infrastructure development due to its versatile application, globally its usage is second to water. For last many years, there are many concerns raised for the continuous increase of cement use because of the reasons that the production of cement causes large amount of carbon dioxide (CO₂) emission and it also consume significant amount of natural rock and minerals that

may lead to deplete at one point of time. Manufacture of one ton of Portland cement (PC) generates about one ton of CO₂ to the atmosphere which constitutes 5% global CO₂ emission [1]. It is reported that the global production of PC contributes about 1.35 billion tons greenhouse gas emissions annually [2], [3]. Due to the manufacture of PC, the CO₂ emission is likely to rise by about 50% from the current levels in 2020 [4]. Recently, to reduce the environmental impact due to cement production, a type of binder is produced from an aluminosilicate precursor activated in high alkali solution. This cementitious binder is known as geopolymer cement.

Many efforts have been done to minimize the use of cement as a binder in concrete production. One of the pozzolanic materials that has been introduced in the construction industry is the fly ash (FA) [2], which is a by-product from coal-fired electric and steam generating plants. References [5] and [6] developed a geopolymer concrete (GC) using the FA as the base material. Prior the introduction of FA as a source material, metakaolin was used as the base material in many studies [7]-[9], however since last decade, much research has been done using FA because it contains high amount of alumina and silica content. The works done on geopolymer technology [5], [7], [10], [11] shows a significant potential for its utilization in concrete industry, particularly low-calcium FA. Consequently, the use of geopolymer as binder in concrete production not only resulted to reduce the CO₂ emission because of elimination of cement, but also utilizes the industrial by-products of aluminosilicate materials to produce environmental friendly construction material [1]-[10]. Placement of fresh concrete in the form-work requires compaction efforts and also involves skilled labor. This compaction primarily aims to minimize the entrapped air in fresh concrete in order to obtain homogeneous mix with no cavities (honey-comb) [11]. While the concrete is placed and compacted at the construction site, normal vibrating concrete may unable to exhibit the required fresh and hardened properties. To obtain adequate compaction in freshly mixed concrete, skilled labor is required. One solution to overcome this problem is the employment of self compacting concrete (SCC) [12]. SCC transforms the concreting operation by complete elimination of vibration during compaction and allows the concrete to flow through sections with congested reinforcement under its own weight alone, filling the

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formwork without segregation of its constituent materials. Such concrete needs a high slump flow [13] that can easily be obtained by superplasticizer addition to a concrete mix and carefully controlled mix proportion. SCC was developed in Japan in the late 1980s because of shortage of skilled labor and emergence of heavily reinforced structure [12]. References [14] and [15] studied the properties of SCC made with low calcium fly ash (Class F). They made experimental investigation on workability, structural and durability properties of self compacting concrete by replacing PC with fly ash up to 35% and 80% respectively. The results showed that SCC made with fly ash increased the workability and enhanced the hardened properties. Adequate compaction of fresh concrete is essential to achieve good consolidation, uniform properties, better quality and durability [16], strong bond with reinforcement [17] and improved interface between the aggregate and hardened paste [18], and enhanced microstructure of concrete. This paper presents the test results of behavior of SCGC in fresh and hardened states containing Class F fly ash to identify the optimized mix proportion and the main objective of this study is to investigate the effects of superplasticizer dosage and molarity of alkaline solution on workability and compressive strength and microstructure properties of SCGC.

II. METHODOLOGY

A. Material Selection

1. Fly Ash

In the experimental work, materials were selected according to the specifications that meet the requirements of British Standards and EFNARC guidelines [11].

Dry low-calcium fly ash obtained from thermo electric power station was used as the base material. American Standard Testing and Material (ASTM C618) classify fly ash into Class F and C depending mainly on CaO content and the fly ash used in the research was Class F with chemical composition, as determined by X-Ray Florescence (XRF) analysis, given in Table I.

2. Aggregates

Coarse aggregate used in this research was crushed granite stone with maximum size of 14 mm (BS 812-103.2 1989). The specific gravity of coarse aggregate is 2.66 with SSD condition while the fine aggregate used is dry clean natural Malaysian sand with the fineness modulus of 2.76, maximum size of 5mm and a specific gravity of 2.61.

3. Alkaline Solution

Alkaline solution plays an important role in geopolymer synthesis for the dissolution of silica and alumina as well as for the catalysis of polymerization reaction [19]. In this experiment, a combination of sodium silicate and sodium hydroxide was chosen as the alkaline liquid.

Na₂SiO₃ (Grade A53) used with a composition of 55.52% water, 29.75% SiO₂ and 14.73% Na₂O. NaOH (99% purity, in the form of pellets) was dissolved in distilled water to avoid the effect of unknown contaminants in the mixing water. The

activator alkaline solution was prepared at least one hour prior to its use. The different concentration NaOH solution was 8M, 10M, 12M & 14M and in order to make 1 Kg of solution, 29.4%, 36.7%, 44.1% and 51.4% of pellets were added to the water respectively.

Super plasticizer (Sika Visco Crete-3430) was used to increase the workability to the extent required for self compactability of Geopolymer Concrete. The utilization of Viscosity Modifying Admixture (VMA) gives more possibilities of controlling segregation (stability) and homogeneity of the mix [20]. The amount of SP used was in accordance with EFNARC 2002 [20]. The water used in the mix was tap water in accordance with B.S. EN 1008:1997.

TABLE I
 CHEMICAL COMPOSITION OF LOW CALCIUM FLY ASH (LCFA) [25]

Compounds	Mass (%)	Requirement as per BS EN 450-1:2005
SiO ₂	51.3	min. 25%
Al ₂ O ₃	30.1	-
Fe ₂ O ₃	4.57	-
SiO ₂ +Al ₂ O ₃ + Fe ₂ O ₃	85.9	min. 70%
CaO	8.73	max. 10%
P ₂ O ₅	1.6	-
SO ₃	1.4	max. 3%
K ₂ O	1.56	-
TiO ₂	-	-

B. Mixing Method

The concrete mixing procedure consists of dry and wet mixings. The solids components of SCGC, i.e. the fly ash and the fine and coarse aggregates, were dry mixed in the pan mixer for about 2.5 minutes. The liquid part of the mixture, i.e. the sodium silicate solution, the sodium hydroxide solution, extra water and the super plasticizer, were premixed thoroughly and then added to the dry mixture. The wet mixing was done for 3 minutes. It was believed that the chemical reaction between alkaline solution, super plasticizer and water took place and the reaction played an important role in giving the required workability for SCC and compressive strength of hardened concrete. The fresh SCGC had a flowing consistency and with high tendency of filling ability, passing ability and resistance to segregation.

C. Casting and Curing

The fresh concrete was filled in 100mmx100mmx100mm steel moulds and allowed to fill all the spaces of the moulds by its self weight (no need to vibrate for compaction). After casting the specimens including the moulds were kept in an oven at a temperature of 70°C for 48 hours duration. The specimens were placed outside the room but protected from direct sunlight and rain and then the specimens were demoulded

TABLE II
MIX DESIGN PROPORTION

Mix Sample	Fly Ash Kg/m ³	Coarse Agg. Kg/m ³	Fine Agg. Kg/m ³	NaOH		Na-Silicate		Extra Water		SP		Curing	
				Kg/m ³	Molarity	Kg/m ³	Kg/m ³	%	Kg/m ³	%	Time hr.	Temp. °C	
S1	400	950	850	57	12	143	48	12	12	3	48	70	
S2	400	950	850	57	12	143	48	12	16	4	48	70	
S3	400	950	850	57	12	143	48	12	20	5	48	70	
S4	400	950	850	57	12	143	48	12	24	6	48	70	
S5	400	950	850	57	12	143	48	12	28	7	48	70	
S6	400	950	850	57	8	143	48	12	24	6	48	70	
S7	400	950	850	57	10	143	48	12	24	6	48	70	
S8	400	950	850	57	14	143	48	12	24	6	48	70	

and tested for direct compression in a digital 2000KN Compression testing machine. The ambient curing preceded by oven curing was adopted for this research to accelerate polymeric reaction at elevated temperature and to improve the compressive strength performance as claimed by Nuruddin et al [21]. The reported compressive strength is the average strength of three specimens. Analysis on ITZ and microstructure properties was conducted using field emission scanning electron microscopy (FESEM) on 28 days concrete sample.

D. Mix Proportion

The mix design proportion adopted in the research and details of these mixtures are shown in Table II. The ratio of sodium silicate to sodium hydroxide solution by mass was 2.5 for all mixture proportion. The mass ratio of fine aggregate to fly ash was 2.125 for all mixture. Mix Samples S₁, S₂, S₃, S₄, and S₅ were prepared to study the effects of superplasticizer dosage on the fresh workability and hardened compressive strength of SCGC. The designed SP dosages were 3%, 4%, 5%, 6% and 7% and all the other test parameters were held constant while the SP dosage varied. Mixes S₄, S₆, S₇ and S₈ were prepared to study the influence of molarity of alkaline solution on the workability and compressive strength of SCGC. The different molarity of NaOH solution was setted as 8M, 10M, 12M and 14M respectively. All the other test parameters were kept constant while the molarity varied.

E. Test Procedure

A concrete mix can only be considered as SCC if the three characteristics for workability are satisfied. The three fresh concrete characteristics mandatory for SCC are filling ability, passing ability and resistance to segregation.

Different test methods have been developed in attempts to measure the three properties of SCC; however, so far no single standard methods is capable of determining all the relevant workability aspects at a time so each mix design should be assessed by more than one test method for the different workability characteristics. Filling ability and passing ability can be measured by the test methods as shown

in Table III. Resistance to segregation can be assessed more or less in all the tests based on observation through visual stability. The European Guidelines EFNARC has proposed different test methods to characterize an SCC mix. Table III shows the test methods and property along with their recommended values given by EFNARC.

TABLE III
TEST METHODS, PROPERTY AND RECOMMENDED VALUES AS PER EFNARC GUIDELINES [20]

S.No.	Methods	Property	Acceptance values as per EFNARC Guide lines	
			Minimum	Maximum
1	Slump flow by Abrams cone	Filling Ability	650mm	800mm
2	T _{50cm} Slump flow	Filling Ability	2s	5s
3	V-funnel	Filling Ability	6s	12s
4	L-Box (H ₂ /H ₁ , Ratio)	Passing Ability	0.8	1.0
5	J-Ring	Passing Ability	0mm	10mm

In this research, the mixes underwent slump flow, T-50, V-funnel, L-Box & J-Ring tests to ascertain their self-compacting capabilities. All those tests are in accordance with EFNARC guidelines. The hardened compressive strength test was performed on one day after curing period in accordance with BS EN 12390-3:2002 using 2000 KN Digital Compressive Testing Machine in the Concrete Laboratory of Civil Engineering Department, Universiti Teknologi PETRONAS. A set of three cubes for each mix were tested for compressive strength measurement.

III. RESULTS AND DISCUSSION

In this section, the experimental results of various fresh properties tested by slump flow test (slump flow diameter and T_{50cm}), J-ring test (J-ring Blocking step (B_j)); L-box test (ratio of heights at the two edges of L-box (H₂/H₁)); V-funnel test (time taken by concrete to flow through V-funnel after 10 s T_{10s}); for various mix compositions are discussed. The results of the workability tests are given in Table IV.

A total of 8 mixtures were made to study the influence of various parameters on the workability and compressive strength. Workability is the main parameter that characterizes SCC as superior workable in attaining self-consolidation and required hardened properties. All the workability tests were performed as per European guide lines EFNARC 2002 for SCC. The test results of the quantitative analysis and visual observations showed that except for Mix Samples S₁, S₂ and S₃, all the other concrete mix samples had the desired fresh properties and were within the EFNARC limits of SCC. The mean compressive strength of the three test cubes for all mix composition is presented in Table IV.

A. Supreplasticizer Dosage, Percentage by Mass

The first five Mixtures S₁, S₂, S₃, S₄ and S₅ have identical mix composition, but different superplasticizer dosages of 3%, 4%, 5%, 6% and 7% respectively. The difference is the amount of superplasticizer added to the mix. The concentration of sodium hydroxide solution was held constant at 12M and 12% extra water by mass for all mix. These mixtures were prepared to study the effects of SP dosage on workability and compressive strength of SCGC. From Table IV, it can be seen that that Mix S₅ with SP dosage of 7% shows highest compressive strength as compared to the other mixes that have SP dosage of 3%, 4%, 5%, 6% and 7%. Mix samples S₁, S₂ and S₃ failed to exhibit the required workability property for SCC due to the lower percentage of superplasticizer. Fig. 1 shows that the maximum performance at 7% SP dosage for all ages. Also the maximum compressive strength achieved at 28 days of age, which is, 53.80MPa. SP is required in geopolymer concrete to improve workability and enhance hardened properties of SCGC such as microstructure and compressive strength. The condensation polymerization that takes place is endothermic in nature therefore supply of heat is required. This process is different from OPC based concrete as geopolymer concrete does not utilize water in its polymeric reaction. Water in the mix plays a vital role in synthesis and acts as a medium for dissolution, condensation and polymerization of Al and Si precursors into polymeric structures [5]. This in turn helps the mixing and casting process to increase the workability fresh concrete along with superplasticizer. During the curing process at elevated temperature, the water was expelled and evaporated from hardened concrete sample. Spaces that were formerly occupied by water remained as micropores within the concrete. These pores resulted a microcrack path which can led to the premature failure of concrete at lower stress level when exposed to compressive load, hence resulting in low compressive strength performance. Fig. 1 shows that as the SP dosage in the mix increased, the compressive strength increased. This was due to the more effective action of the superplasticizer in increasing the workability of the geopolymer concrete. Fig. 1 shows that 7% SP produced the highest compressive strength of 53.80MPa at 28 days after curing. There is a slight increase in the compressive strength for Mix contained 7% SP as compared to Mix with 6% SP.

The results indicated that SCGC with 7% SP resulted in a very small increase in the compressive strength than SCGC with 6% SP. SP dosage of 6% was taken as the optimum due to the fact satisfactory performance was obtained in both fresh and hardened SCGC. This phenomenon is important for the construction industry as far as economy is concerned.

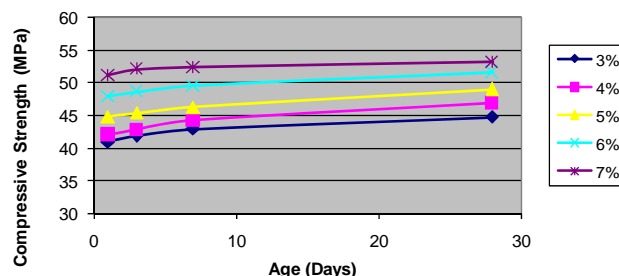


Fig. 1 Effect of SP dosage on compressive strength

B. Molarity of Alkaline Solution

Mixes S₄, S₆, S₇ and S₈ were prepared to study the influence of NaOH solution concentration on workability and compressive strength of SCGC. All the other test parameters were held constant while the concentration of NaOH varied. The workability results of slump, T_{50 cm}, V-funnel, L-Box and J-Ring are presented in Table IV and the results showed that as the molarity of NaOH solution increases the workability of geopolymer concrete decreases. The compressive strength result for 1st, 3rd, 7th and 28th days of testing are shown in Fig. 6. For all days of testing, 12M NaOH solutions showed the highest compressive strength of 47.83, 48.52, 49.44 and 51.52Mpa respectively. It was observed that an increase in compressive strength from 8M to 12M but decreased from 12M to 14M for all days of testing.

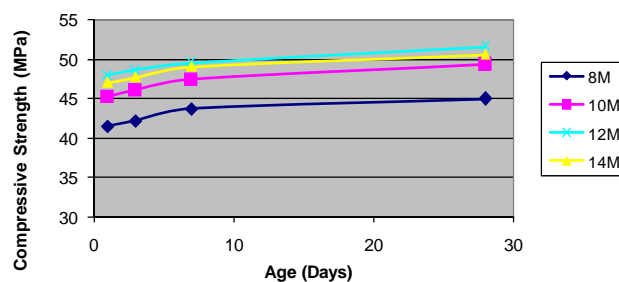


Fig. 2 Effect of NaOH molarity on compressive strength

As the concentration of NaOH solution increased from 12M to 14M, it was observed that a decrease in compressive strength due to the lower rate of polymer formation resulting in the decrease of strength. The highest compressive strength was obtained using a solution of NaOH and sodium silicate as an activator.

TABLE IV
WORKABILITY AND COMPRESSIVE STRENGTH TEST RESULTS

Mix Sample	Workability Test Results					Compressive Strength			
	Slump Flow	T _{50 cm} Slump Flow	V-funnel Flow time	L-Box (H ₂ /H ₁)	J-Ring Blocking Step, B _J	1-Day	3-Days	7-Days	28-Days
	(mm)	(sec.)	(sec)	Ratio	(mm)	(MPa)	(MPa)	(MPa)	(MPa)
S1	625	6.5	15.5	0.84	13	40.85	41.77	42.84	44.69
S2	640	6.0	14	0.88	10	42.02	42.68	44.17	46.86
S3	665	5.0	12.5	0.9	8	44.74	45.28	46.19	48.90
S4	690	4.5	10	0.94	7	47.83	48.52	49.44	51.52
S5	710	4.0	7.0	0.96	5	51.03	51.98	52.26	53.08
S6	700	4.0	9.5	0.96	5	41.45	42.14	43.62	44.87
S7	690	4.0	10	0.95	6	45.19	46.02	47.32	49.28
S8	675	5.0	12	0.90	9	46.96	47.64	48.98	50.46
Acceptance criteria for SCC as per EFNARC [20]									
Min.	650	2	6	0.8	0				
Max.	800	5	12	1.0	10				

Combination of NaOH and sodium silicate is the most suitable as alkaline activator because sodium silicate contains partially polymerized and dissolved silicon which can react easily, incorporate into the reaction products and significantly contribute to improve the characteristics of mortar [22] and also enhances the process of geopolymerization process as claimed by Xu and Deventer [23].

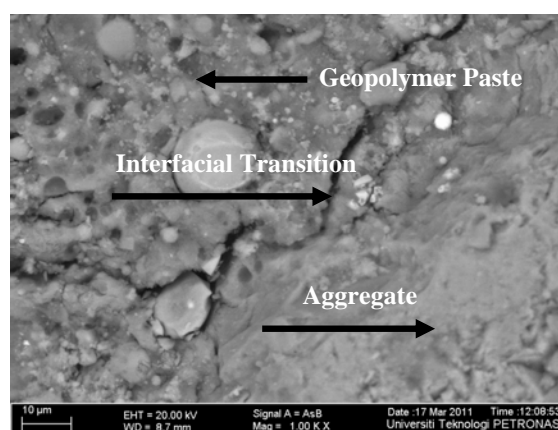
In normal geopolymer concrete as showed by [6], it was evident that an activator with a 12 M of NaOH concentration led to better performances than a 18 M of NaOH concentration due to an excess of OH⁻ concentration in the system involved a strength decrease of the alkali cement.

C. Field Emission Scanning Electron Microscopy (FESEM) Analysis

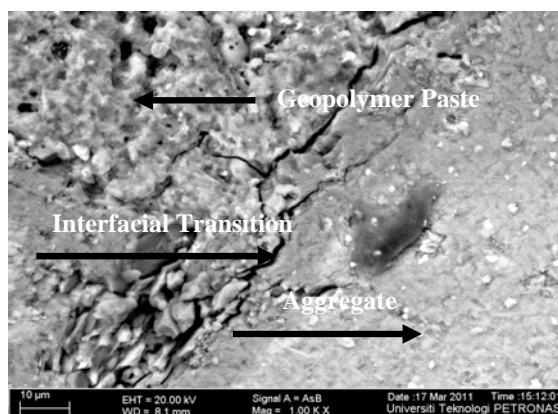
FESEM analysis was carried out to observe the microstructure properties of SCGC mainly on its ITZ. Fig. 3 shows that the ITZ in different SP dosage concrete sample. As the percentage of SP increased, the ITZ thickness decreased. The presence of the gap between the paste and aggregate was due to insufficient dosage to made them less workable and decreased concrete strength. However; when the percentage of SP increased to 6% or 7%, refinement of ITZ was observed and the microcrack was discontinued when entering the ITZ, hence resulting higher compressive strength compared to 3%, 4% and 5% SP dosage.

Fig. 4 shows the ITZ in different NaOH concentration concrete sample. As the concentration of NaOH solution increased, the gap thickness of ITZ decreased. The concrete compressive strength depends on the extent of polymeric reaction occurred during maturing. The degree of aluminosilicate polymerization in the matrix depends on the concentration and type of alkali solution used and the source material crystallinity and Si/Al ratio as claimed by [24]. Improved ITZ with less pore size was observed when the concentration increased to 10M and 12M than 8M.

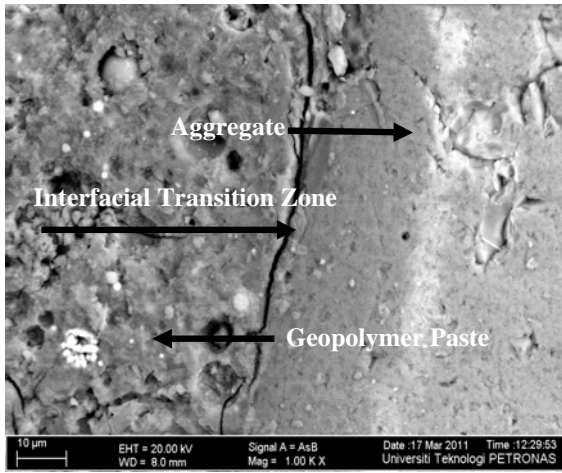
Nanocracks were identified when the concentration increased further to 14M.



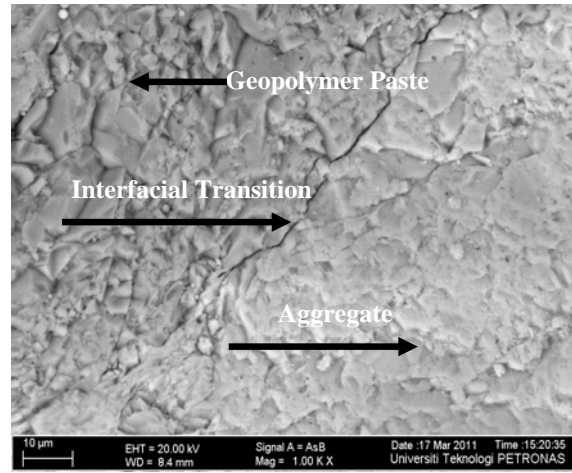
a) 3% SP



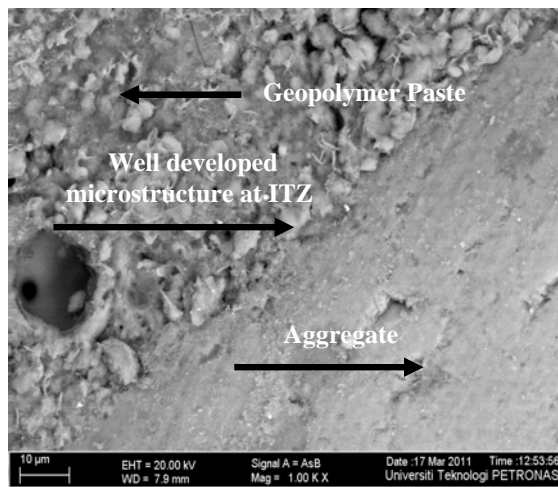
b) 4% SP



c) 5% SP

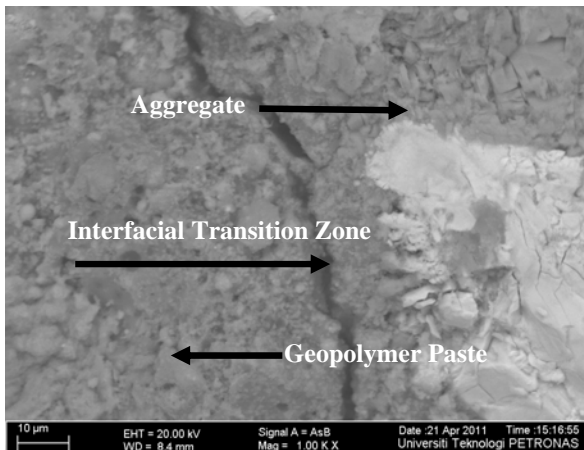


d) 6% SP

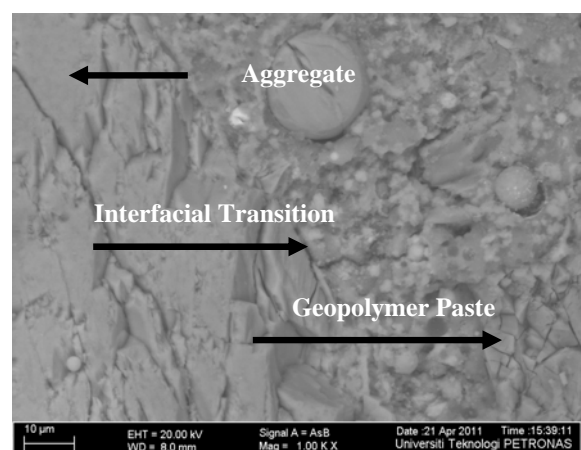


e) 7% SP

Fig. 3 FESEM images of SCGC with different SP dosage



a) 8M of NaOH solution



b) 10M of NaOH solution

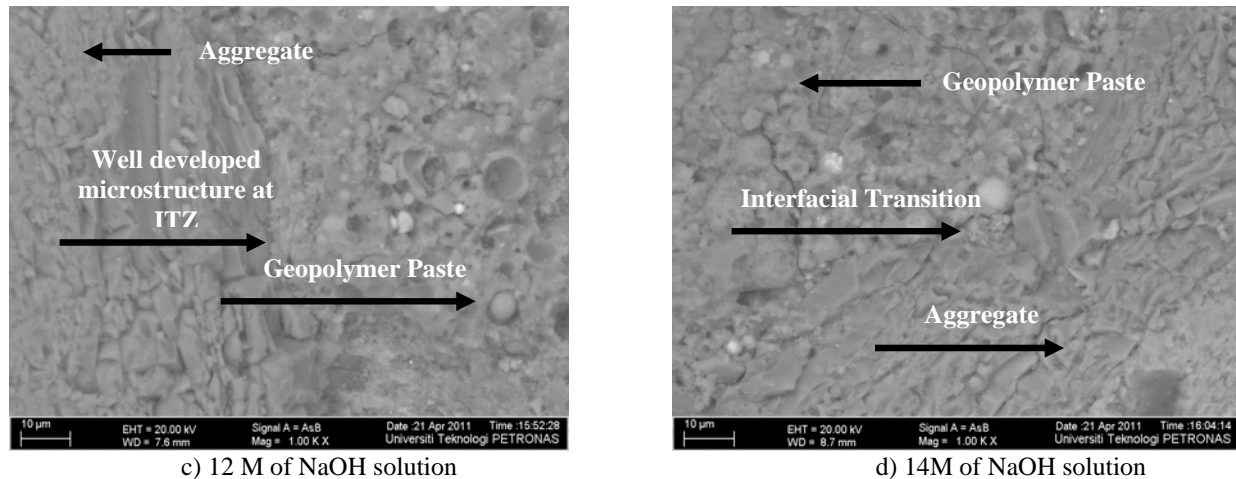


Fig. 4 FESEM images SCGC with different concentration of NaOH solution

IV. CONCLUSIONS

Based on the experimental research on SCGC, the following conclusions are drawn.

1. It can be seen that low SP content namely 3, 4 and 5% had poor filling and passing ability and the workability results were not within the range of EFNARC limits of SCC.
2. The specimens were tested after 48hrs of curing to optimize the SP dosage and molarity needed for SCGC. SP dosage of 6% and NaOH concentration of 12M produced satisfactory performance. Superplasticizer dosage of 6% is taken as the optimum because no significant contribution of SP dosage of 7%. As the concentration of NaOH solution increased from 8M to 12M, the compressive strength of geopolymer concrete was increased. But the strength decreased when the concentration increased from 12M to 14M. Concentration of 12M produced the highest compressive strength for all days of testing.
3. 6% of SP dosage and 12M of NaOH concentration could produce concrete compressive strength up to 51.52MPa tested at 28-days.
4. Improvement of ITZ was also obtained by SCGC sample in 6% SP and 10M of NaOH solution. Well developed microstructure at ITZ led to higher compressive strength.

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